Main actions

In order to find one or more solutions to this problem, all the parties involved in process of production and distribution of clothing need to take responsibility. For this reason, the Plastic Soup Foundation has launched the Ocean Clean Wash campaign, where NGOs, brands, and scientists have come together to look for solutions.

Ocean Clean Wash gathered a coalition of more than 100 organizations from all around the world to put pressure on the fashion industry, as well as other parties involved, to take responsibility for the problem throughout the entire value chain.

The solutions may be included in one of these categories:

- *a washing machine filter, synthetic yarns and fabrics that do not release fibers,*
- *synthetic yarns and fabrics that do not release plastic fibers during washing,*
- *eco-friendly coatings to prevent the release of plastic fibers,*
- *eco-friendly washing detergents with the same purpose, and a yet undiscovered innovation beyond imagination.*

Thanks to the **Mermaids LIFE+** project, we have developed a method that helps us to elaborate a benchmark to measure the amount of fibers that are released during laundry processes.
A benchmark for clothes is a strong communication tool we can use to help and inform consumers when they go shopping for clothes.

For more information,
visit www.oceancleanwash.org and www.life-mermaids.com

mermaids.
OCEAN CLEAN WASH

GOOD PRACTICE GUIDE

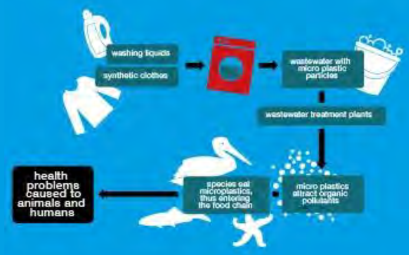
Mitigation of micro plastics impact caused by textile washing processes

(LIFE13 ENV/IT/001069)

Did you know that...

- **Around 60% of the fashion market is selling synthetic clothes.**
- **Europeans do 36 billion loads of washing every year**
- **Every wash of synthetic fabrics discharges plastic fibers less than 1mm in length**
- **Between 6,000,000 and 17,700,000 plastic microfibers are released per wash according to the Mermaids research project**

This is the problem ...



Plastic fibers coming from synthetic clothes do not biodegrade, they fragmentize into smaller pieces. These micro- and nanoplastics are smaller than 5mm and usually not visible to the naked eye. They also cannot be blocked through the waste water treatment plants and, consequently, microplastics end up in rivers, lakes, seas and oceans. Eventually, fish mistake these fibers for plankton when they end up in the oceans and seas. Around 65% of the shrimp in the North Sea contain synthetic fibers. And, guess what? We are at the top of the food chain, so they end up in our plates.

One polyester fleece jacket sheds almost a million fibers per wash.
An acrylic scarf: 300,000 fibers.
A pair of nylon socks: 136,000 fibers.

What can you do?

- Wash less
- Fill up your washing machine to the max: washing a full load results in less friction between the clothes and, therefore, less fibers are released.
- Use washing liquid instead of powder: the 'scrub' function of the grains of the powder result in loosening the fibers of clothes more than with liquid.
- Avoid using detergents with a high pH and oxidizing agents
- Wash at a low temperature: when clothes are washed at a high temperature some fabrics are damaged, leading to the release of fibers.
- Avoid long washings: long periods of washing cause more friction between fabrics, which supposes more tearing of the fibers.
- Dry spin clothes at low revs: higher revolutions increase the friction between the clothes, resulting in higher chances of fibers loosening.
- When cleaning the dryer, do not flush the lint down the drain, throw it in the bin
- Avoid buying synthetic clothes and look for wool, cotton, linen, silk, cashmere or other natural fabrics.

Name of the associated deliverable:

Report on localization and estimation of laundry microplastics sources and on micro and nanoplastics present in washing wastewater effluents

Number of the associated action: A1

Task A1.1. Mapping and estimation of laundry effluent microplastics release.
Task A1.2. Characterization and quantification of micro and nanoplastic contained into different samples.
Task A1.3. Textile origin of microplastic particles.

LIFE13 ENV/IT/001069

Mitigation of microplastics impact caused by textile washing processes

With the contribution of the LIFE financial instrument of the European Comunity:

Name of the report: REPORT TASK 1.1. Mapping and estimation of laundry effluent microplastics release

Number of the associated action: A1

LIFE13 ENV/IT/001069

Mitigation of microplastics impact caused by textile washing processes

With the contribution of the LIFE financial instrument of the European Comunity:

Acronym: MERMAIDS

Title: Mitigation of microplastics impact caused by textile washing processes

LIFE13 ENV IT 1069

REPORT TASK 1.1. Mapping and estimation of laundry effluent microplastics release

Authors :	Laia Piñol , Lola Rodriguez (LEITAT TECHNOLOGICAL CENTER)
Co-authors (alphabetical order):	Marolda Brouta, Marta Escamilla, Rosa Escudero (LEITAT TECHNOLOGICAL CENTER)
Reviewer:	Maurizio Avella (CNR)
ACTION :	A1
Task:	1.1.
Contractual Date of delivery to EC:	The results of this report will be integrated in the deliverable *"Report on localization and estimation of laundry microplastics sources and on micro and nanoplastics present in washing wastewater effluents"* 31/12/2014
Actual Date of delivery to EC:	31/03/2015
Revision number	1

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1. Introduction

European oceans are contaminated by marine litter, mainly by plastics. Contamination by plastic is growing because production trends and usage patterns are changing and global and European population is increasing (population growth rate in Europe was 0.21% in 2012).1 Conventional manufacturing is being replaced by more cost-effective plastic of equivalent or superior design. Currently, it is estimated that the global plastic production is almost 250million t/y, and it is still increasing by 10% per year2. Microplastics are particularly worrying because water treatment plants do not take them into account in their management processes and they are deposited in waterways and sewage sludge.

The European Marine Strategy Framework Directive - MSFD (2008/56/EC) establishes a framework in order for Member States (MS) to contribute to create a Good Environmental Status in the marine environment by 2020. Descriptor 10 of the MSFD tackles the properties and quantities of microplastics.3 Microlitter is specifically considered in the Commission Decision (2010/477/EU): "Trends in the amount, distribution and, where possible, composition of micro-particles (in particular micro- plastics)''.The REACH Directive (EC1907/2006) and the EU Water Framework Directive (2000/60/EC) also address the impact of microplastics.

The European Commission published, in 2013, the Green Paper on a Strategy on Plastic Waste in the Environment. Microplastics were highlighted since *"they are ubiquitous and reach even the most remote areas with a concentration in water some times higher than that of plankton. These micro-plastics, and the chemical additives they contain, if ingested in large quantities by marine fauna may have a high potential for contaminating the food chain through predator-prey interaction".* (Green Paper, p. 6 and 14).[4]

Microplastics were first described by Thompson et al. in 2004 who encountered these microscopic plastic fragments on shorelines and in the water column. Thereafter, NOAA (USA) defined microplastics as plastic particles smaller than 5mm in diameter. After these references, microplastics have been defined as particles smaller than 1mm in diameter by several authors

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[1] CIA World Factbook.

[2] CLAESSENS,Michael. *Occurrence and distribution of microplastics in marine sediments along the Belgian coast*. Marine Pollution Bulletin 62, 2011.

[3] EC. Brussels, 14.10.2011. Relationship between the initial assessment of marine waters and the criteria for good environmental status.

[4]Green Paper on a Strategy on Plastic Waste
http://ec.europa.eu/environment/waste/pdf/green_paper/green_paper_en.pdf

and sources.[5] By this definition, nanoplastics particles (orders of magnitude smaller than microplastics) are included.

The main objective of this report is to gather the state of the art of laundry as an important microplastic source taking into account all the elements involved in clothing and textile washing processes at domestic and industrial laundry level.

Although the problem of microplastics arising from washing wastewater effluents is widely known, it is still slightly documented and registered. In this sense, a big effort has been made in order to gather the state of the art of microplastic release arising from domestic and industrial laundry.

Some evidences have been already published by means of articles and scientific papers and a coherent scheme of how laundry is a marine microplastic pollution vector and how this can be tracked. Nevertheless, there is no constancy on the dimension of the contribution of laundry wastewater microplastic contribution at a regional/national scale.

In order to set the scene of laundry effluent microplastic release, an analysis of the consumption of laundry products as well as clothing and textile garments was carried out within the project participant countries.

In this sense, the following document provides the reader with a comprehensive understanding of the environmental problem of microplastic fibers, households washing habits, consumption of laundry products and clothes (according to consumers), washing machine market in Europe and professional laundry industry. This report is intended to be used in Action 1 tasks in order to extrapolate and estimate laundry waste water microplastic contribution in Europe/MERMAIDS partner countries.

2. Methodology

A literature review was made by means of several research databases in order to gather current and available knowledge and main findings concerning microplastics' impact (mainly in terms of amount of microplastics encountered in riverine and marine environments). All MERMAIDS' partners have contributed to the report with several information sources such as published articles, scientific papers, public communications and reports, among others.

LEITAT attended the workshop "Achievements and future research on micro-plastics in the marine environment" within the 2nd International Ocean Research Conference – IORC (Barcelona, 17-21 November 2014) that took place November 16th 2014. This session provided

[5] M.A. Browne et al. Accumulation of Microplastic on Shorelines Woldwide: Sources and links. Environmental Science & Technology. 45, 9175-9179 (2011). Accessed February 4,2012.

attendees with a comprehensive understanding on microplastic sources, fates, and impacts by means of different group discussions. Within this workshop the amount of information available was censed and future research and international collaborations were discussed. This workshop was very useful to establish contact between different experts on microplastics.

A presentation of the Group of Experts on the Scientific Aspects of Marine Environmental Protection (GESAMP) report in reference to microplastics took place on November 20th, also within the IORC conference. This document [6]was also reviewed.

The COMMON SENSE Project[7], lead by LEITAT, is developing, among others, a sensor to monitor the concentration of microplastics in the water column. In this sense, background information has been reviewed and several deliverables concerning microplastics' marine monitoring have been made available.

Moreover, a survey on households' laundry habits was launched October 2014 and remained open until mid December 2014. *Encuestafacil.com* has been used as the survey platform in order to structure, unify and analyze results. Household databases for a wider dissemination and approach of the questionnaire were also acquired obtaining this way more answers to the questionnaire. The objective of the inquiry was to learn about the different consumer laundry habits and to gather knowledge concerning participant countries households' practices (e.g. washing cycles) and trends. This methodology allowed us to partially quantify the microplastic problem and the awareness among consumers.

The number of questionnaires received was 831. From these, 520 were selected as useful since they were finished by respondents, and a reasonable number of answered questions were gathered. Directives and conventions have not been reviewed within this report since they will be reviewed within Action 3 of the MERMAIDS Project.

3. Environmental problem of microplastic fibers' release.

3.1. Origin of microplastic release

Marine waste consists of 60-80% of plastics (30-60% in the Baltic Sea), and most of the marine plastic litter is less than 5mm in size (microplastics).[8]

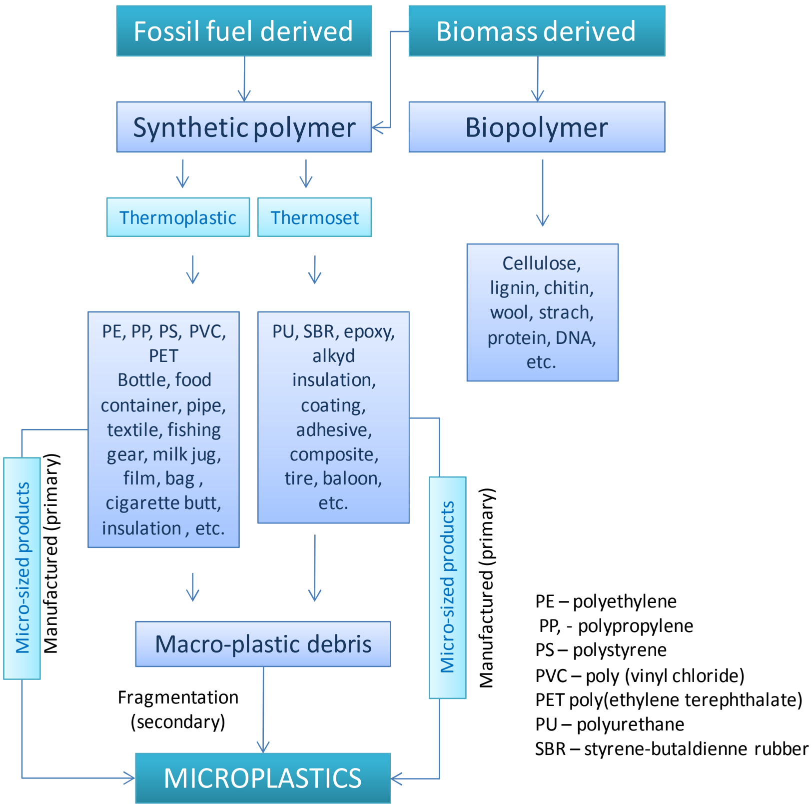
[6] GESAMP (2010, IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection); Bowmer, T. and Kershaw, P.J., 2010 (Eds.), Proceedings of the GESAMP International Workshop on plastic particles as a vector in transporting persistent, bio-accumulating and toxic substances in the oceans. GESAMP Rep. Stud. No. 82, 68pp.

[7] COMMON SENSE project website http://www.commonsenseproject.eu/

[8] HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

The origin of microplastics in the seawater are mainly: (1) introduction of microplastic from runoff, by means of e.g. cosmetics and synthetic sandblasting; (2) wastewater and meso and microplastics weathering breakdown. The following diagram shows the formation of secondary microplastics.

Figure 1. Formation on secondary microplastics by fragmentation of larger "macro-size" debris.[9]



Main land based sources for plastic marine litter are the following: storm water discharges, sewer overflows, tourism-related litter, illegal dumping[10], industrial activities, improper transport, consumer cosmetic products, synthetic sandblasting media or polyester and acrylic fibers from washing clothes. [11]

[9] GESAMP (2010, IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection); Bowmer, T. and Kershaw, P.J., 2010 (Eds.), Proceedings of the GESAMP International Workshop on plastic particles as a vector in transporting persistent, bio-accumulating and toxic substances in the oceans. GESAMP Rep. Stud. No. 82, 68pp.

[10] Liffman M. and Boogaerts (1997) "Linkages between land-based sources of pollution and marine debris" in Marine Debris. Sources, Impacts, Solutions pp. 359-366.

[11] Browne, M.A., Crump, P., Niven, S.J., Teuten, E., Tonkin, A., Galloway, T., Thompson, R. (2011). Accumulation of microplastics on shorelines worldwide: sources and sinks. Environ Sci Technol, 45(21), 9175-9179.

3.1.1 Microplastic release due to laundry wastewater

Microplastic particles of synthetic clothes coming from laundry wastewater have been encountered in marine sediments, ecosystems, runoff and sewage waters. The *"Accumulation of Microplastic on Shorelines Worldwide: Sources and sinks*" study states that "*the source of the microplastic fibres in the sewage treatment plants is most likely to be from washing machine wastewater as the mixture of fibres found in synthetic textiles is similar to the mixture of microplastic fibres found in beaches at disposal sites and in the wastewater of sewage treatment plants.".*

On average, more than 1900 fibres of microplastics can be released by a synthetic garment during one wash[12]. To get this data, washing machine effluents were analysed by means of the following three different front loading models: Bosch WAE24468GB, John LewisJLWM1203 and Siemens Extra Lasse XL 1000) were used (40° C, 600 R.P.M.) . Water discharge was analysed with and without clothes (polyester blankets, fleeces, shirts). No detergent nor conditioner were used since they interfere the filtration process, blocking the filters. *"Cross-contamination was minimized (<33 fibers) at the start of the experiment and in between washes, by running washing-machines at 90°C, 600 R.P.M for 3 cycles without clothes. Effluent was filtered and microplastic counted."*[13]

The main mechanisms responsible for the degradation of plastics in the marine environment are light-induced degradation and biodegradation. High degraded plastics can become brittle enough to fall apart into powdery fragments that can keep degrading even reaching a complete mineralization. This process is retarded in seawater due to the lower temperatures and the lower oxygen concentration[14].

The ecologist Mark Browne who was studying the accumulation of microplastics on shorelines worldwide[8] found that 85% of the human-made material found on the shoreline were microfibers, and matched the types of material, such as nylon and acrylic, used in clothing. In this sense, Browne tried to contact important clothing brands (e.g. Patagonia, Nike and Polartec) claiming for help in order to monitor the flow of synthetic fibers from clothing to the washing machine and to the ocean and to try to develop better textile fibers. However, no support was lent from the companies contacted. Nevertheless, after this bump, he received support from the women's clothing brand *Eileen Fisher*, which it is currently offering 90% of their products made of natural fibers.[15]

3.2. Microplastics toxicity

Scientific papers on microplastics affectation in birds appeared already in 1970s, however the attention of the scientific community increased a decade ago. [16]

[12] Science for Enviromental policy. EC DG Env, new alerts issue 272, 9 February 2012.

[13] http://www.fibershed.com/wp-content/uploads/2012/08/Microplastic-Study.pdf

[14] Andrady, A.L. (2011). Microplastics in the marine environment. Mar Pollut Bull 62.

[15] The Guardian http://www.theguardian.com/sustainable-business/2014/oct/27/toxic-plastic-synthetic-microscopic-oceans-microbeads-microfibers-food-chain?curator=MediaREDEF

[16] GESAMP http://www.gesamp.org/work-programme/workgroups/working-group-40

Filter feeders ranging from nanoplankton to whales have no ill effects due to microplastics since they do not have any enzymatic pathway to break down these particles so they can be neither digested nor absorbed[17]. Nevertheless, microplastics concentrate Persistent Organic Pollutants via partitioning and they can be ingested by marine biota entering in the food web[18]. It is in these dissolved POPs that toxic outcomes arise. The more hydrophobic a chemical is, the greater its affinity for microplastics.[19] In this sense, microplastics have a potential impact on the human health from the consumption of contaminated organisms.

Any toxicity associated with plastics and microplastics can be originated by one or more of the following: *(a) Residual monomers from manufacture present in the plastic or toxic additives used in compounding can leach out of the ingested plastic; (b) Toxicity of some intermediates from partial degradation of plastics; (c) POPs in seawater are slowly absorbed and concentrate in the microplastic fragments. POPs have a very large water-polymer distribution coefficient in favour of the plastic.*[20]

Some studies on the interaction of the biota are non-organic engineered nano-particles that have identified different levels of toxicity to algae, zooplankton, *Daphnea* sp. Zebra fish and bivalves, among others. Pico and nanoparticles are in the size range that can enter into cells by endocytosis.[21], [22]Evidences of microplastics ingestion by the pacific Krill (*Euphasea Pacifica*) have been also found[23]. Some other species such as the *Holoturia fieldana, Holoturia grisea, Cucumaria frondosa, Thyonella gemmata*[24]*, Mytilus edulis*[25] *and Arenicola marina*[26,27] have been reported to ingest plastic microparticles. Several observed biological effects of microplastic exposure are gathered by the "Microplastic Litter in the Dutch Marine

[17] Andrady, A.L. (2011). Microplastics in the marine environment. Mar Pollut Bull 62.

[18] Teuten, E.L., S.J. Rowland, T.S. Galloway, and R.C.Thompson. 2007. Potential for plastics to transport hydrophobic ontaminants. Environmental Science and Technology, 41 : 7759-7764

[19] Leslie, H.A., van der Meulen, M.D., Kleissen, F.M., and Vethaak, A.D. 2011. Microplastic Litter in the Dutch Marine Environment. Deltares, Netherlands.

[20] Andrady, A.L. (2011). Microplastics in the marine environment. Mar Pollut Bull 62.

[21] Templeton, R., Ferguson, P., Washburn, K., Scrivens, W., Chandler, G., 2006. Life- cycle effects of single-walled carbon nanotubes (SWNTs) on an estuarine meiobenthic copepod. Environ. Sci. Technol. 40

[22] Andrady, A.L. (2011). Microplastics in the marine environment. Mar Pollut Bull 62.

[23] Andrady, A.L., 2009. Proceedings of the International Research Workshop on the Occurrence, Effects and Fate of Micro-plastic Marine Debris, Sept 9-11, 2008. NOAA Technical Memorandum NOS-OR&R-30. Arthur, C., Baker, J., and Bamford, H.

[24] Graham, E.R., Thompson, J.T., 2009. Deposit- and suspension-feeding sea cucumbers (Echinodermata) ingest plastic fragments. Journal of Experimental Marine Biology and Ecology 368.

[25] Browne, M.A., Dissanayake, A., Galloway, T.S., Lowe, D.M., 2008. Ingested microscopic plastic translocates to the circulatory system of the mussel, Mytilus edulis . Environ. Sci. Technol 42 (13)..

[26] Thompson, R.C., Olsen, Y., Mitchell, R.P., Davis, A., Rowland, S.J., John, A.W.G., McGonigle, D., Russell, A.E., 2004. Lost at sea: where is all the plastic? Science 304, 838.

[27] Voparil, I.M., Burgess, R.M., Mayer, L.M., Tien, R., Cantwell, M.G., Ryba, S.A., 2004. Digestive bioavailability to a deposit feeder (Arenicola marina) of polycyclic aromatic hydrocarbons associated with anthropogenic particles. Environ. Toxicol. Chem. 23.

Environment" report.[28] Silver-based nanoparticles pose a threat to some living organisms such as bacteria[29].

Awareness and knowledge concerning microplastics impact is currently increasing. Many different studies have been recently published concerning microplastics impact on *Idotea emarginata*[30] , Shore Crab *Carcinus maenas*[31] , marine habitats and the Great Lake[32].

Microplastics food chain transfer from prey to predator has been already demonstrated. Preliminary studies concluded that "*airborne nanoplastics (up to 240 nm) can enter the human blood stream and can cross the human placenta, possibly exposing the developing fetus to these particles. Plastic particles from the nm to the low μm range are likely to be absorbed by human tissue should exposure to nano-and microplastics arise*". [33]

Transport and distribution

There is not enough information about degradation to predict fragmentation and generation of microplastic fibers arising from clothing, ropes and fishing nets. Nevertheless separation of riverine and marine origin of microplastics can be done by using stable isotopes. Many different physical and mechanical processes could influence microplastics distribution (e.g. currents, seabirds, etc.). Current models only account for surface models so there is a need of models that could model non surface water microplastics distribution.[34]

"Most common plastics have specific gravities (SG) from ca. 0.6 to 1.5 but some finished products containing fillers can reach as high as 3.0. PE, PP natural and synthetic rubbers all have SG ranges of less than 1.0 and float on water. Many other common plastic types have an

[28] Leslie, H.A., van der Meulen, M.D., Kleissen, F.M., and Vethaak, A.D. 2011. Microplastic Litter in the Dutch Marine Environment. Deltares, Netherlands.

[29] SCILIFESTYLE. Consequences of the use of Silver Nanoparticles in clothing. Article 2012

[30] Julia Hämer †‡, Lars Gutow †, Angela Köhler †, and Reinhard Saborowski *† . Fate of Microplastics in the Marine Isopod *Idotea emarginata. Environ. Sci. Technol.*, Article ASAP **DOI:** 10.1021/es501385y Publication Date (Web): October 7, 2014 Copyright © 2014 American Chemical Society http://pubs.acs.org/doi/abs/10.1021/es501385y

[31] Andrew J. R. Watts *†, Ceri Lewis †, Rhys M. Goodhead †‡, Stephen J. Beckett §, Julian Moger ‡, Charles R. Tyler †, and Tamara S. Galloway † . Uptake and Retention of Microplastics by the Shore Crab Carcinus maenas. Environ. Sci. Technol., 2014, 48 (15), pp 8823–8830. DOI: 10.1021/es501090e Publication Date (Web): June 27, 2014. http://pubs.acs.org/doi/abs/10.1021/es501090e .

[32] Amanda McCormick †, Timothy J. Hoellein *†, Sherri A. Mason ‡, Joseph Schluep †, and John J. Kelly † . Microplastic is an Abundant and Distinct Microbial Habitat in an Urban River. Environ. Sci. Technol., 014, 48 (20), pp 11863–11871. DOI: 10.1021/es503610r Publication Date (Web): September 17, 2014 http://pubs.acs.org/doi/abs/10.1021/es503610r

[33] http://www.noordzeeloket.nl/images/Microplastic%20Litter%20in%20the%20Dutch%20Marine%20Environment_851.pdf

[34] "Sources and distribution" discussion group. *Workshop "Achievements and future research on micro-plastics in the marine environment". International Ocean Research Conference, Barcelona November 16th , 2014.*

SG of slightly more than 1.0 , e.g. polystyrene but given the higher density of seawater as opposed to freshwater many still float in the marine environment.PVC and POM have much higher SGs at around 1.4 and tend to sink. Finally, some speciality polymers such as polytetrafluoroethylene (PTFE) may have an SG of up to 2.3. The behaviour of different types of plastics in the water column needs further study."[35]

3.3. Quantification of microplastics

3.3.1. Riverine and inland waters

Preliminary study on Synthetic microfibers and particles at a municipal waste water treatment plant [36]

It highlights the lack of standardized methods of sampling and analyzing microplastic that results in a complicated comparability of the results obtained from the studies carried out. In this sense the Helsinki Region Environmental Services Authority has developed a microplastic sampling method targeted at WWTPs and shared it with the State Unitary Enterprise Vodokanal of St. Petersburg within the HELCOM BASE project.

The main goals of the study according to HELCOM BASE project[37] were both to provide know-how of microplastic research to Vodokanal and to conduct a pilot scale microplastic study in Vodokanal. Moreover the pilot study aimed at learning research methods (sampling, filtering and analyzing) and gaining preliminary results about the amount of micro-size litter in wastewater[38].

The amount of microplastics was assessed by sampling the wastewater at the beginning of the process and during and after the purification process.

With these aims in view[39]:

- 50 liters of purified wastewater was collected manually from wastewater stream.
- Water was filtered through different mesh sized filters (300, 100 and 20 µm, respectively) using a filtering device.

[35] GESAMP (2010, IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection); Bowmer, T. and Kershaw, P.J., 2010 (Eds.), Proceedings of the GESAMP International Workshop on plastic particles as a vector in transporting persistent, bio-accumulating and toxic substances in the oceans. GESAMP Rep. Stud. No. 82, 68pp

[36] http://helcom.fi/Lists/Publications/Microplastics%20at%20a%20municipal%20waste%20water%20treatment%20plant.pdf

[37] HELCOM BASE PROJECT - http://helcom.fi/helcom-at-work/projects/base

[38] HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

[39] HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

- 24-hours composite samples were taken from different stages of purification processes.
- Composite samples were filtered and inspected under stereo microscope.
- Microplastics were identified, counted and divided into: particles and fibers (black, blue and red).

The following graphic and tables show the preliminary results from the Vodokanal pilot study.

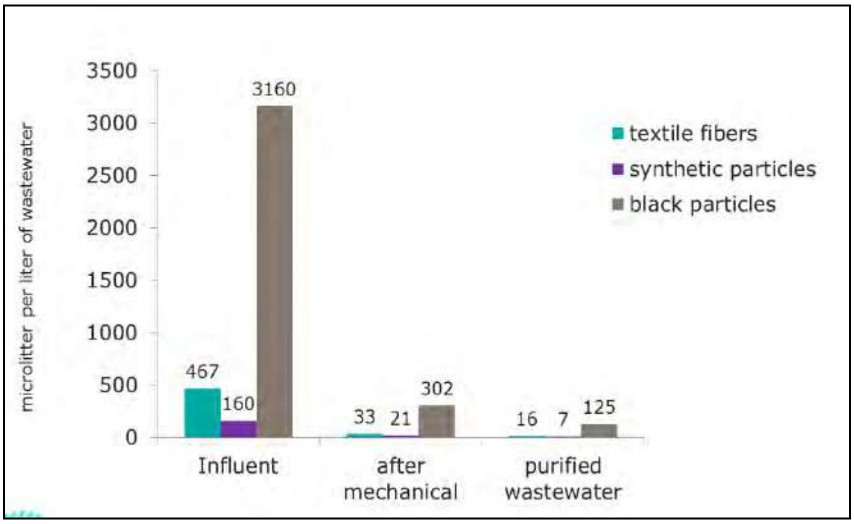


Figure 2. Preliminary results from Vodokanal (HSY). [40].

Table 1. Micro-size litter per liter of wastewater. [41]

Litter type	Influent	After mechanical	After purification	Purification %
Textile fibers	467	33	16	96.5
Synthetic particles	160	21	7	95.63
Black particles	3160	302	125	96.04

Microplastic concentration considerably decreases after the purification process, nevertheless some microplastics still end up in the water environment with purified water, acting as a point source of marine microplastics.

40 HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

41 HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

Table 2. Microplastic concentration in the Baltic Sea (the results were reported as mean values).[42]

Area	Sampling time	Synthetic fiber concentration (number per m^{-3})	Filter mesh size (µm)	Reference
Baltic Sea (Gotland basin)	2008	720	20	Noren et al 2008[43].
Baltic Sea (Gulf of Bothnia)	2008	8870	20	Noren et al 2008[44]
Baltic Sea (coast of Helsinki)	2012	126	20	Talvite et al 2012

Conclusions[45]:

- In the central WWTP of Vodokanal, the largest quantities of microplastics were found in the beginning of the purification process and the amount of microplastic decreased during the process.
- Fiber and particle contents are significantly higher in the purified wastewater than in the sea water. In this sense, WWTPs are possible point sources of microplastics.
- More research is needed.

Proceedings of the GESAMP International Workshop on Microplastic particles as a vector in transporting persistent, bioaccumulating and toxic substances in the ocean[46]

Main findings useful for the MERMAIDS Project:

NOAA has made an effort in order to standardize sampling regime of quantitative methods that provided a good starting point. Existing methods are usually addressed to biological sampling so they are limited to the collection purpose (zooplankton, phytoplankton, etc.).

The workshop discussed the following three scenarios concerning microplastics distribution but consensus was not reached:

- Hypothesis 1; the sorption of PBTs to micro-plastics is reversible.
- Hypothesis 2; for most PBTs, atmospheric transport dominates.

[42] HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

[43] Norén, F. (2008). Small plastic particles in coastal Swedish waters. N-Research report, commissioned by KIMO Sweden.

[44] Ibidem

[45] HSY - http://helcom.fi/Documents/HELCOM%20at%20work/Projects/BASE/BSD_Hazardous%20RT_Microplastics.pdf

[46] GESAMP Joint Group of Experts on the Scientific Aspects of Marine Environment Protection. http://www.gesamp.org/data/gesamp/files/media/Publications/Reports_and_studies_82/gallery_1510/object_1670_large.pdf

- Hypothesis 3; micro-plastics are stable in the surface water

Microplastic pollution in St. Lawrence River sediments[47]

Microplastics (polyethylene microbeads, 0.40–2.16 mm diameter) were reported in the sediments of St. Lawrence River. 10 freshwater sites, along 320Km section of the lake were sampled. Microbeads' abundance and magnitude varied from saple to sample: *"median and mean (±1 SE) densities across sites were 52 microbeads·m−2 and 13 832 (±13 677) microbeads·m − 2, respectively. The highest site density was 1.4 × 105 microbeads·m − 2 (or 10 3microbeads·L − 1), which is similar in magnitude to microplastic concentrations found in the world's most contaminated marine sediments. Mean diameter of microbeads was smaller at sites receiving municipal or industrial effluent (0.70 ± 0.01 mm) than at non-effluent sites (0.98 ± 0.01 mm), perhaps suggesting differential origins*."

High-levels of microplastic pollution in a large, remote, mountain lake[48]

This study evaluates the abundance, distribution and composition of pelagic microplastics in the *Lake Hovsgol* (Mongolia), encountering, among other plastics. an average microplastic density of 20,264 particles km^2. No plastic films and few pellets were encountered while the most abundant microplastic types were fragments and films (see the distribution in the table below). Microplastics pollution decreased when distancing from the most populated shore.

Table 3. Average density (particles Km2) and proportion of microplastics by type and site. [49]

Plastic type	Average microplastic density (particles km^{-2})				
	0.333–0.999 mm	1.000–4.749 mm	>4.75 mm	Total	Percent (%)
Fragment	5950	1876	335	8160	40
Film	881	4164	2740	7786	38
Line/fiber	1237	2044	702	3984	20
Foam	219	0	0	219	1
Pellet	0	58	57	115	1
Total	8287	8142	3834	20,264	
Percent (%)	41	40	19		

Microplastics in the Marine Environment: Investigating Possible Sources, Presence and Abundance Chrysoula Sofra.[50]

The objective of the study was to assess the presence of microplastic particles in skin cleansers and define how WWTP are a source of microplastic particles into the environment. Riverine

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47 Rowshyra A. Castañeda, Suncica Avlijas, M. Anouk Simard, and Anthony Ricciardi. Microplastic pollution in St. Lawrence River sediments. NRC Research Press. 2014. http://www.academia.edu/8209188/Microplastic_pollution_in_St._Lawrence_River_sediments

48 Christopher M. Freea, Olaf P. Jensen , Sherri A. Mason, Marcus Eriksen, Nicholas J. Williamson,

Bazartseren Boldgiv. High-levels of microplastic pollution in a large, remote, mountain lake. Marine Pollution Bulletin. 2014.

49 Christopher M. Freea, Olaf P. Jensen , Sherri A. Mason, Marcus Eriksen, Nicholas J. Williamson,

Bazartseren Boldgiv. High-levels of microplastic pollution in a large, remote, mountain lake. Marine Pollution Bulletin. 2014.

50 Microplastics in the Marine Environment: Investigating Possible Sources, Presence and Abundance Chrysoula Sofra , Dr Bradley Clarke, Dr Geoffrey Fowler

https://workspace.imperial.ac.uk/ewre/Public/MSc%20posters%202010/Sofra%20FOWLER.pdf water samples were collected from locations around the area of Greater London, in order to quantify microplastics. Samples named as A and B were collected in a river that passes through a WWTP. The following table shows the number and weight of the particles extracted. Particles found in locations C and D possibly contain PE and PP, with PE in excess, and the presence of polymer mixture, possibly PE and PP, within the particles recovered from location D.

Microplastic presence in the Thames river samples, before and after the discharge of a treated effluent of a WWTP were found despite they were not identified as scrubbers' microplastics. Samples collected other sources constitute a more significant source of Microplastics to the marine environment than WWTPs.

Table 4. Number, weight and concentration of particles extracted from the riverine samples from locations A, B, C and D and the blank sample. [51]

	Number	Total weight	Concentration	
		(mg)	(particles·l-1)	(mg·l-1)
Location A	ND*	ND*	<0.05	-
Location B	ND*	ND*	<0.05	-
Location C	188	6.59	9.9	0.35±0.01
Location D	60	0.69	3.3	0.04±0.00

<u>Microplastic pollution in the surface waters of the Laurentian Great Lakes.</u> [52]

Water samples were collected at 21 stations, along a 1300Km expedition in July 2012 in the Laurentian Great Lakes of the United States. The method used was a a manta trawl with 333lm mesh. The average abundance of microplastic particles/km was approximately 43,000, station 20, but downstream from two major cities, samples contained over 466,000 particles/km, greater than all other stations combined. Many microplastic particles were multi-colored spheres, which were related to microbeads from consumer products containing microplastic particles of similar size, shape, texture and composition (see the table below). The presence of microplastics was likely from nearby urban effluent and coal burning power plants.

[51] Microplastics in the Marine Environment: Investigating Possible Sources, Presence and Abundance Chrysoula Sofra , Dr Bradley Clarke, Dr Geoffrey Fowler

https://workspace.imperial.ac.uk/ewre/Public/MSc%20posters%202010/Sofra%20FOWLER.pdf

[52] Marcus Eriksen, Sherri Mason, Stiv Wilson, Carolyn Box, Ann Zellers, William Edwards, Hannah Farley, Stephen Amato. Microplastic pollution in the surface waters of the Laurentian Great Lakes. Marine Pollution Bulletin, 2013.

Abundance of plastic particles (count/km^2) by type and size			
	0.355–0.999 mm	1.000–4.749 mm	>4.75 mm
Fragment	247,106.5	123,906.2	11,219.8
Film	3943.5	1332.2	4006.1
Foam	54,340.9	18,208.4	1810.5
Pellet	430,029.8	5614.1	420.9
Line	1328.9	2571.9	449.0
Count/km^2	736,749.6	151,632.9	17,906.3
% Of total	81%	17%	2%

Table 5. Abundance and type of particles in three size classes. [53]

3.3.2. Marine environment

Even though some references and information are available on the amounts of marine litter that are found in the marine environment, it is very difficult to quantify both the amounts and sources of this litter. Many experiences exist on the quantification and classification of marine litter on beaches but very few on the quantification of marine litter, and especially micro litter, on the water surface and the water column.

Although most of the plastic materials are persistent and do not biodegrade easily, under the influence of solar UV radiations, high temperature, wave and air friction, do degrade and fragment into small particles, termed microplastics. Our oceans eventually serve as a sink for these small plastic particles and in one estimate, it is thought that 200,000 microplastics per km2 of the ocean's surface commonly exist.[54]

Studies showed that microplastics have accumulated in marine regions and in oceans and sediments worldwide with maximum concentrations reaching 100.000 particles per cubic meter.[55]

In relation to the quantification of micro litter in the marine environment, the following information is provided.

- **BALTIC SEA**

The **amount of micro-particles in water column** has been surveyed in Sweden by Norén and Magnusson (2010) and KIMO (2007) and in Finland by Setälä et al. (unpublished). The concentrations ranged from 340 to 14620 >10 µm fibres m-3 (2.8 fibers of >300 µm per m3 in average) and from 760 to 104.780 non-fibers m-3 of the same size (2.5 non-fibers of >300 µm

[53] Marcus Eriksen, Sherri Mason, Stiv Wilson, Carolyn Box, Ann Zellers, William Edwards, Hannah Farley, Stephen Amato. Microplastic pollution in the surface waters of the Laurentian Great Lakes. Marine Pollution Bulletin, 2013.

[54] Valavanidis, A., Vlachogianni, T., (2014). Microplastics in the marine environment: Ubiquitous and persistent pollution problem in the world's oceans threatening marine biota. http://www.chem.uoa.gr/scinews/Reports/PDF/MICROPLASTICS-REVIEW.pdf

[55] Valavanidis, A., Vlachogianni, T., (2014). Microplastics in the marine environment: Ubiquitous and persistent pollution problem in the world's oceans threatening marine biota. http://www.chem.uoa.gr/scinews/Reports/PDF/MICROPLASTICS-REVIEW.pdf

per m3 in average). In Finland, the recent survey in 2012 found similar concentrations of fibers and "other particles". Of the Swedish micro-particles, 23% were plastic and the majority of organic material (Norén/Magnusson 2010, Kinell et al. 2012). For unknown reasons the amounts were highest in the Gulf of Bothnia.[56]

Among the gaps, the document mentions the following:

- Amounts and consistency/composition, and transport, origin and impacts of marine litter on the sea floor and in the water column (floating litter, micro-particles).

- Transfer of toxic chemicals with micro-particles, and toxicity of marine litter.

- Input pathways of marine litter, especially regarding micro-particles (role of cosmetics, textile fibers, ash and road traffic) and inputs from rivers.

The following results from field study carried out in the **Swedish coast**[57] are available:

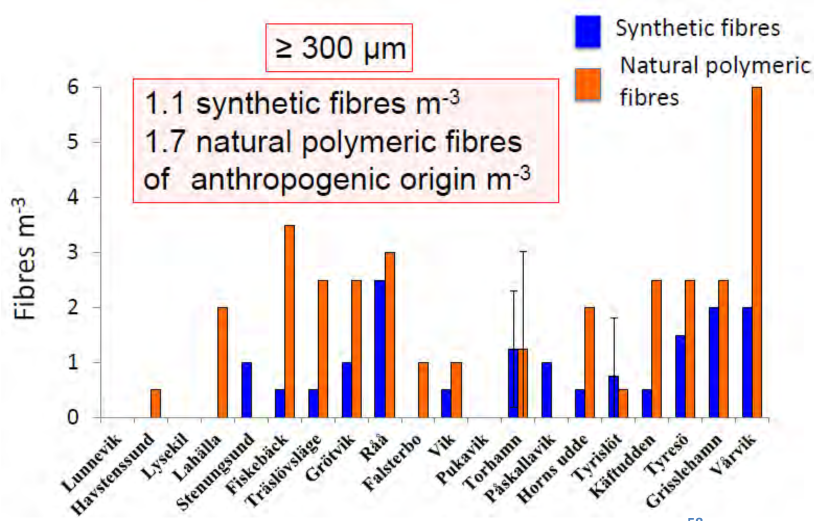

Figure 3. Results from field study along the Swedish coast[58]

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56 HELCOM MONAS (2014) Marine Litter in the Baltic Sea: sources, monitoring approaches, posible common indicators and first lines of thinking on measures.

57 Magnusson, Kerstin (2013) Microscopic anthropogenic particles – methods for monitoring and results from a survey. http://www.google.es/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0CCMQFjAA&url=http%3A%2F%2Fgesreg.msi.ttu.ee%2Fdownload%2F20130206_Kerstin%2520Magnusson.pdf&ei=zvVyVPaJC9DiaPvDgagN&usg=AFQjCNGKDTEewLcM0q8l0hSGFeW5bHLQiA&bvm=bv.80185997,d.d2s

58 Magnusson, Kerstin (2013) Microscopic anthropogenic particles – methods for monitoring and results from a survey. http://www.google.es/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0CCMQFjAA&url=http%3A%2F%2Fgesreg.msi.ttu.ee%2Fdownload%2F20130206_Kerstin%2520Magnusson.pdf&ei=zvVyVPaJC9DiaPvDgagN&usg=AFQjCNGKDTEewLcM0q8l0hSGFeW5bHLQiA&bvm=bv.80185997,d.d2s

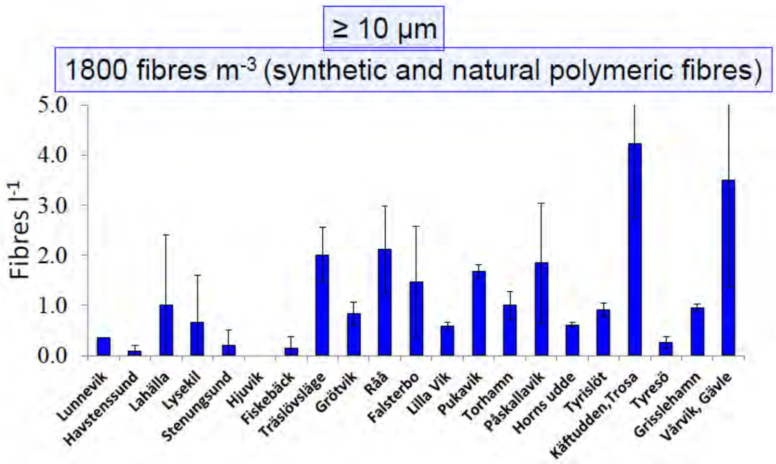

Figure 4. Results from field study along the Swedish coast[59]

	Particle conc. Number per m³
Swedish coastal waters (*our study*)	1.1
California, coastal, before a storm	0.6
California, coastal, after a storm	18
California, off shore, before a storm	0.1
California, off shore, after a storm	1

Figure 5. Results from Swedish waters compared to a Californian study (plastic particles ≥300 µm in surface water (0-1.5 m))[60]

Furthermore, the role of **waste water treatment plants (WWTPs) as entrance routes for microplastic particles to the sea** has been evaluated in 2014 in Sweden by the IVL Swedish Environmental Research Institute.

WWTP concentration in incoming water was 15 000 microplastic particles per m3, resulting in an inflow of 3 200 000 particles per hour. Retention in sewage sludge was > 99 %, and 1 770 microplastic particles per hour left the WWTP with the effluent water. The microplastic concentration in the recipient of the effluent tube was elevated compared to background levels; 1.1 -1.8 plastic particles m-3were found in the effluent plume compared to 0.45 m-3in the reference area. Higher particle concentrations were found close to the mouth of the tube compared to 200 m downstream. For further information on this matter, please access report.[61]

[59] Magnusson, Kerstin (2013) Microscopic anthropogenic particles – methods for monitoring and results from a survey. http://www.google.es/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0CCMQFjAA&url=http%3A%2F%2Fgesreg.msi.ttu.ee%2Fdownload%2F20130206_Kerstin%2520Magnusson.pdf&ei=zvVyVPaJC9DiaPvDgagN&usg=AFQjCNGKDTEewLcM0q8l0hSGFeW5bHLQiA&bvm=bv.80185997,d.d2s

[60] Magnusson, Kerstin (2013) Microscopic anthropogenic particles – methods for monitoring and results from a survey. http://www.google.es/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&ved=0CCMQFjAA&url=http%3A%2F%2Fgesreg.msi.ttu.ee%2Fdownload%2F20130206_Kerstin%2520Magnusson.pdf&ei=zvVyVPaJC9DiaPvDgagN&usg=AFQjCNGKDTEewLcM0q8l0hSGFeW5bHLQiA&bvm=bv.80185997,d.d2s

[61] Magnussen K., Norén, F., IVL Swedish Environmental Research Insitute (2014). Screening of microplastic particles in and down-stream a wastewater treatment plants.

- **NORTH SEA**

In the North Sea area only a handful of studies of the occurrence of microplastics in seawater and marine sediments have been performed to date. They show that microplastics are present in these matrices (table below). Reported concentrations range from 1 to 400 microplastic particles/kg dry sediment and from 0.01 to 102,000 particles/m3 in seawater (the last figure representing a 'hotspot', Norén 2008). Elsewhere in the world, many more studies have demonstrated the ubiquitous nature of microplastic pollution at low background levels to high levels at hotspots (table below). [62]

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http://www.ivl.se/download/18.1acdfdc8146d949da6d4852/1417761985912/C55+NV+screening+Microlitter+reportKM.pdf

[62] Deltares (2011) Microplastic Litter in the Dutch Marine Environment – providing facts and analysis for Dutch policymakers concerned with marine microplastic litter.

Table 6. Microplastics concentrations observed in seawater surface samples from the North Sea Area, greater Atlantic Ocean and Pacific Ocean (CPR, continuous plankton recorder).[63]

Sampling mesh size	Occurrence	Location	Reference
North Sea area			
127 mm^2 aperture in the CPR on to a scrolling 280 µm-mesh silkscreen	Microplastics in CPR records increased since 1960, peak: 0.04 - 0.05 fibres/m^3 (1980s).	Samples collected at 10 m over 40-year period on standard shipping routes	Thompson et al. 2004
80 µm	150-2400 particles/m^3	Harbour and ferry locations in Sweden, depth 0-0.3 m	Norén 2008
450 µm	0.01 to 0.04 particles/m^3	Harbour and ferry locations in Sweden, depth of 0-0.3 m	Norén 2008
0.5-2 mm	102,000 polyethylene particles/m^3	Harbour near polyethylene plant	Norén 2008
10-500 µm although method optimal for 10-300 µm	Microplastic fibres in samples same concentration as control (0.2 to 1 particle/L)	Skagerrak, Norwegian South coast	Norén & Naustoll 2011
Continuous Plankton Recorder studies	Microplastics widely detected over the North Atlantic Ocean.	UK coastal areas and North Atlantic Ocean	Edwards et al. 2011
Atlantic Ocean			
333 µm, between 30 and 600 m^3 seawater sampled per trawl	Polystyrene spherules (<2 mm) 0.04 and 2.58 particles/m^3 (max 14/m^3)	North-Eastern coastal waters USA	Carpenter et al. 1972
Surface plankton net	n=247 samples, 62% contained plastic particles	Cape Cod USA to the Caribbean	Colton et al. 1974
A neuston net 0.4x0.4 m opening; 308 µm mesh size	3.5 particles/km^2	20 transects (length 1.85 km, sampling approx. 740 m^2 each transect) (200 km E of N.S., Canada)	Dufault & Whitehead 1994

[63] Deltares (2011) Microplastic Litter in the Dutch Marine Environment – providing facts and analysis for Dutch policymakers concerned with marine microplastic litter.

Sampling mesh size	Occurrence	Location	Reference
Atlantic Ocean			
330-µm mesh manta net	142 mg microplastic/g dry weight seawater. Microplastics between 0.33 and 5 mm.	Baltimore Harbour, USA	Arthur et al. 2009c
335-µm mesh plankton net	Time series 1986 – 2008: 60% of 6136 surface tows collected buoyant microplastic pieces; highest microplastics incidence observed between 22° and 38°N.	N. Atlantic Subtropical Gyre	Lavender Law et al. 2010
Pacific Ocean			
Neuston net mesh size 3.0 mm and 0.333 µm	Concentration microplastic particles/ km^2 in Bering Sea 80±190; in Subarctic North Pacific 3370±2380; in Subtropical North Pacific 96100±780000.	Bering Sea, Subarctic and Subtropical North Pacific	Day & Shaw 1987
Net of mesh size 0.053 µm (Sameoto neuston sampler)	Most plastic fragments fell into the 0.5 mm size class (22 locations, 81.5%).	27 locations in the North Pacific Ocean	Shaw & Day 1994
330 µm plankton net	5114 particles/km^2. 98% were thin films, PP/ monofilament line or unidentified plastic.	11 neuston samples North Pacific Gyre	Moore et al. 2001
Manta trawl lined with 333 µm mesh	Average plastic density: 8 pieces/ $m^{-3;}$ density after the storm was 7x higher than prior.	5 locations offshore of San Gabriel River (California, USA)	Moore et al. 2002
10 L of seawater collected per sample, filtered over 1.6 µm glass microfiber filter	PE, PP and PS microplastic (1-2 particles/10 L when detected; 35% of samples <LOD) in surface microlayer samples (top 50-60 µm) and subsurface layer (1 m).	2 locations on north and south sides of in Singapore Island coastal waters. 20 samples total	Ng & Obbard 2006
Neuston net (mouth opening 50 x 50 cm; side length 3 m; mesh size 330 µm)	Plastics detected at 72% of locations; mean mass of 3600 g/km^2 and mean abundance of 174,000 particles/km^2. Dominant size class: 3 mm.	76 stations in the Kuroshiro Current area (North Pacific Ocean)	Yamashita & Tanimura 2007
Manta net neuston sampler	Detectable microplastics at 56-68% of stations; average size 2.3-2.6 mm. Median concentrations range 0.011–0.033 particles/m^3 in different years, with a maximum of 3.141 particles/m^3.	California current system - California Cooperative Oceanic Fisheries Investigations. Winter sampling in 1984, 1994, 2007	Gilfillan et al. 2009

➢ MEDITERRANEAN SEA

The 2010 scientific expedition undertaken by the European program called Mediterranean En-Dangered (MED) reveals that 250 billion **microplastics** could be **found in the Mediterranean Sea**.[64],[65] In general, micro debris floating on the Mediterranean Sea reached 115,000 particles per square kilometre with a maximum of 892,000 particles.[66]

The **ratio of microplastics to zooplankton in neustonic waters of the Northwestern Mediterranean basin** has been investigated and the following information has been made available[67]:

Forty neustonic samples were collected between July 9th and August 6th, 2010, in the North-western Mediterranean Basin. The samples were collected at sea. A greater sampling effort was made across the Liguro-Provençal Front.

Ninety percent of the 40 stations contained microplastic particles (size 0.333–5 mm) of various types: e.g., filaments, polysty-rene, or thin plastic films. Thirty percent of the samples contained more than 0.1 particles/m2. A total of 4371 microplastic particles, with a total dry weight of 7.9 g, were collected. The mean weight of the microplastics was 1.81 mg per particle, with a mean concentration of 2.02 mg/m2. An average concentration of 0.116 particles/m2 was observed over the total area investigated (table below).

Table 7. Synthesis of the survey results (mean microplastic particles and zooplankton)

	Microplastics		Zooplankton dry weight
	particles/m^2	mg/m^2	mg/m^2
Mean value	0.116	0.202	0.438
Minimal value	0	0	0.041
Maximal value	0.892	2.28	1.81
East part (mean)	0.164	0.306	0.406
Western part (mean) (Storm)	0.050	0.060	0.480

Studies in 2010 showed that 90% of the samples contained plastic debris (sizes 0.3-5 mm), which were mostly fibres, polystyrene fragments and plastic films. Microplastics concentrations increased 5 times before a strong wind event than after, suggesting that wind stress might redistribute plastics in the upper layers of the water column and prevent them from being sampled by the surface tows.[68]

[64] Galgani, François (2014), Mediterranean Conference on Combating Marine Litter in the Adriatinc macroregion https://www.youtube.com/watch?v=QNcNrUkVHuU

[65] Mongabay.com http://news.mongabay.com/2011/0105-fidenci_microplastics.html#sthash.l45e3pQv.dpbs

[66] Mongabay.com http://news.mongabay.com/2011/0105-fidenci_microplastics.html#sthash.l45e3pQv.dpbs

[67] Collignon, A., et al. Neustonic microplastic and zooplankton in the North Western Mediterranean Sea. Mar. Pollut. Bull.(2012), doi:10.1016/j.marpolbul.2012.01.011

[68] Valavanidis, A., Vlachogianni, T., (2014). Microplastics in the marine environment: Ubiquitous and persistent pollution problem in the world's oceans threatening marine biota. http://www.chem.uoa.gr/scinews/Reports/PDF/MICROPLASTICS-REVIEW.pdf

Another survey for microplastics in the Mediterranean Sea collected 41 samples (from September 2011 until August 2012, in the Gulf of Lion, Eastern Spanish coast, Balearic Islands, Sardinia, Corsica). The concentrations of micro-plastic debris (0.33-5 mm) were measured in term of particles number and particles mass per surface unit. The average values of micro-plastics measured were 130,000 parts/km2 and 58,000 mg/km2 respectively. A qualitative analysis revealed the presence of fragments (77% in mass), thin films (13%), foams (7%), pellets (2%) and lines (2%). The average concentrations of meso-plastic debris (5-50 mm) reached the number of 5, 500 parts/km2 and the mass of 120,000 mg/km2, mostly fragments (57%) and thin films (34%). The results revealed that plastic debris are widely distributed in Western Mediterranean Sea and are of the same order of magnitude with those measured in the North Atlantic or North Pacific subtropical gyres.[69]

4. Households washing habits

4.1. Available data according to the International Association for Soaps, Detergents and Maintenance Products (AISE)

AISE carried out a survey in 2008 and in 2011 in order to assess laundry habits and practices (including the frequency and number of washing cycles). On the other hand the Prof. R. Stamminger (University of Bonn) lead a study including 2300 online surveys on laundry habits to households of 10 different EU countries. Both studies used different methodologies but had similar results in terms of washing temperatures. The average washing temperature across Europe is 40,9ºC. Accentuated differences are noticed by country from 2008 to 2011 (see the table below).

[69] Valavanidis, A., Vlachogianni, T., (2014). Microplastics in the marine environment: Ubiquitous and persistent pollution problem in the world's oceans threatening marine biota. http://www.chem.uoa.gr/scinews/Reports/PDF/MICROPLASTICS-REVIEW.pdf

Table 8. Wash temperatures[70].

Country	Average washing temperature (ºC)
Austria	43
Belgium	41,2
Bulgaria	42,4
Czech Republic	44,3
Denmark	43
Finland	45,1
France	39,7
Germany	42,2
Greece	41,5
Hungary	41,8
Ireland	39,7
Italy	40,4
Netherlands	41
Norway	45,2
Poland	44
Portugal	36,5
Romania	42,8
Slovakia	43,5
Spain	33,9
Sweden	45,3
Switzerland	42,8
Turkey	42,5
United Kingdom	39
Average (Europe)	**40,9**
Average (5 campaign countries)	**38,9**

According to AISE, 35.60 billion laundry loads per year are done in Europe (2011) with an average wash frequency of 3.2 times per week, slightly less than in 2008. UK and Ireland are among those EU countries with a highest wash frequency (4 washed/week) while Turkey and Czech Republic have an average wash frequency below 3 washes/week (see the following graph). Stamminger survey (2011) identified an average wash frequency of 3,5 cycles per week (182 cycles per household/year). This trend started to decrease in 2002 since washing loads are increasing.

[70] AISE. The case for the " A.I.S.E. low temperature washing initiative" Substantiation dossier October 2013.

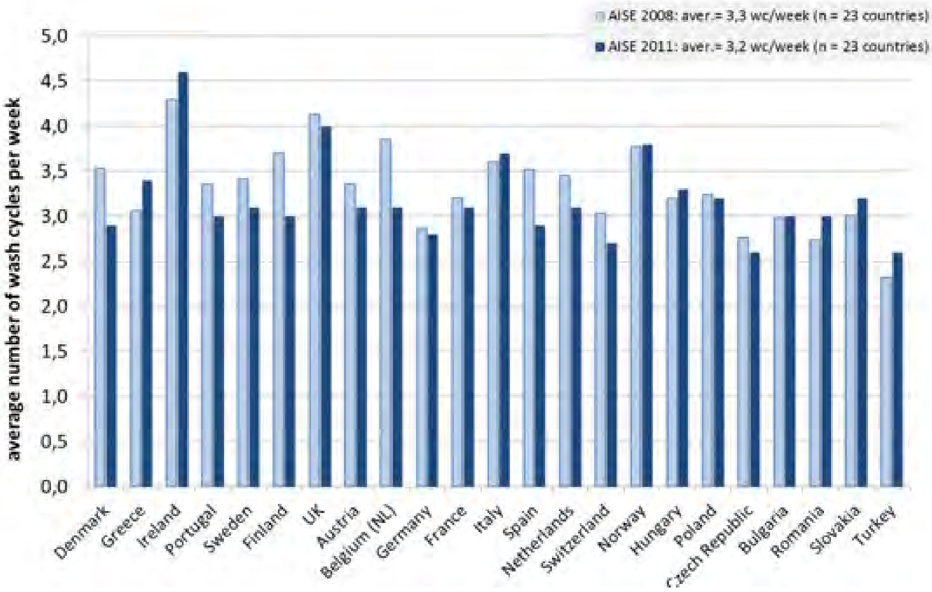

Figure 6. Average number of wash cycles[71]

According to the ISE Appliances[72], in Europe, over 21 million washing machines are now consumed per year. 18 million washing machines were produced in 2005[73].In the future, a large part of the market will draw on the replacement of old appliances, as currently in Europe, about 40 million washing machines are older than 10 years.

Concerning machine loading, the majority of the interviewees surveyed in both studies, claimed to load the washing machine up to 75-100%. No differences were detected between 2008 and 2011. New washing machines have increase load capacity and some of them have sensors in order to detect the degree of wash loading adapting wash conditions dosing and heating the water according to the detected load. [74]

According to the ISE Appliances, the average capacity of the household washing machines has changed from about 4,8 kg in 1997 to approximately 5,4 kg in 2005; models of 4 or 5 kg capacity have been replaced by 5 or 6 kg or new models with 7kg capacity. Related to water consumption, while in 1997 the majority of machines had a water consumption of 75 litres, this value is now about 50 litres per cycle. When comparing the average water consumption per kg, has decreased from 13.9 l/kg in 1997 to 9.6 l/kg in 2005.

[71] AISE. The case for the " A.I.S.E. low temperature washing initiative" Substantiation dossier October 2013.

[72] ISE Appliances http://www.iseappliances.co.uk/

[73] EEA. Discussion Report: EU Ecolabel for Washing Machines, 2009

[74] AISE. The case for the " A.I.S.E. low temperature washing initiative" Substantiation dossier October 2013.

Moreover according to A.I.S.E. "I prefer 30º" campaign, washing machines represent around 6.4% of the households EU electricity consumption. It is also estimated that the worldwide laundry electricity consumption is around 1.000 TWh.

DEFRA carried out a study with households in UK in 2012[75] concluding that washing machines use around 5% of the total UK annual electricity consumption. This study took into account the six main domestic appliances (cold, wet, cooking lightening, consumer electronics and computing).

AISE and R. Stamminger experiences will be contrasted in the next section with the survey carried out in the framework of the MERMAIDS project.

4.2. Households' laundry habits

Data included in this section arises, unless stated otherwise, from the Household's washing habits survey carried out within the MERMAIDS project framework from October to December 2014. The number of questionnaires received was 831. From these, 520 were selected as useful since they were finished by respondents, and a reasonable number of answered questions were gathered.

a. <u>Personal data</u>

Respondents' age was above 18 years old and the balance between the different age ranges was equilibrated, highlighting respondents from 35 to 44 years old (see figure below).

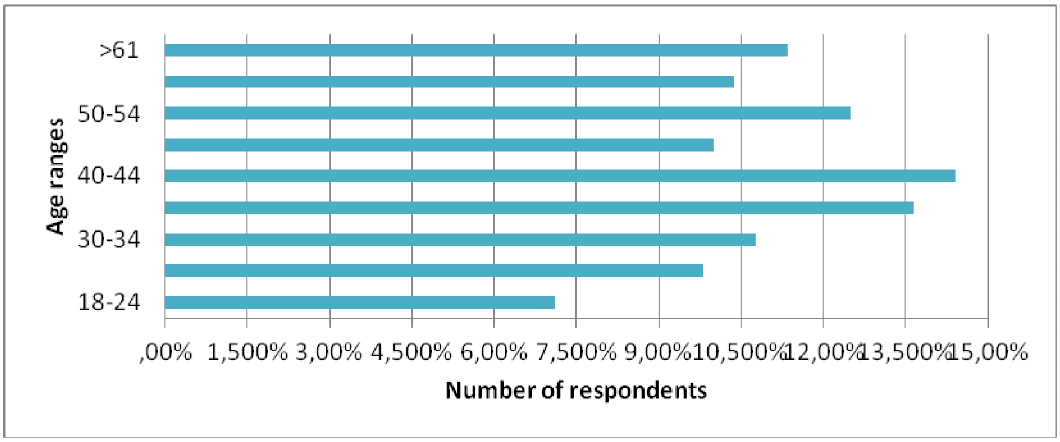

Figure 7. Age of respondents.[76]

Concerning respondents' gender, 63% were women and 37% were men (see figure below).

[75] DEFRA "Powering the Nation – Household electricity survey: A study of domestic electric product usage", Report R66141, UK DEFRA, may 2012

[76] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

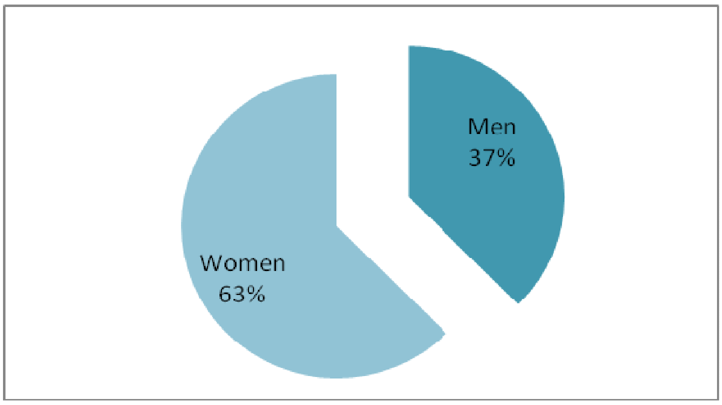

Figure 8. Gender of the respondents. [77]

Answers from all around Europe were received but a greater effort has been made in the MERMAIDS' partner countries and France. The figure below shows the country of residence of the respondents of the questionnaires received, highlighting (in order decreasing order of respondents) Spain, France, Italy, Netherlands, Belgium, Portugal and Germany (see figure below).

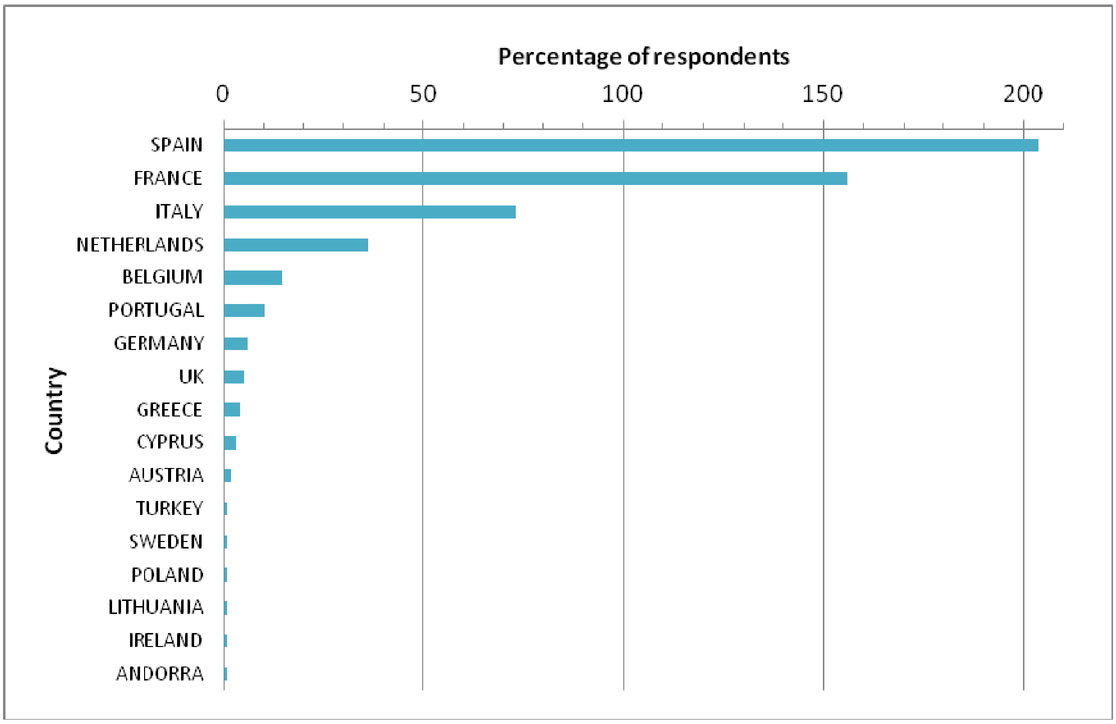

Figure 9. Country of residence. [78]

[77] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[78] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

Regarding the number of people living together in the respondents' household, most of the answers received (79%) pointed 2-4 people living together in the same household (see the figure below).

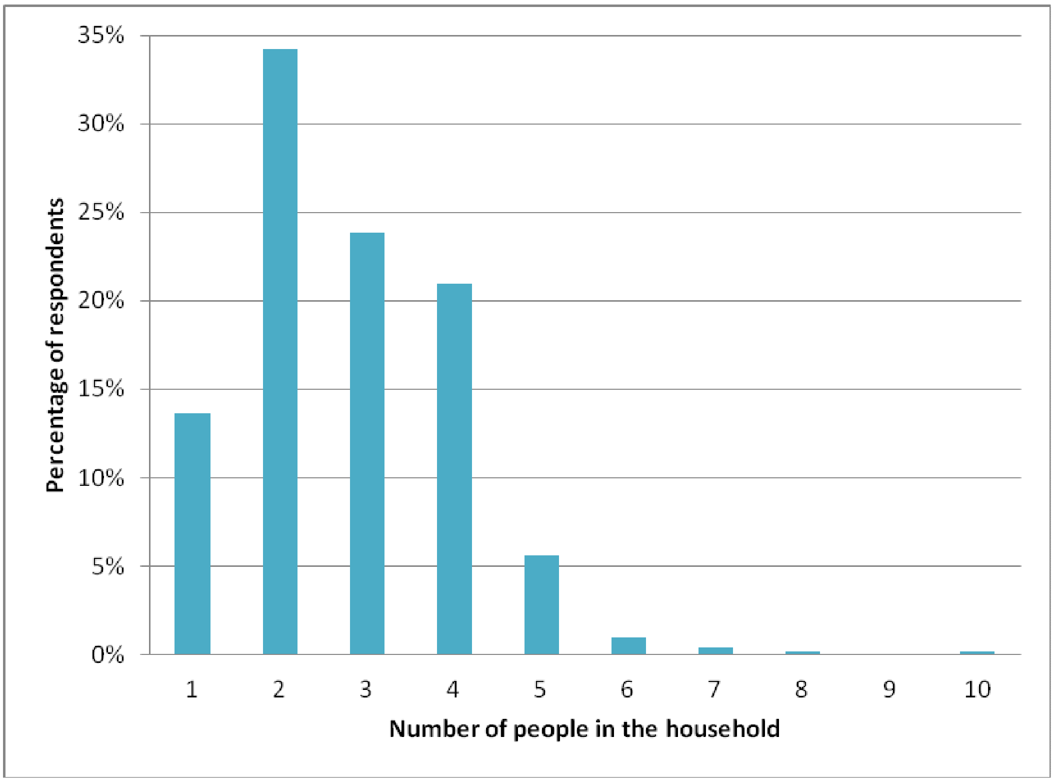

Figure 10. People living in the household. [79]

EUROSTAT defined, according to Lacovou and Skew methodology[80], different household typologies according to the number and ages of people as follows:

[79] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[80] Iacovou, M. and Skew, A.J. (2010), "Household Structure in the EU", Eurostat methodologies and working papers, Eurostat, Luxembourg.

Table 9. EU households typology[81]

Single person < 65	A single person under age 65
Single person >= 65	A single person aged 65 or over
Couple both < 65	A couple (married or cohabiting) both aged under 65
Couple, at least one >= 65	A couple (married or cohabiting), one or both of whom is aged 65 or over
Couple + dependent child/ren	A couple with one or more of their own children, including at least one child aged under 18.
Couple + adult child/ren	A couple living with one or more of their own children, all of whom are aged 18 or over.
Lone parent + dependent child/ren	A single adult plus one or more of his or her own children, including at least one child aged under 18.
One parent + adult child/ren	A household consisting of one parent plus one or more of his or her own children, all of whom are aged 18 or over.
Extended family	Non-nuclear households whose members all belong to the same family. Most of these are either three-generation families, or households including a parent and an adult child with a partner or spouse.
Other households	Other households, incl. lodgers, unrelated sharers, etc.

b. Washing machine brand and capacity

As mentioned in section 8 *"Washing machine market in Europe"* of this report, the most common washing machine brands are Bosch, Whirpool, LG, Indesit and AEG and 90% of the respondents indicated that their washing machine capacity is between 5 and 8 Kg.

c. Washing program

Taking into account Europeans domestic habits, the following five groups of washed clothes were established: mix clothes (washes including different types of clothes), colour/dark clothes, white clothes, delicates and bed linen/towels washes. Regarding these different groups established, respondents had to indicate the washing machine program used to wash every kind of clothes. The figure below shows the percentage of respondents using the different programs to wash the different groups of clothes. Cotton program was pointed out as one of the most used for all kind of clothes except for delicates.

[81] Iacovou, M. and Skew, A.J. (2010), "Household Structure in the EU", Eurostat methodologies and working papers, Eurostat, Luxembourg.

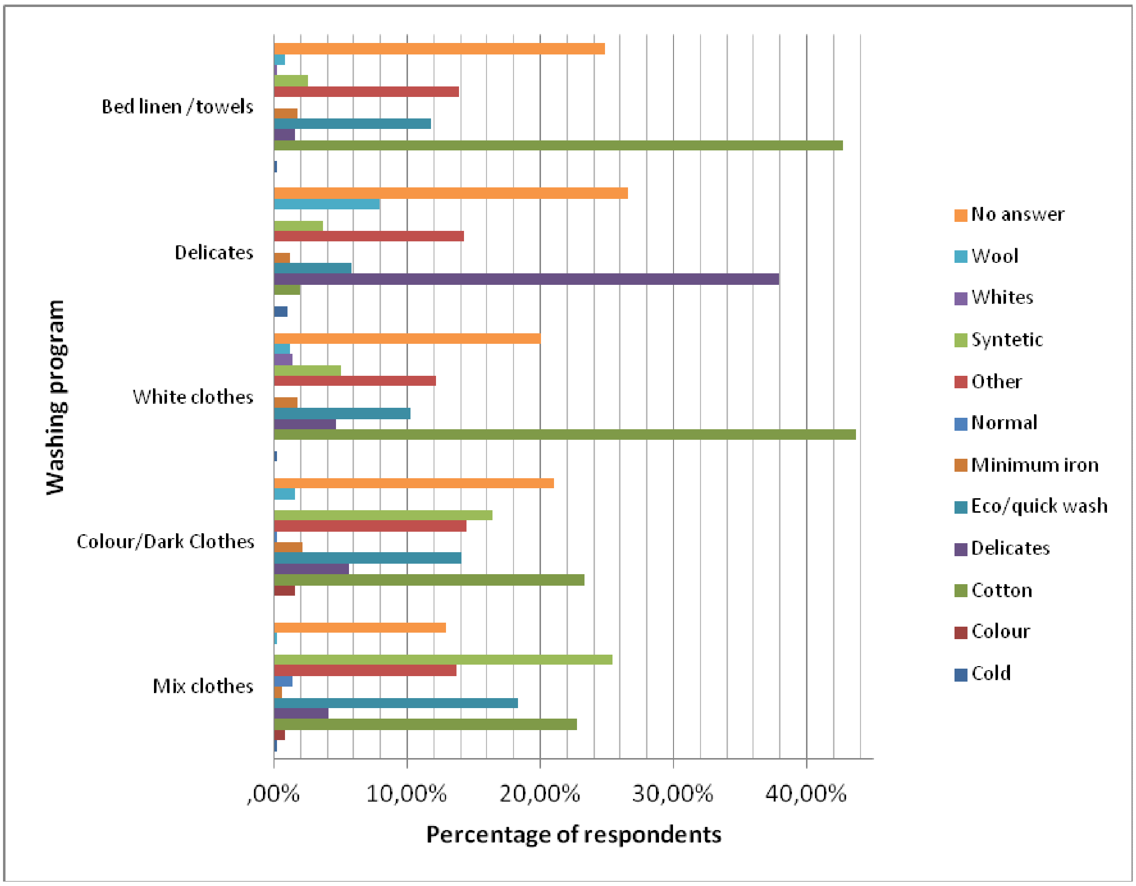

Figure 11. Washing program used for different type of clothes. [82]

The same data can be expressed in a different way, taking into account that microplastic release depends, among others on the type of clothes and the aggressively of the washing program/centrifugation used. The figure below shows the type of clothes usually washed taking into account the different washing machine programs.

[82] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

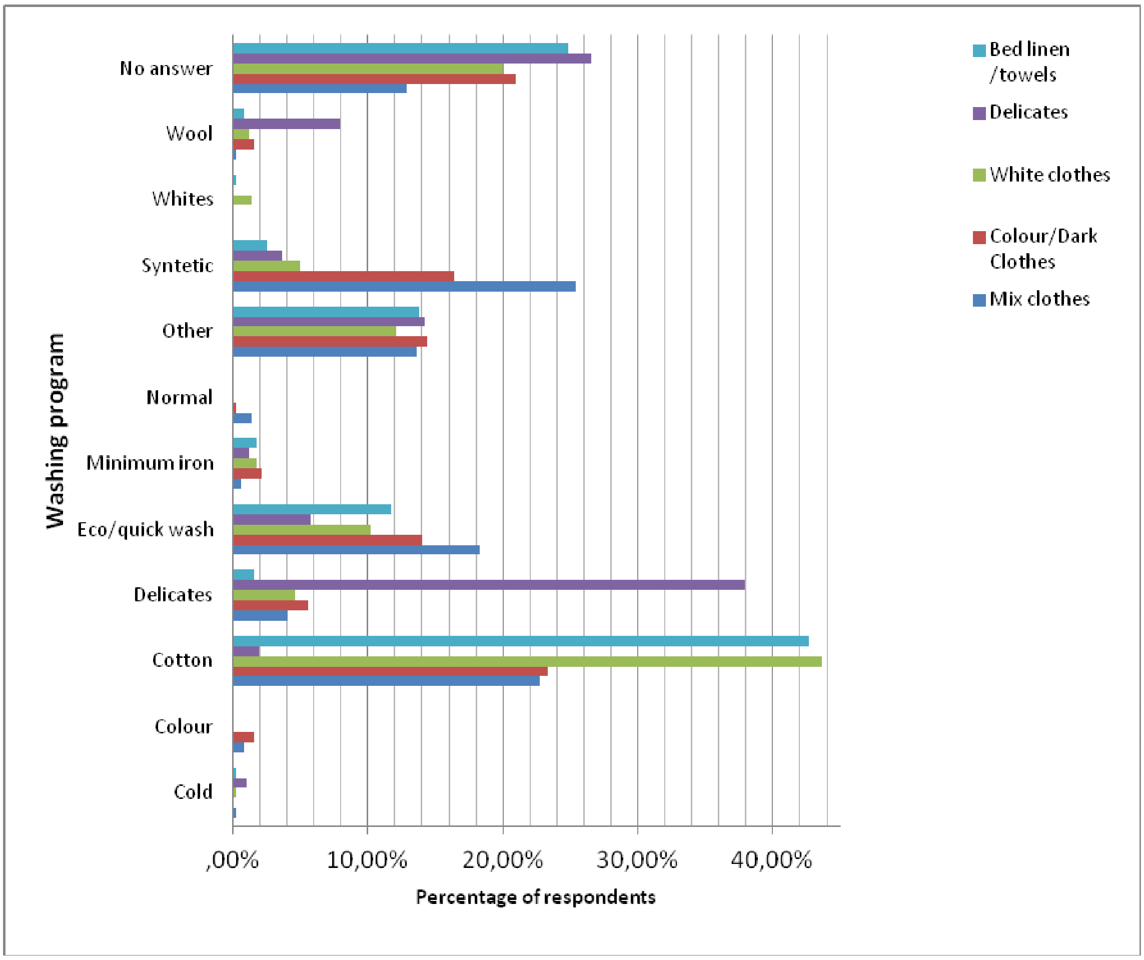

Figure 12. Typology of clothes washed using the different types of programs.[83]

d. Washing temperature

Concerning washing temperature, the following table and figure show the temperatures used for the different groups of clothes according to the survey respondents.

Table 10. Washing temperature used for the different type of clothes (percentage of respondents)[84].

Program / Temperature	COLD	30ºC	40ºC	50ºC	60ºC	>60ºC	Other	No answer/I don't know	TOTAL
Mix clothes (Normal clothes)	14%	38%	32%	2%	3%	0%	1%	11%	100%
Mix clothes (Stainedclothes)	0%	30%	37%	4%	6%	0%	1%	22%	100%
Colour/Dark clothes (Normal clothes)	0%	34%	27%	2%	2%	0%	15%	21%	100%
Colour/Dark clothes (Stained clothes)	11%	28%	33%	4%	4%	0%	1%	20%	100%
White clothes (Normal clothes)	10%	19%	27%	4%	18%	5%	0%	18%	100%
White clothes (Stained clothes)	10%	15%	26%	6%	17%	6%	0%	20%	100%
Delicates (Normal clothes)	28%	36%	7%	1%	1%	0%	1%	27%	100%
Delicates (Stained clothes)	19%	0%	34%	14%	3%	5%	0%	24%	100%
Bed linen/towels (Normal clothes)	7%	15%	23%	5%	22%	4%	0%	24%	100%
Bed linen/towels (Stained clothes)	10%	14%	16%	6%	22%	6%	0%	25%	100%

[83] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[84] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

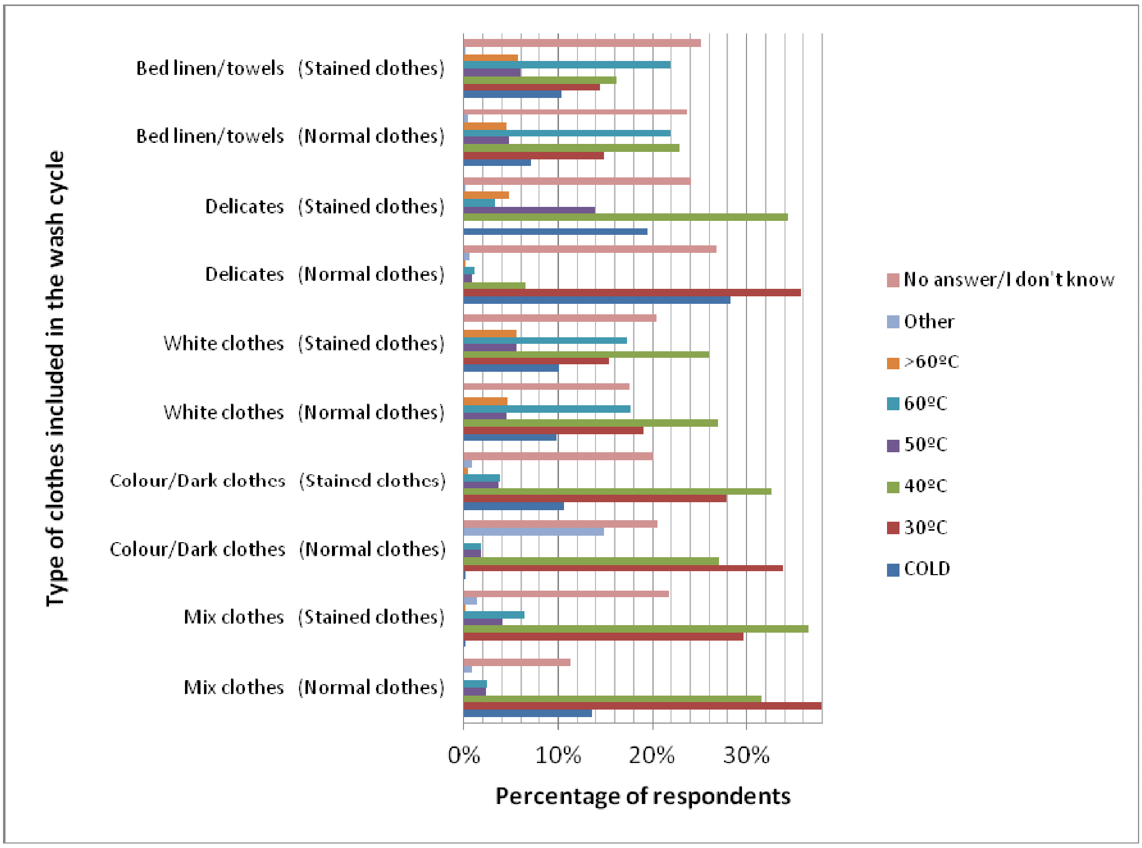

Figure 13. Washing temperature used for the different type of clothes[85].

e. Wash duration

As it is shown in the table below, domestic wash cycles usually last between one hour and one hour and thirty minutes followed by washes between 30 and 45 minutes of duration.

Table 11. Washing temperature by different wash duration[86].

Wash duration	30min-45min	1h-1h30min	1h45 - 2h15min	2h30min - 3h	/I don't know	TOTAL
Mix clothes	31%	43%	13%	3%	11%	100%
Colour/Dark clothes	25%	41%	11%	2%	21%	100%
White clothes	18%	39%	19%	6%	19%	100%
Delicates	45%	22%	5%	1%	28%	100%
Bed linen/towels	15%	35%	18%	7%	25%	100%
	27%	36%	13%	4%	21%	100%

[85] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[86] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

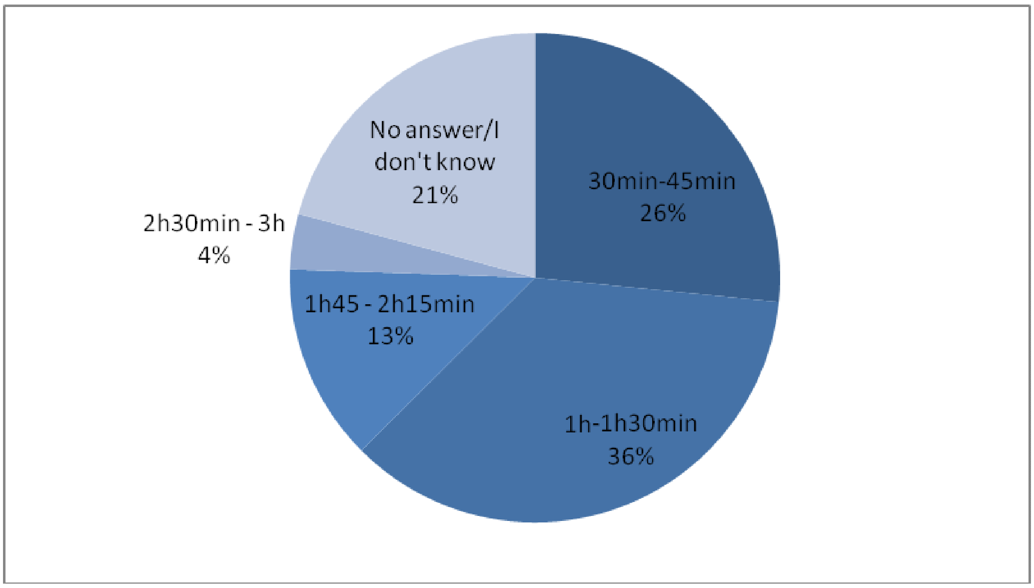

Figure 14. Washing temperature by different wash duration[87].

Regarding wash duration by different typology of clothes, delicates seem to be washed with shorter washes than the rest of clothes and bed linen/towels and white clothes are the two groups washes with longer cycles (see figure below).

[87] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

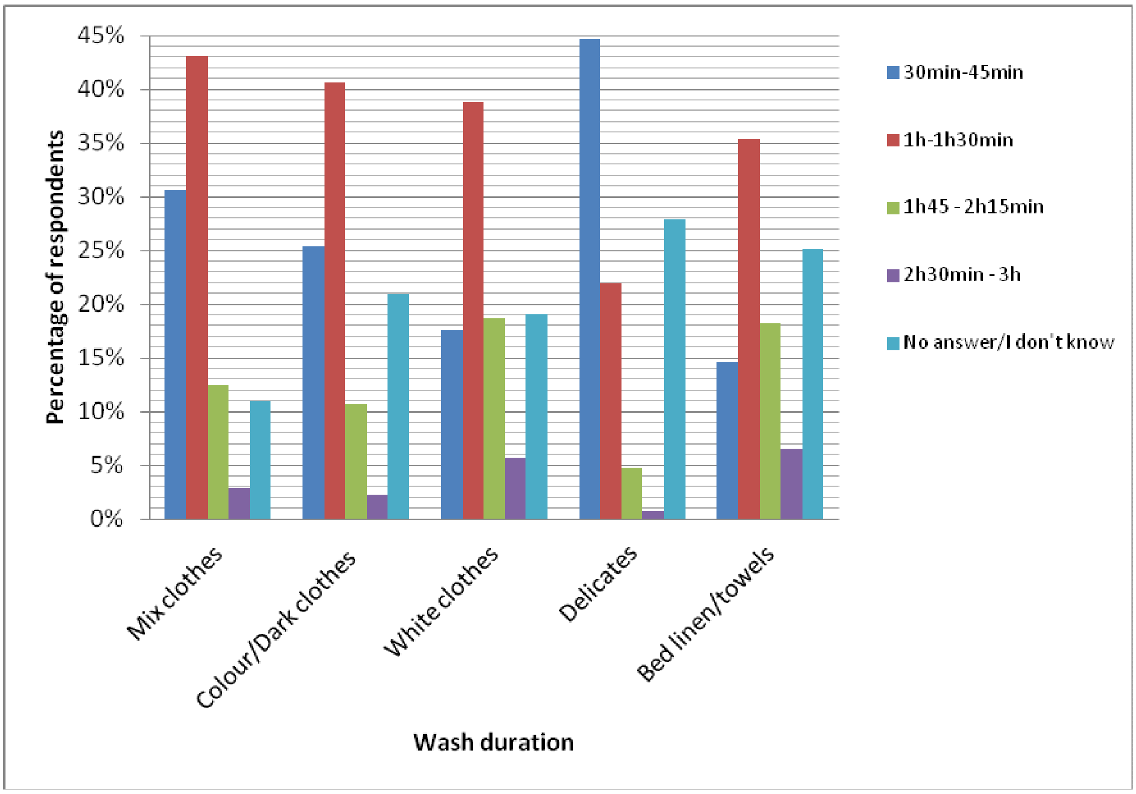

Figure 15. Wash duration by different typology of clothes[88].

f. Pretreatment

Pre-treatment washing option is more used for white clothes and bed linen/towels than for the other type of clothes. Nevertheless, 12-13% of the respondents stated that they use the pre-treatment option also for mix clothes, colour/dark clothes and delicates (see table and figure below).

Table 12. Pre-treatment option use. [89]

Pretreatment	Yes	No	No answer	Total
Mix clothes	13%	73%	14%	100%
Colour /Dark clothes	13%	69%	18%	100%
White clothes	22%	61%	18%	100%
Delicates	12%	62%	26%	100%
Bed linen/towel	16%	61%	22%	100%

[88] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[89] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

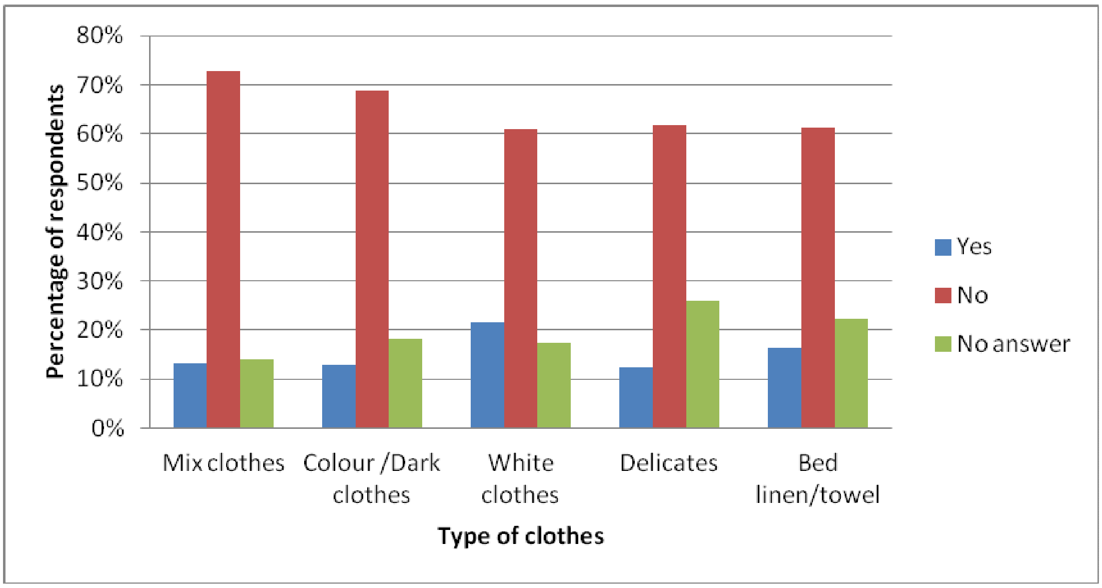

Figure 16. Pre-treatment option use. [90]

g. Centrifugation

Most centrifugation programs used are between 800 and 1000 rpm. Nevertheless, delicates centrifugation is mostly done at 400 rpm (see table and figure below).

Table 13. Centrifugation used in domestic wash cycles (r.p.m.). [91]

Centrifugation (rpm)	400	500	600	700	800	900	1000	>1000	No answer	Total
Mix clothes	9%	0%	17%	1%	17%	7%	21%	14%	13%	100%
Colour /Dark clothes	9%	1%	14%	0%	18%	5%	19%	13%	21%	100%
White clothes	8%	1%	10%	0%	18%	6%	23%	14%	21%	100%
Delicates	27%	1%	15%	1%	13%	3%	6%	4%	30%	100%
Bed linen/towel	5%	0%	10%	0%	15%	5%	22%	17%	26%	100%

[90] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[91] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

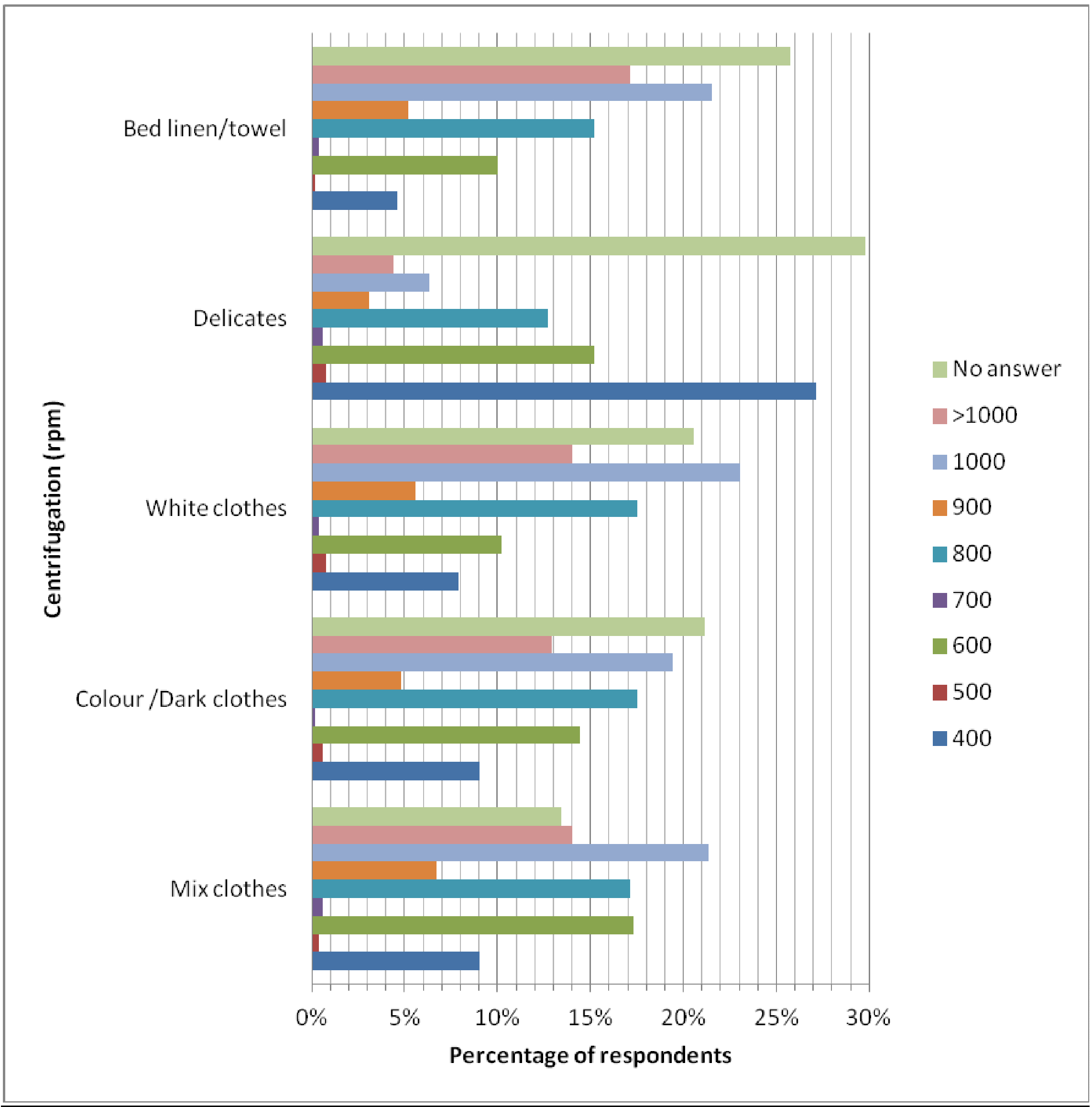

Figure 17. Centrifugation used in domestic wash cycles (r.p.m.). [92]

h. Washing machine average load

Concerning washing machine load, most respondents stated that they fill their washing machine until it is full, independently of the type of clothes. However, delicates are treated as special since 40% of the respondents stated to fill half of the washing machine.

[92] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

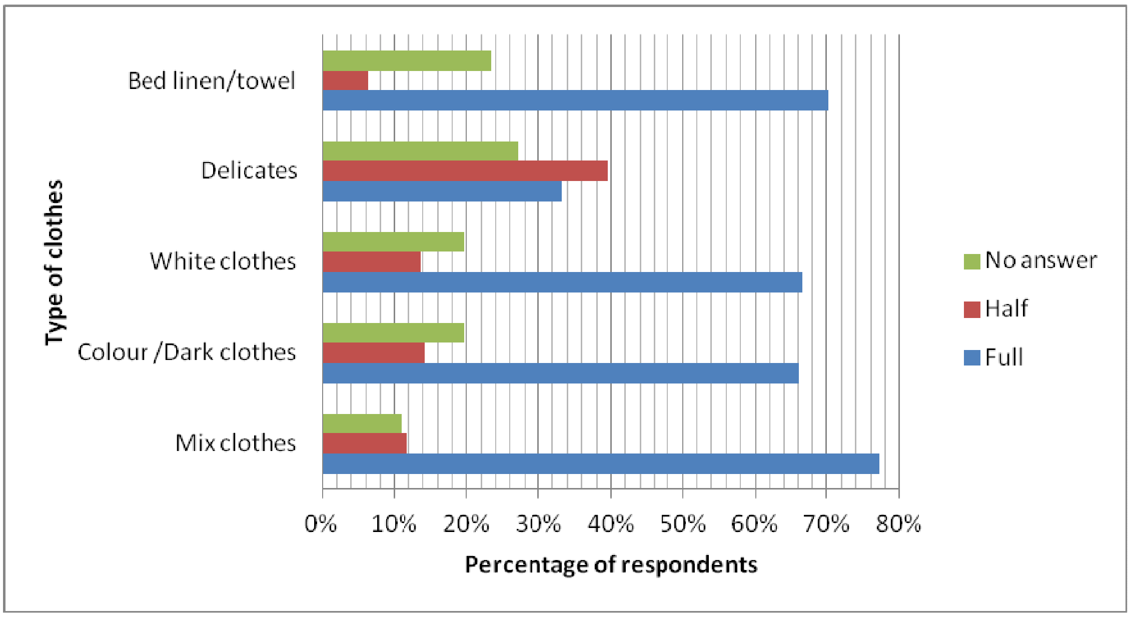

Figure 18. Washing machine average load. [93]

i. Water used per cycle

Most respondents of the MERMAIDS' survey stated that they consume between 30 and 50 liters per wash cycle.

Table 14. Water used in domestic wash cycles (liters). [94]

Water used per cycle (liters	<30	30	40	50	60	70	>70	I don't know	No answer	Total
Mix clothes	0%	7%	6%	6%	2%	1%	0%	49%	29%	100%
Colour /Dark clothes	1%	4%	6%	6%	2%	1%	0%	45%	35%	100%
White clothes	0%	4%	4%	6%	3%	2%	1%	45%	35%	100%
Delicates	0%	8%	6%	3%	1%	0%	1%	40%	42%	100%
Bed linen/towel	0%	2%	4%	4%	3%	3%	1%	42%	40%	100%

j. Washes per week

MERMAIDS' survey respondents were asked about the number of washes per week/month they usually do and for which type of clothes. According to the answers received the average of washes per household and per year is 352,54, that is, 6,7 washes/household*week (see table below).

[93] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[94] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

Table 15. Number of washes per year. [95]

	Once per month - Mix clothes	Once per month - Colour /Dark clothes	Once per month - White clothes	Once per month - Delicates	Once per month - Bed linen /towels	Twice per month - Mix clothes	Twice per month - Colour /Dark clothes	Twice per month - White clothes	Twice per month - Delicates	Twice per month - Bed linen /towels	Once per week - Mix clothes	Once per week - Colour /Dark clothes	Once per week - White clothes	Once per week - Delicates	Once per week - Bed linen /towels	2 times per week - Mix clothes
Total	27	40	72	179	85	37	70	72	105	109	170	200	212	120	207	126
Washes per year	324	480	864	2148	1020	888	1680	1728	2520	2616	8840	10400	11024	6240	10764	13104
Washes per year (%)	0,2%	0,3%	0,5%	1,2%	0,6%	0,5%	0,9%	0,9%	1,4%	1,4%	4,8%	5,7%	6,0%	3,4%	5,9%	7,1%

2 times per week - Colour /Dark clothes	2 times per week - White clothes	2 times per week - Delicates	2 times per week - Bed linen /towels	3 times per week - Mix clothes	3 times per week - Colour /Dark clothes	3 times per week - White clothes	3 times per week - Delicates	3 times per week - Bed linen /towels	4 times per week - Mix clothes	4 times per week - Colour /Dark clothes	4 times per week - White clothes	4 times per week - Delicates	4 times per week - Bed linen /towels	5 times per week - Mix clothes	5 times per week - Colour /Dark clothes	5 times per week - White clothes	5 times per week - Delicates	5 times per week - Bed linen /towels
97	69	39	66	64	49	38	16	23	23	20	15	6	10	20	8	10	5	3
10088	7176	4056	6864	9984	7644	5928	2496	3588	4784	4160	3120	1248	2080	5200	2080	2600	1300	780
5,5%	3,9%	2,2%	3,7%	5,4%	4,2%	3,2%	1,4%	2,0%	2,6%	2,3%	1,7%	0,7%	1,1%	2,8%	1,1%	1,4%	0,7%	0,4%

6 times per week - Mix clothes	6 times per week - Colour /Dark clothes	6 times per week - White clothes	6 times per week - Delicates	6 times per week - Bed linen /towels	7 times per week - Mix clothes	7 times per week - Colour /Dark clothes	7 times per week - White clothes	7 times per week - Delicates	7 times per week - Bed linen /towels	More than 7 times per week - Mix clothes	More than 7 times per week - Colour /Dark clothes	More than 7 times per week - White clothes	More than 7 times per week - Delicates	More than 7 times per week - Bed linen /towels	TOTAL	Average per household
6	6	9	6	2	11	3	2	2	1	5	7	3	0	1	2476	
1872	1872	2808	1872	624	4004	1092	728	728	364	1820	3640	1560	0	520	183.320,00	352,54
1,0%	1,0%	1,5%	1,0%	0,3%	2,2%	0,6%	0,4%	0,4%	0,2%	1,0%	2,0%	0,9%	0,0%	0,3%	100%	

k. Drying machine brand and capacity

As it is mentioned in section 8 *"Washing machine market in Europe"* of this report, the most common drying machine brands are Bosch, ZANUSSI, LG, BALAY, AEG, Whirpool, Siemens, Miele, Samsung and Indesit and 50,8% of the respondents indicated that their washing machine capacity is between 5 and 7 Kg.

Most of the respondents stated to fill the drying machine until it is full (40-58% depending on the type of clothes), from 10% to 24% of the respondents stated (depending on the type of clothes) that they load it until the half of its capacity (see figure below).

[95] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

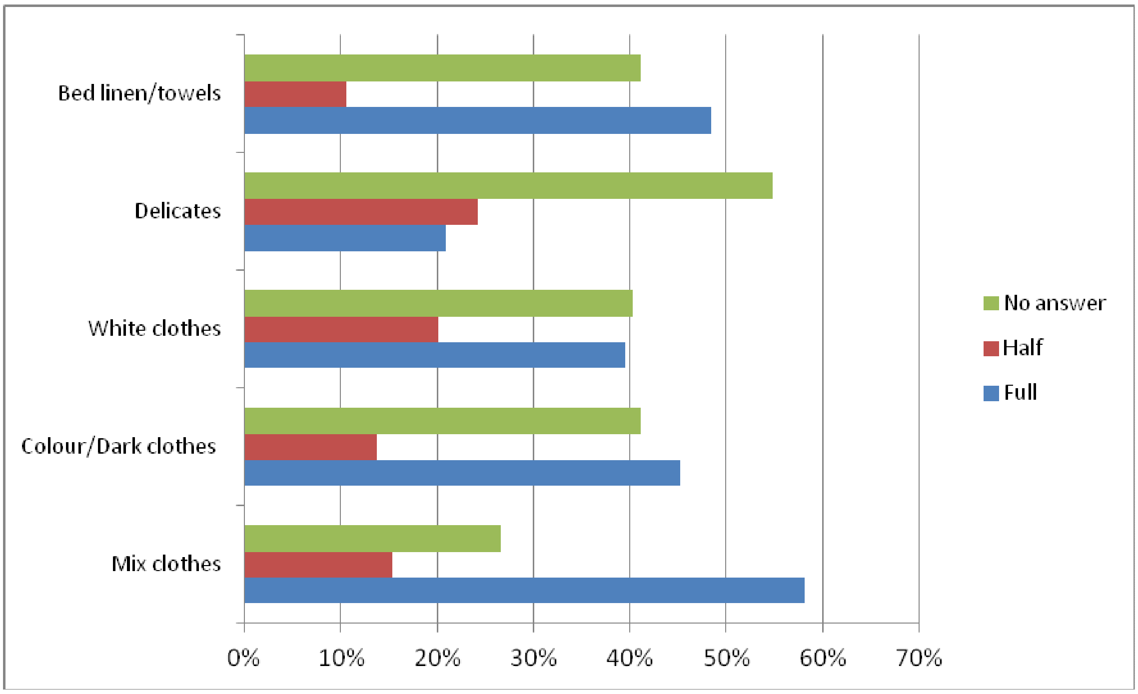

Figure 19. Drying machine average load. [96]

I. Drying machine program

The normal drying program followed by Air Dry and Synthetic programs are the most used by the survey respondents. For delicates, the delicates program predominates and the cotton/towels program is the most preferred for bed linen /towels after the normal program, (see table and figure below).

Table 16. Drying machine program according to the type of clothes. [97]

	Mix clothes	Colour/Dark clothes	White clothes	Delicates	Bed linen/towels
Air Dry	9,7%	8,1%	5,6%	10,5%	4,8%
Cotton / Towels	10,5%	2,4%	12,1%	0,8%	25,0%
Delicates	2,4%	4,0%	2,4%	19,4%	0,0%
Eco/quick dry	4,0%	2,4%	4,0%	0,0%	0,0%
Normal	37,1%	35,5%	29,8%	11,3%	27,4%
Synthetic	11,3%	7,3%	3,2%	0,0%	0,0%
Other	10,5%	7,3%	8,1%	6,5%	8,1%
No answer	14,5%	33,1%	34,7%	51,6%	34,7%

[96] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[97] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

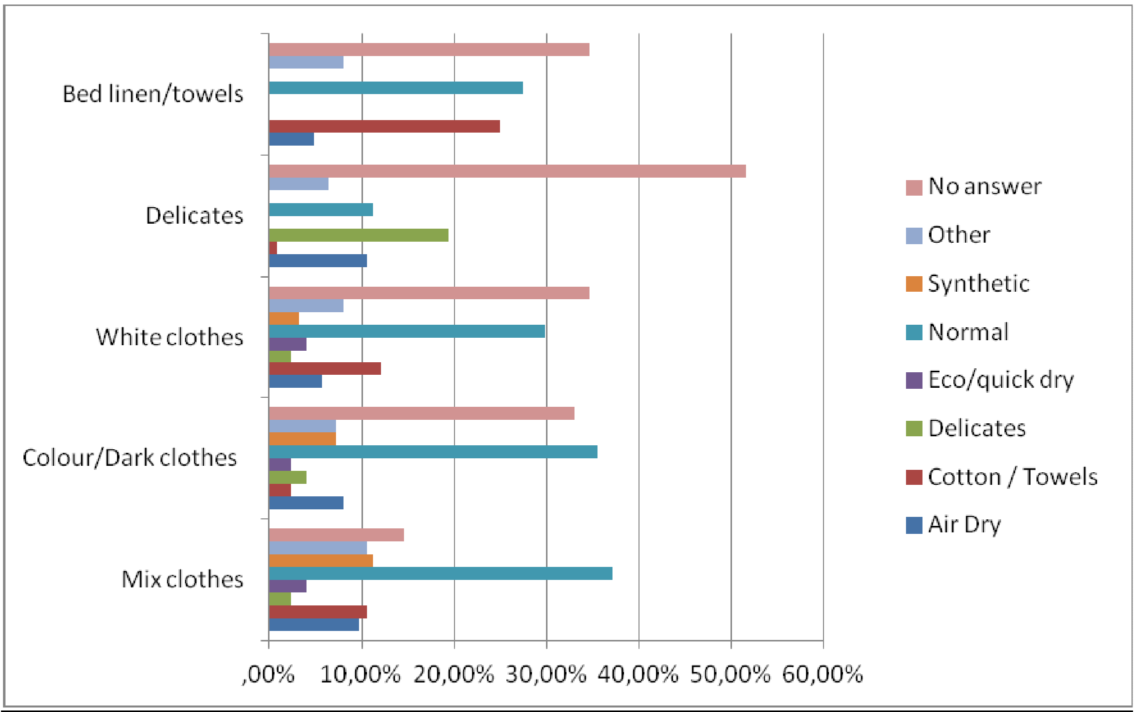

Figure 20. Drying machine program according to the type of clothes. [98]

Concerning drying program duration, this is usually comprised between 1h and 1h and 45min for bed linen /towels, colour/dark clothes and mix clothes. Regarding delicates and white clothes, the programs used are usually shorter, around 15-45min (see figure below).

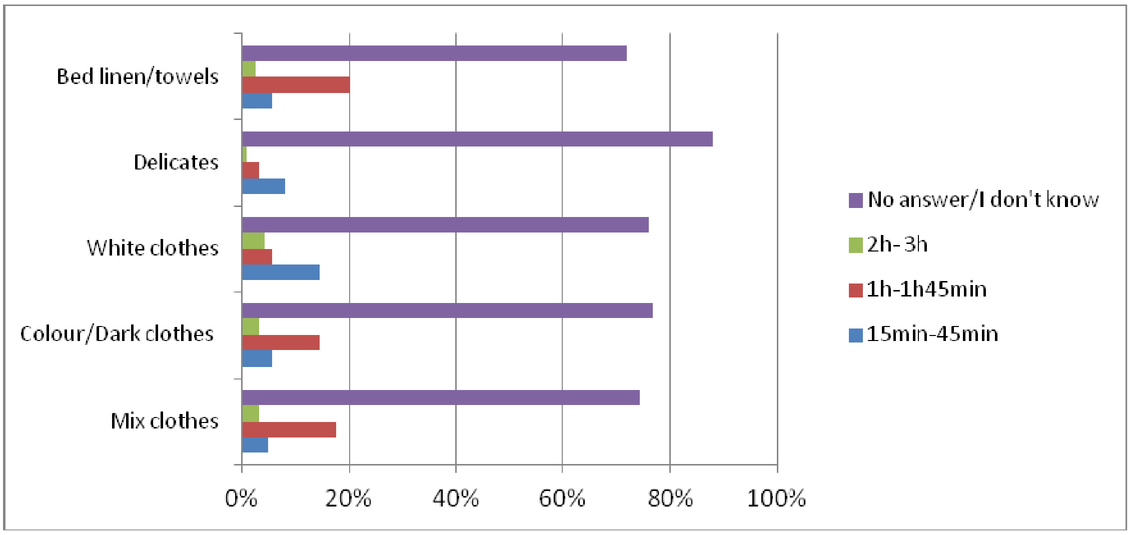

Figure 21. Drying machine program duration. [99]

Regarding special programs, 29-32% of the respondents never use iron aid program (or similar) while 13%-24% always use it. Refresh clothes program is not very used and 33-43% of the

[98] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[99] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

respondents stated that they never use it. The anti-crease program it is not highly used but a high percentage of the respondents did not answer this question (see tables below attached).

Table 17. Use of the iron aid program (or similar). [100]

	Always (70%-100%)	Sometimes (40%-70%)	Very Little (	Never (0%)	No answer/I don't know
Mix clothes	24,2%	9,7%	4,0%	35,5%	26,6%
Colour/Dark clothes	15,3%	9,7%	3,2%	32,3%	39,5%
White clothes	20,2%	8,1%	2,4%	32,3%	37,1%
Delicates	12,9%	5,6%	2,4%	29,8%	49,2%
Bed linen/towels	15,3%	5,6%	1,6%	36,3%	41,1%

Table 18. Use of the refresh clothes program (or similar). [101]

	Always (70%-100%)	Sometimes (40%-70%)	Very Little (	Never (0%)	No answer/I don't know
Mix clothes	11,3%	9,7%	8,1%	43,5%	27,4%
Colour/Dark clothes	8,1%	8,9%	6,5%	39,5%	37,1%
White clothes	8,1%	8,1%	2,4%	39,5%	41,9%
Delicates	4,8%	4,8%	4,8%	33,1%	52,4%
Bed linen/towels	5,6%	6,5%	2,4%	43,5%	41,9%

Table 19. Use of the anti-crease program (or similar). [102]

	Always (70%-100%)	Sometimes (40%-70%)	Very Little (	Never (0%)	No answer/I don't know
Mix clothes	2,4%	4,8%	0,8%	20,2%	71,8%
Colour/Dark clothes	2,4%	4,0%	1,6%	20,2%	71,8%
White clothes	4,0%	3,2%	0,0%	19,4%	73,4%
Delicates	1,6%	3,2%	0,8%	12,9%	81,5%
Bed linen/towels	1,6%	4,0%	0,0%	22,6%	71,8%

5. Professional/industrial laundry

Definitions

The following distinction can be made within the laundry industry[103]:

- According to their location:
    - On Premise-Laundries (OPLs): laundries on the same site as the customer they serve. These are typically found in hotels, hospitals, cruise ships, residential homes, factories, etc.
    - Central Laundries (CL): laundries located away from their customers.
- According to their property:
    - Linen Suppliers: laundries which rent out textiles.
    - Laundries which primarily wash the customers' own textiles (which can be both On Premise and Central Laundries).
- According to their dimension:
    - Heavy Duty Laundries (HDLs): the largest industrial laundries.
    - Professional Laundries (PLs): the smallest institutional laundries.

100 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

101 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

102 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

103 Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

"The limit between HDLs and PLs is at approximately 15 metric tons of work per week. Professional Laundries are typically college laundries and coin operations, where the machines are smaller than in Heavy Duty Laundries, but not as small as in the households. Machine sizes (measured on washer extractors) in the Professional and Heavy Duty Laundries range from 5 kg. up to 450 kg. per batch." [104]

- According to their production methods:
    - pool-laundries: primarily wash clothes in large pools of similar, non-customer-specific textiles (equivalent to stock production in other industries)
    - portion-laundries: primarily wash the textiles in customer-specific-portions (the equivalent to make-to-order production).

The most typical textiles washed by the laundry industry are the following:

Table 20. Textiles in all shapes and sizes: examples of uses. [105]

Carrying	Hygienic	Protection	Functional	Orna-mental	Production
nightwear	towels	duvet covers	sails	curtains	dying
underwear	towel rolls	pillowcases	straps	gobelins	bleaching
skirts	washcloths	sheets	parachutes	neck ties	embossing
socks	bath mats	barrier sheets	flags	bow ties	finishing
gowns	diapers	operating table covers	rucksacks		impregnation
petticoats	handkerchiefs	burial sheets	sleeping bags		
trousers	wash leathers	shroud	body bags		
t-shirts	dishtowels	table cloths	straitjackets		
shirts	dishcloths	table mats			
jerseys	floor cloths	napkins			
blouses	mops	carpets			
coats	yarns	mats			
gloves	protective masks	upholstery			
scarfs	caps	blankets			
uniforms	hairnets	shower curtains			
cloak	smocks	aprons			
cape					
chasubles					

Global industrial laundry market

Global industrial laundry market is not very balanced between Eastern and Western countries. Out of approximately 5.500 production units in the western countries, the majority are located in Germany and the USA. The major part of the 18.500 production units in the Eastern countries are located in China. The following graphics show the current relative global distribution of industrial laundries (production units).[106] The European countries included in this study (Germany, UK, France, Italy, Spain, Netherlands, Sweden, Denmark, Norway and Finland) account for 77% of production units, that is, 4.235 production units.

104 Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

105 Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

106 Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. Danish edition, 2004. English edition, 2014

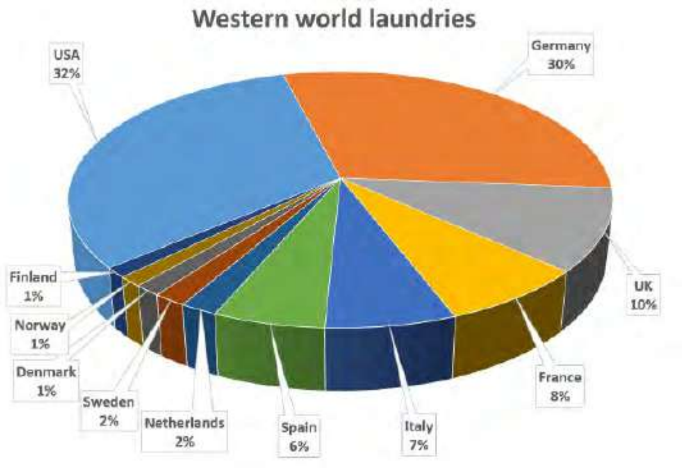

Figure 22. The distribution of industrial laundries in production units in the western world. [107]

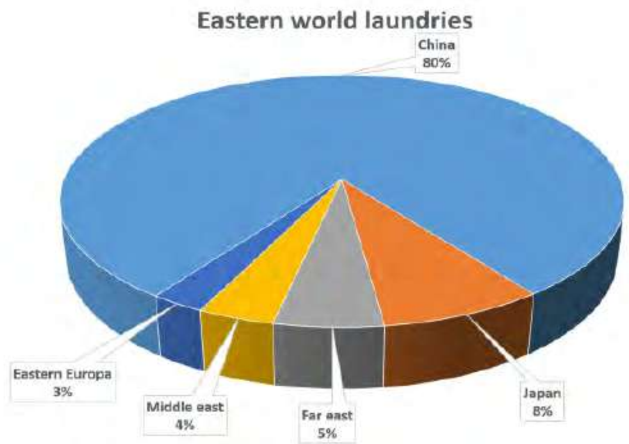

Figure 23. The distribution of industrial laundries in production units in the Eastern world. [108]

"In total, there are an estimated 24,000 heavy-duty laundry production units globally (i.e. laundry operations with a production volume of more than 15 tons per week), which together wash some 33 million tons of textiles annually. In addition, at least some 65,000 professional laundries (units producing less than 15 tons weekly) wash approximately 21 million tons of textiles annually, and a, albeit un-known, very large number of private household washing machines." 500 million m³ of clean, fresh drinking water is used every year for washing processes.[109]

[107] Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

[108] Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

[109] Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

Concerning the laundry industry, the following figures show the turnover and employees at different scales:

- A typical western laundry production unit turns over approximately €3 million per year, with approximately 60 employees in the production.[110]
- The variable costs in the laundry average at approximately 70% of the turnover. Depreciation costs in average approx. 5%.[111]
- In Europe alone, industrial laundries turnover approximately €10.7 billion together and employ approx. 134,000 people.[112]
- In the Western world, industrial laundries turnover some €15 billion annually and employ approximately 350,000 employees.[113]
- Worldwide, the industrial laundries turnover approximately €25 billion and employ some 1.1 million employees.[114]

The main associations in Europe are the following:

- **TSA** - Textile Services Association, Great Britain http://www.tsa-uk.org/
- **ETSA** – European Textile Services Association, Europe http://www.etsa-europe.org
- **WIRTEX** - Wirtshaftsverband Textil Service, Germany http://www.wirtex.de
- **GEIST** - Groupement des Entreprises Industrielles de Services Textiles, France. http://www.geist.fr
- **ASSOSISTEMA** – Associazione Sistema Industriale Integrato Servizi Tessili e Medici Affini, Italy, http://www.assosistema.it
- **FTN -** Federatie Textielbeheer Nederland, Holland http://www.ftn-nl.com
- **FLB** - Fédération des Loueurs de Linge de Belgique, Belgium http://www.fbt-online.be
- **ST** - Sveriges Tvätteriforbund, Sweden http://www.tvatteriforbundet.se
- **BVT** – Brancheforeningen for vask og tekstiludlejning, Denmark www.danskevaskerier.di.dk
- **NRV** – Norsk Renseri- & vaskeriforening, Norway http://www.nrv.no/
- **ADELMA -** Asociación de empresas de Detergentes y de productos de Limpieza, Mantenimiento y Afines (household and industrial) http://www.adelma.es **TRSA** - Textile Rental Services Association, USA http://www.trsa.org/

Rental textile market size[115]

[110] Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

[111] Jensen Group - http://www.jensen-group.com/

[112] ETSA 2011. http://www.textile-services.eu/

[113] Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

[114] Steen Søgaard, Laundry Logics, on. Laundry Operations. Published in the UK in association with TSA - Textile Services Association. English edition, 2014

[115] Deloitte. Quantifying the opportunity European Market Sizing Study for ETSA (June 2014)

The European trade association for the textile services industry (ETSA) represents textile rental companies, suppliers of detergents, fabrics and machinery, textile service associations and working research institutes.

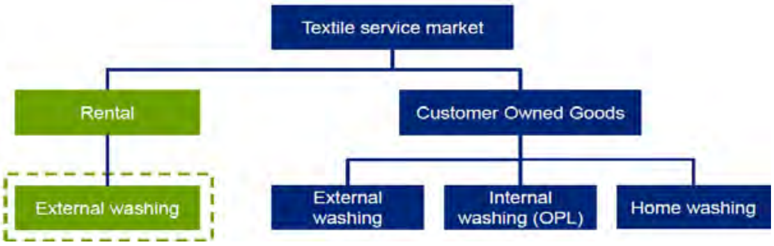

Figure 24. Textile service market. [116]

The potential rental textile market size in Europe was estimated by Deloitte in 2014. The scope included:

- 30 countries: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, United Kingdom.
- 4 market segments: hotels, restaurants, healthcare, industry/trade/services – ITS.
- 5 product ranges: flat linen, workwear, washroom, dust control, cleanroom.

Data was provided by ETSA members, and the total European textile rental market was estimated to be between €10.5 and €11.5bn (see graphic below).

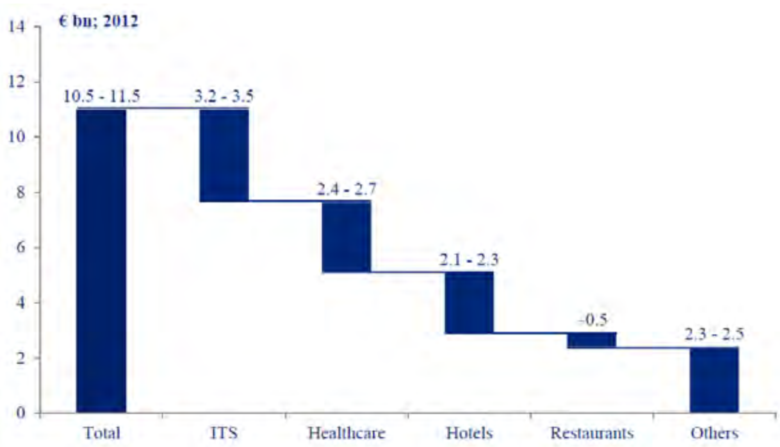

Figure 25. European textile rental market by segment.[117]

[116] Deloitte. Quantifying the opportunity European Market Sizing Study for ETSA (June 2014)

[117] Deloitte. Quantifying the opportunity European Market Sizing Study for ETSA (June 2014)

The tables below show rental textile market current size and potential growth and trend by segment service and the European textile rental market by segment and product.

Table 21. European textile rental market figures by segment 118

**Note: CAGR: Compounded average growth rate. CAGRs, obtained from companies' data, may be overvalued due to acquisitions completed during the period. CAGRs and trends based on data given by ETSA member companies.*

Segment	What does this segment include?	Current size (billion Euro)	Growth* (2008-2012)	Trend (2012-2016)
Industry/Trade/Services	ITS consists of flat linen, industrial, corporate and services workwear and cleanroom but excludes washroom, mats and mops and dry cleaning. ITS is the largest segment and represents 30% of the overall textile rental market.	3.2-3.5	4%	Favourable outlook
Healthcare	Healthcare designates products for hospitals, operating rooms and retirement and elderly care. It consists mostly of flat linen products but also includes staff and kitchen work wear.	2.4-2.7	1.7%	Favourable outlook
Hotels	As its name suggests, hotels designates products for the hotel industry. It consists mainly of flat linen and corporate workwear	2.1-2.3	1.1%	Favourable outlook
Restaurant	The restaurant segment consists almost exclusively of flat linen and workwear.	0.5	2.3%	Favourable outlook
Others	Others" includes all the items that do not fit into the first four segments (e.g. washroom, mats, wipers, etc.).	2.3-2.5	-	-

[118] Deloitte. Quantifying the opportunity European Market Sizing Study for ETSA (June 2014)

Table 22. European textile rental market figures by product[119]

**Note: CAGR: Compounded average growth rate. CAGRs, obtained from companies' data, may be overvalued due to acquisitions completed during the period. CAGRs and trends based on data given by ETSA member companies.*

Product	What does this segment include?	Current size (billion Euro)	Growth* (2008-2012)	Trend (2012-2016)
Flat linen	Flat linen is a category of products that includes bedspreads, sheets, pillowcases, mattress pads, blankets, towels and shower curtains, table linen, table skirting, napkins, place-mats and kitchen towels.	4.3-4.9	2.1%	Favourable outlook
Workwear	The workwear category consists of industrial, services and corporate workwear. It excludes protective equipment such as helmets, eye protection, shoes and cleanrooms.	3.5-3.8	3.8%	Favourable outlook
Washrooms	Washroom products include textile towels and other products and services such as soap dispensers, feminine hygiene, toilet paper or air fresheners.	1.3-1.4	0.3%	Weak outlook
Dust control	Dust control includes products related to floor cleaning (e.g. mats and mops).	1	3.3%	Favourable outlook
Cleanroom	A cleanroom is an environment that has a low level of environmental pollutants such as dust, airborne microbes, aerosol particles and chemical vapors. Cleanroom products are suited for specific types of customers (e.g. hospitals, pharmaceutical sector, micro-electronics, etc.).	0.35	7.5%	Favourable outlook

[119] Deloitte. Quantifying the opportunity European Market Sizing Study for ETSA (June 2014)

Within the framework of this study, Deloitte calculated the following two different scenarios for potential European textile market:

1. Conservative scenario: potential market between €21.5 bn and €26bn.
2. Aggressive scenario: potential market between € 38 bn and € 46 bn.

Textile rental service industry has a significant economic growth and an important contribution to the employment creation at EU level. This sector is currently mostly located in the western European countries.

Workwear and flat linen laundry resources consumption

ETSA carried out a study in 2012 in order to assess workwear and flat linen laundries in 2012. Resources analyzed were: water, electricity, chemicals and oil or gas for laundering. Production volumes were taken into account in order to calculate the average consumption. With this aim in view they have carried out a survey among 96 workwear plants and 63 flat linen plants, covering consumption for the year 2011. Although there is some variations in the textile process in both laundries, results were gathered as one group[120]:

- Workwear laundry includes: workwear (trousers and jackets), protective personal clothing, business wear, shirts, underwear and gloves. The main groups of clients are: industries and shops (representing different levels of soiling).
- Flat linen laundry includes many different kind of textiles, e.g. sheets, towels and table cloths. The main groups of clients are: hotels, restaurants and hospitals.

The following table shows the results of the average consumption of workwear and flat linen laundry industries taking into account production volumes.

Table 23. ETSA survey results on workwear and flat linen laundry plants consumption.[121]

2011 survey, average for all plants	Units	Workwear, 2011		Flat Linen, 2011	
		Weighted average	*Number of plants*	Weighted average	*Number of plants*
Water per kg	l/kg	14,4	*96*	10,6	*63*
Electricity per kg	kWh/kg	0,28	*96*	0,21	*63*
Chemicals per kg	g/kg	37	*81*	17,9	*43*
Oil/gas per kg	kWh/kg	1,46	*96*	1,18	*63*
Delivery:					
Fuel per kg laundry	l/kg	0,04	*26*	0,02	*29*
Driving efficiency	l/100 km	10,8	*26*	9,3	*10*
Delivery distance	km/kg	0,34	*49*	0,20	*21*

120 ETSA Survey on resource consumption in workwear laundries and flat linen laundries in 2011 – 'WECO 3' . FINAL 8 January 2013 Prepared by EcoForum / Henrik Grüttner.

121 ETSA Survey on resource consumption in workwear laundries and flat linen laundries in 2011 – 'WECO 3' . FINAL 8 January 2013 Prepared by EcoForum / Henrik Grüttner.

The consumption of production of laundry flat linen is significantly lower than the consumption for processing workwear most likely due to the higher soiling level. On the other hand, tunnel washers or continuous batch washers are efficient equipment used by the flat linen industry.

Workwear plants were previously surveyed in 2007 and results are shown in the table below. As it can be observed laundry processes performance have generally improved in terms of energy consumption but chemicals and water used have increased by 18% and 2% respectively. Concerning chemical use, explanations pointed by ETSA include the differences in chemistry (e.g. water content, bleach type, active components, etc.), changes in applied technology and/or a higher quality market demand.

Table 24. Comparing resource consumption for the group of laundries which participated in both the 2011 and 2007 surveys, illustrating the changes in the weighted averages. Note: this table only includes the 32 laundries that were surveyed in both 2011 and 2007. [122]

Workwear, comparing 2011 and 2007	Units	Weighted average 2011	Weighted average 2007	% difference
Water per kg	l/kg	15,1	14,8	+2%
Electricity per kg	kWh/kg	0,39	0,41	-5%
Chemicals per kg	g/kg	43,6	37,1	+18%
Oil/gas per kg	kWh/kg	1,38	1,59	-13%

A similar survey was carried out by ETSA in 2001 and a Life Cycle Assessment in 1999 showing, respectively, a water consumption of 19 l/kg. and 18 l/kg. Taking into account the data mentioned above, water consumption has stabilized around 14-15l/Kg.

Nevertheless, there are efficiency differences among workwear laundries. The best 20% laundries in terms of performance show a stable water consumption of 10 l/Kg between 2007 and 2011.

This study also gathered information on water supply and waste water of a third of the surveyed laundry plants. In this sense, they concluded that the majority of laundries use public supply water and approximately 40% of the workwear laundries have installations for pH neutralisation, and around 50% have a kind of extended wastewater treatment. Concerning flat linen laundries, around 25% have pH neutralisation and around 20% have some form of extended wastewater treatment. Wastewater treatment installations usually recycle part of the wastewater consumed but increase electricity consumption.

Some commitments of ETSA that can be taken into account when analyzing the data presented above, are the following:

- *"ETSA members strive to use environmentally-friendly washing detergents. Only detergents which minimise the impact on the environment are selected.*

[122] ETSA Survey on resource consumption in workwear laundries and flat linen laundries in 2011 – 'WECO 3' . FINAL 8 January 2013 Prepared by EcoForum / Henrik Grüttner.

- *ETSA member companies ensure that their waste and wastewater fully meet the legal regulations in their respective countries. The sludge from the wastewater treatment is disposed of in a responsible manner.*
- *The main waste from textile rental is the disposed textiles themselves. ETSA members strive to re-use textiles whenever possible. Otherwise, textiles are recycled or used as raw materials in energy production. ETSA members monitor their waste volumes closely.*[123]

6. Consumption of laundry products according to consumers

According to AISE, laundry products market (EU28 plus Norway and Switzerland) accounted for, based on Euromonitor International data, €13,745 million in 2013 with a growth of 0,9% between 2012 and 2013. This figure is distributed in the following products:

- Powder detergents: 3,411 million Euro
- Liquid detergents: 4,238 million Euro
- Unit Doses 936 million Euro
- Fabric Conditioners 2,335 million Euro
- Laundry Aids, Others 2,825 million Euro[124]

Households' laundry care products include laundry detergents, fabric softeners, carpet cleaners and laundry aids. Concerning industrial laundry products, these comprise laundry detergents, fully formulated detergents, powder/liquid detergents, pre-wash additives, boosters, pH-adjustment, water hardness regulators, bleach additives, disinfectant detergents/additives for hygienic laundry (hospital, food industry), fabric softeners, starch finishing, ironing aid, fragrance rinse, etc.[125]

Concerning the global/international laundry care products industry, it is estimated to reach a revenue of $85 billion in 2017 with a compound annual growth rate of 5.1% during the next 5

123 ETSA : http://www.textile-services.eu/our_commitment/

124 AISE http://www.aise.eu/our-industry/market-and-economic-data.aspx

125 AISE http://www.aise.eu/documents/document/20131014165516-product_categories_2013.pdf

years. This growth is attributed to rising standards of living, population increasing and purchase growing.[126]

According to the results received by means of the MERMAIDS' survey answers, although many different brands are bought by European consumers, Ariel, Dash and Skip are the most used detergent brands (see table below).

Table 25. Brant of detergent bought by European consumers. [127]

DETERGENT	NUMBER OF RESPONDENTS	%
CARREFOUR	4	1%
Día	4	1%
FAIRY	4	1%
lidl	4	1%
No answer	4	1%
MIR	5	1%
Omino Bianco	5	1%
Robijn	5	1%
xtra	6	1%
Colon	7	1%
marseille	8	2%
Ecover - froggy	9	2%
persil	10	2%
Wipp	10	2%
omo	11	2%
Le chat	16	3%
Cheapest / on sale	17	3%
Dixan	24	5%
Bosque verde	30	6%
Skip	39	8%
Dash	46	9%
Ariel	103	20%
other	149	29%
	520	100%

Concerning the format of the detergent used, liquid is preferred, followed by powder and tablets (see figure below).

[126] Business Wire. Research and Markets http://www.businesswire.com/news/home/20130425006772/en/Research-Markets-Global-Laundry-Care-Products-Industry#.VG8JtsmmVZo

[127] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

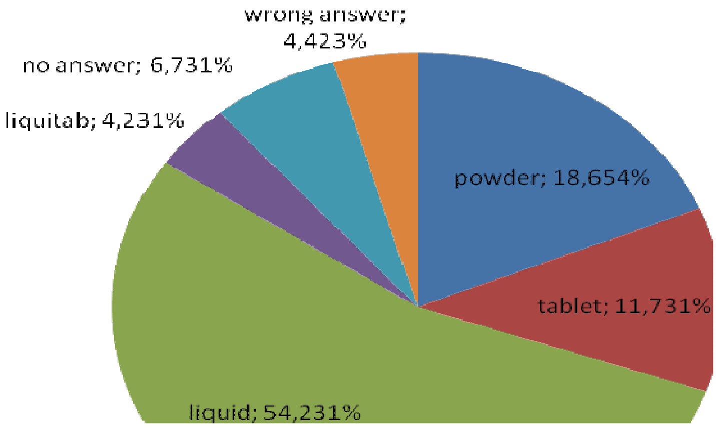

Figure 26. Detergent format. Wrong answer indicates that the respondent answered none of the possible categories.[128]

Concerning the laundry products used by European citizens, they depend on the different type of clothes. Regarding mix clothes detergent is used by most of the respondents while starch and clothes refreshers are rarely used. Softener is used by 47% of the respondents (see table and figure below).

Table 26. Frequency of laundry products used for washing mix clothes. [129]

	Mix clothes						
	Detergent	Softener	Bleach	Stain Remover	Clothes refreshers	Starch	Other products
Always (70-100%)	88%	47%	1%	2%	2%	0%	3%
Sometimes (40-70%)	4%	12%	9%	17%	2%	1%	2%
Very Little (1-40%)	1%	7%	15%	27%	3%	2%	2%
Never (0%)	2%	19%	45%	25%	56%	59%	41%
No answer	5%	15%	31%	29%	37%	38%	53%

128 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

129 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

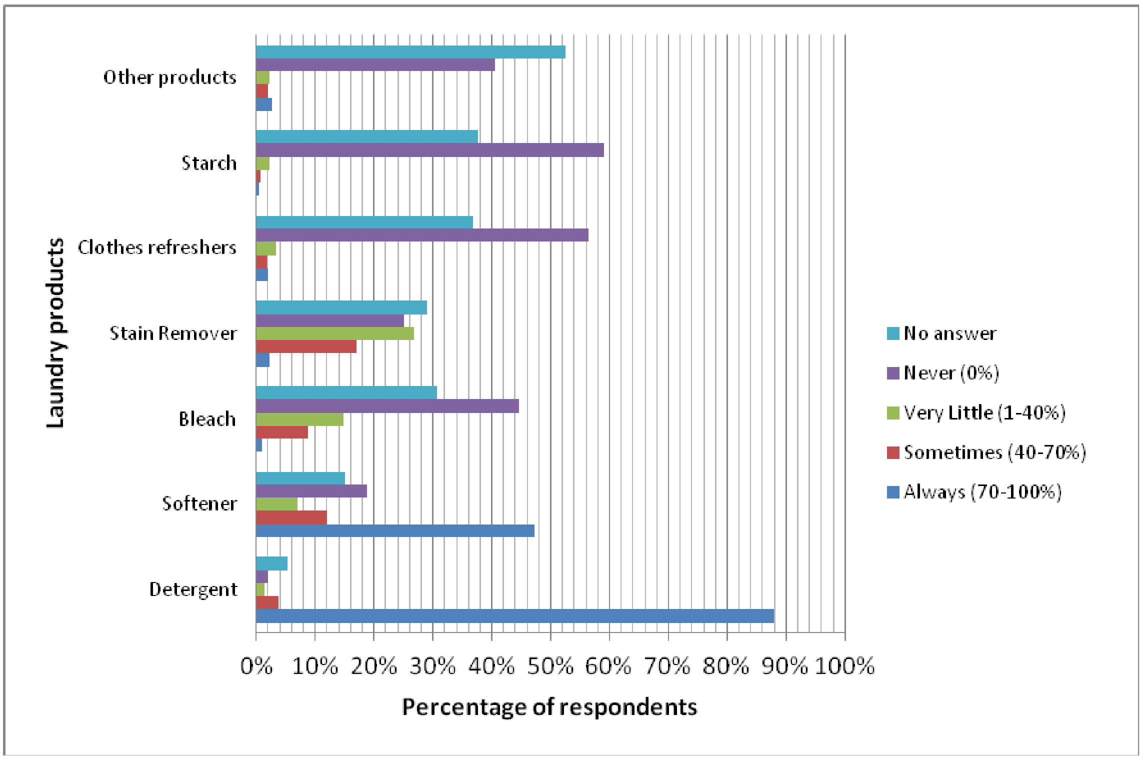

Figure 27. Frequency of laundry products used for washing mix clothes. [130]

Most respondents (84%) use detergent when washing colour/dark clothes and 45% use softener. Bleach, stain remover and clothes refreshers are not used a lot and starch is rarely used (see table and figure below).

Table 27. Frequency of laundry products used for washing colour/dark clothes. [131]

	Colour dark clothes						
	Detergent	Softener	Bleach	Stain Remover	Clothes refreshers	Starch	Other
Always (70-100%)	84%	45%	1%	1%	2%	0%	2%
Sometimes (40-70%)	3%	12%	7%	13%	2%	1%	1%
Very Little (1-40%)	3%	7%	10%	19%	57%	2%	2%
Never (0%)	3%	19%	48%	32%	3%	61%	48%
No answer	7%	17%	35%	34%	36%	37%	48%

130 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

131 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

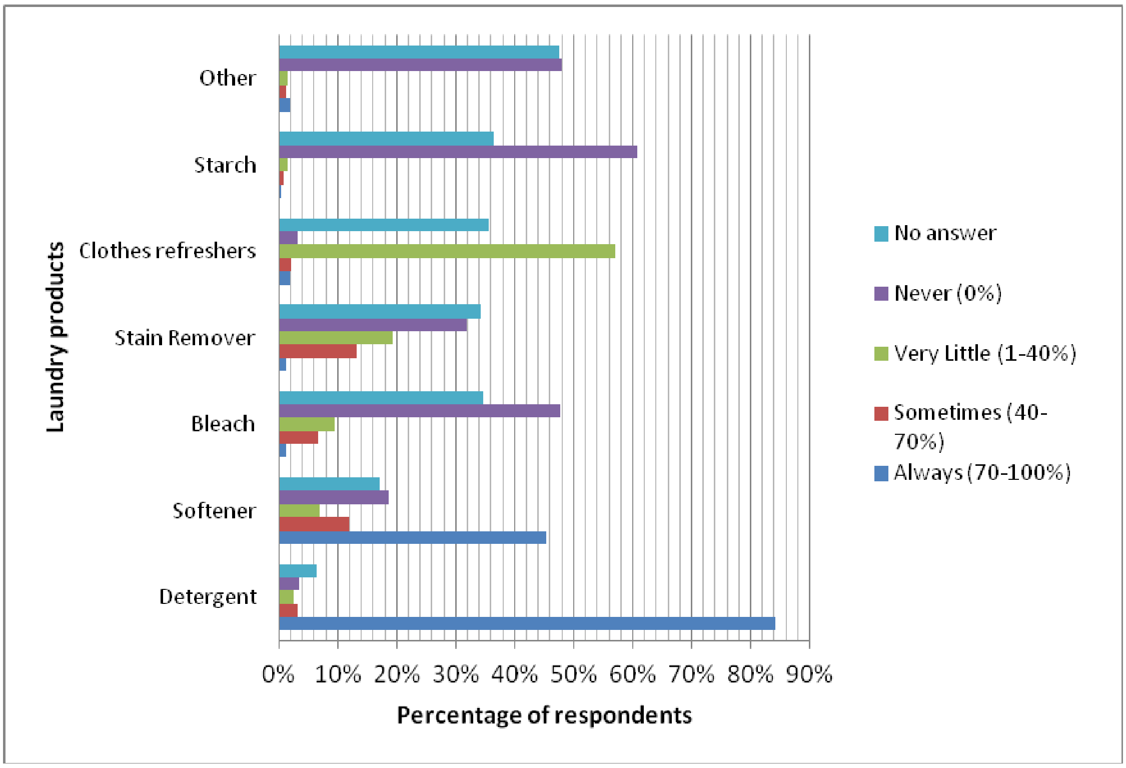

Figure 28. Frequency of laundry products used for washing colour/dark clothes. [132]

Concerning white clothes, the answers received pointed out that most of the people (84%) use detergent and 47% use softener. Bleach and stain remover are not used a lot and clothes refreshers and starch are barely used (see table and figure below).

Table 28. Frequency of laundry products used for washing white clothes. [133]

	White clothes						
	Detergent	Softener	Bleach	Stain Remover	Clothes refreshers	Starch	Other
Always (70-100%)	84%	47%	4%	3%	2%	0%	2%
Sometimes (40-70%)	4%	11%	16%	21%	3%	2%	2%
Very Little (1-40%)	2%	5%	20%	23%	3%	2%	2%
Never (0%)	3%	20%	29%	23%	55%	55%	39%
No answer	8%	18%	31%	31%	37%	41%	55%

132 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

133 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

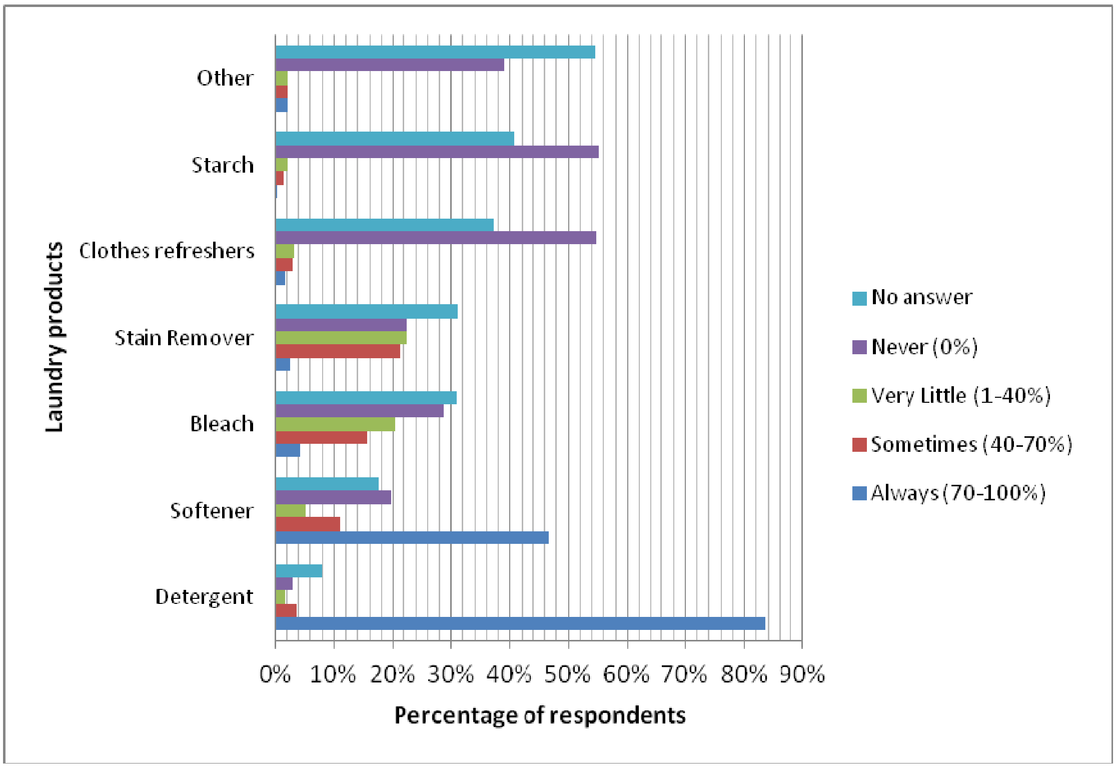

Figure 29. Frequency of laundry products used for washing white clothes. [134]

Detergent is always used by 79% of respondents and softener for 43% of respondents (see the table and figure below).

Table 29. Frequency of laundry products used for washing delicates. [135]

	Delicates						
	Detergent	Softener	Bleach	Stain Remover	Clothes refreshers	Starch	Other
Always (70-100%)	79%	43%	1%	1%	2%	0%	2%
Sometimes (40-70%)	4%	12%	7%	12%	2%	1%	1%
Very Little (1-40%)	3%	6%	10%	15%	3%	2%	2%
Never (0%)	4%	18%	44%	34%	51%	53%	45%
No answer	10%	21%	38%	38%	43%	44%	50%

134 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

135 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

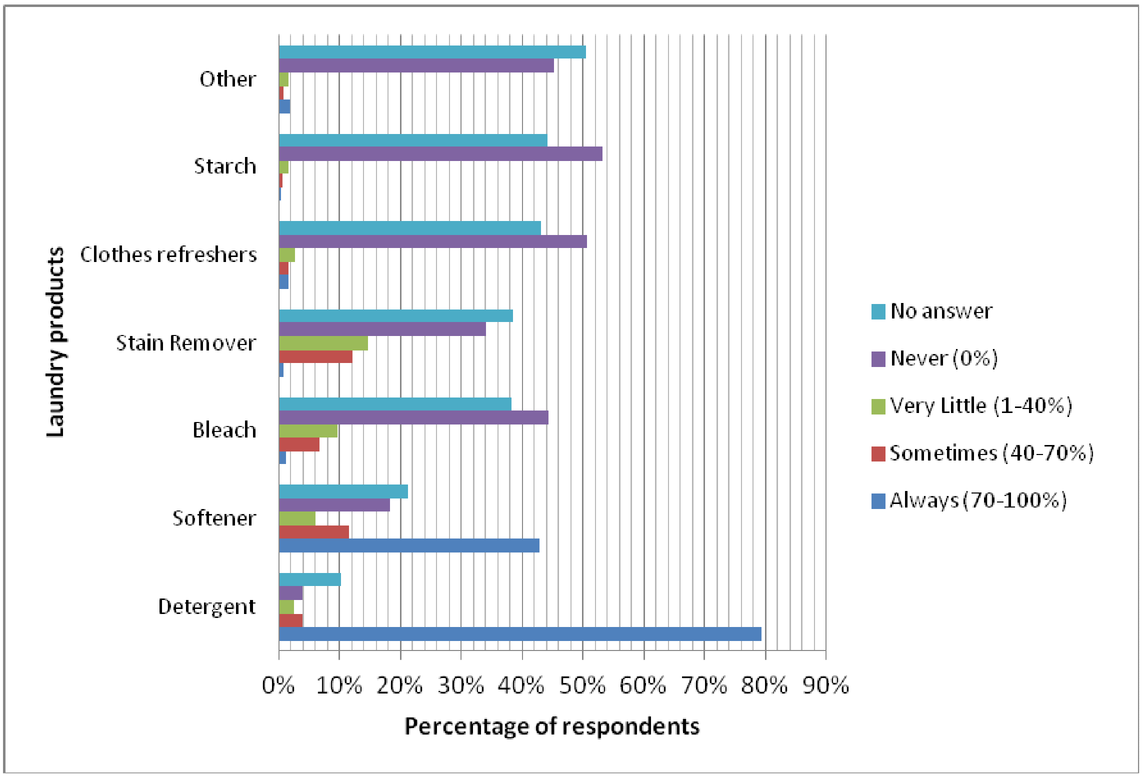

Figure 30. Frequency of laundry products used for washing delicates. [136]

84% of respondents always use detergent for bed linen/towels washing and 49% always use softener. The other laundry products are barely used (see table and figure below).

Table 30. Frequency of laundry products used for washing bed linen/towels. [137]

	Bed linen /towels						
	Detergent	Softener	Bleach	Stain Remover	Clothes refreshers	Starch	Other
Always (70-100%)	84%	49%	2%	2%	3%	0%	2%
Sometimes (40-70%)	4%	10%	11%	17%	2%	1%	2%
Very Little (1-40%)	1%	4%	18%	19%	4%	2%	2%
Never (0%)	3%	17%	37%	28%	52%	55%	42%
No answer	8%	19%	32%	33%	39%	42%	53%

136 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

137 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

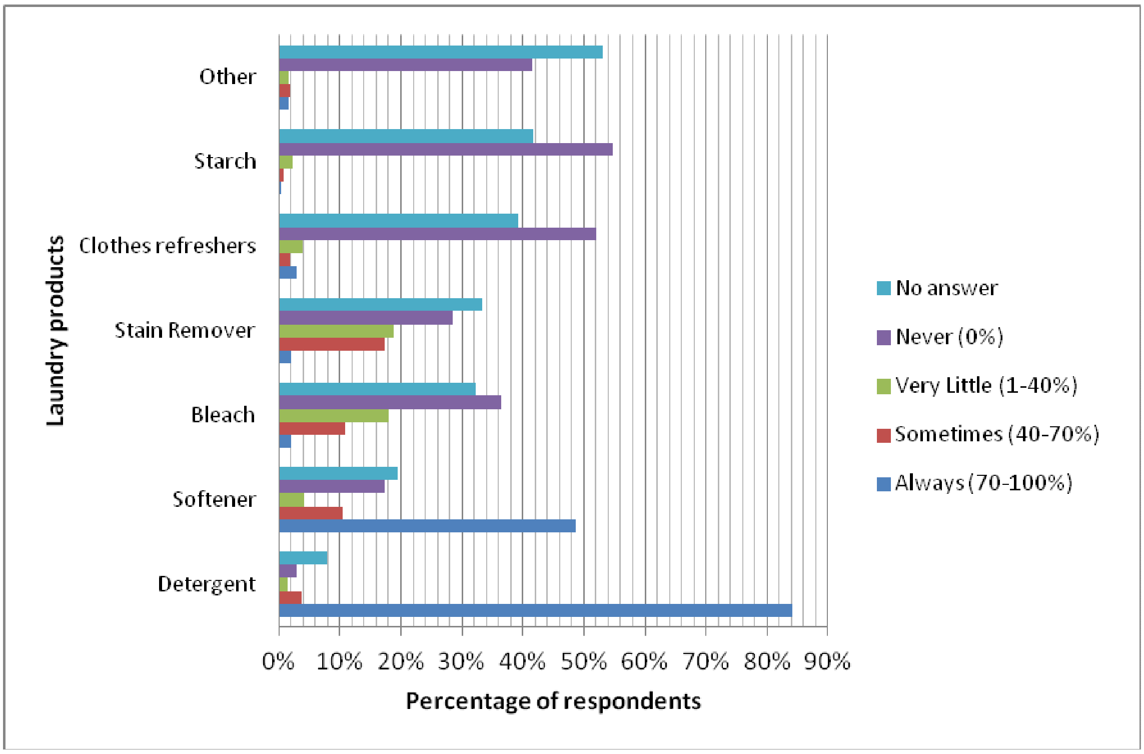

Figure 31. Frequency of laundry products used for washing bed linen/towels. [138]

7. Consumption of clothing and textile garments

Textile fibres

Global textile fibres production in 2006 was of 74.7 million metric tons distributed as shown in the figure below. Synthetic fibre production accounts for 41.4 million metric tons.

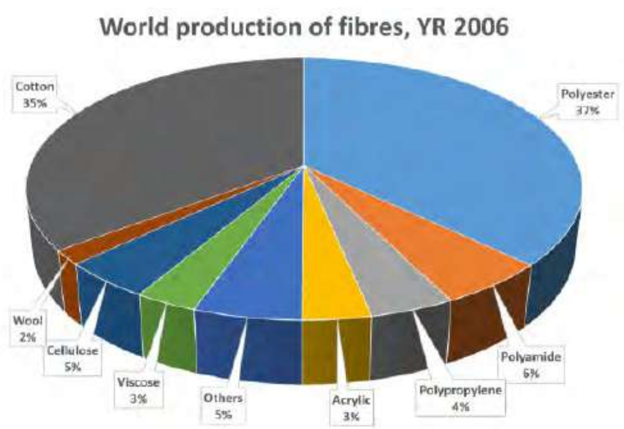

Figure 32. The relative distribution of textile production in the world. [139]

138 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

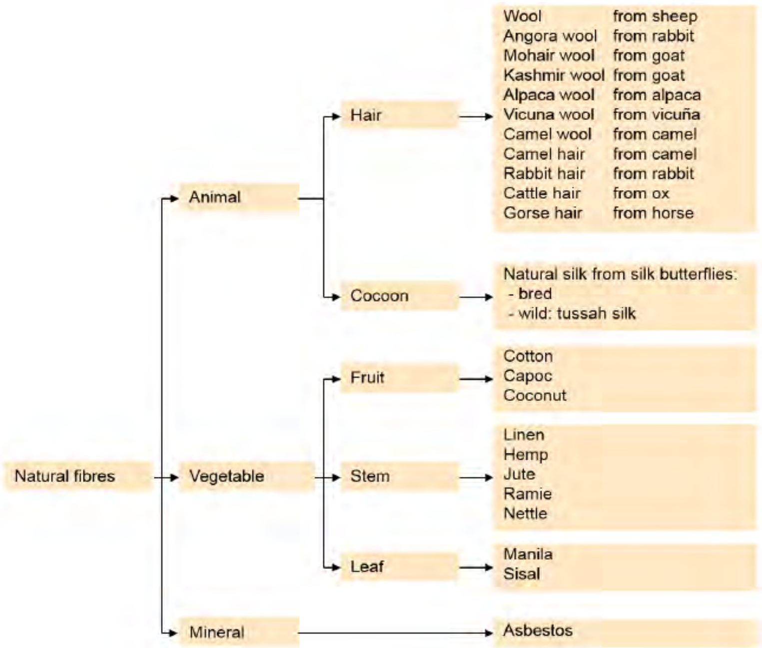

Figure 33. Natural fibers

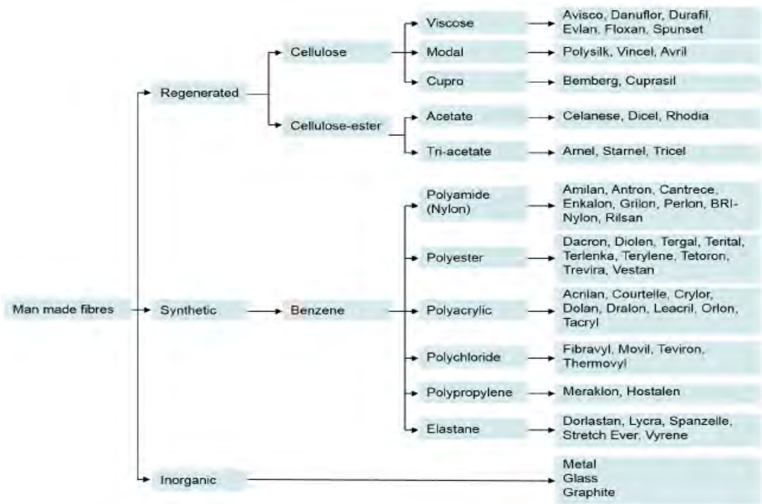

Figure 34. Man-made fibers

Clothing industry

Clothing and footwear accounted for 5,4% of the EU-27 households' expenditures in 2011(800 Euros per capita). However, clothing is among those households' expenditures most affected by crisis.[140] Nevertheless, an increase of clothing consumption is expected by 2020.[141]

139 Oerlikon Saurer Textile

140 EUROSTAT. Analysis of EU-27 houshold final consumption expenditure. 2013.

141 Technopak. Global Changes In Clothing Consumption By 2020, And Their Impact On Fibre-Manufacturer Supply Chains. 2012.

Despite large efforts made to improve the environmental impacts in the manufacturing and consumption of textile and clothing over the last 25 years, the overall volume of production and consumption of these products has increased. Moreover, the deliberately limited lifespan of these products due to the fast cycles of fashion have shortened their lifecycle.[142]

The textile and clothing industry plays an important role to the EU-27 economy and well-being. According to the latest structural data available*," in 2006 there were 220.000 companies employing 2.5 million people and generated a turnover of €190 billion. The textile and clothing sector accounts for 3% of total manufacturing value added in Europe".*[143]

Spain and Italy are among the EU-27 biggest producers in textile and clothing (T&C) industry. As mentioned, southern EU countries (e.g. Italy and Portugal) contribute more to clothing production while northern countries (e.g. UK and Germany) contribute more to the textile production.[144]

"The largest activity within the textile and clothing sector (at NACE Division level) was the manufacture of textiles which accounted for a little under one half (46.3 %) of sectoral value added in 2006." In 2009, the EU-27 textile manufacturing sector was comprised by 60.000 enterprises, employing 700.000 persons with a turnover of 70.000€m.[145]

The textile and clothing market is completely globalized. Extra-EU27 trade data for textile and clothing reflects that *Imports* accounted for 92.007 million Euros and Exports accounted for a 37.408 million Euro, in 2011. Nevertheless, the growth percentage of imports is 14,4% and 2,4% of exports.[146] EU T&C industry has had serious difficulties competing with other countries with less stringent legislation requirements. Production processes have been generally subcontracted or relocated to other countries with lower labour costs. In this sense, European clothing industry has undertaken a modernization and restructuring process in order to increase companies' competitiveness. The main competitiveness advantage of the European clothing industry is the design and the quality of clothing as well as added value products.

The interconnectedness of the industry means that changes in the trade and production of textiles and clothing can have significant positive and negative impacts on national economies, and/or the growth or subsidence of the industry. In general, the T/C industry can be considered

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142 European Commision. An end to fast fashion? Consumer-focused, sustainable alternatives, 2012.

143 European Commission. Enterprise and Industry, 2006. http://ec.europa.eu/enterprise/sectors/textiles/

144 European Commission. The EU-27 textiles and clothing industry. http://ec.europa.eu/enterprise/sectors/textiles/single-market/eu27/

145 EUROSTAT. Textile, clothing, leather and shoe production statistics - NACE Rev. 1.1. http://epp.eurostat.ec.europa.eu/statistics_explained/index.php/Textile,_clothing,_leather_and_shoe_production_statistics_-_NACE_Rev._1.1

146 European Commission. Statistics on textiles and clothing 2006-2011. http://ec.europa.eu/enterprise/sectors/textiles/files/statistics/textiles_en.pdf

a catalyst for economic growth in developing countries, due to its suitability for people with limited skills and education. Despite the health risks and low income, it is often the most economically viable source of employment in areas where other occupations are either not available or do not provide sufficient income.

Conventional textile finishing techniques are wet chemical modifications where water and chemicals are used in large quantities and wastewaters need to be processed before discharging effluent, whereas the most problematic factor are ecological impacts to the environment and effects to human health. Manufacturers have introduced new finishes based on fluorochemicals with shorter fluorinated side-chains, which eliminate PFOA (perfluorooctanoic acid) contamination and, as a result, have a much lower environmental and toxicological impact. Furthermore, a number of companies have developed softening treatments for apparel which allow huge savings on water and energy. Such improvement helps to stimulate market demand by providing opportunities for introducing greater novelty, innovation, higher quality and greater added value to finished materials. In the proposal it will take an eco-moment to consider the environmental friendliness of the textile finishing compounds which will be used. Conventional solvent-based finishes such as nitrocellulose lacquer and varnishes provide a durable, high quality finish at a reasonable cost. However, they can also be significant sources of volatile organic compounds (VOCs) and hazardous air pollutants (HAPs) both in the application and cleanup stages. Solvents released to the air include xylene, toluene, ketones, methanol, methylene chloride, and various mineral spirits.

Clothing consumption

According to the results received by means of the MERMAIDS' survey answers, (see table below), 25% of respondents stated to have 70-80% of their garments/home linen made of natural fibers while 15% stated to have 50% of both kind of textiles/fibers. 58% of the respondents have more natural fibers made garments/home linen while 26,5% have more synthetic fibers made garments/home linen.

Table 31. Percentage of the garments/home linen that respondents have at home by type of fabric (natural/synthetic). [147]

	Nº of respondents	%
0% synthetic; 100% Natural	27	6,5%
1% synthetic; 99% Natural	6	1,4%
2% synthetic; 98% Natural	1	0,2%
5% synthetic; 95% Natural	14	3,4%
10% synthetic; 90% Natural	31	7,5%
15% synthetic; 85% Natural	15	3,6%
20% synthetic; 80% Natural	54	13,0%
25% synthetic; 75% Natural	9	2,2%
30% synthetic; 70% Natural	50	12,0%
35% synthetic; 65% Natural	9	2,2%
40% synthetic; 60% Natural	22	5,3%
45% synthetic; 55% Natural	3	0,7%
50% synthetic; 50% Natural	64	15,4%
60% synthetic; 40% Natural	33	8,0%
70% synthetic; 30% Natural	29	7,0%
75% synthetic; 25% Natural	6	1,4%
80% synthetic; 20% Natural	25	6,0%
90% synthetic; 10% Natural	9	2,2%
95% synthetic; 5% Natural	2	0,5%
100% synthetic; 0% Natural	6	1,4%

43,8% of respondents said that they always check the composition of the garments when shopping, 30,8% do it sometimes, 19,4% very little and 6% of respondents never check clothes composition. When they were asked about the garment composition that they usually buy, a great variety of answers came up (see table below). 36,35% of respondents stated to buy mainly cotton garments but as shown in the table below, most respondents buy a mixture of synthetic and natural fibers.

Table 32. Garment composition usually bought. [148]

	Number of respondents	**%**
Cotton	189	36,35%
Syntetic	34	6,54%
Natural fibers	33	6,35%
Natural and synthetic fibers	5	0,96%
Wool	7	1,35%
Cotton and synthetic	50	9,62%
Cotton and poliester	18	3,46%
Cotton and wool (and in some cases silk/viscose/linen)	36	6,92%
Acrylic and cotton	6	1,15%
Acrylic, silk and cotton	1	0,19%
No answer/Don't know	97	18,65%
All kind	43	8,27%
leader	1	0,19%
	520	100,00%

[147] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

[148] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

8. Washing machine market in Europe

Laundry processes use a certain amount of resources. It has been estimated that "*the global energy and water consumption for automatic laundry washing is at about 100 TWh of electricity and 20 km^3 of water per year*" [149]. On the other hand laundry detergent market is estimated to value 77 billion US$ a year[150].

Washing machines/washes.

According to the ISE Appliances, in Europe over 21 million washing machines are now consumed per year. 18 million of washing machines were produced in 2005151.In the future a large part of the market will draw on the replacement of old appliances, as currently in Europe, about 40 million washing machines are older than 10 years. The average capacity of the household washing machines has changed from about 4,8 kg in 1997 to approximately 5,4 kg in 2005; models of 4 or 5 kg capacity have been replaced by 5 or 6 kg or new models with 7kg capacity. Related to water consumption, while in 1997 the majority of machines had a water consumption of 75 litres, this value is now about 50 litres per cycle. When comparing the average water consumption per kg, has decreased from 13.9 l/kg in 1997 to 9.6 l/kg in 2005.

Initiatives

HERA (www.heraproject.com) is a voluntary industry initiative, lead by AISE, that includes Life Cycle Assessment studies on different ingredients included in household cleaning products. The HERA partnership was established in 1999 and includes household cleaning products manufacturers (from A.I.S.E.) and chemical industry suppliers (CEFIC). In 2013 A.I.S.E. published a reference study on solid (powder) detergents.[152]

Cleanright initiative held in 2008 (www.cleanright.eu - webportal) aims at providing information of laundry products (soups, maintainers to consumers, etc.). Cleanright does not discuss specific brands but provides information on different types/families of products and the benefits each type of product offers.

AISE's campaign "I prefer 30º"[153] aimed at reducing washing temperature used by households by 3º.

[149] Pakula, C. and Stamminger R.: Electricity and water consumption for laundry washing by washing machine worldwide. Energy Efficiency 3 (2010) 365 –382.

[150] Smallwood, P.: Data for 2010 from Euromonitor (private communication, 2012)

[151] .EEA. Discussion Report: EU Ecolabel for Washing Machines, 2009

[152] AISE. The case for the " A.I.S.E. low temperature washing initiative" Substantiation dossier October 2013.

[153] http://iprefer30.eu/

Concerning detergents, AISE has lead many different initiatives such as the AISE Code for Good Environmental Practice (2003) and EU Commission Recommendation 98/480/EC. The Code for Good Environmental Practice enabled to reduce the use of laundry detergents, packaging and poor softeners.

A motion accepted by the majority of the Dutch Parliament (Government in Netherlands) in November 2012, established that microplastics, pharmaceutical residues and chemicals in clothing can be a threat to the quality of surface and drinking water or can increase the costs of water purification. In this sense, The Government in Netherlands requested to advance agreements at European level to reduce these effects; and to Requests government also, in the context of corporate social responsibility:

- *To sit down with market stakeholders to discuss how microplastics can be reduced.*
- *To sit down with market stakeholders to discuss how the release of chemicals from clothing during washing can be prevented.*
- *Confer with hospitals and other health institutions to reduce directly or indirectly pharmaceutical waste to water.*[154]

Regarding the answers received within the survey carried out in the MERMAIDS' project[155], the figure below shows the washing machine capacity indicated by respondents. Around 46% of respondents' washing machines have a capacity between 5 and 6Kg and 44% between 7 and 8 Kg.

154 Vaststelling van de begrotingsstaten van het Ministerie van Infrastructuur en Milieu (XII) voor het jaar 2013. Voorgesteld 27 november 2012. https://zoek.officielebekendmakingen.nl/kst-33400-XII-47.html

155 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

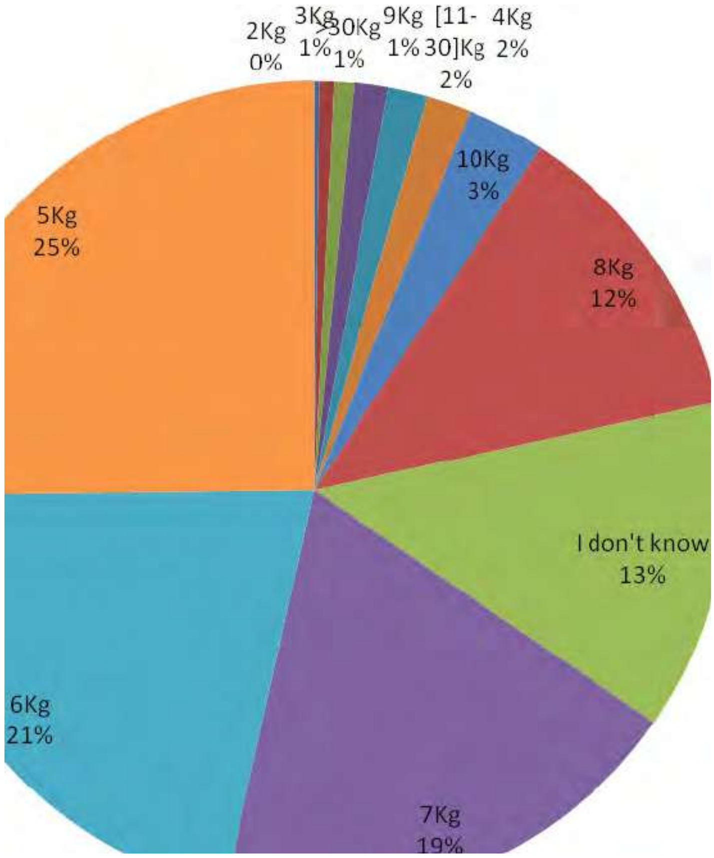

Figure 35. Washing machine capacity. [156]

Regarding the most common washing machine brands bought by European consumers are Bosch, Whirpool, LG, Indesit, AEG, Siemens, Candy, Balay, ZANUSSI, Samsung, Miele, Fagor and Electrolux as shown in the figure below.

156 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

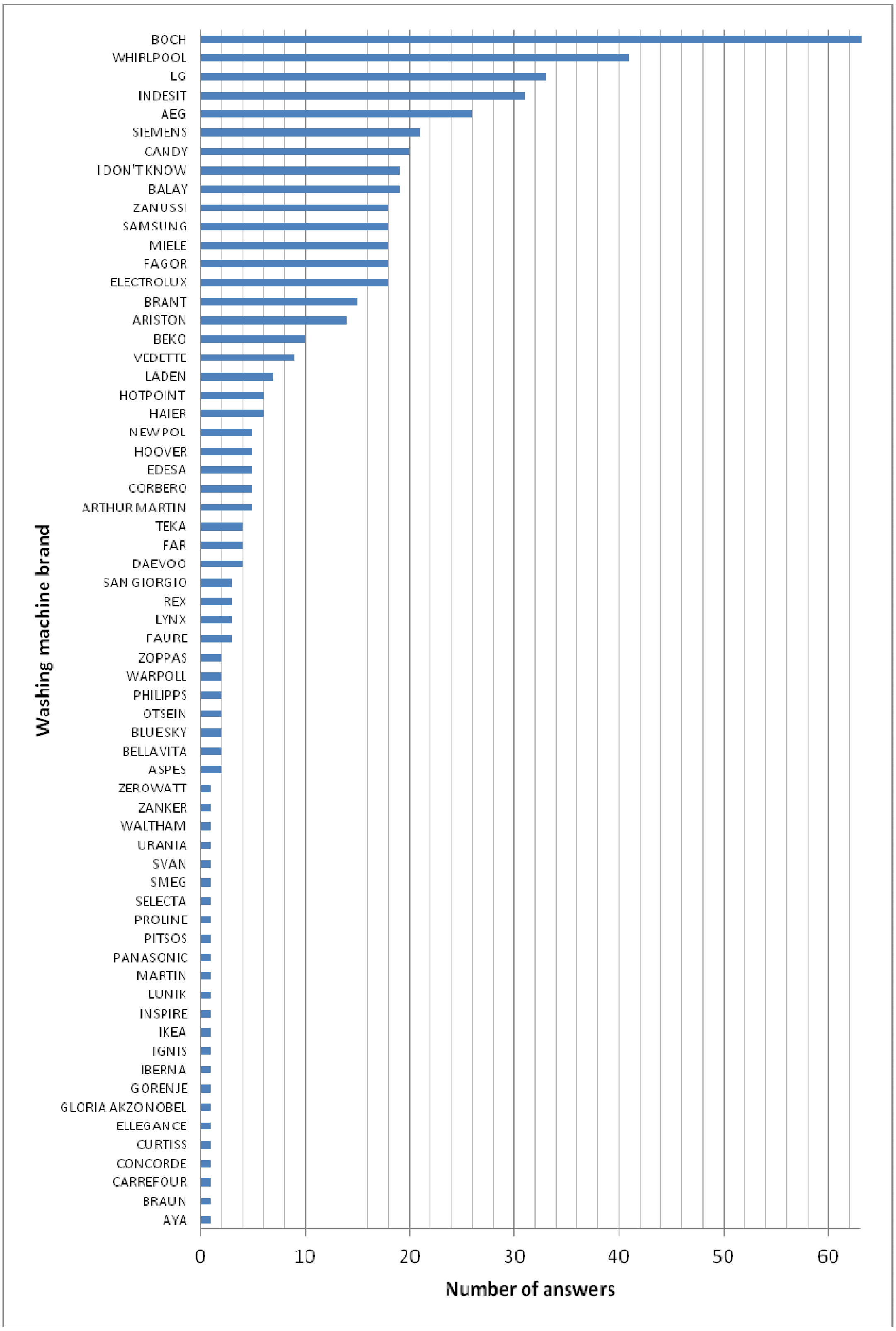

Figure 36. Washing machine brand indicated by consumers. [157]

[157] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

Drying machine

Regarding the answers received within the survey carried out in the MERMAIDS' project[158], 124 out of the 520 respondents answered the drying machine related questions so it has been assumed that they have a drying machine or a washing drying machine.

The figure below shows the drying machine capacity indicated by respondents. Around 50,8% of respondents' washing machines have a capacity between 5 and 7Kg.

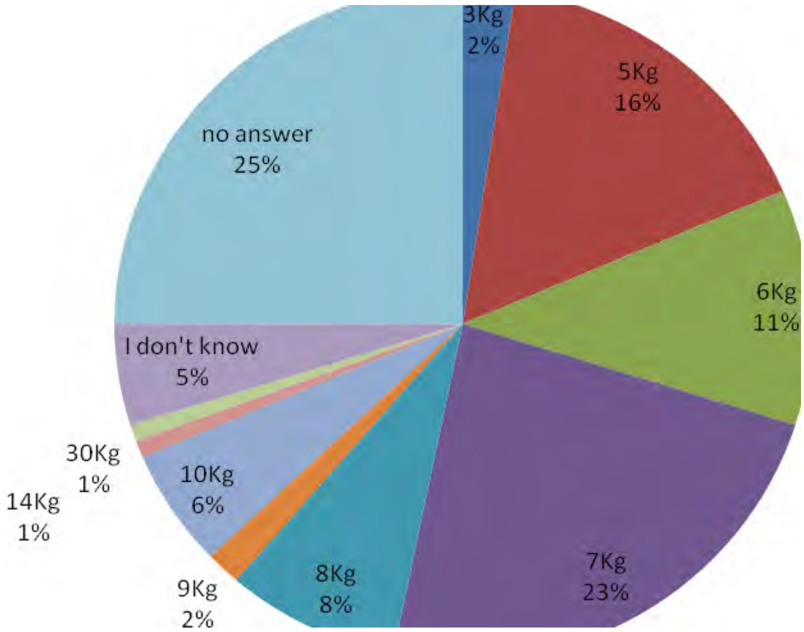

Figure 37. Drying machine capacity. [159]

Regarding the most common drying machine brands bought by European consumers are Bosch, ZANUSSI, LG, BALAY, AEG, Whirpool, Siemens, Miele, Samsung and Indesit, as shown in the figure below.

158 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

159 MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

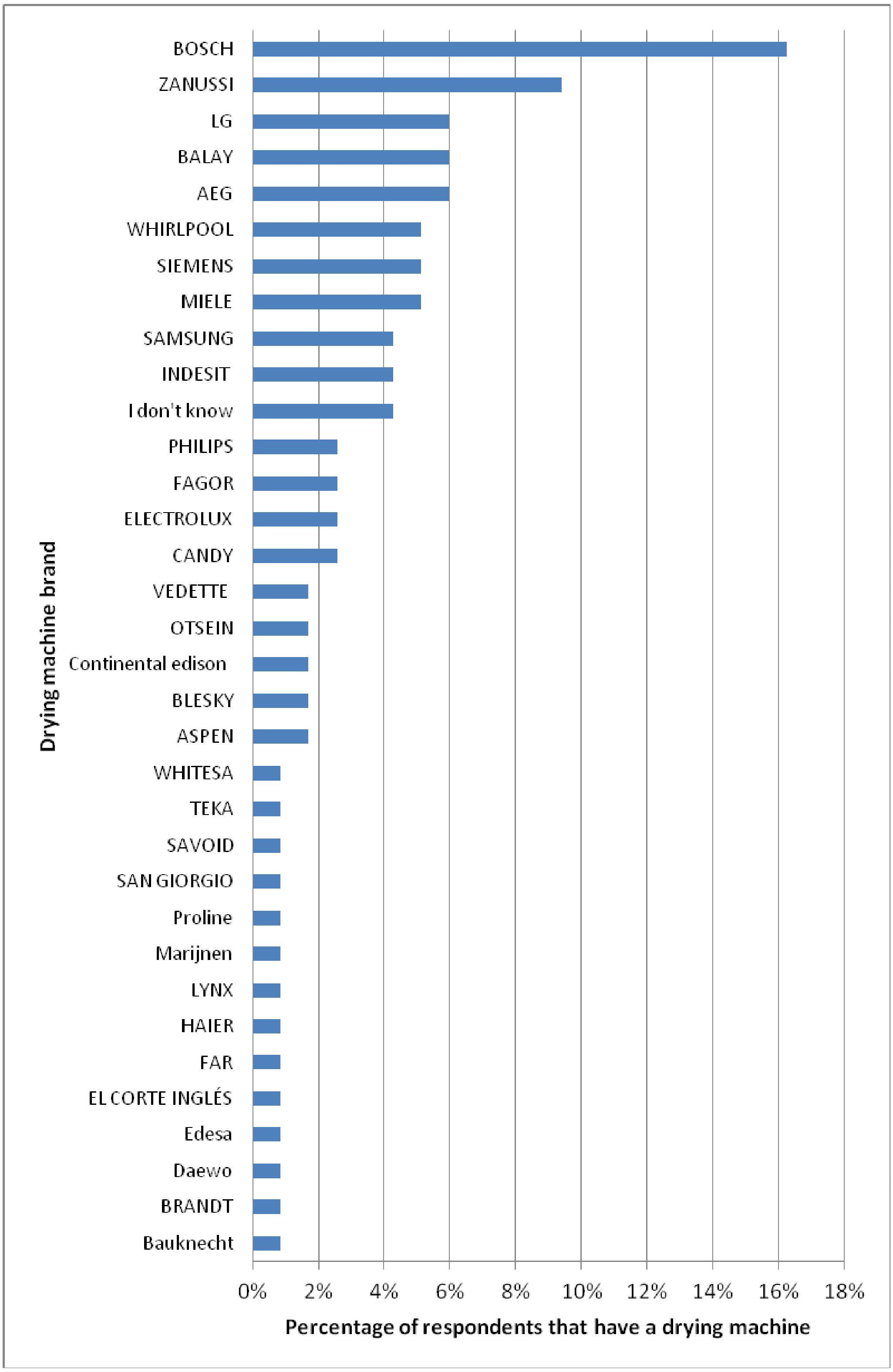

Figure 38. Drying machine brand. [160]

[160] MERMAIDS Project. Survey on domestic washing habits (October-December 2014).

9. Conclusions

General conclusions

1. A global assessment of microplastics is necessary and could be beneficial at this time because there is a public awareness concerning this issue and there is a need of using this information by means of, among others, the MSFD monitoring programs implementation and measure programs definition/implementation.

2. EU representatives highlighted the *"the need to identify and develop global environmental standards as well as to select a small number of broadly applicable indicators, with which to benchmark these standards."*. Seabirds are one of the most easily measured. [161]

3. Policy makers should be fully informed on the distribution of microplastics and their potential impact on marine/riverine ecosystems and species in order for them to assess this problem making decisions and defining regulatory and non regulatory measures.

4. There is concern that microplastics could reach marine/riverine environments, as well as WWTP, by means of water runoff/discharge or atmospheric deposition.

5. Physical harm of microplastic particles of different sizes entering the body, organs and cells of organisms is highly un-quantified.

6. More work is required in order to quantify the amounts and the sources of marine litter, especially micro litter, that is found in the marine environment mainly on the water surface and the water column. Increased monitoring activities addressing indicator 10.1.3 of the Marine Strategy Framework Directive (MSFD) "Trends in the amount, distribution and, where possible, composition of micro-particles (in particular micro- plastics)" will contribute to the implementation of the MSFD by 2020.
7. A multi-stakeholder approach, including textile and clothing garments' producers and laundry industries should be tackled in order to address laundry microplastics problem. Workshops and conferences organised within the MERMAIDS' Project should try to tackle these professional communities.

8. There is a concern that microplastics can transport chemicals which would not otherwise reach the oceans by mean of, for instance, atmospheric transport.[162]

[161] GESAMP (2010, IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection); Bowmer, T. and Kershaw, P.J., 2010 (Eds.), Proceedings of the GESAMP International Workshop on plastic particles as a vector in transporting persistent, bio-accumulating and toxic substances in the oceans. GESAMP Rep. Stud. No. 82, 68pp.

[162] GESAMP (2010, IMO/FAO/UNESCO-IOC/UNIDO/WMO/IAEA/UN/UNEP Joint Group of Experts on the Scientific Aspects of Marine Environmental Protection); Bowmer, T. and Kershaw, P.J., 2010 (Eds.), Proceedings of the GESAMP International Workshop on plastic particles as a vector in transporting persistent, bio-accumulating and toxic substances in the oceans. GESAMP Rep. Stud. No. 82, 68pp.

9. It is necessary to work with the industry in order to recover, reduce, reuse and/or recycle plastics and this should not be a stop of industrial practices rather than a change on industrial practices.[163]
10. A comprehensive understanding of domestic washing habits has been extracted from the survey carried out within the MERMAIDS project framework from October to December 2014, highlighting the following main findings:
    a. Most common washing machine brands are Bosch, Whirpool, LG, Indesit and AEG and 90% of respondents indicated that their washing machine capacity is between 5 and 8 Kg.

    b. Cotton program was pointed as one of the most used for all kind of clothes except for delicates.

    c. Concerning washing temperature, most of the different types of clothes are washed between 30ºC and 40ºC.

    d. Domestic wash cycles usually last between one hour and one hour and thirty minutes followed by washes between 30 and 45 minutes of duration. Regarding wash duration by different typology of clothes, delicates seem to be washed with shorter washes than the rest of clothes and bed linen/towels and white clothes with longer cycles.

    e. Pre-treatment washing option is mostly used for white clothes and bed linen/towels than for the other type of clothes.

    f. Most centrifugation programs used are between 800 and 1000 rpm. Nevertheless, delicates centrifugation is mostly done at 400 rpm.

    g. Most respondents stated that they fill their washing machine until it is full, independently of the type of clothes. However, delicates are treated as special since 40% of respondents stated to fill a half of the washing machine.

    h. Water consume was mostly stated between 30 and 50 litters per wash cycle.

    i. The average of washes per household and per year is 352,54, that is, 6,7 washes/household*week. This figure is much higher than the average wash frequency identified by the Stamminger survey (2011) (3,5 cycles per week, i.e. 182 cycles per household/year).

    j. The most common drying machine brands are Bosch, ZANUSSI, LG, BALAY, AEG, Whirpool, Siemens, Miele, Samsung and Indesit and 50,8% of the respondents indicated that their washing machine capacity is between 5 and 7 Kg.

[163] "Solutions" discussion group. *Workshop "Achievements and future research on micro-plastics in the marine environment". International Ocean Research Conference, Barcelona November 16th , 2014.*

k. The normal drying program followed by Air Dry and Synthetic programs are the most used by survey respondents. For delicates, the delicates program predominates and the cotton/towels program is the most preferred for bed linen /towels after the normal program.

l. Concerning drying program duration, this is usually comprised between 1h and 1h and 45min for bed linen /towels, colour/dark clothes and mix clothes. Regarding delicates and white clothes, the programs used are usually shorter, around 15-45min.

m. Although many different brands are bought by European consumers, Ariel, Dash and Skip are the most used detergent brands.

n. Concerning the format of the detergent used, liquid is the most preferred one, followed by powder and tablet.

o. Laundry products used by European citizens depend on the different type of clothes, however, most of the respondents use detergent when washing any kind of clothes and half of them use softener.

p. 25% of respondents stated to have 70-80% of their garments/home linen made of natural fibers while 15% stated to have 50% of both kind of textiles/fibers. 58% of respondents have more natural fibers made garments/home linen while 26,5% have more synthetic fibers made garments/home linen.

q. 43,8% of respondents said that they always check the composition of the garments when shopping, 30,8% sometimes, 19,4% very little and 6% of respondents never check clothes composition.

Specific conclusions regarding MERMAIDS' next steps to be taken

1. It will be necessary to extrapolate the amount of microplastics arising from laundry wastewaters taking into account washing habits described in this report and samples assessed by means of A1 actions.
2. A multi-stakeholder approach, including textile and clothing garments' industry and appliance industry should be tackled in order to address microplastics problem.
3. Concerning laundry industry associations, these could be approached by means of Action A3 (through specific consultations/interviews) and Action D6 (by inviting them to the events organized within the project framework).

Name of the Report:

Task A1.2. Characterization and quantification of micro and nanoplastic contained into different samples.
Task A1.3. Textile origin of microplastic particles.

Number of the associated action: A1

Name of the associated deliverable:
Report on localization and estimation of laundry microplastics sources and on micro and nanoplastics present in washing wastewater effluents

Mitigation of microplastics impact caused by textile washing processes

With the contribution of the LIFE financial instrument of the European Comunity:

Acronym: MERMAIDS

Title: Mitigation of microplastics impact caused by textile washing processes

LIFE13 ENV IT 1069

REPORT: TASK A 1.2. Characterization and quantification of micro and nanoplastic contained into different samples. TASK A 1.3 Textile origin of microplastic particles.

Authors :	Sara Gavignano, Alessio Montarsolo, Raffaella Mossotti (CNR ISMAC - BIELLA)
Reviewer:	Maurizio Avella (CNR)
ACTION :	A1
Task:	A1.2, A1.3
Contractual Date of delivery to EC:	The results of this report will be integrated in the deliverable *"Report on localization and estimation of laundry microplastics sources and on micro and nanoplastics present in washing wastewater effluents"* 31/12/2014
Actual Date of delivery to EC:	30/06/2015
Revision number	2

Contents

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1. Executive Summary

Microplastics comprise a very heterogeneous group of pieces that vary in size, shape, colour, specific density, chemical composition, and other characteristics. Microplastics deriving from textiles washing effluents are expected to be mostly constituted by synthetic fibers widely produced by textile industry that are mainly polyester, acrylic, polypropylene, polyethylene, polyamide.

Because of the novelty of the problem targeted by the Project, the partners evaluated the importance of defining specific methodological criteria to estimate the abundances and composition of microplastics in the washing effluents. Thus, different morphological identification and chemical/physical characterization methods were used to establish standardized procedures to define analytical protocols (TASK A1.2). Microplastics released from the washing of synthetic fabrics may be mainly identified by SEM analysis on the basis of the shape of the original fibers, since they are actually fiber snippets. Neverthless the smallest fragments cannot be identified with this identification and classification methodology. However, the largest fiber fragments will generate thousands of smaller fragments when released in the ambient, so that targeting on the reduction of the fiber snippets represents an effective way of mitigation of microplastics impact caused by textile washing processes.

The protocols setup will be useful for the implementation actions to determine the decrease of microplastics release by using innovative textile additives (ACTION B1) and innovative detergents additives (ACTION B2).Moreover this deliverable includes the evaluation of textile properties related to the microplastic release (TASK A1.3). This evaluation aims to define the textile characteristics that could affect mostly the amount of microplastics released (see in particular paragraph 3.2.3.2), in order to obtain information useful for the implementation Actions B.

This report contains the result achieved both for TASK A1.2 and A1.3 that are strongly linked.

1.1. State of the art of the Characterization and the Quantification of Micro and Nanoplastic contained into different samples

The term "microplastics" was first used in the year 2004 and is associated with a classification based on size. There is no general consensus about a specific size nomenclature, although it has been suggested that microplastics should be defined as particles <5 mm. In a recent document (2013) by the European MSFD (Marine Strategy Framework Directive) Working Group on Good Environmental Status (WG-GES) a more specific definition is given based on size and shape of microplastics: Large Microplastics (1-5 mm diameter), Small Microplastics (20 μm - 1 mm diameter), Particles (including nanoparticles, diameter lower than 20 μm). The scientific literature available on this topic is almost exclusively dedicated to samples recovered from marine sediments. Currently there are few scientific articles on the characterization and identification of fibers fragments released in textile washing processes [1]. For what concerns the qualitative analysis, in all reviewed studies, visual sorting of residues from marine sediments was necessary to separate the plastics from other materials, such as organic debris. This is done by direct examination of the sample by the naked eye or with the aid of a dissecting microscope. In the reviewed studies several methods have been employed to identify microplastic polymers. Identification based on infrared (IR) spectroscopy was used in several studies [2-4]. This method compares the IR spectrum of an unknown plastic sample with spectra of known polymers. Also, a differential scanning calorimeter was used, where temperature is applied simultaneously to an unknown sample and a reference. Quantitative data were reported in many reviewed studies: for sediment samples the most commonly used units for mass were "grams of microplastics per m^2" and for abundance "microplastic items per m^2" (or items cm^{-2}). No information were found on specific methods for the qualitative and quantitative characterization of microplastics deriving from washing processes of textiles, thus confirming the lack of data and scientific studies on this topic.

2. Materials and Methods

It was decided to first operate in standard conditions, to avoid the influence due to further variables, using synthetic standard fabrics (defined as "ready to dye") that are commonly used in quality control laboratories to test different textile properties. The standard woven textile chosen are made of 100% polyamide 6.6, 100% polyacrylic, 100% polyester, 100% polypropylene and polyester/cotton blend (65%/35%); the knitted ones are made of 100% polyacrylic, 100% polyamide, 100% polyester. The standard fabric chosen for washing tests were characterized in order to achieve textile characteristics that could interfere with the fiber release such as count (according to UNI EN ISO 2060:1997), weight (gram/sqm), fiber diameter (µm), number of fibers per section and pilling evaluation with Martindale (according to UNI EN ISO 12945-2:2002).

The washing processes were studied both with an industrial and a domestic approach. Industrial washing was carried out according to the UNI EN ISO 105-C12 standard method, which simulates the stresses caused by 5 to 10 industrial washings, with a good reproducibility. Industrial washing processe were carried out with and without the standard detergent (only water), in order to achieve for each sample the reference values (blank test). Domestic washing was carried out according to the UNI EN ISO 105-C06 standard method. Both methods employ the LINITEST laboratory equipment as washing machine.

Then, textile materials (market products) were purchased selecting garment containing polyester, polyacrylic and polyamide fibers (pure or in blend with other fibers) according to the most representative abundance on the market; clothes were subjected to standard washing (according to the UNI EN 26330-5A standard), using a frontloading machine Wascator FOM71MP-Lab from Electrolux, that is officially acknowledged as a standard reference for washing tests of fabrics. The commercial clothes used for Wascator tests were characterized with chemical and physical methods, with light microscopy observations, pilling test with Martindale (according to UNI EN ISO 12945-2:2002), tenacity with bursting strength test method (ASTM D3787). The microplastic fibers were separated from the washing machine effluents by filtration. In the case of wastewater collected from the LINITEST, different filter pore sizes were tested: 0,1 µm, 0,2 µm, 5 µm for membrane filters made of PVDF (Polyvinylidene fluoride), 30 µm for non woven PP filters and random pore sizes for paper filters. The hydraulic system used for filtrations was constituted by a peristaltic pump (Mettler Toledo, flow rate 100 ml/min) connected with Tygon tubes. For the filtration of Wascator effluents also a sieve with 40 µm pores (Giuliani Setacci, Italy) was used, in order to obtain a larger amount of fiber residue that was dried and weighed. The material collected from

filtration was characterized with qualitative and quantitative methods such as Light Microscopy, Scanning Electron Microscopy (SEM, LEO 435VP Oxford UK), Fourier Transformed Infrared Spectroscopy (FT-IR ATR), Differential Scanning Calorimetry (DSC), Thermo Gravimetric Analysis (TGA). Optical Emission Spectroscopy coupled with Mass Spectrometry (ICP-OES) for the determination of heavy metals deriving from dyes requires a minimum quantity of solid sample (1g) to be subjected to extraction with acid perspiration solution, as reported in the standard method (DIN 54020) commonly adopted in our laboratory. Thus, this technique was not considered suitable because of the small amount (in the order of mg) of residue obtained from the washing processes. As a general remark, the characterization techniques listed in the project proposal (Task A1.2) were adopted depending on the material properties, selecting the more suitable ones for the action purposes.
A scheme of the analytical process followed for Wascator tests is reported in Figure 1.

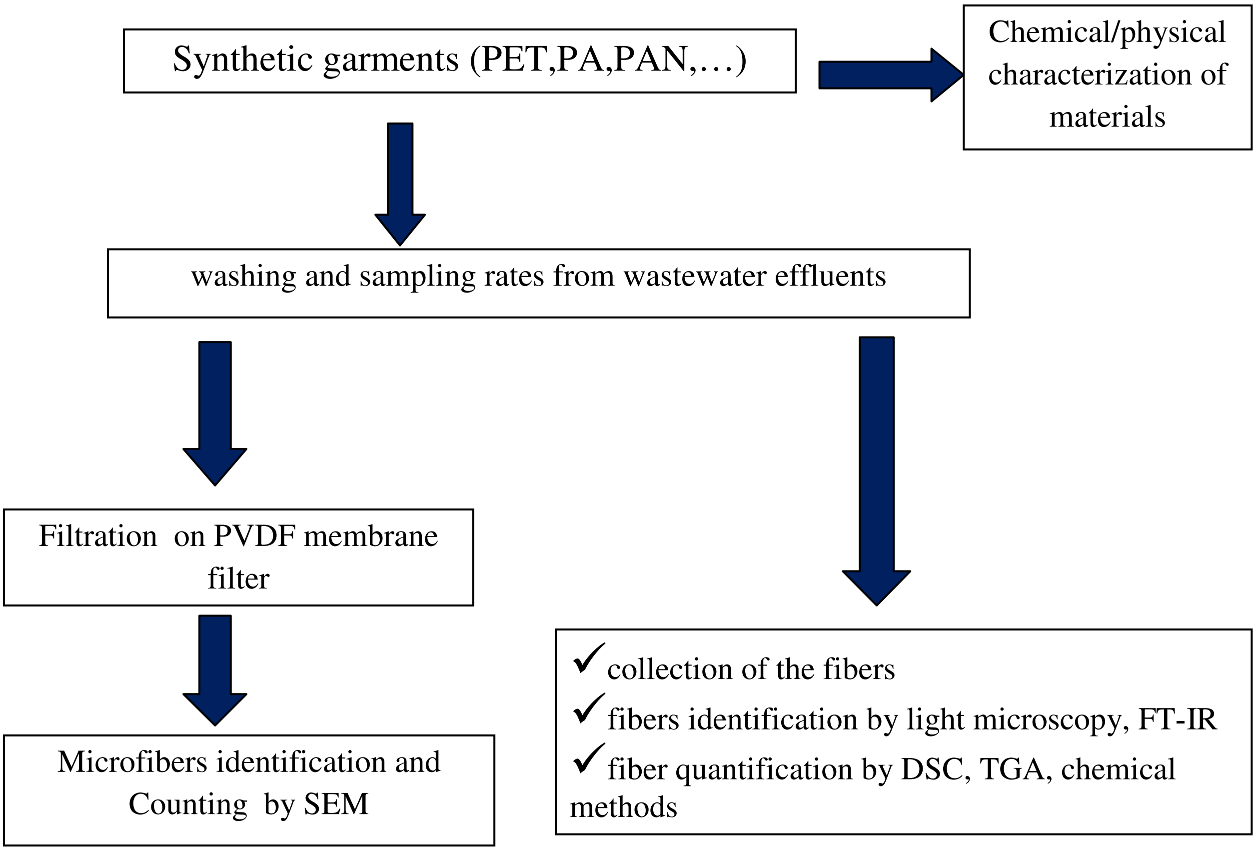


Figure 1. Flow chart of the experimental phase in Wascator machine.

3. Results and achievement

3.1. Major results and achievements

The tests performed with the Linitest aim to provide a standardized method for the identification and the quantification of microplastics released from textiles, but they can also give information about the connection between fabric textile characteristics and fiber losses. The tests performed with the Wascator aim to determine the release of microplastics due to domestic washing and they allow to collect a greater amount of microplastics necessary to carry on further qualitative and quantitative investigations.

The main results achieved are listed as follows:

- ✓ Setup and optimization of water effluents filtration
- ✓ Setup of a statistical method for the numerical quantification of microplastics by SEM analysis

- ✓ Identification of microplastics from washing processes of textiles containing synthetic fibers (pure or in blend with natural or other man-made fibers) by means of light microscopy, FT-IR ATR spectroscopy, DSC and TGA techniques
- ✓ Quantification by weight and by means of DSC and TGA techniques of the filtration residues

3.2. Technical progress of the work

3.2.1. Setup and optimization of water effluents filtration

Different kinds of filters have been tested, in order to choose the most suitable one; the key variable we had to choose were pore size and filter material, in order to collect also the smallest fragments of microplastic avoiding pore obstruction.
Indeed we started to make filtration tests with standard polyacrylic fabrics using ECE with perborate as detergent (standard ISO C105 C06) at 75 °C (using also steel balls). This is mainly to test the filtration system (XX4304700 Millipore in-line filter holder connected to a peristaltic pump). We tested filters with different pore size: PVDF 0,22 µm (Figure 2), PVDF 5µm, PP 30µm. The following SEM images are taken from a sample of 1cm^2 cut from a 0,22µm filter after the filtration of the washing solution (Figures 3,4,5). The images show fragments of polyacrylic fibers with a diameter of about 15µm. However it can be also noticed a large amount of material that covers the surface of the filter and fill the pores that is mainly due to the detergent fouling. We obtained similar results using the filter with 5µm pores and also another method of filtration (with a vacuum pump).

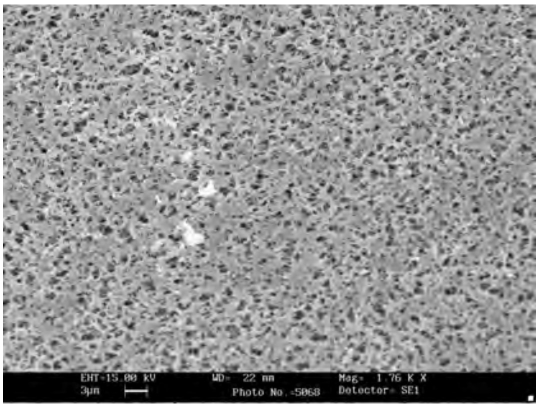


Figure 2. PVDF filter 0,22µm before filtration (1,76KX)

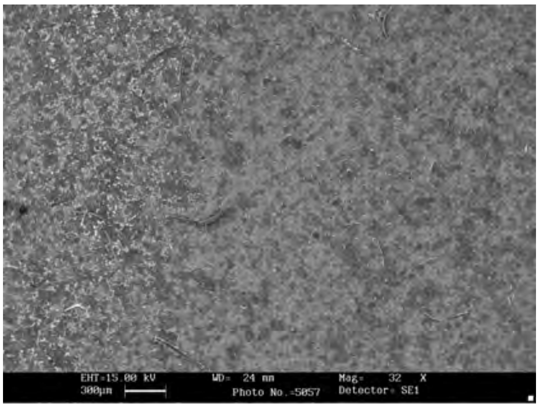
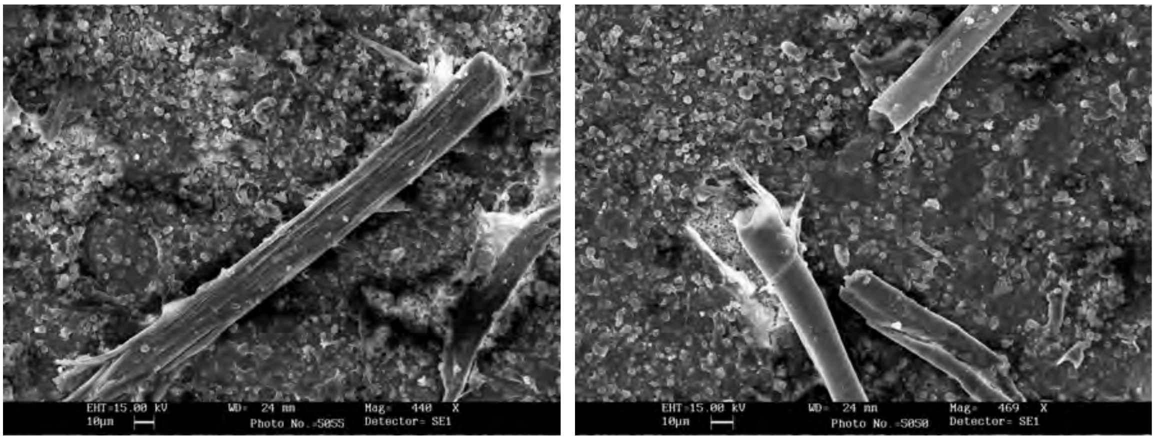


Figure 3. PVDF filter 0,22µm after filtration with washing solution (32x)



Figures 4-5. PVDF filter 0,22µm after filtration with washing solution (440x , 469x)

Considering the problem we tried to pre-filter the solution on a laboratory paper filter (with a buchner) and then on the PVDF filters. The following images show the paper filter (Figure 6) and the 5µm PVDF filter (Figure 7) after filtration. The 5µm filter appears not filled with material deriving from the solution and fragment of fibers are visible.

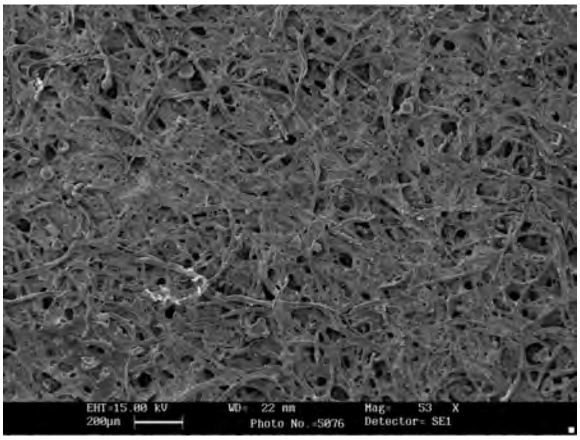


Figure 6. Paper filter

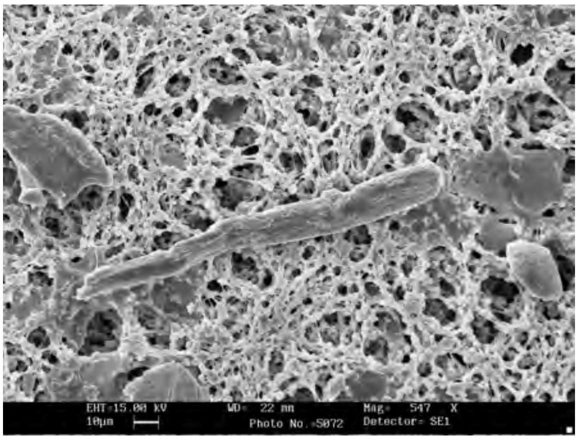


Figure 7. PVDF 5 µm filter

However, also this method is not suitable because probably also fibers and fragments (microplastics) are blocked in the first pre-filtration.
Thus further filtration tests with the peristaltic pump and 5 µm filters were performed; in this case we fluxed clean water after the filtration of the washing solution in order to re-solubilize detergent components and avoid the filling of the filter pores (Figure 8). There is always a lot of material on the filter other then fiber fragments, but the filter pores are not completely filled. We performed also a test filtering only the detergent solution (without the fabric) and we found the same kind of particles observed previously, thus confirming the presence of detergent components blocked by the filter (Figure 9).

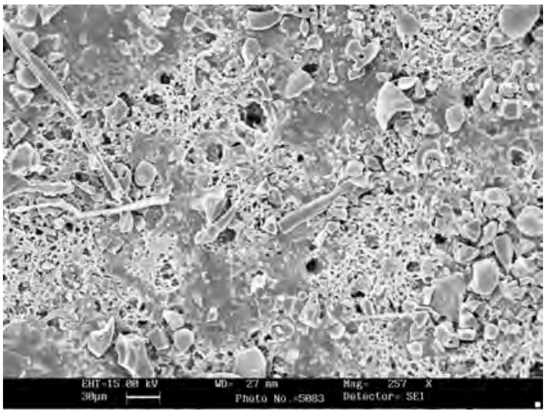


Figure 8. PVDF 5 µm filter after filtration of washing solution

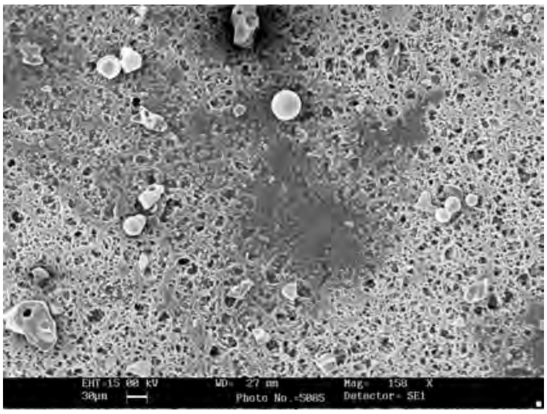


Figure 9. PVDF 5 µm filter after filtration of detergent solution

We also tried Polypropilene (PP) filters of 30 µm size, but they are not suitable because, observing them with SEM, they are not membranes but they are constituted by a non-woven structure of fibres (Figure 10 and 11).

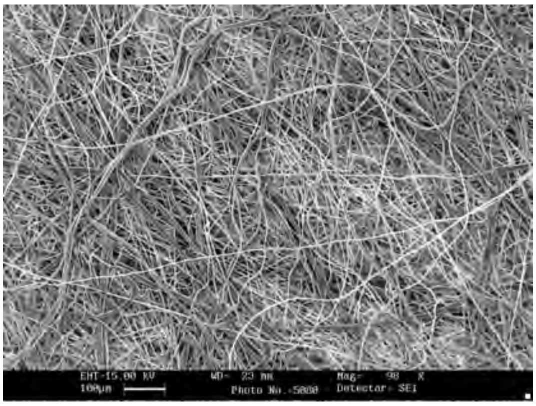


Figure 10. PP filter, 30 µm pore size

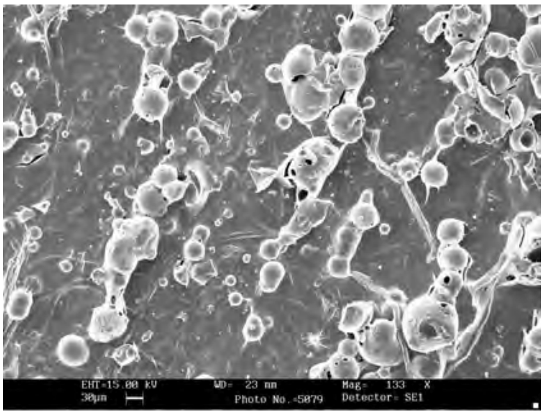


Figure 11. PP filter after filtration

In conclusion we consider that working with detergent may cause the filling of the filters pores and for this reason 5µm PVDF filters are more useful than 0,1 and 0,22 µm filters. Through the SEM observations we found that microplastics deriving from the washing of synthetic fabrics are fragments that maintain the shape of the starting fibers.

3.2.2 Setup of a statistical method for the numerical quantification of microplastics by SEM analysis

Because of the small amount of fiber released by Linitest experimental phase, after trials made by weighing dry filter before and after washing solution filtration, it was judged that the most accurate technique to quantify the microfibers coming from Linitest was the Scanning Electron

Microscopy. SEM direct observation of the filter surface, due to its high resolution, depth field and high magnification, allows the fiber identification and a statistical count with evaluation of errors depending on the number of fields read in the microscope. As expected, this technique is useful also for the characterization of microplastics deriving from Wascator tests. The counting procedure is based on the direct observation of a part of the whole surface of the filter. The sampling was carried out according to two different methods that confirmed the reproducibility and reliability of the measures. In the following paragraphs these methods are explained in details.

3.2.2.1 Counting method N. 1

This SEM detection analysis method used for filter readings was borrowed by a standard method used for asbestos and metallic particulate filter counting in automotive sector (ISO 16232). In the very first attempts of the study, we collected square samples from the center or from the peripheral areas of the filter with 1 cm^2 area. Then the readings were made to cover a 5 mm^2 area (30 readings each stub, 250x of magnitude). In that way, we could determine a strong inhomogeneous fiber deposition, due to random effects involved in filtration, probably caused by flow pulse given by peristaltic pump (usually used for filtration at laboratory scale). The major density was often observed in center areas, but it wasn't noticed a circular symmetry.
The main purpose of the study was to realize a completely random and representative of the whole filter method of sampling to improve the measurement accuracy. Then it was decided to consider not only a square portion of filter, but to divide it into eight triangle sectors and randomly choice four of them reading the fields on the bisector of each triangle (Figure 12). In this way, considering the filtration area (except the circular ring occupied by the seal), the bisector length is multiple of the field length at 250x; then the bisector was divided in 40 square fields and 20 of them were read, skipping one alternately.
To avoid double counts of the fibers (microplastics), some rules were used:

- count 1 for each fiber entirely contained in the square field
- count 0,5 for each fiber partially contained
- count 1 for each fiber that crosses the whole field.

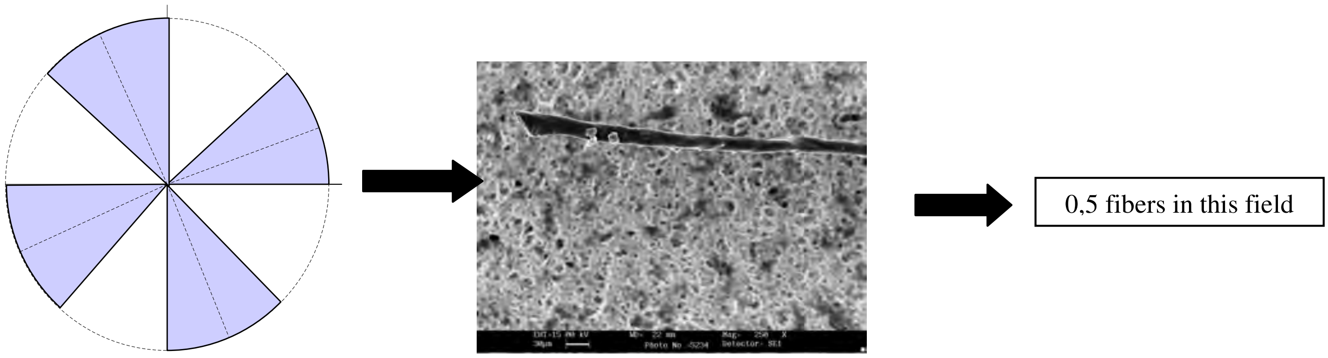


Figure 12. Exemplification of the reading method developed

The data collected with this method were then elaborated to give useful information regarding the total amount of fibers fragments (microplastics) released.

According to the statistical approach initially adopted (assuming the lack of a circular symmetry and the lack of a preferential path imposed by the pump to the fluid), each field was assumed to be representative of the whole area filter. Thus, the total number of fibers observed was reported to the whole filtering area, as follows:

$$N = \frac{n}{a} \cdot A$$

N : number of fibers on the whole filter area

n : number of fibers observed in the 80 square fields (20 for each triangular sector)

a : total area observed (area of the 80 square fields)

A : area of the whole filter (1385,44 mm^2, excluding the circular ring occupied by the seal).

Since the sample tested hasn’t the same weight per square meter, to provide a data based on the weight of the fabric tested, the values coming from the observations by SEM were expressed on a weight basis, as follows:

$$Conc_{Avg,i} = A \cdot \frac{n_i}{a_i} \cdot \frac{1}{m}$$

$$Conc_{Avg} = \frac{\sum_{i=0}^{4} Conc_{Avg,i}}{4}$$

$Conc_{Avg}$: Average concentration of fibers on the whole filter on the basis of the sample weight [fibers/gram]

A : area of the whole filter (1385,44 mm^2, excluding the circular ring occupied by the seal)

n_i : number of fibers on the bisector of each triangle sector (where i:1,..,4 , running from the 1st to the 4th triangle sector)

a_i : total area of the 20 fields read contained in each triangle sector (where i:1,..,4 running from the 1st to the 4th triangle sector)

m : weight of the fabric sample [grams].

3.2.2.2 Counting method N. 2

The second SEM detection analysis method tested is based on the acquisition of several electron micrographs of the filter surfaces using a scanning electron microscope, SEM, FEI Quanta 200 FEG. SEM observations were performed in low vacuum mode (PH_2O = 0.7 torr), using a large field detector (LFD) and an acceleration voltage of 5-20 kV.

The observations were performed on the whole filter without applying metal coating.

Taking into account the geometry and the dimensions of the used filters, 21 SEM micrographs were acquired for each filter. Every micrograph represents a squared area (A_S) of the filter surface, equal to 7.8 mm^2 (see Figure 13).

SEM micrographs were acquired along two orthogonal diameters of the circular filter (see Figure 14).

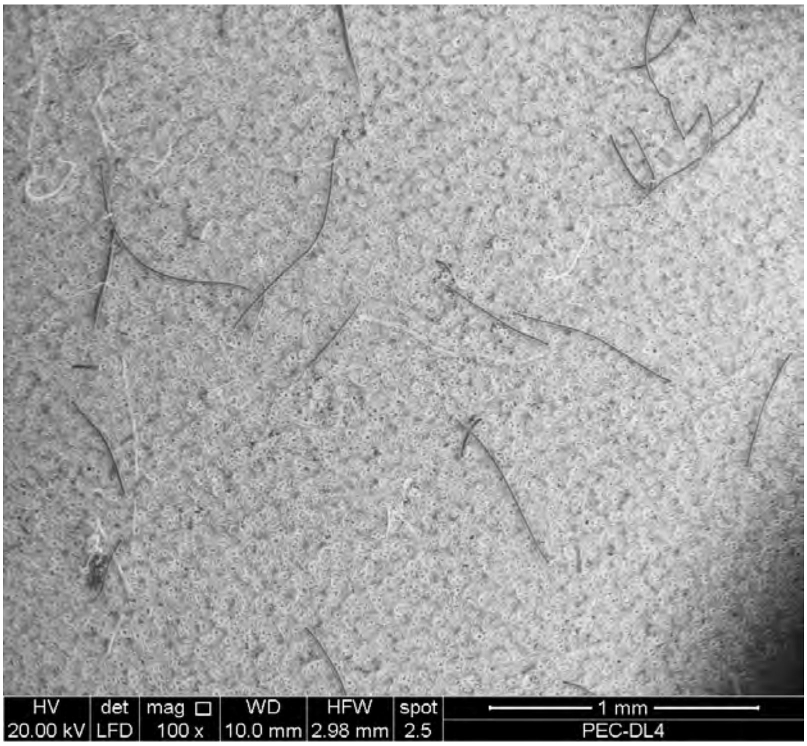


Figure 13. SEM micrograph of a squared area (AS = 7.8 mm^2) of a filter containing PEC fibres.

It was hypothesised that the fibre distribution is equal along all the traceable diagonals, thus 6 concentric circles were taken into account (see Figure 15):

- The first circle is right in the centre of the filter and it circumscribes the central square;
- The other 20 squares are included, four by four, in 5 annuli.

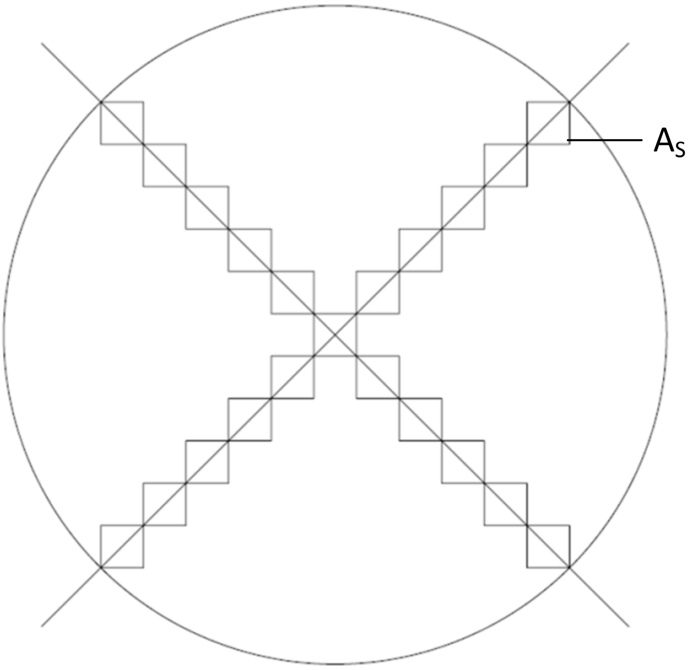


Figure 14. Position of the images along the filter.

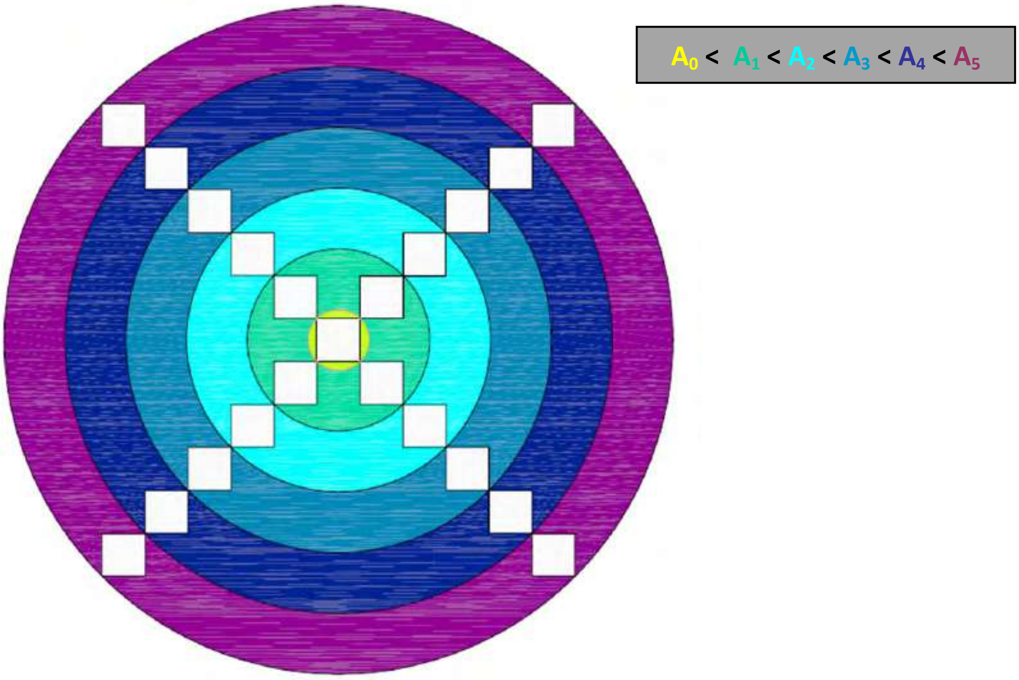


Figure 15. Concentric circles.

The counting develops in 4 steps explained below:

1. Counting of the number of fibres in the first image in the centre of the filter (n_0) and division of this number for the area of the single square (A_S = 7.8 mm^2). In this way it is possible to obtain the number of fibres per area ($n_0/A_s = C_0$). Considering the area of the circle (A_0) that circumscribes the central square and multiplying this value for C_0, the number of fibres contained in the first circle is easily obtained ($N_0 = C_0 \times A_0$).

2. In the first annulus, the number of fibres is counted in each of the four squares (n_1, n_2, n_3, n_4) and an average value is calculated $\left(\frac{n_1 + n_2 + n_3 + n_4}{4} = n_A\right)$. As done in the previous step, this value is divided for A_S ($n_A/A_S = C_1$) and multiplied for the area of the considered annulus (A_1), obtaining the number of fibres contained in the first annulus ($C_1 \times A_1 = N_1$).

3. The previous step is repeated for the other 5 annuli, obtaining N_2, N_3, N_4, N_5.

4. Finally N_0, N_1, N_2, N_3, N_4 and N_5 are added together giving the total number of fibres on the filter surface.

3.2.2.3. Selection of the counting method

The two counting methods above illustrated displayed comparable results ranging in the same order of magnitude, confirming the reproducibility of the whole procedure. Nevertheless, considering the overall area of the filter observed by using each procedure, the counting method N. 1 covers an area of almost 13.78 mm^2 whereas the N. 2 allows to analyse an area of about 163.80 mm^2. Comparing these values with the whole area of the filter, the percentages of the observed areas are 1% and 10% for N. 1 and 2, respectively. Then, the N. 2 is more representative of the whole filter, while the N. 1 has to be improved by comprising the acquisition of a higher number of square areas in order to increase to whole scan from 1 to almost 10%.

3.2.3. Identification and quantification of microplastics

3.2.3.1. Linitest industrial washing tests

As mentioned before the tests were first performed in Linitest with standard fabrics, in order to provide a standardized protocol for microplastics release able to give quantitative information and a correlation between fabric characteristics and the amount of fiber loss.
For this purpose, we decided to first operate in standard conditions, to avoid the influence due to further variables, using synthetic standard fabrics (defined as "ready to dye"); the whole washing process was carried out in standard conditions according to UNI EN ISO 105-C12 normative, which specify the operations to simulate an industrial washing process.
An important goal is to setup a complete procedure for the characterization of microplastics released by a garment after washing, by a qualitative and a quantitative point of view.
The fabrics were treated according to the standard method with a Linitest equipment (Figure 16) with an alkaline detergent solution (pH 12-12,5) with 5 g/l of surfactant and 1 g/l of sodium hydroxide.
The surfactant (according to 1S method of UNI EN ISO 105 C12) is composed as follow:

Substance	Concentration [%W]
Sodium alkylbenzene sulfonate	0,425 %
Non ionic surfactants	6%
Sodium citrate dihydrate	5%
HEDP	1%
metasilicate anhydrous	42,3%
Polymaleic acid	2%
Foam inibitors (phosphoric acid esters)	3%
sodium carbonate	39,5%
Wet %	0,475%

The fabrics were cut in 16 x 8 cm rectangles to provide, after being sewn, a "bag" of 8 x 8 cm; 25 stainless steel balls were placed inside bags and other 25 outside, in the metallic specimen containers (Figure 17). The bath ratio indicated by the normative is 1:15, that corresponds to 15 ml of solution every gram of fabric. Washing operative conditions are 75°C for 1 hour.
Then the solution was filtered in PVDF Millipore filters, with pore size of 5 µm; pore diameter was chosen to obtain a compromise between the dimension of fibers and their possible clogging.
Indeed it was experimentally pointed out that with lower pore sizes it was shown a clogging by the detergent that made difficult the microscopic reading.
For the filtration we used a peristaltic pump with tygon tubes. After filtration 400 ml of hot micro filtered water were fluxed in the filtration system; the amount of water was found optimal to avoid an excess of detergent on the filter surface.
Also blank washing solutions (only detergent solubilized in water) were prepared to evaluate the influence of the surfactant on the filtration.

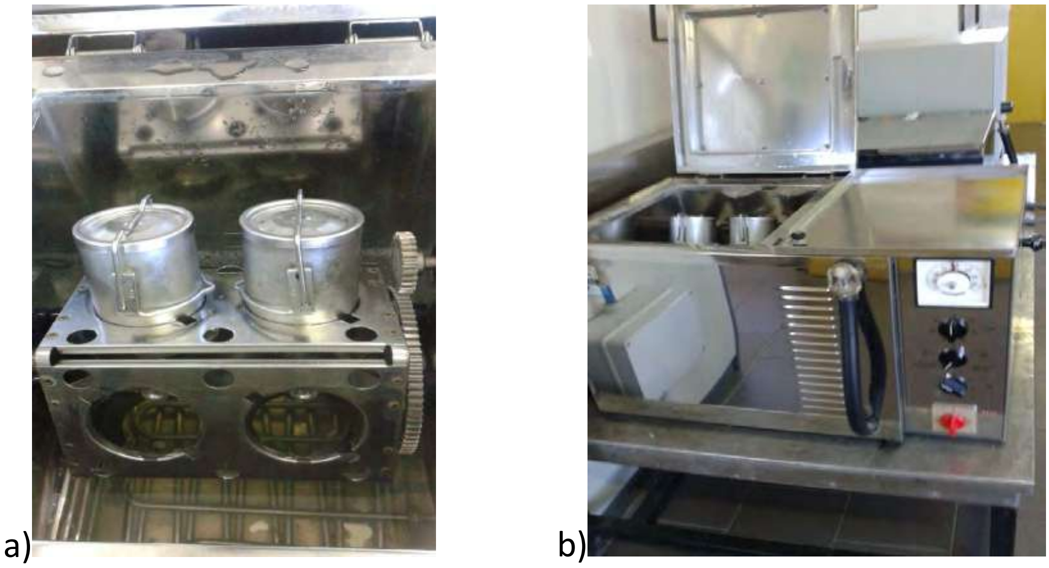


Figure 16. Linitest apparatus (a); specimen containers inside Linitest (b).

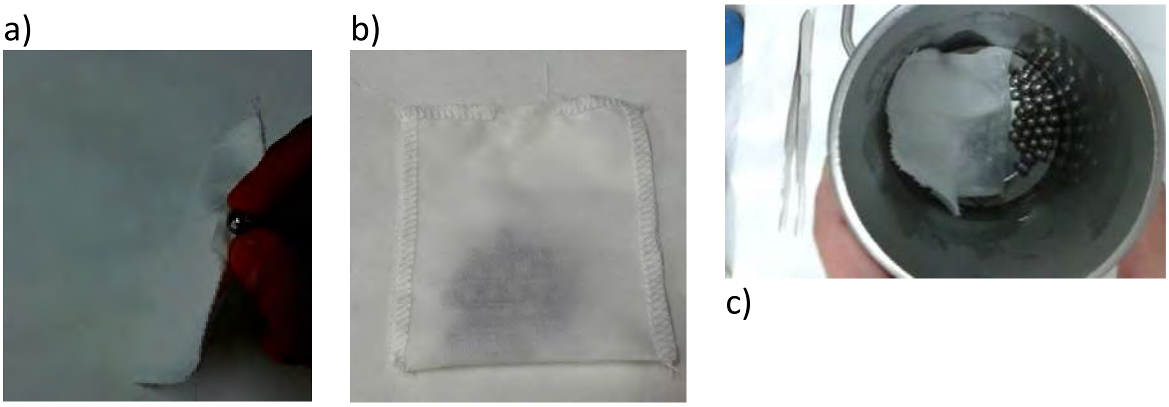


Figure 17. preparation of fabric samples; a) insertion of stainless steel in the fabric b) fabric bag with 25 stainless steel inside c) view of the inside part of the specimen container before washing process.

SEM microscopy analysis on filter allows to identify microplastics released by the standard fabrics. From these observation, we can say that microplastics from the fabrics are constituted by fiber fragments, usually longer than 40 µm, conserving the cross sectional area shape, and normally not strongly damaged by the mechanical and physical action involved during tests.

Figure 18. Peristaltic pump used for filtration.

Figures 19, 20 and 21 report photograph of the fiber, taken with the SEM.

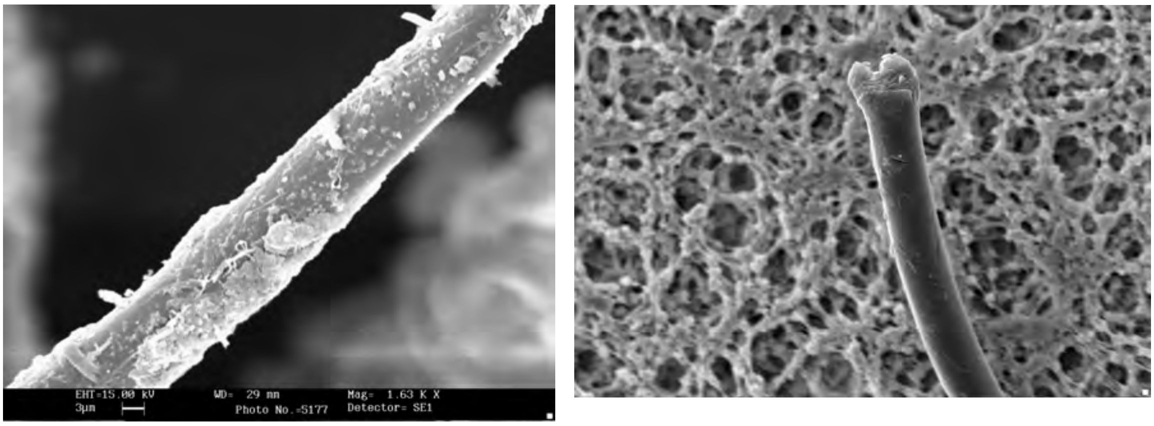


Figure 19. SEM photos of polyamide microplastics from woven standard fabric on filter; tests made with and without detergent.

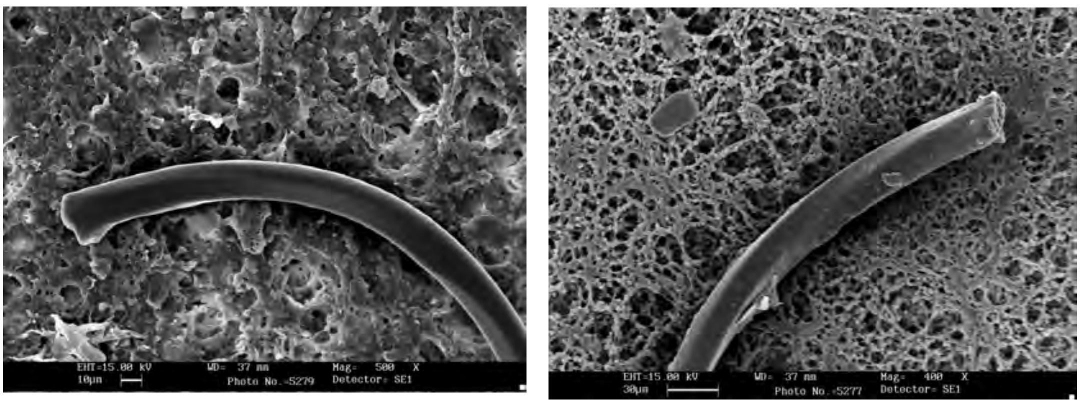


Figure 20. SEM photos of polyester microplastics from knitted standard fabric on filter.

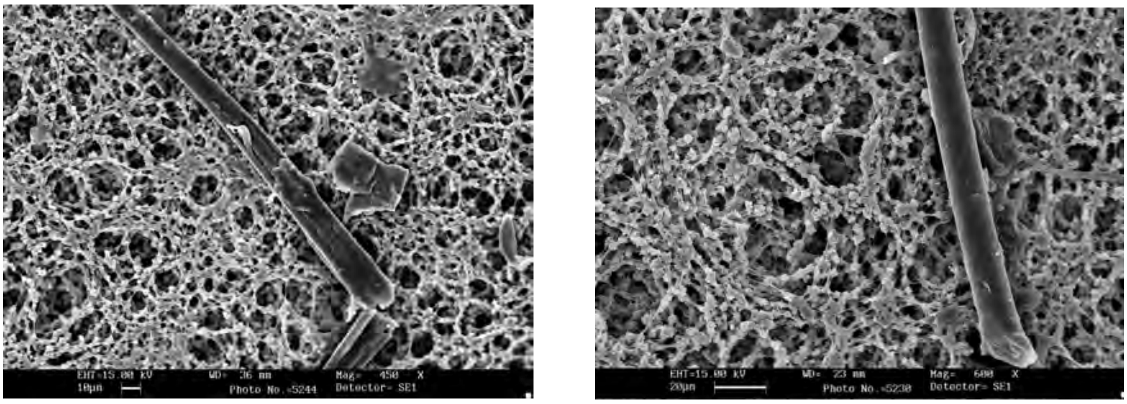


Figure 21. SEM photos of polypropylene microplastics from knitted standard fabric on filter.

After filtration, as explained before, the filter was divided into eight circular sections and for the readings were chosen four of them. Twenty fields for each section were counted with a total number of eighty fields in the whole filter area.
After the microscopic readings, the data were collected and elaborated to provide an estimation of the real release of microplastics during washing. The elaboration can't be extrapolated from the operative conditions and reported to a single process, because the normative allows to simulate 5 to 10 washings; that means that isn't possible to express a concentration on liter basis, because of the small amount of solution used, but it is preferable to express this value on fabric weight, to have an estimation not linked to bath ratio value.
In the tables below are shown the results:

Composition	***Washing***	***Conc. $_{Avg}$ [fibers/g]***	***Conc. $_{Avg}$ [fibers/filter]***
100% Polyester	*Detergent solution*	1389	2412
	Water only	145	251
100%Polyamide	*Detergent solution*	2470	4221
	Water only	2352	4020
100% Acrylic	*Detergent solution*	1359	2663
	Water only	786	1541
100% Polypropylene	*Detergent solution*	687	1608
	Water only	513	1005
Blend 65%Polyester 35%Cotton	*Detergent solution*	1041	1357
	Water only	771	1005

Table 1. Concentration of fiber released by woven fabrics in detergent and blank (only water) solution.

Knitted fabrics			
Composition	**Washing**	**Conc. $_{Avg}$ [fibers/g]**	**Conc. $_{Avg}$ [fibers/filter]**
100% Polyester	Detergent solution	349	804
	Water only	174	402
100%Polyamide	Detergent solution	885	2663
	Water only	384	1156
100% Acrylic	Detergent solution	3118	6433
	Water only	2371	4891

Table 2. Concentration of fiber released by knitted fabrics in detergent and blank (only water) solution.

As expected, the results collected are extremely variable, due to fabric intrinsic textile characteristics.
In order to understand the variability of the experimental data and to evaluate the results obtained, the standard fabrics used for Linitest experimental phase were subjected to a physical characterization.
The textile characteristics measured are represented in the tables below:

Composition	*100% Polypropylene*	*100% Polyamide*	*100% Acrylic*	*100% Polyester*	*Blend 35%Cotton 65%Polyester*
Woven fabric construction	Warp and weft	Warp and weft	Warp and weft	Warp and weft	Warp and weft
Fiber and yarn characteristics	**Short staple- Ring Spun- Circular cross section (warp and weft twisted yarn 2 ends)**	**Polyamide 6.6 - Short staple- Ring Spun- Circular cross section (warp yarn twisted 2 ends)**	**Short staple- Ring Spun- Dog bone cross section (warp and weft yarn twisted 2 ends)**	**Short staple- Ring Spun- Circular cross section (warp yarn twisted 2 ends)**	**Short staple- Ring Spun- Circular cross section (not twisted yarn warp and weft)**
Weight [g/m2]	170	190	190	126	104
fiber d $_{Avg}$ weft [µm]	22,63	14,98	20,98	11,98	11,15
fiber d $_{Avg}$ warp [µm]	22,84	15,35	20,39	11,43	11,25
Yarn weft count [Tex]	65,12	20,13	41,72	22,41	14,15
Yarn warp count [Tex]	63,96	39,76	41,71	41,21	13,43
Fibers per yarn Warp	173	188	86	291	64
Fibers per yarn Weft	180	100	88	144	68

Table 3. Textile characteristics of woven standard fabrics.

Pilling (Martindale UNI EN ISO 12945-2)					
Composition / N° of cycles	*100% Polypropylene*	*100% Polyamide*	*100% Acrylic*	*100% Polyester*	*Blend 35%Cotton 65%Polyester*
125	2	1/2	3	1/2	4
500	1	1	2	1	2/3
1000	n.d.	n.d.	1	n.d.	1
2000	n.d.	n.d.	n.d.	n.d.	n.d.
7000	n.d.	n.d.	n.d.	n.d.	n.d.

Table 4. Textile characteristics of woven standard fabrics: pilling test with Martindale (UNI EN ISO 12945-2); the tests are performed with increasing number of cycles until the achievement of the worst value (1= high values of pilling).

Composition	*100% Polyamide*	*100% Acrylic*	*100% Polyester*
Knitted fabric construction	**Double knit piqué**	**Jersey**	**Double knit piqué**
Fibers characteristics	**Polyamide 6,6 - Continuous texturized filament**	**Short Staple- Open End- dog bone like cross section**	**Continuous texturized filament**
Weight [g/m^2]	260	143	200
Count [Tex]	17,91	27,83	18,69
fiber d_{Avg} [μm]	17,66	17,96	23,34
fiber d_{Min} [μm]	13,20	14,65	17,50
fiber d_{Max} [μm]	20,14	21,08	29,59
Fiber diameter CV%	7,65	7,80	12,68
Fibers/filaments per yarn	64	166	32

Table 5. Textile characteristics of knitted standard fabrics.

Pilling (Martindale UNI EN ISO 12945-2)			
Composition / N° of cycles	*100% Polyamide*	*100% Acrylic*	*100% Polyester*
125	5	4	5
500	5	3	5
1000	5	1/2	5
2000	5	1	5
7000	5	n.d.	5

Table 6. Textile characteristics of knitted standard fabrics; Pilling test with Martindale (UNI EN ISO 12945-2) the tests are performed with increasing number of cycles until the achievement of the worst value (1= high values of pilling).

Tables 4 and 6 show the values of pilling measure according to UNI EN ISO 12945-2 (modified Martindale method); fuzzing and pilling are assessed visually after defined stages of rub testing, in accordance with a grading range from 5 (no change) to 1 (severe pilling and pills covering the whole specimen surface). The fabric evaluation were made at 125, 500, 1000, 2000 and 7000 rubs.

The experimental data shows that polyamide and polyester knitted fabrics, because of their compact structure (double knit fabric) and the continuous filament component, don't present changes after 7000 rubs. However acrylic knitted fabric shows intense pilling over the whole sample surface already after 2000 rubs. These values are strongly connected with fiber release (3100 fiber each gram of sample in acrylic knitted fabric tested compare to 850 and 350 for polyamide and polyester respectively).

Correlating experimental data with textile characteristics measured, may be certified the importance of some variables that influence the amount of microplastics released. The identification of these variables will be extremely important for the next actions, implementation ones, because it allows to work on the characteristics that influences more the phenomenon.

Indeed the more important variables for fiber release are:

- ✓ Detergent presence: in almost all cases, the presence of the detergent involves an increase in fiber losses, probably due to the increase of fabric wettability;
- ✓ Textile construction characteristics: higher values are collected for acrylic jersey fabric made of short staple fiber, rather than fabrics made with continuous filaments and with a less flexible geometry;
- ✓ Pilling propensity: as a consequence of the geometrical structure and fiber constitution of the fabric, pilling higher values are connected to the fiber release increase;
- ✓ Chemical and physical properties of the fiber: for example, polyamide shows high values of fiber released and the data from blank and detergent tests are similar; this aspect is probably due to the higher hydrophilicity of the polyamide in comparison with other synthetic fibers.

3.2.3.2. Wascator domestic washing tests

After the first experimental phase dedicated to the realization of a protocol of analysis for determination of microplastics release, the second part of the preparatory actions proceeded with the identification and characterization of laundry wastewater from Wascator washing tests.
With this purpose, we performed the experiments in Wascator with commercial clothes of different composition, containing the most common synthetic fibers used in textile field (polyester, polyacrylic and polyamide).
For the tests we used the standard method UNI EN 26330-5A, that specifies the conditions to be used to simulate a standard domestic washing process. This kind of experiments, as expected, allow to collect a larger amount of microplatics which can be characterized with further methods such as Optical Microscopy, FT-IR ATR, DSC, TGA and chemical methods, based on selective solvents. Moreover, we also used the filter readings method with scanning electron microscopy following the protocol of analysis previously setup with Linitest. These techniques are complementary because they could give quantitative and qualitative information, and some of them are more effective according to the kind of polymer investigated. For simplicity, we decided to proceed testing synthetic garments containing polyester, polyamide and polyacrylic fibers separately; to reach the minimum Wascator load (2 kgs) polyester standard weights were used. The fabrics were also characterized before the washing test in Wascator, in order to determine the textile characteristics that could affect the fiber release.

In Table 7 and 8 the physical data collected before the washing tests are shown:

Type	*Bordeaux jumper 100%Acrylic*	*Green Jumper 100% Acrylic*	*Grey cardigan 100% Acrylic*	*Grey pile scarf 100%Polyester*	*Woman underwear jumper PA*	*Black leggings 94%PA 6%EA*
Woven fabric construction	Knitted	Knitted	Knitted	Woven	Knitted	Knitted
Fiber and yarn characteristics	Short Staple- RingSpun- Circular cross section	Short- RingSpun- Circular cross section	Short Staple- RingSpun- Circular cross section	Short Staple- Continuous filament- Circular cross section	Short Staple- Continuous filament- Circular cross section	Short Staple- Continuous filament- Circular cross section
Composition (96/74/CE Directive)	100% Acrylic	100% Acrylic	100% Acrylic	100% Polyester	100% Polyamide	94% Polyamide 6% Elastomer
Tenacity (puncture resistance of the textile ASTM D3787-07)	Mean value[N]:807 CV%:2	Mean value[N]:808,7 CV%:1,8	Mean value[N]:683,5 CV%:1,3	Mean value[N]: 487,3 CV%:7,3	Mean value[N]: 690,5 CV%:12	Mean value[N]: 425,3 CV%:8,2

Table 7. Textile characteristics of the commercial clothes tested.

Pilling (Martindale UNI EN ISO 12945-2)					
Composition / N° of cycles	*Bordeaux jumper 100%Acrylic*	*Green Jumper 100% Acrylic*	*Grey cardigan 100% Acrylic*	*Woman underwear jumper PA*	*Black leggings 94%PA 6%EA*
125	2/3	2/3	3/4	4/5	5
500	1/2	1/2	2	4/5	5
1000	1	1	1	4/5	5
2000	n.d.	n.d.	n.d.	4	5
7000	n.d.	n.d.	n.d.	1	4

Table 8. Textile characteristics of the commercial clothes tested.

Table 8 shows the pilling values of commercial clothes tested; it can be noticed that the worst values were obtained for acrylic jumpers (grade 1 reached already after 1000 rubs); the woman underwear jumper and leggings showed grade 1 and 4 after 7000 rubs. From these data we expected a larger amount of microplastics released by acrylic jumpers. Table 7 shows mechanical and textile characteristics of the fabric; to evaluate the tenacity of the clothes we choose the bursting strength test according to ASTM D3787, because this kind of tenacity analysis is more representative in case of a fabric. Higher values of bursting strength were obtained for acrylic jumpers. The commercial clothes were also characterized before washing tests with light microscopy; in Figure 21, 22 are shown fiber images.

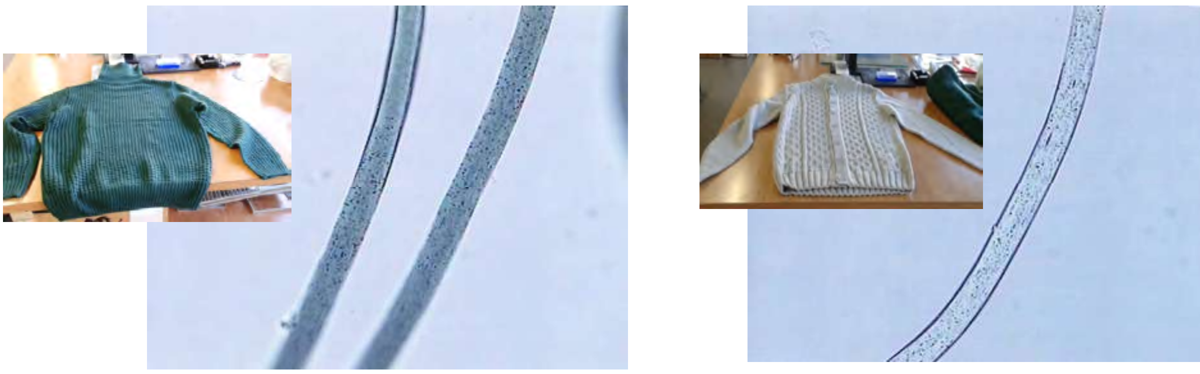


Figure 22. Light Microscopy photos of fibers from 100% acrylic jumper used for Wascator tests, 200X; green and grey cardigan.

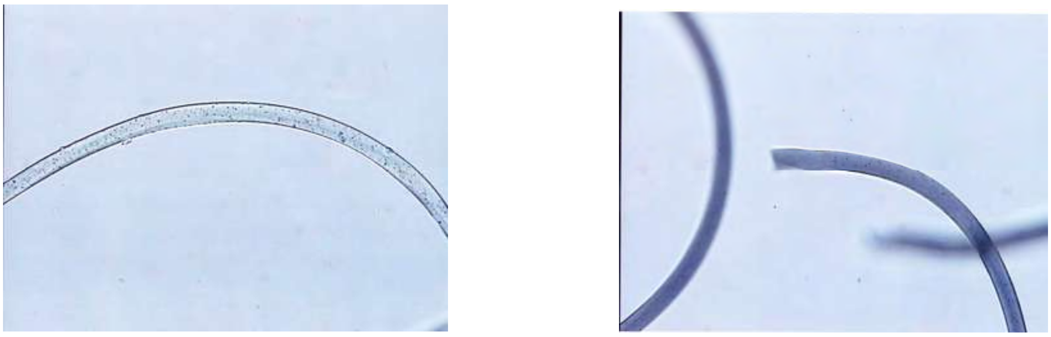


Figure 23. Light Microscopy photos of fibers from 100% polyamide underwear woman jumper and 94% polyamide 6% elastomer black leggings used for Wascator tests, 200X; neutral reactive.

Figure 24. Light Microscopy photos of fibers from 100% polyester scarf; 200X.

Figure 25. Electrolux Wascator FOM 71MP-Lab Washing Machine; CNR-ISMAC equipment.

Prova	Agitazione durante riscaldamento, lavaggio e risciacquo	Carico totale (massa secca)	Lavaggio				1° risciacquo		2° risciacquo			3° risciacquo			4° risciacquo		
			Temperatura 4)	Livello del bagno 5) 9)	Durata del lavaggio a temperatura 8)	Raffreddamento 6)	Livello del bagno 5)	Durata del risciacquo 7) 8)	Livello del bagno 5)	Durata del risciacquo 7) 8)	Durata di centrifugazione 8)	Livello del bagno 5)	Durata del risciacquo 7) 8)	Durata di centrifugazione 8)	Livello del bagno 5)	Durata del risciacquo 7) 8)	Durata di centrifugazione 8)
N°		kg		cm	min		cm	min	cm	min	cm	cm	min	min	cm	min	min
1A	Normale[2)]	4	92 ± 3	10	12	SI	13	3	13	3	1	13	2	1	13	2	6
2A	Normale[2)]	4	60 ± 3	10	12	SI	13	3	13	3	1	13	2	1	13	2	6
3A	Ridotta	2	60 ± 3	10	8	SI	13	3	13	3	-	13	2	1	13	2	2 o sgocciolamento
4A	Ridotta	2	50 ± 3	10	8	SI	13	3	13	3	-	13	2	1	13	2	2 o sgocciolamento
5A	Normale[2)]	4	40 ± 3	10	12	NO	13	3	13	3	1	13	2	1	13	2	6
6A	Ridotta	2	40 ± 3	13	6	NO	13	3	13	3	-	13	2	1	13	2	2 o sgocciolamento
7A	Ridotta[3)]	2	40 ± 3	13	3	NO	13	3	13	3	1	13	2	6			
8A	Ridotta[3)]	2	30 ± 3	13	3	NO	13	3	13	3	-	13	2	2			
9A	Ridotta	2	92 ± 3	10	8	SI	13	3	13	3	-	13	2	-	13	2	2 o sgocciolamento
Lavaggio a mano simulato	Ridotta[3)]	2	40 ± 3	13	1	NO	13	2	13	2	2						

1) *Per le macchine a tamburo di tipo A2, possono essere necessarie alcune modifiche per i cicli di lavaggio del prospetto 1 al fine di ottenere risultati comparabili a quelli eseguiti con le macchine di tipo A1.*
2) *Agitazione ridotta durante il riscaldamento.*
3) *Senza agitazione durante il riscaldamento.*
4) *Tutte le temperature di riempimento per il lavaggio e per il risciacquo sono di 20 ± 5 °C.*
5) *Il livello del bagno è misurato dal fondo del cestello dopo 1 min di funzionamento della macchina e 30 s di arresto.*
6) *Raffreddamento: riempire con acqua fredda fino al livello di 13 cm ed agitare per 2 min supplementari.*
7) *La durata del risciacquo è misurata quando è stato raggiunto il livello del bagno.*
8) *Le durate sono indicate con una tolleranza di ± 20 s.*
9) *I volumi del bagno corrispondenti ai livelli indicati sono determinati con una prova distinta per la quale si usa un recipiente di misura graduato.*

Table 9.Wascator washing conditions according to UNIEN 26330:1996; the conditions chosen are shown in 5A modified with 2 kgs of load.

The washing conditions used for all the Wascator tests are:

- Time: 60 minutes
- Temperature: 40°C
- 1 g/l ECE standard detergent
- 4 rinse after the main wash

Substance	Concentration [%w]
Sodium alkylbenzene sulfonate	8 %
Ethoxylate Alcool	2,9%
Sodic surfactant	3,5%
Sodium polyphosphate	43,7%
Sodium silicate	7,5%
CMC	1,2%
Magnesium silicate	1,9%
EDTA	0,2%
Sodium sulfate	21,2%
Wet %	9,9%

Table 10.ECE standard detergent to UNI EN 26330:1996; the conditions chosen are shown in 5A modified with 2 kgs of load.

During the Wascator tests, were sampled 20 ml from the main wash and 20 ml for each rinse. The total amount of washing solution collected is 100 ml that were then filtered on PVDF membrane and characterized by SEM.
One liter of washing solution from each step (1 l from the main wash and 1 l for each rinse) were collected separately, filtered with sieve, dried at 105°C in oven and then weighed.

Then the residues were characterized by light microscopy, FT-IR spectroscopy, DSC calorimetry and TGA thermogravimetry.

<u>An example of Wascator tests performed with 100% polyacrylic is composed as follows</u>:

- ✓ Bordeaux man jumper 616,84 g
- ✓ Green man jumper 522,48 g
- ✓ Grey man cardigan 570,07 g

In Figure 26 is shown a SEM image of a fiber fragment (microplastic) obtained by filtration of 100 ml washing effluent on PVDF membrane, as explained before.

The microplastics concentration obtained from SEM count is about 4000 fibers per liter of washing solution.

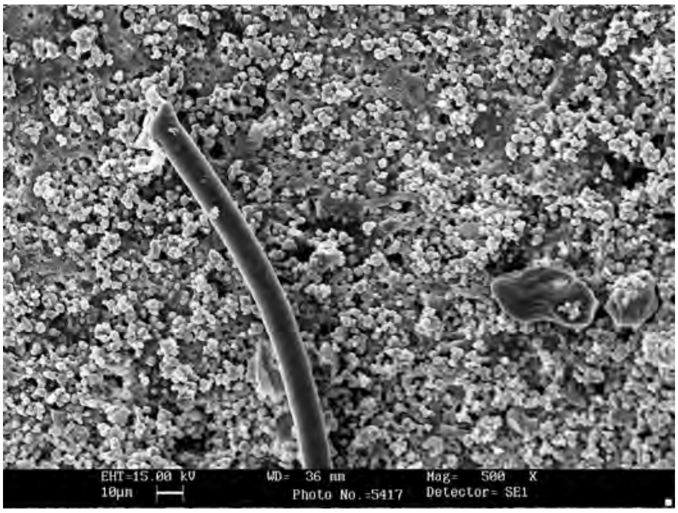


Figure 26. SEM image of acrylic fiber on filter.

Figures 27 shows the images of the filtration residue obtained by light microscopy analysis

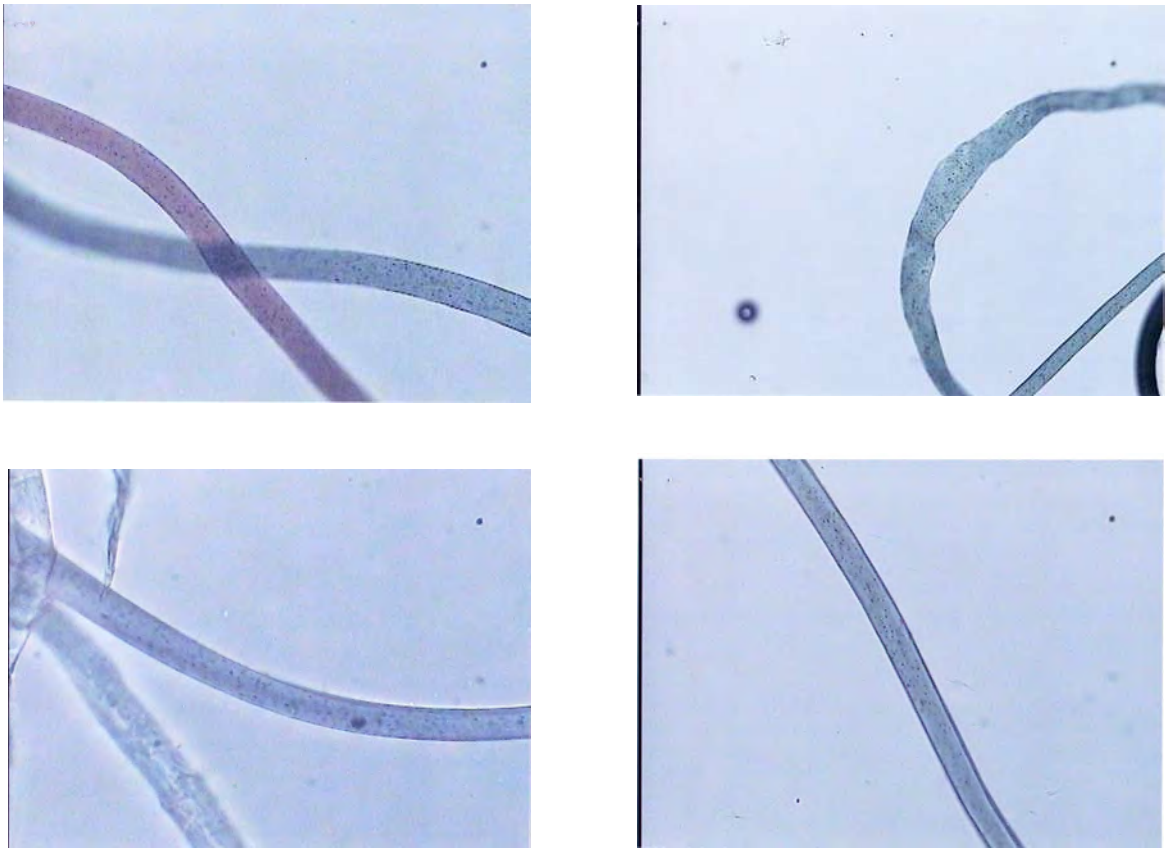


Figure 27. Light microscopy images of acrylic microplastics found in residues after filtration.

The residue obtained from sieve filtration was also analysed with FT-IR spectrometer equipped with an ATR microreflection device.

The sample spectra were collected in attenuated total reflectance mode (ATR), using an ATR accessory (Specac Ltd.) equipped with a ZnSe crystal with a 45° angle of incidence.

Figure 28 shows the spectra of the residue compared to the acrylic standard fabric (reference). It can be noticed the presence of the characteristic peak absorption of acrylic CN group around 2200 cm^{-1}. However, in the residue spectra peak absorption related to cellulose impurity presence around 1000-1100 cm^{-1} are visible. This signals are due to the presence of cotton impurities that are not eliminated even by running washing machine cycles without textiles. On the other hand, the presence of impurities of textile origin is useful to verify better the accuracy of characterization methods chosen, because it can better simulate a real domestic washing process.

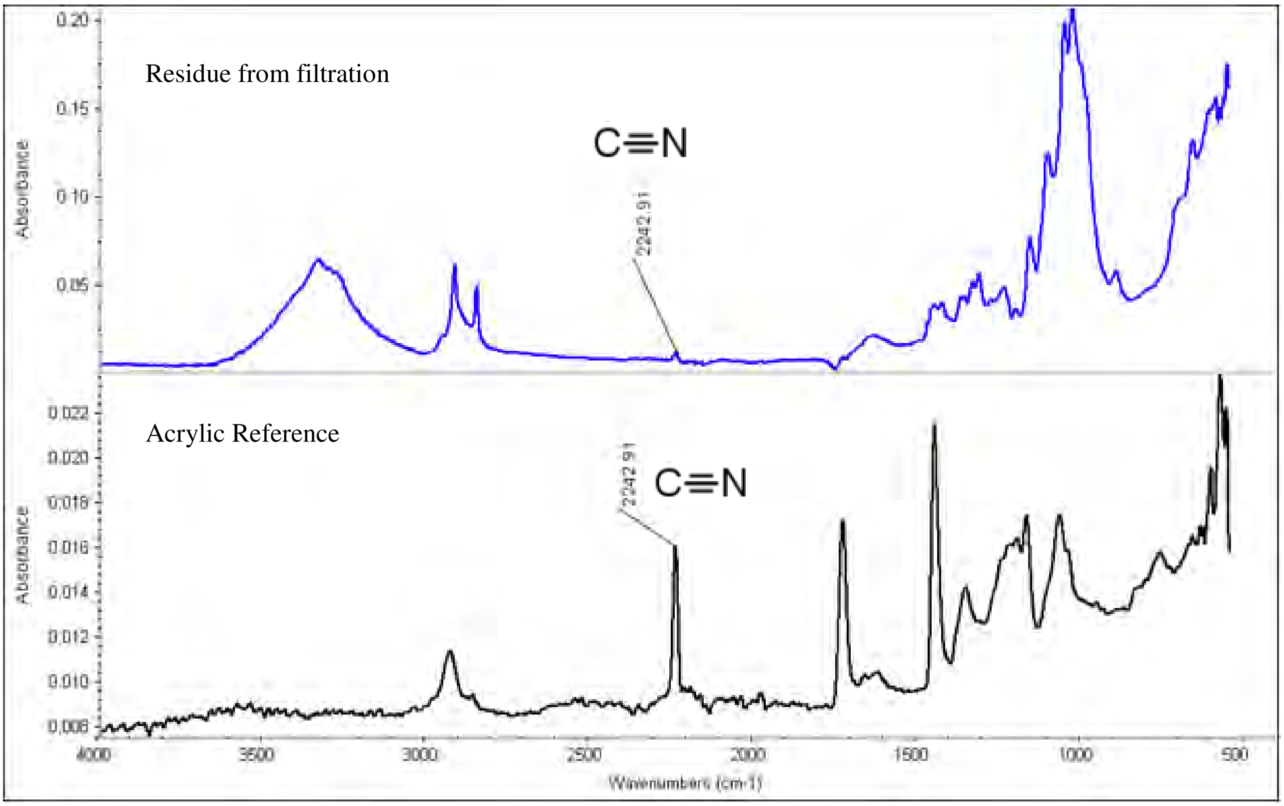


Figure 28. FT-IR ATR Spectra; the upper image shows the residue spectrum, the lower one shows the acrylic reference spectrum.

To quantify the polyacrylic microplastics deriving from washing test, thermal analyisis was first tried. Since the polyacrylic polymer doesn't melt it was decided to carry out a thermogravimetric analysis (TGA/DSC 1, Mettler Toledo) to quantify its content in the residue. A temperature program was set between 30-500°C with a heating rate of 10°C/min. It was possible to build a calibration curve using the loss weight of 4 polyacrylic standard knitted fabric samples by integrating the respective TGA curves (Figure 29). Unfortunately, the TGA analysis seems to be not suitable for our purpose, since it can be noticed, in Figure 29, that the decomposition curve of cotton (that is an impurity) completely overlap the decomposition of acrylic microplastics in the range 280°C - 340°C.

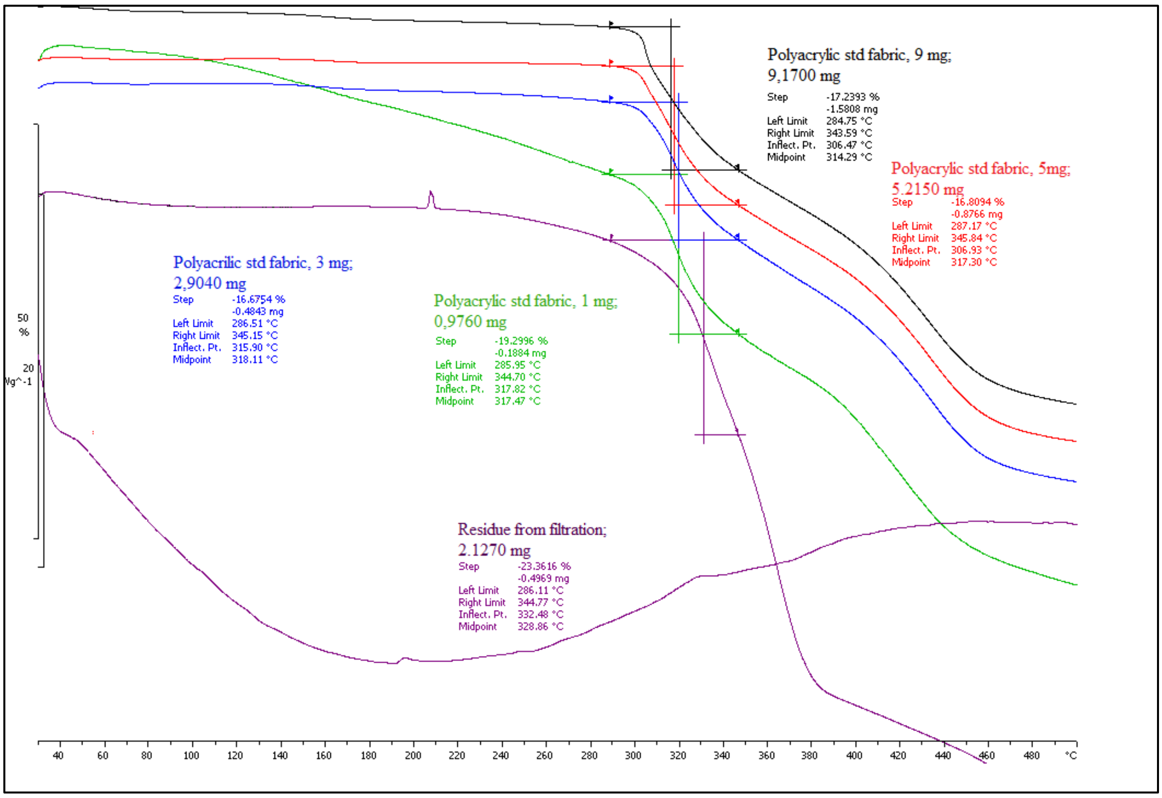


Figure 29. TGA thermograms of Polyacrylic standard fabric and residue from filtration.

To overcome this problem, we setup a chemical method (according to the methods considered in the Italian law of 26th November 1973, N°883) to quantify the presence of the acrylic fragments with a selective solvent (dimethylformamide). Then the quantification was made by weighing the dry sample before and after the treatment with hot dimethylformamide in order to eliminate only acrylic fibers. The residues collected by sieve filtration weight were 0.33 mg, 0.58 mg, 0.40 mg, 0.33 mg and 0.33 mg for each washing step with a total amount of 1.97 mg. The acrylic content in the residue was about 1 mg per 5 liter of wastewater. Thus, since one washing cycle employs 124 l of water, including rinses, the total amount of microplastics released is about 25 mg.

<u>An example of Wascator tests performed with polyamide clothes is composed as follows:</u>

- ✓ Black leggings 83,78 g
- ✓ Underwear woman jumper 182,51 g

In Figure 30 is shown a SEM image of polyamide microplastic, obtained by filtration on PVDF membrane of 100 ml washing effluent.

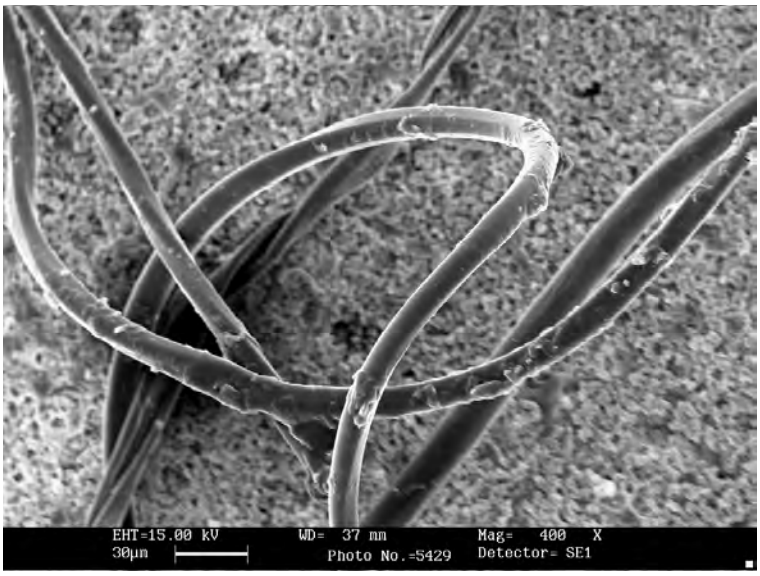


Figure 30. SEM image of polyamide fiber on PVDF filter.

The microplastics concentration obtained from SEM analysis is about 2500 fibers per liter of washing solution. In Figures 31 is shown a light microscopy image of the filtration residue.

Figure 31. Light microscopy image of polyamide found in the filtration residue.

Figure 29 shows the spectrum of the polyamide residue obtained with sieve filtration in comparison with polyamide reference spectrum. The bands observed at around 1532 $cm^{-1}$and 1634 $cm^{-1}$are attributed to the amide II and amide I respectively. These groups are characteristic of polyamide fibers. However, also in this case, we can notice characteristics band absorption due to cellulosic material impurities (1000-1100 cm^{-1}).

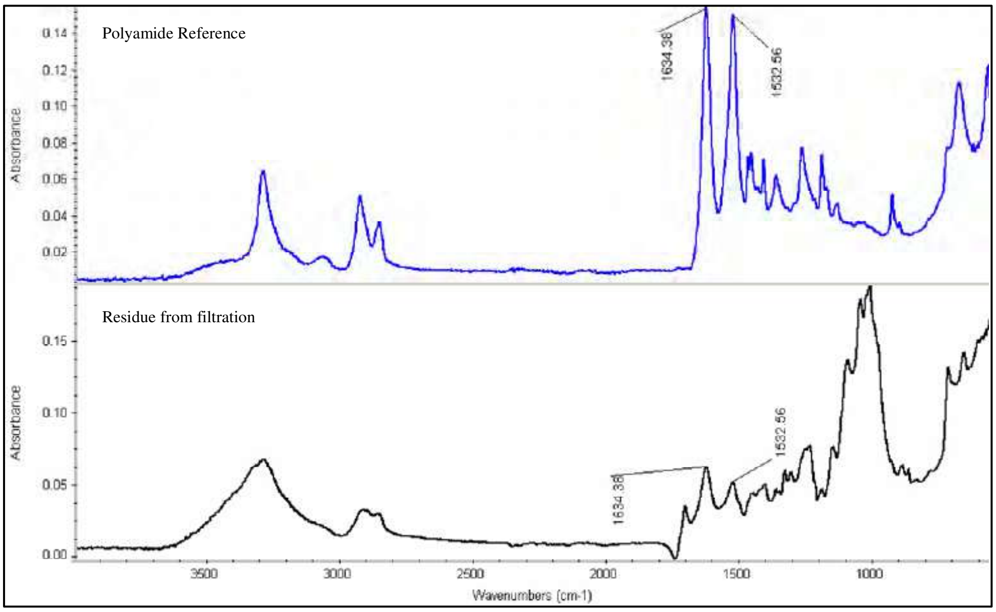


Figure 32. FT-IR ATR spectra of polyamide reference and residue of filtration.

In the case of Polyamides, DSC (DSC 821, Mettler Toledo) was considered a useful technique for the quantification of material released by the washing process, because for this kind of polymers specific melting points are available in data bases (Table 11). The temperature program was set in the range 30-500°C with a heating rate of 10°C/min.

The weights of the residues collected by sieve filtration of 1 liter of laundry effluent deriving from all the washing steps (main wash and rinses) were 0.33 mg, 0.25 mg, 0.13 mg, 0.061 mg and 0.031 mg, with a total amount of 0,802 mg.

In order to quantify the amount of polyamide microplastics contained in the sample, the melting temperatures for standard polyamide 6,6 (around 260°C) and for standard polyamide 6 (around 220°C) were considered, mainly because the market products may contain different kind of polyamides. From the integration of the signals the fusion enthalpy (H) was obtained (Figure 33 and 35) and two calibration curves were built (Figures 34 and 36). In the case of polyamide 6 endothermic signals show in some case a shoulder referable to a polymorphism (hexagonal crystallites melt at a lower temperature respect to the monoclinic fraction). The reliability of calibration curves was successfully checked analysing a known quantity of polyamide knitted fabric that contained both PA6 and PA 6,6 (see black curve in Figure 34).

The total amount of polyamide from the filtration residue, calculated with the calibration curves, was about 0,19 and 0,07 mg for polyamide 6.6 and polyamide 6, respectively. The amount was calculated on the basis of 10 liter of laundry effluents; this means that the amount of microplastics deriving from the whole washing process is about 2,35 mg and 0,87 mg for polyamide 6.6 and polyamide 6, respectively.

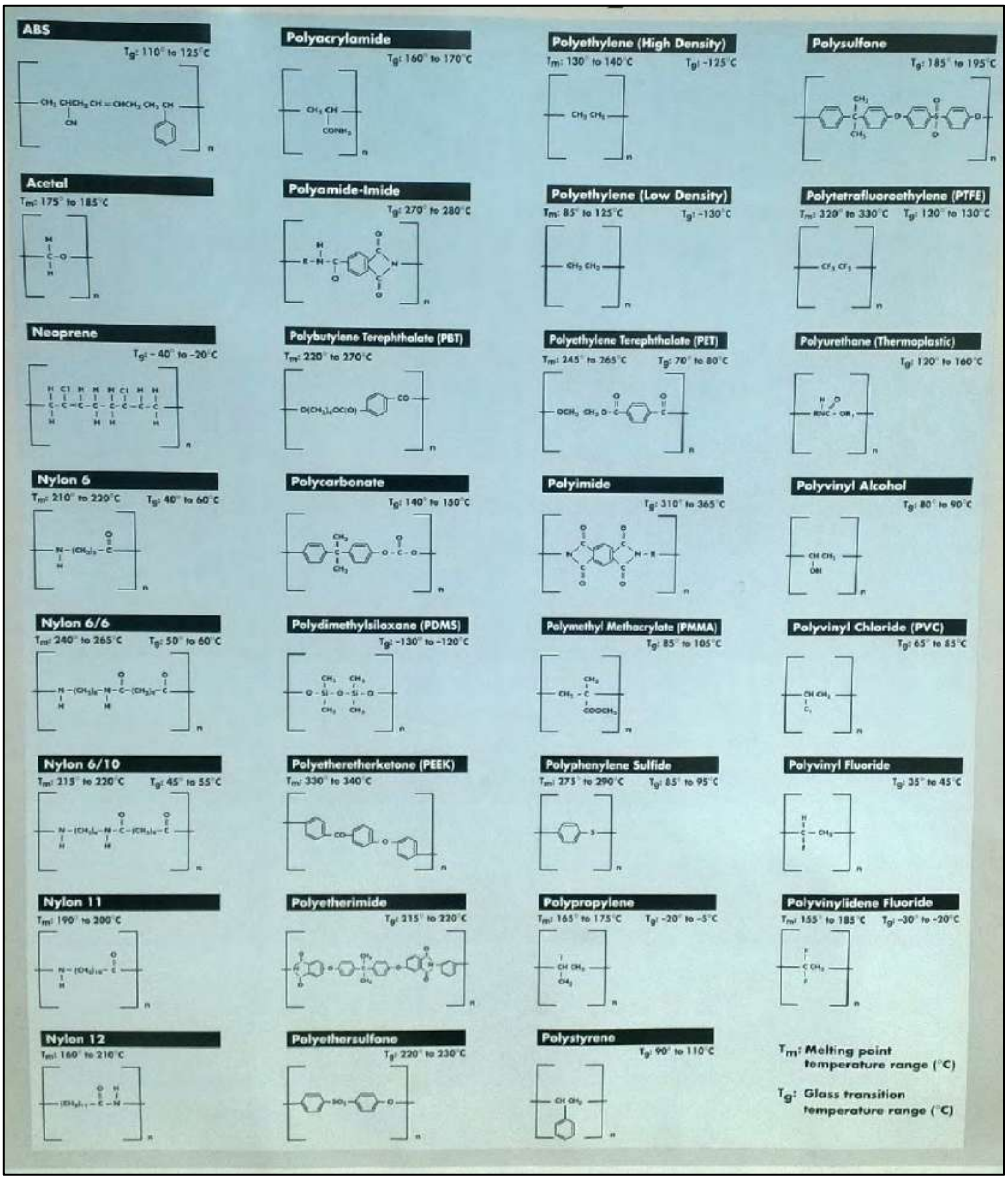


Table 11. Table used as a reference for DSC analysis, for the identification of the melting point of the different polymers.

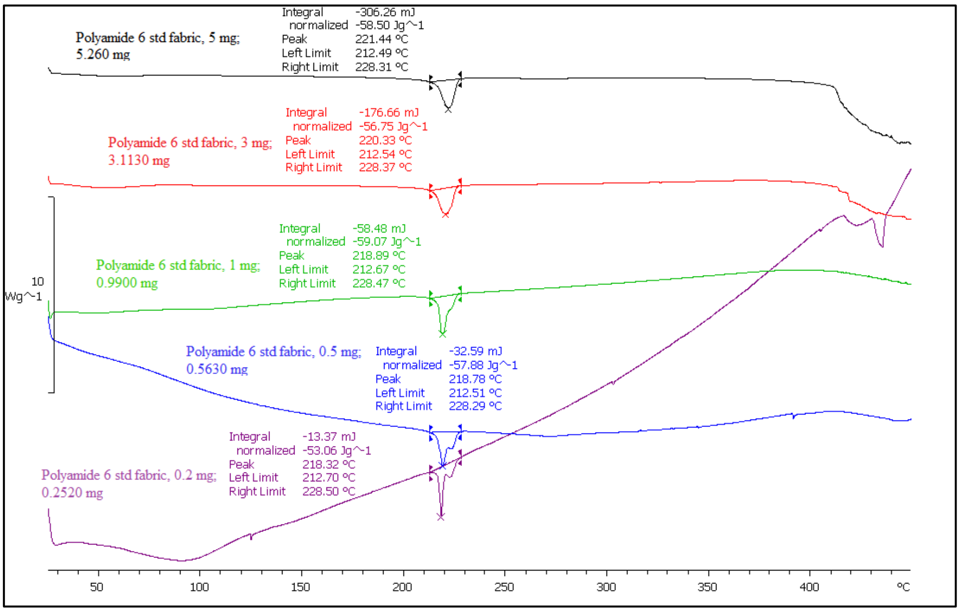


Figure 33. DSC thermograms of polyamide 6 used for the construction of the calibration curve.

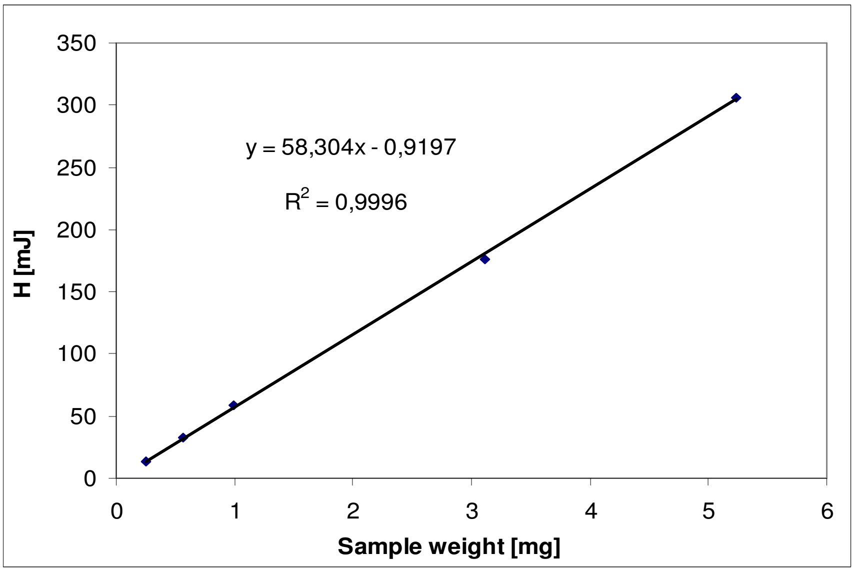


Figure 34. Calibration curve of polyamide 6.

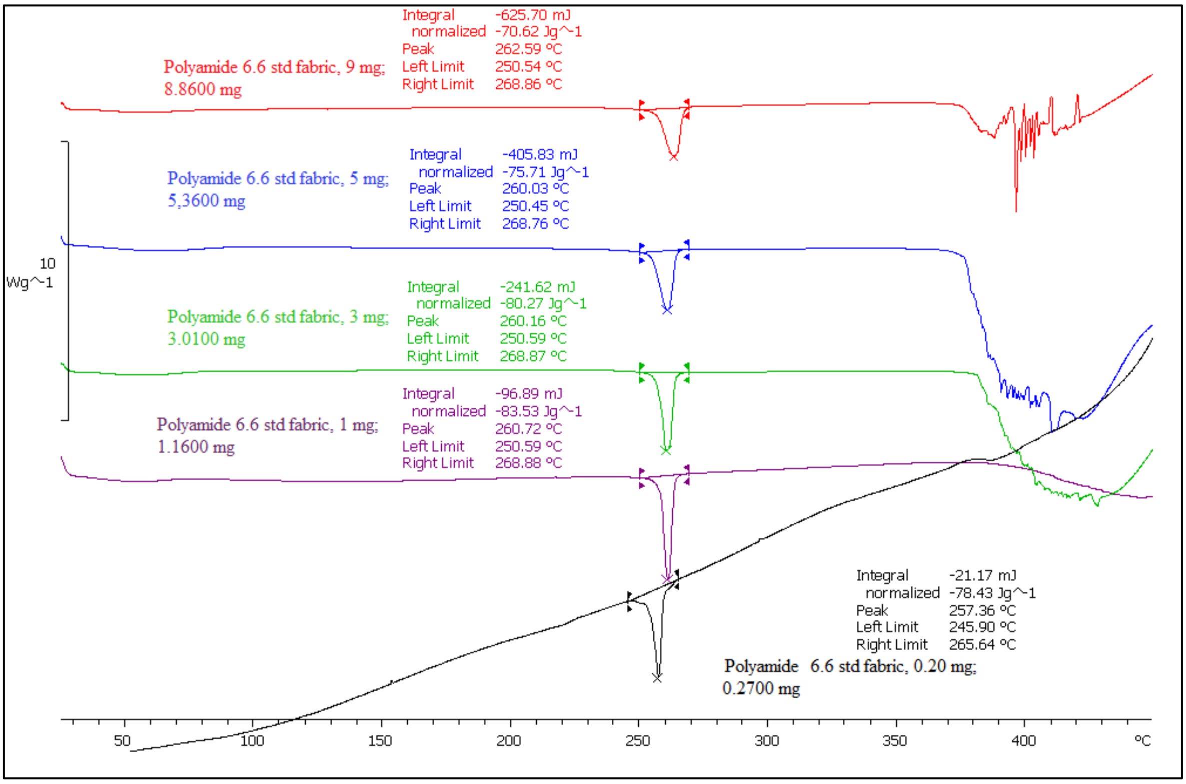


Figure 35. DSC thermograms of polyamide 6.6 used for the construction of the calibration curve.

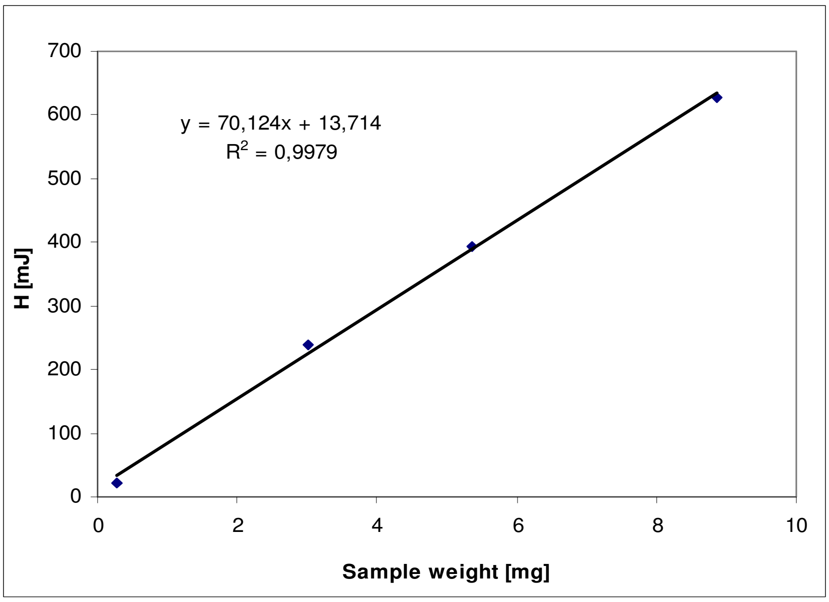


Figure 36. Calibration curve of polyamide 6.6.

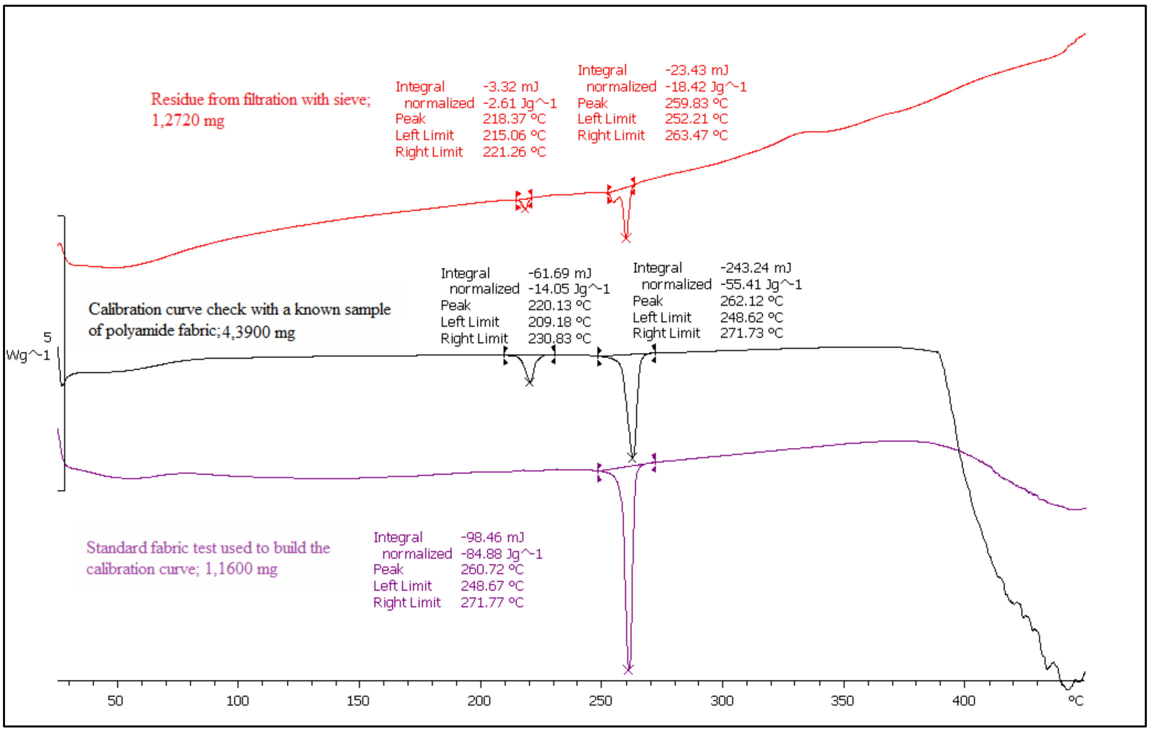


Figure 37. DSC thermograms of polyamide; residue from filtration with sieve in comparison with a known sample used for the calibration curve check and with a standard fabric used for the calibration curve construction.

<u>**An example of Wascator test performed with 100% polyester clothes is composed as follows**</u>:

- ✓ Purple leggings 134,4 g
- ✓ Light blue chinese dress 219,54 g
- ✓ Striped leggings 100,41 g
- ✓ Rose dressing gown 224,95 g
- ✓ Pile blanket 480 g
- ✓ Micropile jumper 200 g

Figure 38 shows a SEM image of a fiber fragment (microplastic) obtained by filtration on PVDF membrane of 100 ml washing effluent as explained before.

Figure 38. SEM image of polyester fiber on filter.

In Figures 39 the light microscopy image of the filtration residue is shown. The microplastics concentration obtained from SEM analysis is about 11000 fibers per liter of washing solution.

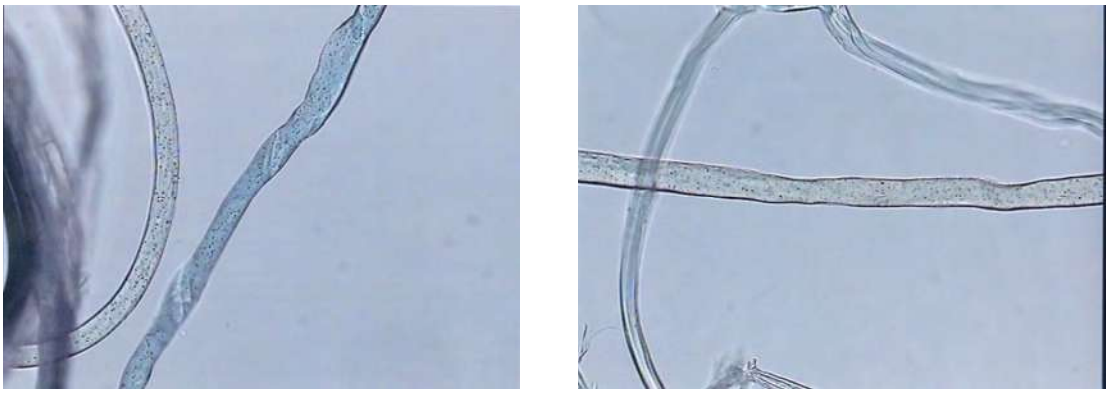


Figure 39. Light microscopy image of polyester found in the filtration residue.

Figure 40 shows the spectra of the polyester residue obtained with sieve filtration in comparison with polyester reference spectrum.

The presence of polyester fibers (microplastics) in the sample is only confirmed by the weak absorption observed at around 1720 cm^{-1}, due to carbonilic group, because the other characteristic bands (aromatic ring for example) are overlapped by the cellulose groups.

The residue collected by sieve filtration weight is 1,232 mg, 1,800 mg, 1,410 mg, 1,151 mg and 1,106 mg for each washing step with a total amount of 6,699 mg; this residue contains the polyester with cotton impurities. In order to quantify the amount of polyester fiber contained in the sample DSC analysis was carried out. Polyesters melting points are expected to be found between 245°C and 265°C.

The calibration curve was built analysing standard fabrics.

From the fusion enthalpy of residue from filtration, it was possible to obtain the weight of polyester contained in the washing effluent; the amount of polyester contained in 5 liters of washing solution is about 0,27 mg, that corresponds to 7 mg for the whole washing process.

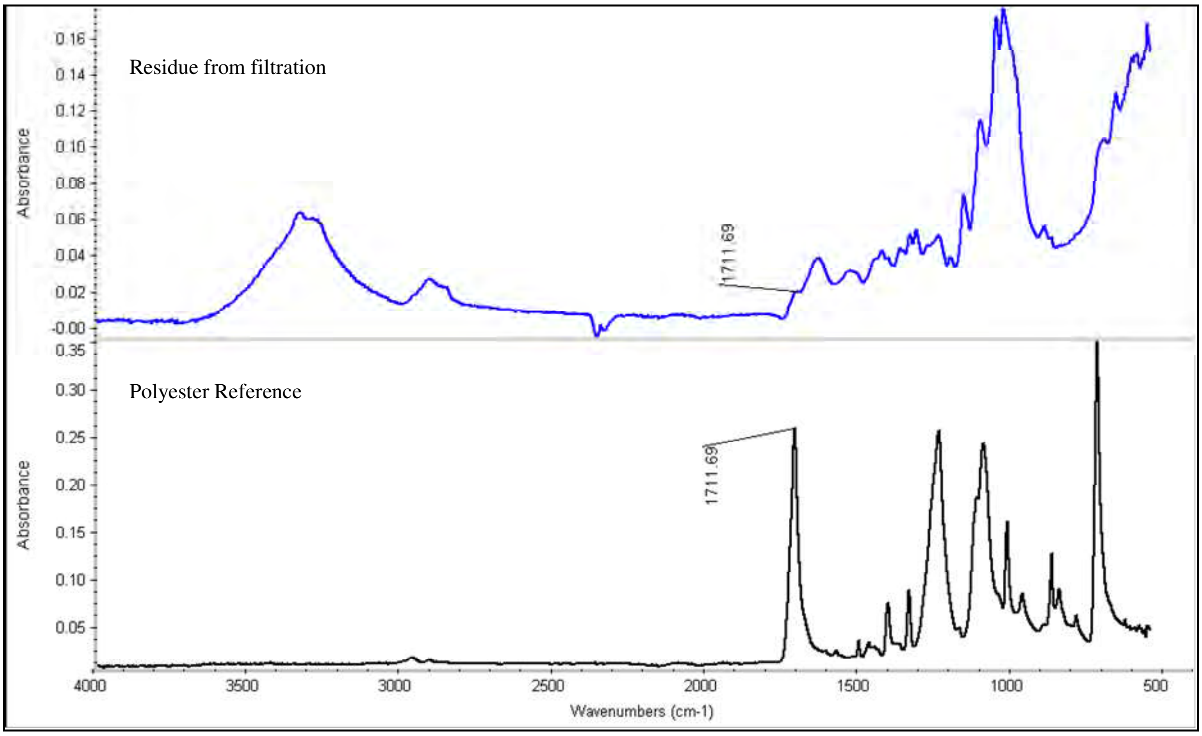


Figure 40. FT-IR ATR spectra of polyester reference and residue from filtration.

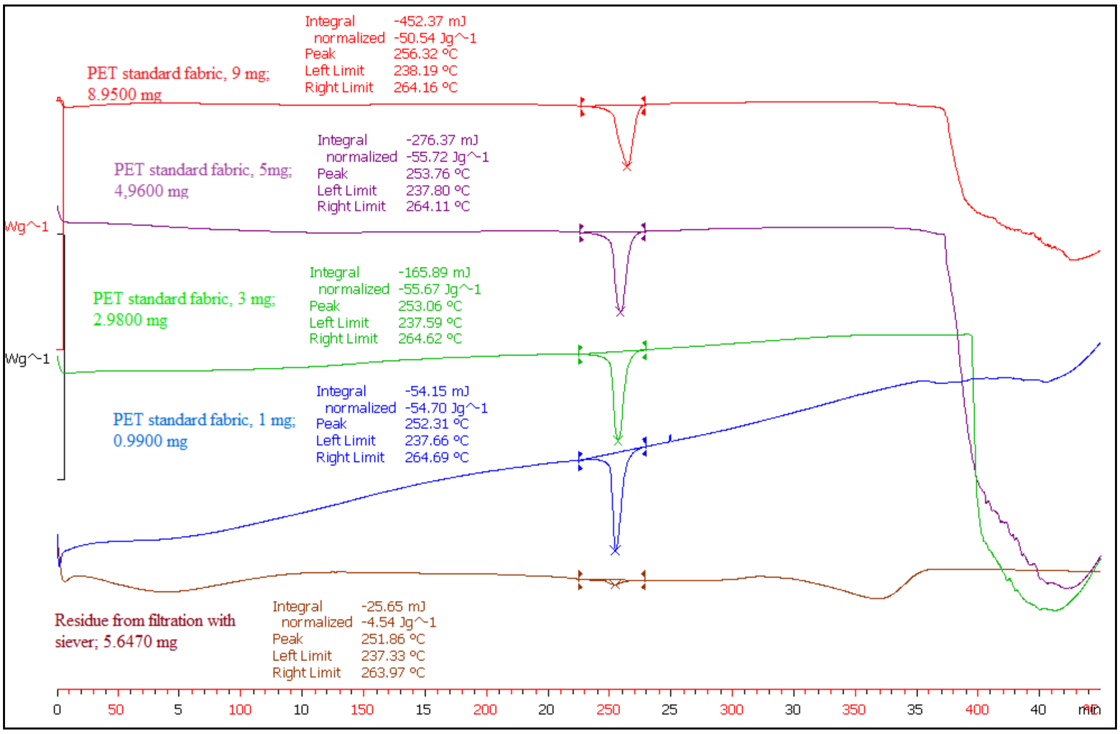


Figure 41. DSC thermograms of polyester; standard fabric used for the building of the calibration line and residue from filtration.

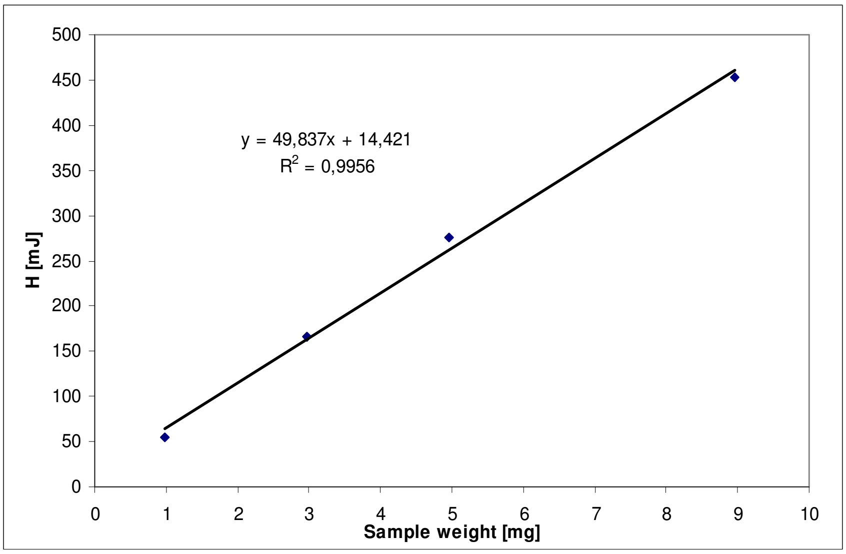


Figure 42. Calibration curve of polyester.

4. Counting Repeatability

In order to confirm the repeatability of the tests performed on standard fabrics, CNR-ISMAC made several experiments with the same test conditions.

The fibers contained in the washing effluents were collected by filtration on PVDF membranes; the filters were analyzed by Scanning Electron Microscopy, taking into account the assumption of a random distribution of the fibers on filter (as already considered in the previous tests).

Table 12 shows an example of the comparison of the data from different experiments, carried out according to the ISO 105-C12 normative. The elaboration of the experimental data confirmed a good reproducibility comparing different samples, since the difference between the values of the coefficient of variation of different tests resulted low.

Composition	*Sample*	*Sample weight [g]*	*Conc.$_{Avg}$ [fibers/g]*	*Conc.$_{Avg}$ [fibers/filter]*	*Mean value between different trials [fibers/g]*	*CV% between different trials [fibers/g]*
100% Polyester woven fabric	*1°sample*	*1,736*	1389	2412	1982	26
	2°sample	*1,820*	2292	4171		
	3°sample	*1,733*	2264	4121		
100%Polyamide woven fabric	*1°sample*	*1,709*	2470	4221	2749	14
	2°sample	*1,709*	3028	5176		
100% Acrylic woven fabric	*1°sample*	*1,960*	1359	2663	1200	19
	2°sample	*2,173*	1041	2261		

Table 12. Repeatability tests performed on polyester, polyamide and acrylic woven fabrics (Ausiliari Tessili srl). The experiments were carried out following the ISO105-C12, with WOB standard detergent.

5. Analysis On Stakeholder's Fabrics

5.1. Summary

On April 9th a meeting has been organized with Radici Group, one of the most important italian chemical companies at international level.

In line with the Mermaids objectives, the meeting, that was held in Biella, represents an opportunity to establish a network with a strategic stakeholder that is also involved in a Life+13 project.

Indeed the company, partner of Officine Maccaferri S.p.A., presented in 2013 an Environmental Life+ project, InnoProWire; the project aims to design an innovative process to produce extruded steel wire for protection and containment structures with a polyamide coating instead of the traditional and more polluting PVC one.

The participants to the meeting were technical and marketing managers, also involved in the InnoProWire project.

The reunion begun with the description of the Company mission, which consists in producing and commercializing synthetic filaments and fibers mainly based on polyester and polyamide polymers, commodities and high value-added products (for example the polyamide 6.10, obtained from green sources such as sebacic acid).

During the meeting the actions and the objectives of the Mermaids and the technical progress till April were exhaustively explained.

The meeting led to some strategic agreements for the Mermaids project, such as the opportunity to test some of the company’s products, to evaluate the influence of further variables on fiber release phenomenon, testing fabrics from various yarns made of polyester and polyamide, raw or dope dyed, such as continuous filament and short staple yarn, textured or air textured filaments.

The tests on the company’s yarn were performed in May by CNR-ISMAC, following the ISO 105-C12 for industrial washing; the data collected were then elaborated to provide a concentration per gram of fabric tested.

5.2. Materials And Methods

In order to investigate the influence on fiber release due to the yarn structure, polyamide and polyester knitted samples has been prepared by Radici for each of the following samples:

- PES#1: Polyester raw textured yarn (3-5 dTex)
- PES#2: Polyester dope dyed textured yarn (3-5 dTex)
- PES#3: Polyester cone dyed textured yarn (3-5 dTex)

- PES#4: Polyester air textured yarn (3-5 dTex)
- PES#5: Polyester microfiber textured yarn (<1 dTex)
- PES#6: Polyester short staple yarn
- PA#1: Polyamide raw textured yarn (3-5 dTex)
- PA#2: Polyamide dope dyed textured yarn (3-5 dTex)
- PA#3: Polyamide cone dyed textured yarn (3-5 dTex)
- PA#4: Polyamide air textured yarn (3-5 dTex)
- PA#5: Polyamide microfiber yarn (<1 dTex).

The study on microplastics release for each sample was carried out by CNR-ISMAC BI, performing industrial washing processes according to the UNI EN ISO 105-C12 standard method, which simulates the stresses caused by 5 to 10 industrial washings, with a good reproducibility. The washing processes were performed with Linitest laboratory equipment, with and without a standard detergent (only water) in order to achieve the reference values with the blank test.
The washing solution coming from each sample, were filtered and the fibers were collected on PVDF membranes. The quantification of fibers released was performed by Scanning Electron Microscopy at a 250X magnification, 80 fields.

5.3. Results And Achievements

In Table 13 and 14 are presented the values obtained from the elaboration of the data achieved from the microscopic observations.

Composition	Washing Conditions	Conc. $_{Avg}$ [fibers/g]
PES #1	Detergent solution	423
	Water only	130
PES #2	Detergent solution	228
	Water only	100
PES #3	Detergent solution	n.d.*
	Water only	800
PES #4	Detergent solution	4566
	Water only	468
PES #5	Detergent solution	2444
	Water only	197
PES #6	Detergent solution	2839
	Water only	310

Table 13. Fibers released by polyester knitted samples expressed per gram of fabric tested. The data were collected by SEM observations of 80 field at 250X on the PVDF membrane used for the filtration of the washing solution collected from the industrial washing processes in Linitest (ISO 105-C12).
*** The amount of fibers collected on the filter can't be determined due to the high quantity of fibers released.**

Composition	*Washing Conditions*	*Conc. $_{Avg}$ [fibers/g]*
PA #1	*Detergent solution*	2725
	Water only	475
PA #2	*Detergent solution*	1055
	Water only	292
PA #3	*Detergent solution*	n.d.**
	Water only	n.d.**
PA #4	*Detergent solution*	5774
	Water only	195
PA #5	*Detergent solution*	1719
	Water only	605

Table 14. Fibers released by polyamide knitted samples expressed per gram of fabric tested. The data were collected by SEM observations of 80 field at 250X on the PVDF membrane used for the filtration of the washing solution collected from the industrial washing processes in Linitest (ISO 105-C12).
**** The value for sample "PA #3" , because the knitted sample was weak to industrial washing mechanical stress.**

Correlating the experimental data with the yarn characteristics, the importance of some parameters which can affect the amount of the microplastics released resulted very clear. In this phase of the preparatory actions the importance of some variables, such as the fiber characteristics and the detergent presence, were confirmed.
The experimental data shows that the highest values were achieved with air textured yarns, both polyester and polyamide ones. Another parameter that can affect the fiber release is the kind of yarn tested; indeed the short staple yarn shows higher fiber losses compared to the textured continuous yarn (except the air textured one).
Other important variables for fiber release are:

- ✓ Detergent presence: in all cases, the presence of the detergent causes an increase in fiber losses, probably due to the increase of fabric wettability (compared with the blank tests, only water);
- ✓ Dyeing process: the cone-dyeing process shows an increase in the value of fiber released.

Figures 43, 44, 45, 46, 47 show the SEM analysis of the fibers coming from the Linitest washing solution collected on PVDF membranes.

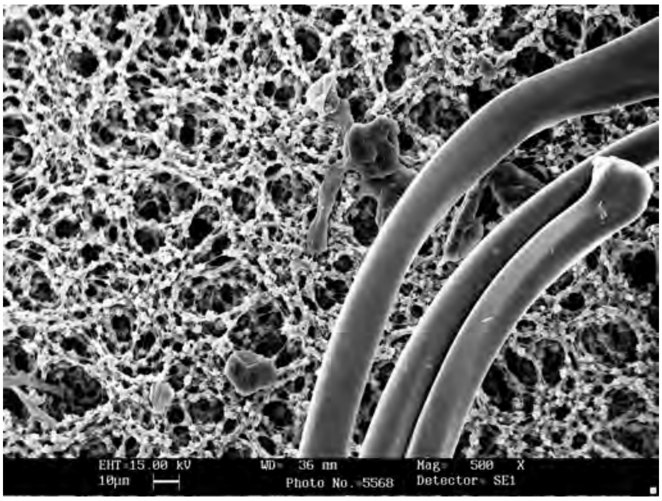


Figure 43. Fibers released by raw white textured polyamide sample (PA#1).

Figure 44. Fiber released by raw white textured polyester sample (PES#1).

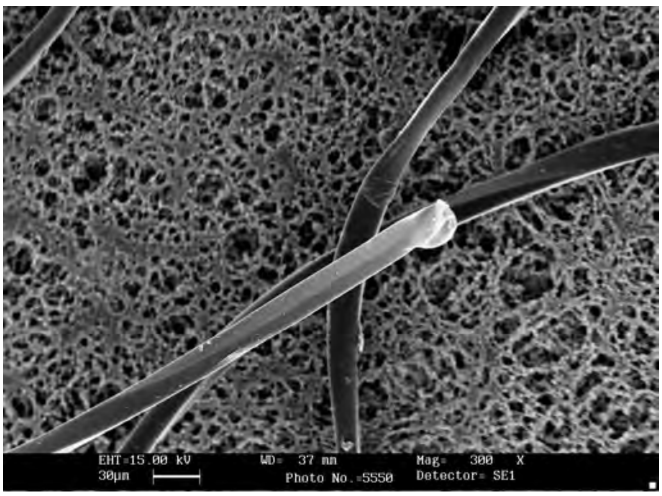


Figure 45. Fibers released by cone dyed textured polyester sample (PES#3).

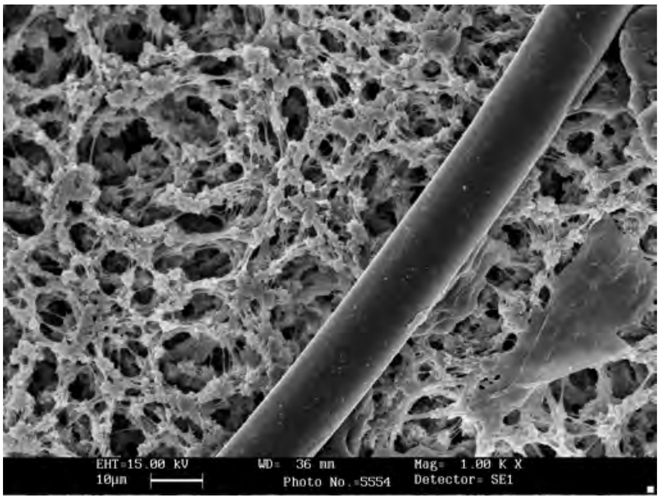


Figure 46. Fiber released by raw white air textured polyester sample (PES#4).

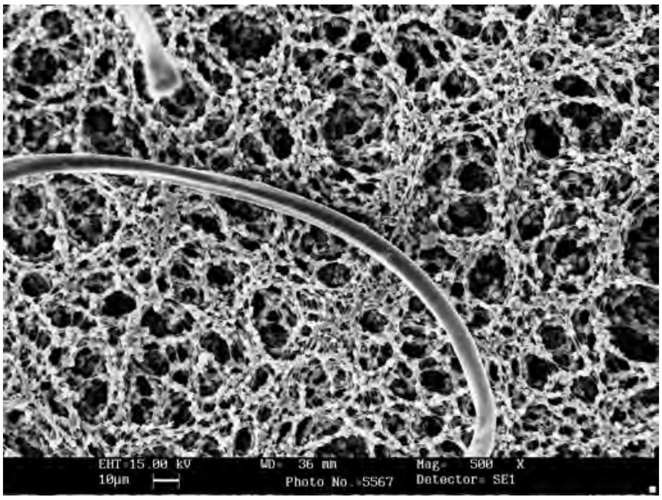


Figure 47. Fiber released by raw white textured microfiber polyester sample (PES#5).

Regarding the sample PES#3 (cone-dyed textured yarn), a numerical quantification by SEM analysis was not possible, due to the high amount of fibers released during the washing test. However, the quantification of microplastics was carried out gravimetrically (dry weight 105°C). The weight of fibers obtained after sieve filtration is 0,61 mg for each gram of fabric. The chemical composition of fibers was determined by FT-IR ATR analysis in order to check the correspondence with the fabric tested (polyester).
The bands were assigned as follows: 1713 cm^{-1} (C=O), 1242 cm^{-1} (stretching C-O bonds), 1095 cm^{-1}, C-O bond vibration crystalline fraction (stretching), and 724 cm^{-1} (aromatic ring). As shown in Figure 48, the absorption bands of the fabric tested (reference sample) and the microplastic residue ones, are completely overlapped.
In order to confirm the correspondence between the residue and the fabric reference, DSC analysis was carried out.
Figure 49 shows the thermogram of polyester residue deriving from the industrial washing effluents. Polyesters melting points are expected to be found between 245°C and 265°C. The characteristic endothermic peak of polyester resulted to be around 252°C.

Figure 50 shows the light microscopy images of polyester fiber residue deriving from the fabric tested.

Wawelenght [cm^{-1}]	**Peak absorption**
1713	C=O Bond
724	Aromatic ring vibration
1340-1177	–CH2 Bending Vibration
1242	C-O bond vibration (stretching)
1095	C-O bond vibration crystalline fraction (stretching)

Table 15. Absorption peaks of polyester fabric.

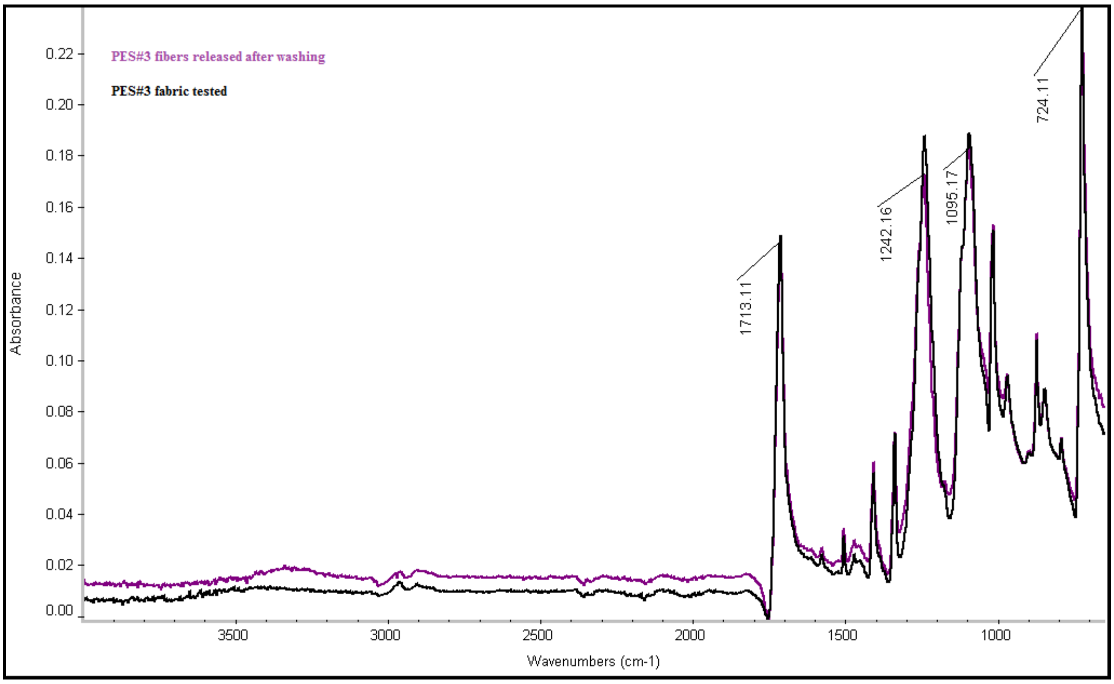


Figure 48. FT-IR ATR spectra of the cone-dyed textured polyester; comparison between polyester fabric tested (reference, black line) and polyester fibers lost during the washing process, collected on sieve and dried at 105°C (purple line).

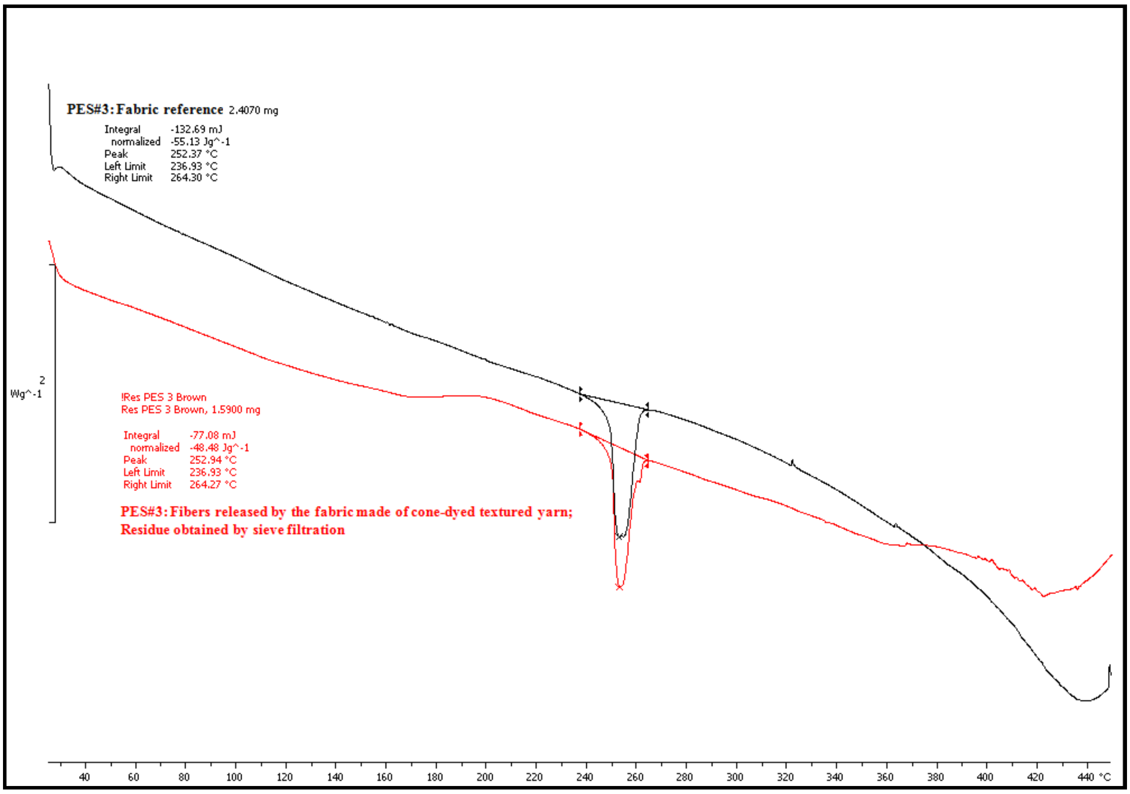


Figure 49. DSC thermogram of polyester fibers released by the washing test of the fabric made of cone-dyed textured polyester yarn (Linitest ISO105-C12).

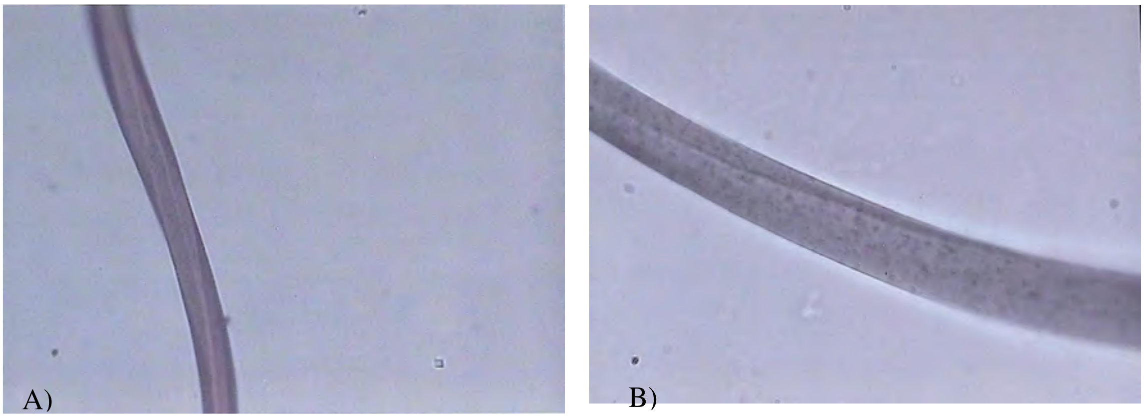


Figure 50. Light microscope photos of polyester fibers released by the washing test of the fabric made of brown cone-dyed textured polyester yarn (Linitest ISO 105-C12); A) 200X magnitude, neutral reactive B) 400X magnitude, neutral reactive.

6. Conclusions

The major achievements of Preparatory Action A - Task A 1.2 were the setup of a statistical method for the numerical quantification of microplastics by SEM analysis. Through the SEM observations we also highlighted an important factor: microplastics released from the washing of synthetic fabrics may be mainly identified on the basis of the shape of the original fibers, since they are actually fiber snippets. Neverthless the smallest fragments cannot be identified with this identification and classification methodology. However, the largest fiber fragments will generate thousands of smaller fragments when released in the ambient, so that targeting on the reduction of the fiber snippets represents an effective way of mitigation of microplastics impact caused by textile washing processes. Moreover the work carried out allowed identifying microplastics from washing processes of textiles containing synthetic fibers (pure or in blend with natural or other man-made fibers) by means of light microscopy, FT-IR ATR spectroscopy, DSC and TGA techniques. Finally, the quantification by weight of the filtration residues and by means of DSC and TGA techniques was carried out.

For Task A1.3 the choice of textile products to be subjected to washing tests was done considering representative samples of the European market. The labelled fibre composition was confirmed at the quality control laboratory of CNR ISMAC following the rules of 96/73/EC directive. The information have been correlated with the results obtained in task A1.2 in order to determine which kind of textiles represents the main cause of contamination of effluents arising from domestic washing. Evaluating the results, parameters such as the fabric construction and the fiber length, which may affect the release of microplastics during washing, have also been considered. From these evaluations it can be stated that the more important variables for microplastics release are linked to the detergent used, textile construction characteristics, pilling propensity and chemical and physical properties of the fiber. For the development of the implementation Actions (Action B), we can start from these achievements, using additives, detergents and textile treatments in order to modulate some textile characteristics (such as pilling propensity) that are responsible for increased microplastics release.

The end of the Preparatory Actions was postponed by three months; from April 2015 to June 2015 the major activity regarded the confirmation of the experimental data, the validation of the counting method, carried on with run trials between CNR-ISMAC and CNR-IPCB and the analysis of stakeholders' fabrics. The comparison among the two counting methods confirmed the reproducibility of the whole procedure used to quantify the microfibers release, since the obtained results displayed the same order of magnitude. Few modifications will be applied to the Method N. 1 in order to make it as representative as the N. 2 of the whole filter area.

CNR-ISMAC carried out further tests to confirm the reproducibility of the experiments, using polyester, polyamide and polyacrylic standard fabrics; a good repeatability was confirmed, in the experimental conditions and statistical assumptions made (random distribution of the fibers on filter).

Furthermore, CNR-ISMAC carried out some experiments on stakeholders samples, in order to quantify the fiber losses from fabrics made of non standard yarns, coming from production. This step also aimed to establish a connection between different yarns, whose fabrics were knitted on the same knitting machine, and the amount of fiber released. The experimental data show that the higher values were obtained with air textured and cone dyed yarns. The data collected with the stakeholder's fabric confirm the same order of magnitude of the values deriving from the tests performed on standard fabrics. However it can be noticed a raise only in air textured yarn and cone dyed in comparison with the standard textile tested.

7. Bibliography

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mermaids. OCEAN CLEAN WASH

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mermaids. OCEAN CLEAN WASH

LIFE13 ENV/IT/001069

Mitigation of microplastics impact caused by textile washing processes

Contents

TASK A.2.2.1 - Study of the different types of commercial laundry additives and their contribution to the reduction of fibre breakage (Market Study).

1. Introduction[1]

Soaps, detergents and maintenance products are used every day by millions of consumers. They make it possible to keep our homes clean and hygienic. They remove stains and dirt from clothing and help us maintain our personal possessions in good condition. They ensure cleanliness in public spaces, which has a highly beneficial effect on health by combating the spread of germs. Last but not least, a clean and healthy environment provides a sense of well-being to all who live or work there.

Maintaining a level of cleanliness and hygiene necessary for healthy living, both inside and outside the home, demands regular attention and an understanding of good cleaning practices. The soaps, detergents and maintenance products industry offers a wide range of products which, when used in accordance with the usage and safety instructions on the labels, help get rid of dirt, combat germs and keep your belongings looking as good as new.

The Part I of this report is a market study of common detergent products for laundry in Europe in order to get an idea of the principal ingredients used in their elaboration. The next steps are to study how some of the products analyzed in this report can increase or decrease the release of micro-fibers in the water before a number of machine washings. Thereby, the influence of some ingredients in the fiber care is expected to be determined, as well as its improvement adding new compounds in the detergent formulation.

1.1. Economic Perspective[2]

Total market value of the overall detergents and maintenance products Industry (for both household and Industrial & Institutional products) for 2013 in the EU28 plus Norway and Switzerland is estimated to have reached 35.1 billion euro. The overall total household value in 2013 is estimated to have reached 28.5 billion euro and shows a growth of approximately 1.0% (based on fixed exchange rates). The 48.2% of this value corresponds specifically to Laundry Care, this is 13,745 million euro. In the following table the share of each type of detergents is shown:

Table 1. Market value of each type of detergent for laundry

Laundry Care	Million euro
Powder Detergents	3,411
Liquid Detergents	4,238
Unit Doses	936
Fabric Conditioners	2,335
Laundry Aids, Others	2,825
Total	13,745

2. Household Laundry Products[3]

Laundry products currently on the market in various parts of the world, and particularly in Europe, can be classified into the following groups from a product standpoint:

2.1. Heavy-duty detergents

This category includes those detergent products suited to all types of laundry and to all wash temperatures. They are offered in the delivery forms of conventional powders, extrudates, tablets, bars, and liquids and pastes.

Liquid heavy-duty detergents are distinctive because of their usually higher surfactant content recently (up to ca. 50%). Unlike powdered detergents, they rarely contain builders such as zeolite or triphosphate and are generally devoid of bleaching agents, the latter for reasons of loss of active oxygen and stability problems with enzymes during storage. They are most effective in removing greasy and oily soil, especially at wash temperatures below 60 ºC. In the following table a general comparison of the usual composition between powder and liquid detergents is shown.

Table 2. Comparison of the usual composition between powder and liquid heavy-duty detergents

Ingredient	Powder	Liquid
Anionic and non-ionic surfactants	✓	✓
Soap	✓	✓
Builders	✓	✓
Solubilizer	×	✓
Alcohols	×	✓
Cobuilders	✓	×
Bleaching agents	✓	×
Bleach activators	✓	×
Antiredeposition agents	✓	✓
Stabilizers	✓	✓
Foam regulators	✓	×
Enzymes	✓	✓
Optical brighteners	✓	✓
Soil repellents	✓	✓
Fillers/processing aids	✓	×
Fragrance	✓	✓
Minors	✓	✓
Water	✓	✓

2.2.Special detergents

These are products developed for washing specific types of laundry. Such detergents are generally used in washing machines but also for hand washing. They can be characterized by the fact that fabric care ranks high and is a more important feature than with heavy-duty detergents, for which soil and stain removal is the key characteristic. These products are offered both as powder and liquid format, although liquid form is increasing.

Detergents specially designed for delicate and colored laundry contain neither bleach nor fluorescent whitening agents, which may adversely affect sensitive dyes used with some of these fabrics. Most products contain cellulases, which helps fabrics look new longer and keep colors bright. Dye transfer inhibitors are added to some special detergents. In the following table a general comparison of the usual composition between powder and liquid special detergents is shown.

Table 3. Comparison of the usual ingredients between powder and liquid special detergents

Ingredient	Powder	Liquid
Anionic and non-ionic surfactants	✓	✓
Soap	✓	✓
Zeolite A	✓	✓
Solubilizer	×	✓
Hydrotropes	×	✓
Sodium perborate	✓	×
Sodium silicate	✓	×
Sodium polycarboxylate	✓	×
Antiredeposition agents	✓	×
Enzymes	✓	✓
Optical brighteners	×	×
Dye transfer inhibitors	✓	✓
Fragrance	✓	✓
Water	✓	✓

2.3.Laundry aids

Laundry aids are products developed to meet the varying needs of often widely divergent washing habits and practices in different parts of the world. In Europe, most powered heavy-duty detergents contain oxygen active ingredients. Depending on their use, laundry aids can be divided into the following categories:

- Pretreatment aids
  The most important are soil and stain removers and water softeners. The first ones are frequently products with high surfactant contents which helps removing greasy and/or bleachable stains. The latter are added to prevent build-up of lime deposits upon multicycle washing due to water of medium to high hardness.

- Boosters
  Boosters are products that can be added separately to detergents to exert specific influences on the washing process and thereby improve its effectiveness. The principal types of boosters are bleaching agents and laundry boosters. Bleaching agents are available in both powder and liquid form; powder bleaching agents generally contain sodium perborate or sodium percarbonate, whereas most liquid bleaching agents are dilute solutions of sodium hypochlorite or hydrogen peroxide. Laundry boosters contain either sodium silicate, sodium citrate or sodium carbonate, usually in combination with surfactants and enzymes.

- Aftertreatment aids
  The goal of these products is to increase the usefulness of laundry by restoring textile characteristics that have suffered the course of the wash. To achieve that, products can be classified in fabric softeners and stiffeners.
  Fabric softeners provide fabrics of soft sensation and pleasant fragrance. Also, they impart good antistatic properties and make fabrics easier to iron. Their principal active ingredients are usually cationic surfactants of the quaternary ammonium type.
  Stiffeners are used to get stiffness and body in fabrics. Usual agents for this purpose include natural starch derived from rice, corn or potato, although synthetic polymeric stiffeners are also used. In addition they contain substances such as poly(vinyl acetate) partially hydrolyzed to poly(vinyl alcohol).

3. Composition of laundry detergents[4]

Laundry detergents are composed of a range of ingredients that give the final product its different characteristics. The most common ingredients are listed alphabetically below (not all of them are necessarily present in a given product).

Anti-redeposition agents

Anti-redeposition agents are added to prevent the dirt that is removed from the clothing from reattaching itself. Accordingly they also prevent the clothing from taking on a grayish color. Commonly occurring anti-redeposition agents in laundry detergents are CMC (carboxymethyl cellulose), CEC (carboxyethyl cellulose), starch-based compounds or other polymers.

Bleaching agents

Bleaching agents are added to remove or decolorize (whiten or lighten) a type of stain that is not removed by the surfactants, i.e. colored stains such as red wine or tea. Bleaching agents work by cutting the stain molecule into smaller pieces that are more easily dissolved and removed. Alternatively, bleaching agents may work by removing the color of the stain so it becomes invisible. These two processes may work simultaneously on a given stain. Bleaching agents used in laundry detergents include borate, perborate, percarbonates, hydrogen peroxide and sodium hypochlorite.

Buffering agents

Buffering agents work by upgrading or protecting the cleaning efficiency of the surfactant. As washing proceeds, the alkalinity or pH drops. Under acidic (low pH) conditions, cleaning is reduced. Buffering agents stabilize the pH, and keep this drop from being too severe and reducing the cleaning.

Builders/sequestering agents

Builders/sequestering agents are primarily added to bind calcium in the water and in the soils on the clothing. The binding of calcium allows the surfactants better access to clean the clothes, as the soil molecules are often bound to the fabric by calcium ion bridging. The binding of calcium also prevents the ions to react with other detergent ingredients and precipitate from the solution. Builders are thus a very important ingredient in laundry detergents in areas in which hard water is used for washing. The builders used in laundry detergents include e.g. phosphate, phosphonate, zeolite, silicates, carbonate and citrate. Often more builders are used in one product, creating a builder system. There is wide variation in the builders used in the European countries. One reason for this is the difference in the number of households connected to treatment plants and whether the treatment plants are capable of removing builders from the water. Another reason is the difference in water hardness in different regions.

Colorants

Coloring agents are added to laundry detergents for aesthetic reasons and/or to aid the marketing of the products. Coloring agents have no effect on the ability of the product to clean clothes.

Corrosion-inhibiting substances

Corrosion-inhibiting substances protect the washing machine against corrosion. Silicates are the most commonly used, and they also function as stabilisers for perborates (see bleaching agents).

Dye-transfer inhibitors (DTIs)

A DTI is a substance that prevents the transfer of dyes from one textile to another during the wash. DTI's are either polymers or copolymers; the most commonly used DTI is PVP (Polyvinylpyrrolidon).

Enzymes

Enzymes are proteins that are used in laundry detergents to break down particularly difficult stains. Enzymes also help to improve wash results at low temperatures. Enzymes function by breaking down difficult stains into smaller parts which can then be more readily removed. The enzymes do not lose the functionality after use on a given stain. Enzymes used in laundry detergents and stain removers include protease, lipase and amylase. Each enzyme has its own defined target, i.e. fat stains, protein stains etc.
Enzymes can replace large quantities of chemicals with the same function.

Fabrics whitening agents (optical brighteners)

Fabrics whitening agents or optical brighteners are fluorescent whitening agents that reflect the ultra-violet rays of the sun as white, visible light. This gives the clothing an impression of whiteness and accordingly cleanness. Optical brighteners are frequently already present in clothing, and the quantity left by the washing process helps to give an impression of cleanness. Optical brighteners/fabric whiteners do not bleach or remove color from the textile.

Fillers

Fillers are added to laundry detergents to give the product structure. In powder products sodium sulphate is often used as filler - in liquid products the filler is water.

Fragrances (perfume)

Fragrances are added to give the product a particular smell or to conceal an unpleasant smell in the raw materials used in the products. Fragrance has no effect on the ability of the product to clean the clothes. Fragrance is a mixture of many different aromatics. Commonly little information is available on the environmental effects of fragrance, but generally fragrances are regarded as environmentally harmful. Allergies to fragrances are common.

Hydrotropes

Hydrotropes help increase the solubility of other ingredients in liquid products.

Preservatives

Preservatives are frequently added to liquid products to prevent the growth of bacteria in the product and thereby extend its durability. Surfactants sometimes have a preservative effect.

Soap

Soap is used for cleaning the fabric by reducing surface tension and loosening, dispersing, and suspending the soil. Soap also softens fabrics naturally. Soap may also be used as a suds inhibitor.

Solvents

Organic solvents are primarily used in liquid products to dissolve the ingredients. Typical solvents are alcohols. Alcohols also add an anti-freeze property so that the detergent may be shipped and stored in cold climates.

Suds inhibitors

Suds inhibitors are added to reduce the quantity of suds (foam) in the washing machine. Suds inhibitors in laundry detergents include silicone or surfactants with this particular property.

Surfactants

Surfactants are added for a number of reasons, the main purposes being their washing and surface active properties. They remove soils from the clothing and keep it suspended in the washing solution. Some surfactants perform other functions such as foam inhibition. Examples of surfactants used in laundry detergents include alkylether
sulphates, alkyl sulphates, alcohol ethoxylates and alkylphenol ethoxylates.

4. Products in the European market

4.1. Objectives

The objective of this study is to perform a detailed evaluation of liquid, powdered and special detergents in the European market, as well as laundry aids. Thereby, a global vision about the ingredients more used in this market can be exposed. Therefore, an analysis of a wide number of detergents in European markets has been carried out considering products from the principal multinational such as Henkel, Procter & Gamble, Reckitt Benckiser and Unilever, as well as Eco products and generic brands.
The main ingredients to be assessed are:

- Surfactants
- Polymers
- Alkaline agents

4.2. Information search

Information related to the ingredients has been obtained from the following public webs:

- General composition (Clean right):
  http://uk.cleanright.eu/index.php?option=com_product&task=section&Itemid=195

- Henkel:
  http://www.henkelconsumerinfo.com/products/Ctrl?_h=henkel.products.ChangeLanguage&lang=en

- Procter & Gamble
  http://info-pg.com/

- Reckitt Benckiser:
  http://www.rbeuroinfo.com/

- Unilever:
  http://www.unilever.com/PIOTI/EN/p1.asp

- L'Abre Vert:
  http://www.arbrevert.fr/index.php/produits-ecologiques-pour-la-maison-l-arbre-vert/lessives-ecologique-l-arbre-vert.html

- Ecover:
  http://us.ecover.com/product-category/laundry/

- Carrefour:
  http://www.info-detergent.com/

- AC Marca:
  http://www.grupoacmarca.com/ingredientes/

- Delta Pronatura – Dr. Beckmann:
  http://www.deltapronatura.de/dp_neu/media/files/Inhaltsstoffe_Ingredients_04_02_2014.pdf

- KH Lloreda:
  http://www.khlloreda.com/es/productos/index.shtml?p=2

4.3.Liquid Heavy-duty Detergents

4.3.1.General Composition

In order to get a global vision about the general composition of liquid heavy-duty detergents in Europe, some tables from the web site *Clean Right* are shown in this report. There, the ingredients of this type of products are classified according their family and their type. Besides, the common range in which they appear is also described.
The general composition of each type of liquid heavy-duty detergent in Europe is very similar. In the following tables a typical composition of each subgroup is shown (Table 4: Regular liquid; Table 5: Compact liquid; Table 6: Liquid Unit Dose).

Table 4. Typical composition of a regular liquid heavy-duty detergent

Regular Liquid (typical dosage 120 ml)		
Family	**Type**	**Range (%)**
Builders	Soap	1 – 5
Enzymes	Amylase Cellulase Lipase Mannanase Other enzymes Protease	0.2 – 0.5
Fragrances		0 – 0.5
Optical Brighteners	Optical Brighteners	0 – 0.2
Sequestrants	Phosphonates	< 0.2 (some 0.2 – 0.5)
Solvent	Ethanol Glycol Others	1 – 5
Surfactants	Anionic Surfactants Cationic Surfactants Nonionic Surfactants	5 – 15 0 – 5 1 – 5 (some 5 – 15)
Water		Balance to 100

Table 5. Typical composition of a compact liquid heavy-duty detergent

Compact Liquid (typical dosage 75 ml or lower)		
Family	**Type**	**Range (%)**
Builders	Soap or Citrate	5 – 15
Enzymes	Amylase Cellulase Lipase Mannanase Other enzymes Protease	0.2 – 0.5
Fragrances		0 – 1.5
Optical Brighteners	Optical Brighteners	0 – 0.1
Polymers		1 – 5
Sequestrants	Phosphonates	0.2 – 0.5 (some 0.5 – 1)
Solvent	Monoethanolamine (MEA) Ethanol Glycol Others	1 – 5
Surfactants	Anionic Surfactants Cationic Surfactants Nonionic Surfactants	5 – 15 (some 15 – 30) 0 – 5 5 – 15 (some 15 – 30)

Table 6. Typical composition of a liquid unit dose heavy-duty detergent

Liquid Unit Dose		
Family	**Type**	**Range (%)**
Builders	Soap or Citrate	5 – 15
Enzymes	Amylase Cellulase Lipase Mannanase Other enzymes Protease	0.2 – 0.5
Fragrances		0.2 – 1.5
Optical Brighteners	Optical Brighteners	0 – 0.5
Sequestrants	Phosphonates	0.2 – 0.5 (some 0.5 – 1)
Solvent	Monoethanolamine (MEA) Ethanol Glycol Others	5 – 10
Surfactants	Anionic Surfactants Cationic Surfactants Nonionic Surfactants	5 – 15 (some 15 – 30) 0 – 5 5 – 15

4.3.2. European Market

In this section some representative liquid heavy-duty detergents of the European market have been analyzed regarding its composition. These products are the following:

- From Henkel
  - German Market:
    - Persil Universal Gel
    - Persil Universal Mega-Caps
    - Persil Duo-Caps Universal
  - Spanish Market:
    - Dixan Gel Eco
    - Wipp Express Gel Activo
    - Wipp Express Duo-Caps
- From Procter & Gamble
  - Spanish Market:
    - Ariel Líquido Básico
    - Ariel Excel Gel Actilift
    - Ariel Excel Tabs Actilift

- From Reckitt Benckiser
  - Spanish Market:
    - Colon Intelligence Plus Gel
    - Colon Power Gel
  - Italian Market:
    - Sole Perle di Pulito (Gel Caps)
    - Sole Bianco Solare Liquido
- From Unilever
  - French Market:
    - Skip Liquide Active Clean
    - Skip Petit & Puissant Active Clean
    - Skip Capsules Active Clean
- From eco brands
  - L'Abre Vert (Ecolabel):
    - Lessive Liquide au Savon Vegetal Ecologique
  - Ecover:
    - Laundry Liquid
- From generic brands
  - Carrefour:
    - Ultra Concentrated Laundry Detergent Liquid with Marseille Soap

4.3.3.Results

An evaluation of the main ingredients that can increase or decrease the release of microfibers has been carried out. The ingredients analyzed are: surfactants (anionic and non-ionic), polymers and alkaline agents. The results are shown in the following graphs, where the different types of each ingredient used in the brands selected can be appreciated, as well as the frequency in which they are used. The X axis corresponds to various types of the ingredient analyzed and the Y axis is the amount of products of each company which contains the corresponding ingredient. A complete description of the composition of each product can be found in ANNEX 1.
In the case of anionic surfactants (Figure 1), sodium laureth sulfate appears in most of companies. However, the tendency of each company is to use their own surfactants. The brand *Ariel* from Procter & Gamble is the one with more anionic surfactants in its composition, being *Ariel Básico* the most complete with seven different anionic surfactants.

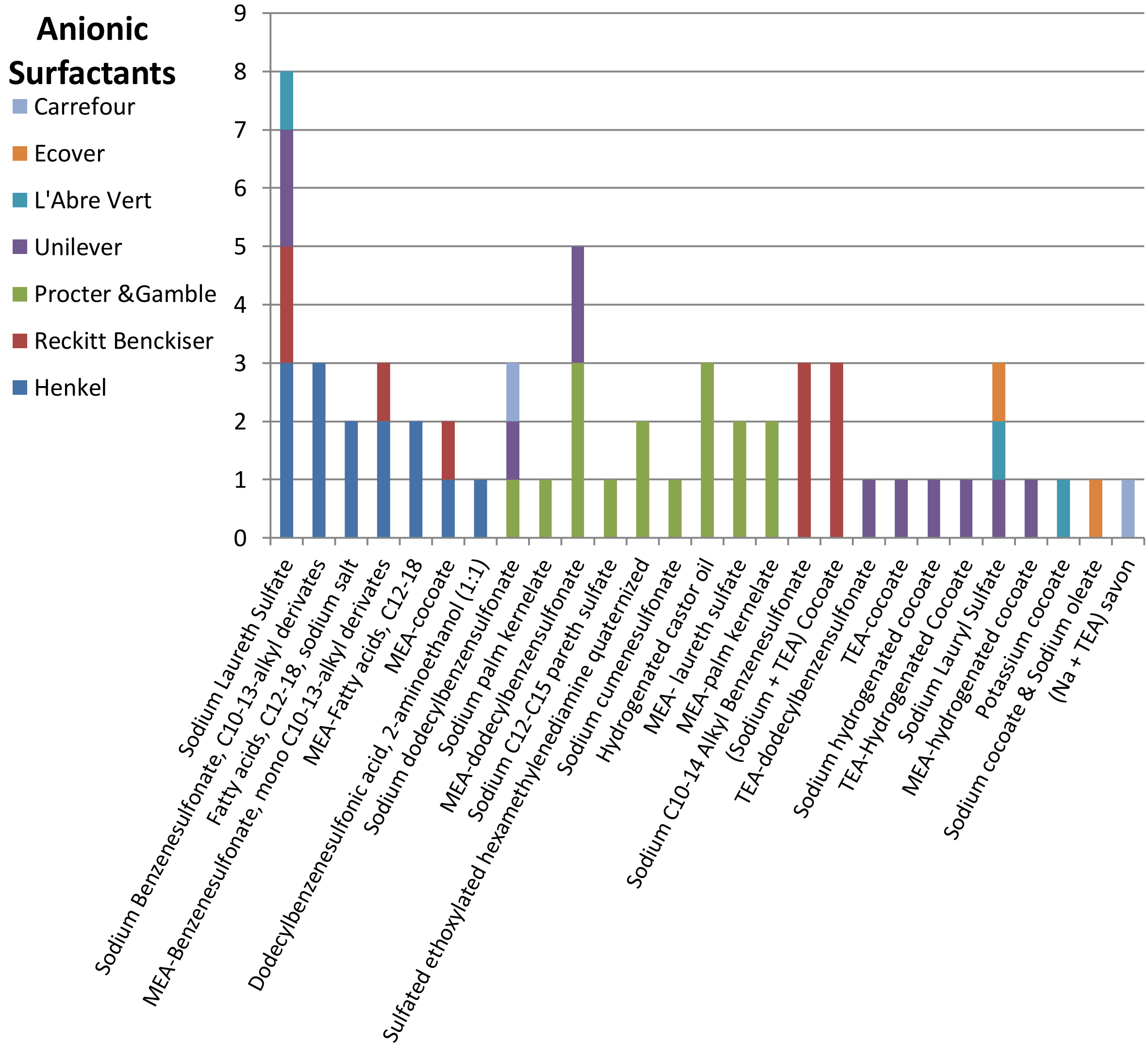

Figure 1. Common anionic surfactants used in some representative products from the European market

Regarding non-ionic surfactants, each company also trends to use their own. Most of their products contain only one non ionic surfactant, except Ariel and Eco brands which use two different non ionic surfactants in each formulation (Figure 2).

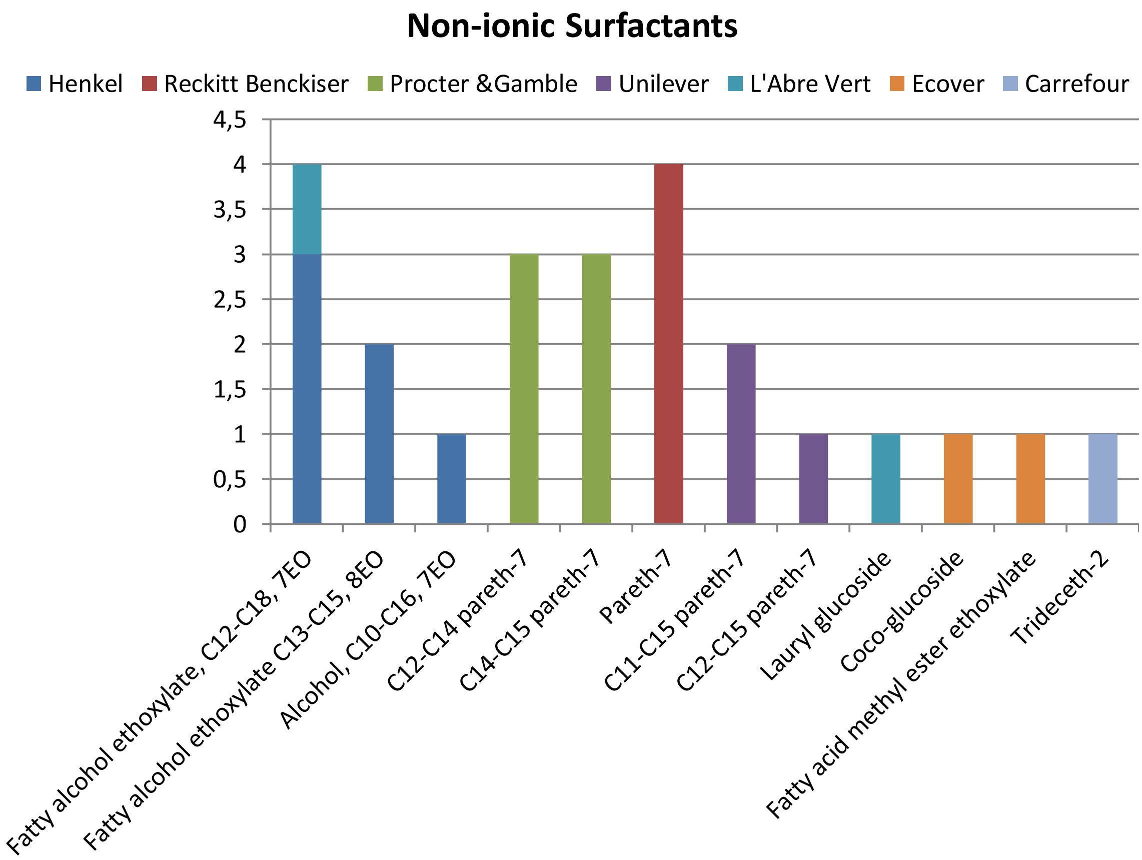

Figure 2. Common non-ionic surfactants used in some representative products from the European market

In the case of polymers (Figure 3), styrene/acrylates copolymer is the more used, followed by polypropylene terephtalate. *Ariel Básico* is again the brand that includes more polymers (four different ones) in an unique formulation.

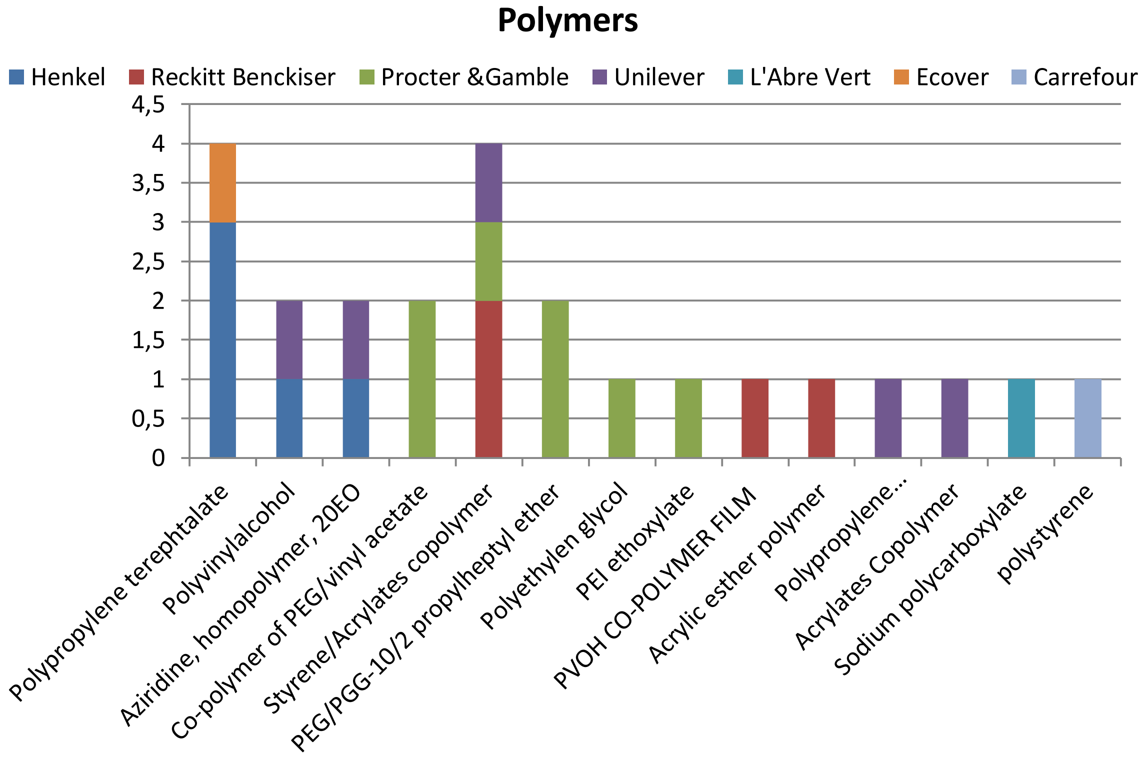

Figure 3. Common polymers used in some representative products from the European market

Finally, only three alkaline agents are used in all selected products of this report (Figure 4). They are: sodium hydroxide, ethanolamine and triethanolamine. These ingredients can appear as unique alkaline agent or combined with another of these ones. The more used is sodium hydroxide, while the rest are typical in concentrated formulations.

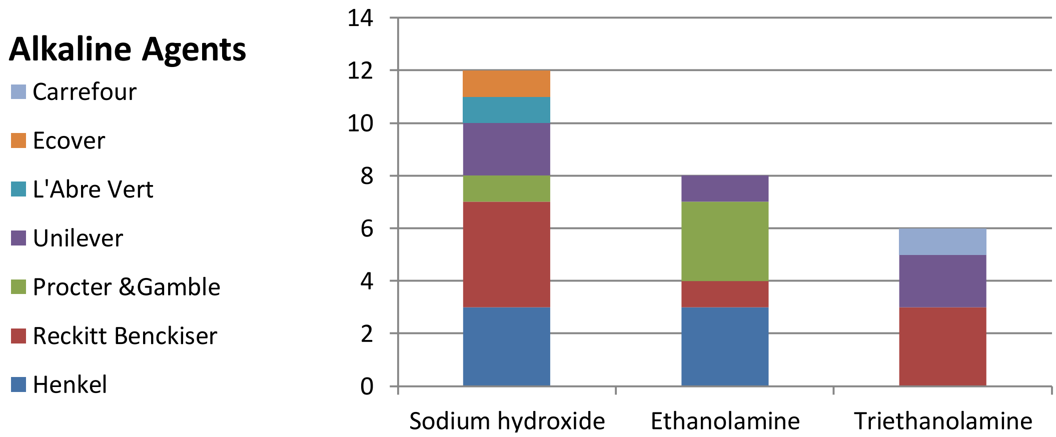

Figure 4. Common alkalyne agents used in some representative products from the European market

4.4.Powdered Heavy-duty Detergents

4.4.1General Composition

In order to get a global vision about the general composition of powdered heavy-duty detergents in Europe, some tables from the web site *Clean Right* are shown in this report. There, the ingredients of this type of products are classified according their family and their type. Besides, the common range in which they appear is also described.
The general composition of each type of powdered heavy-duty detergent in Europe is very similar. In the following tables a typical composition of each subgroup is shown (Table 7: Powder Regular; Table 8: Powder Compact; Table 9: Powder Tablet).

Table 7. Typical composition of regular powdered heavy-duty detergents

Powder Regular		
Family	**Type**	**Range (%)**
Alkalinity sources	Carbonate, Silicate	15 - 30
Bleach precursors	Bleach activators	1 – 5
Builders	Polycarboxylates Soap Zeolite	1 – 5 1 – 5 1 – 15
Enzymes	Amylase Cellulase Lipase Mannanase Other enzymes Protease	0.2 – 0.5
Fragances		0.2 – 0.5
Optical brighteners	Optical brighteners	0.2 – 0.5
Oxidising agents	Oxygen-based bleaching agents	5 – 15
Processing agent	Sodium sulphate	Balance to 100
Sequestrants	Phosphonates	0.2 – 0.5
Surfactants	Anionic surfactants Non ionic surfactants	5 – 15 1 – 15

Table 8. Typical composition of compact powdered heavy-duty detergents

Powder Compact		
Family	**Type**	**Range (%)**
Alkalinity sources	Carbonate, Silicate	15 - 30
Builders	Polycarboxylates Soap Zeolite	5 – 15 (1 – 5) 15 – 30
Enzymes	Amylase Cellulase Lipase Mannanase Other enzymes Protease	0.2 – 0.5
Fragances		0 - 0.2 – 0.5
Optical brighteners	Optical brighteners	0.1 – 0.5
Oxidising agents	Bleach activators Oxygen-based bleaching agents	1 – 5 5 – 15 (some 15 – 30)
Sequestrants	Phosphonates	0.2 – 0.5
Surfactants	Anionic surfactants Non ionic surfactants	5 – 15 5 – 15 (some 1 – 5)

Table 9. Typical composition of powder tablets

Powder Tablet		
Family	**Type**	**Range (%)**
Alkalinity sources	Carbonate, Silicate	15 - 30
Bleach precursors	Bleach activators	1 – 5
Builders	Polycarboxylates Soap Zeolite	5 – 15 1 – 5 15 – 30
Enzymes	Amylase Cellulase Lipase Mannanase Other enzymes Protease	0.2 – 0.5
Fragances		0.2 – 0.5
Optical brighteners	Optical brighteners	0.2 – 0.5
Oxidising agents	Oxygen-based bleaching agents	5 – 15 (some 15 – 30)
Sequestrants	Phosphonates	1 – 5 (0.2 – 0.5)
Surfactants	Anionic surfactants Non ionic surfactants	5 – 15 5 – 15 (some 1 – 5)

4.4.2.European Market

The representative powdered heavy-duty detergents of the European market selected to be analyzed regarding their composition are the following:

- From Henkel
  - German Market:
    - Persil Tabs
  - Belgian Market:
    - Persil Universal Powder
  - Spanish Market:
    - Dixan Business Line
    - Wipp Express Polvo Business Line
- From Procter & Gamble
  - Spanish Market:
    - Ariel Básico
    - Ariel Actilift Excel Tabs
- From Reckitt Benckiser
  - Spanish Market:
    - Colon Intelligence Plus Polvo
    - Colon Easy Clean
  - Italian Market:
    - Sole Pulito e Igiene Polvere
    - Sole Bianco Solare Polvere
- From Unilever
  - French Market:
    - Skip Poudre Active Clean
    - Skip Tablettes Active Clean
- From eco brands
  - L'Abre Vert (Ecolabel):
    - Lessive Poudre au Savon Vegetal Ecologique
    - Doses Lessives Hydrosolubles (poudre)
  - Ecover:
    - Laundry Bio Washing Powder
    -

- From generic brands
  - Carrefour:
    - Oxy White Fabric Brightening Tablets
    - Detergente en Tabletas

4.4.3.Results

The same evaluation as the case of liquid heavy-duty detergents has been carried out for powdered heavy-duty detergents. The ingredients analyzed are: surfactants (anionic and non-ionic), polymers and alkaline agents. The results are shown in the following graphs, where the X axis corresponds to the various types of the ingredient analyzed and the Y axis is the frequency they are used in the products selected. A complete description of the composition of each detergent can be found in ANNEX 2.

In the case of anionic surfactants, sodium dodecylbenzenesulfonate is the most common which is used by five from the seven companies analyzed. Sodium C12-C18 sulfate is used by 3 companies, while most of the rest appears in the composition of products of just one company (Figure 5). The brands with more anionic surfractants (four) are: *Ariel Actilift* from Procter & Gamble and *L'Abre Vert Lessive Pouder au Savon Vegetal Ecologique*. On the other hand, the hydrosoluble tab version of L'Abre Vert and *Carrefour Oxy-White Fabric Brightening* do not contain any anionic surfactant.

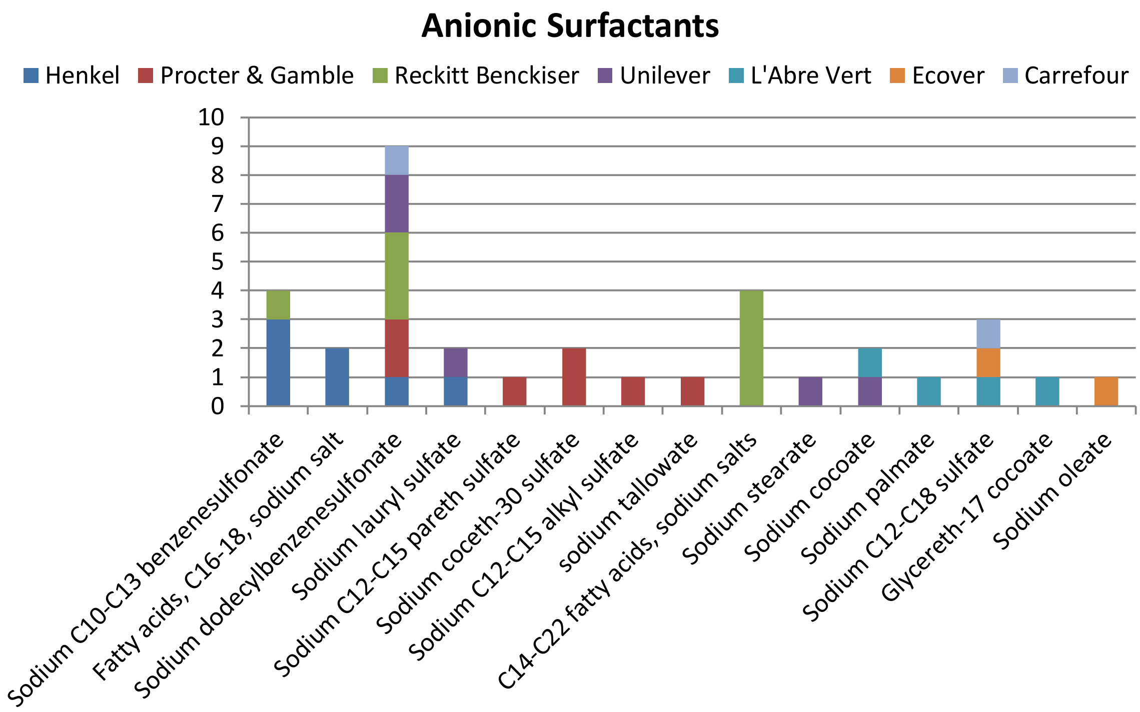

Figure 5. Types of anionic surfactants used in some powdered heavy-duty detergents from the European market

Regarding non ionic surfactants, in general each company uses their own, being lauryl glucoside and C12-C15 pareth-7 the only ones which appears in two different companies (Figure 6). In this case the brand *Persil Tabs* from Henkel is the one with more different non ionic surfactants (three).

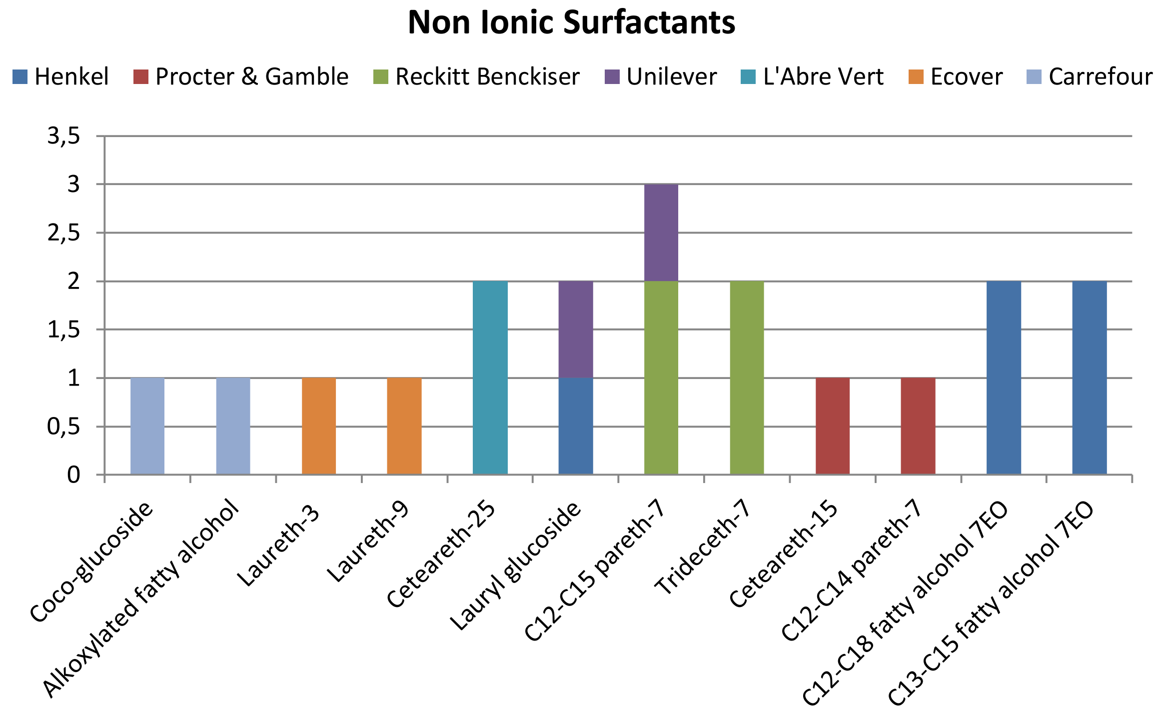

Figure 6. Non ionic surfactants used in some powdered heavy-duty detergents from the European market

The general trend in polymers is that each company uses different types. Only sodium acrylic acid/MA copolymer and sodium polyacrylate appear in four and three companies of the seven analyzed, respectively (Figure 7). The brand Skyp Poudre Active Clean from Unilever is in this case the one which contain more different polymers (six), followed by *Ariel Actilift* from Procter & Gamble and *Persil Tabs* from Henkel with five different polymers.

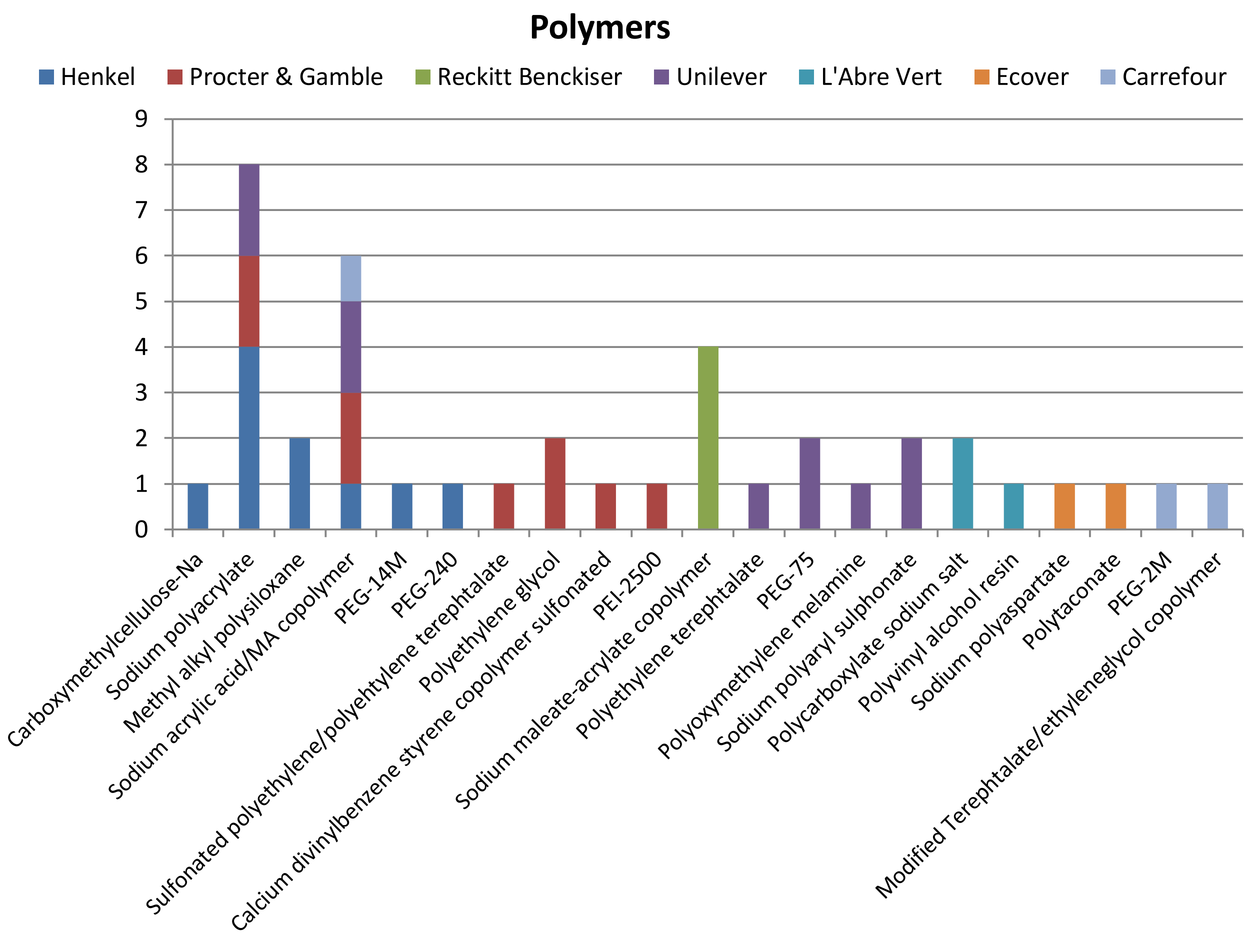

Figure 7. Polymers used in some powdered heavy-duty detergents from the European market

In the case of alkaline agents, sodium hydroxide is the only one used in these powder detergents (Figure 8. Alkaline agent used in some powdered heavy-duty detergents from the European market).

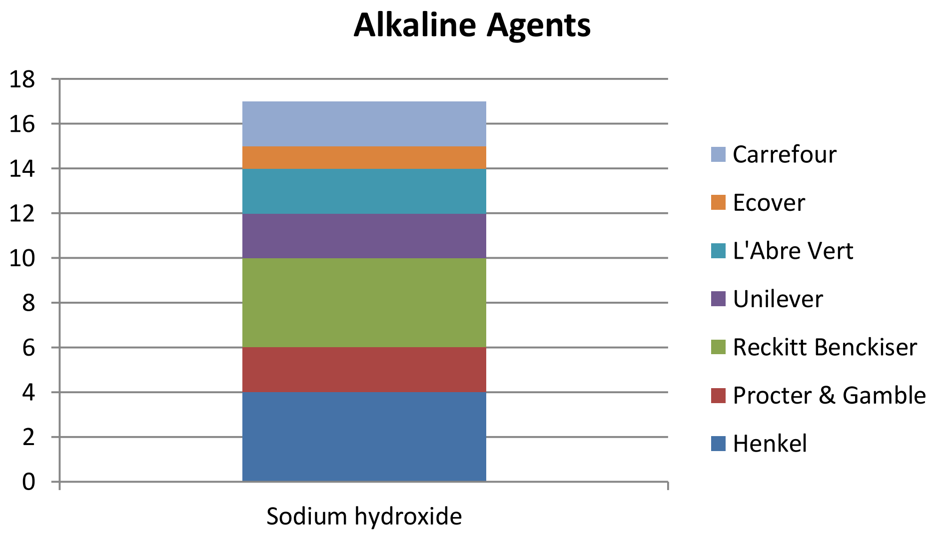

Figure 8. Alkaline agent used in some powdered heavy-duty detergents from the European market

4.5.Special Detergents

4.5.1.General Composition

In order to get a global vision about the general composition of special detergents in Europe, some tables from the web site *Clean Right* are shown in this report. There, the ingredients of this type of products are classified according their family and their type. Besides, the common range in which they appear is also described.

The general composition of each type of special detergent in Europe is very similar. In the following tables a typical composition of each subgroup is shown (Table 10: Liquid; Table 11: Powder).

Table 10. Typical composition of liquid special detergents

Wool/Silk Laundry Detergent - Liquid		
Family	**Type**	**Range (%)**
Alkalinity sources		Low
Bleach precursors	Bleach activators	
Builders	Soap	1 – 5
Fragances		0.2 – 0.5
Oxidising agents	Oxygen-based bleaching agents	
Sequestrants	Phosphonates	0.2 – 0.5
Surfactants	Anionic surfactants Non ionic surfactants	5 – 15 1 – 15

Table 11. Typical composition of powdered special detergents

Wool/Silk Laundry Detergent - Powder		
Family	**Type**	**Range (%)**
Alkalinity sources	Carbonate	Low
Builders	Zeolites Soap	15 – 30 1 – 5
Fragances		0.2 – 0.5
Sequestrants	Phosphonates	0.2 – 0.5
Surfactants	Anionic surfactants Cationic surfactants Non ionic surfactants	5 – 15 0 – 5 1 – 15

4.5.2.European Market

The representative special detergents of the European market selected to be analyzed regarding their composition are the following:

- From Henkel
  - German Market:
    - Persil Color Gel
    - Persil Color Mega-Caps
    - Persil Color Duo-Caps
  - Spanish Market:
    - Micolor Gel
    - Perlán Máquina Pétalos de Tiaré
  - Finnish Market:
    - Perlana Color & Delicates
    - Perlana Wool & Delicates
- From AC Marca
  - Spanish Market:
    - Norit Cuidado Delicado Máquina
    - Norit Cuidado Color
- From Reckitt Benckiser
  - Italian Market:
    - Sole Lana e Delicati Detersivo Liquido
    - Sole Perle Proteggicolore (gel caps)
    - Lip Woolite Mix Noir
    - Lip Woolite Mix Color
    - Lip Woolite Lana e Delicati (gel caps)
- From eco brands
  - L'Abre Vert (Ecolabel):
    - Lessive Lana et Textiles Delicats Ecologique
  - Ecover:
    - Delicate Laundry Liquid
    - Color Washing Powder
- From generic brand
  - Carrefour:
    - Lana e Capi Delicati
    - Detergente Lana

4.5.3.Results

Again, an evaluation of the surfactants, polymers and alkaline agents present in some representative special detergents in the European market has been carried out. The results are shown in the following graphs, where the X axis corresponds to the various types of the ingredient analyzed and the Y axis is the frequency they are used in the products selected. A complete description of the composition of each detergent can be found in ANNEX 3.

The most common anionic surfactant in the selected companies is sodium laureth sulfate (Figure 9), which appears in four of the six ones, followed by sodium dodecylbenzenesulfonate. The rest of surfactants are used only by one company, or two as much. Most of products studied contain two or three anionic surfactants, except *Carrefour Detergente Lana* with four.

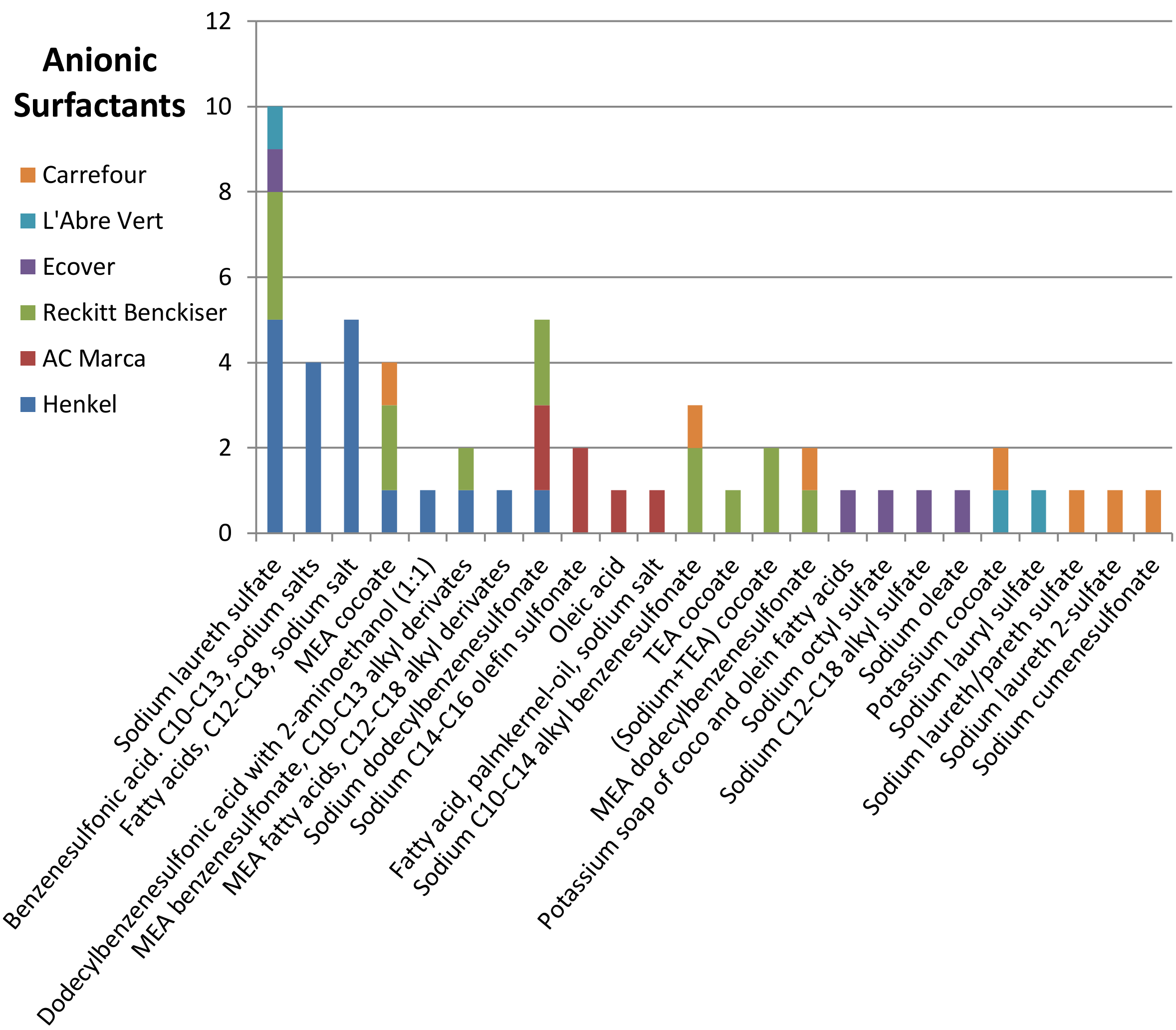

Figure 9. Common anionic surfactants used in some representative special detergents from the European market

In the case of non ionic surfactants, each company uses their own class (Figure 10). There is not any surfactant appearing in more than one company. All products studied contain one or two different non ionic surfactants.

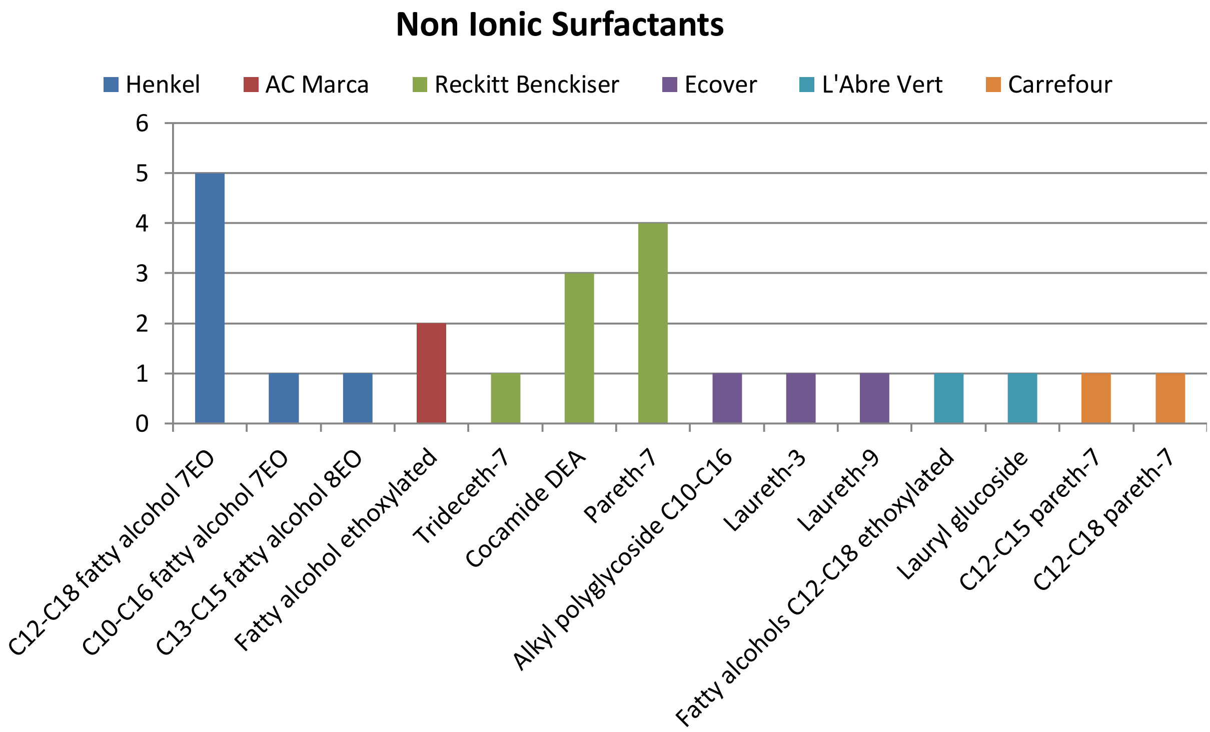

Figure 10. Common non ionic surfactants used is some representative special detergents from the European market

The polymer most commonly used is styrene/acrylate copolymer, which appears in three of the six companies selected (Figure 11). The vinylpyrrolidone/vinylimidazole copolymer and the polyvinylalcohol polymer are used in two companies (Henkel and Reckitt Benckiser). The rest of them are typical of only one company. The products containing more different polymers (four) are from the brand *Perlana* in the Finnish market from Henkel. Meanwhile, other products do not contain polymers: *Micolor* from Henkel, *Norit* from AC Marca, *Ecover Delicate Laundry Liquid*, *L'Abre Vert* and *Carrefour Detergente Lana*.

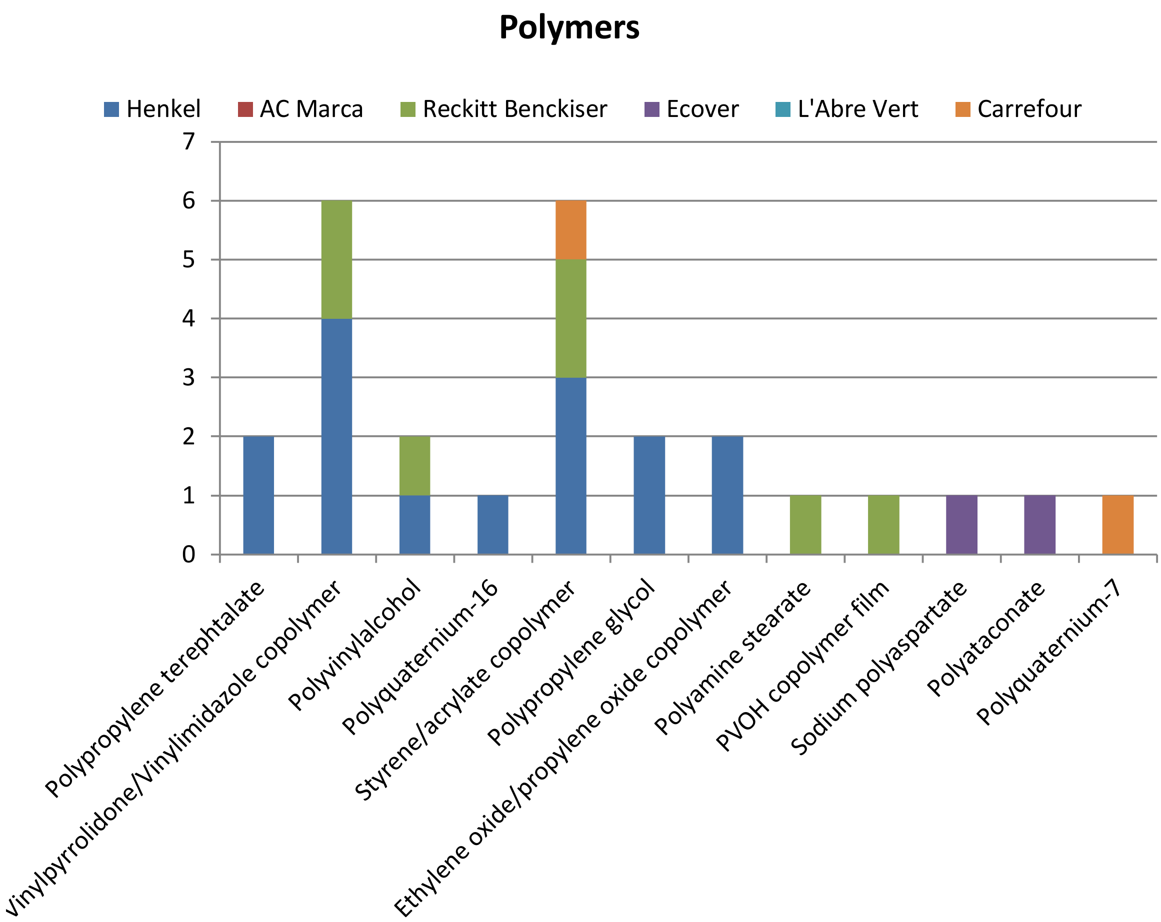

Figure 11. Common polymers used in some representative special detergents from the European market

Regarding alkaline agents, all companies use sodium hydroxide in their products. Ethanolamine appears in three of the six ones selected and potassium hydroxide is commonly used in eco brands (Figure 12).

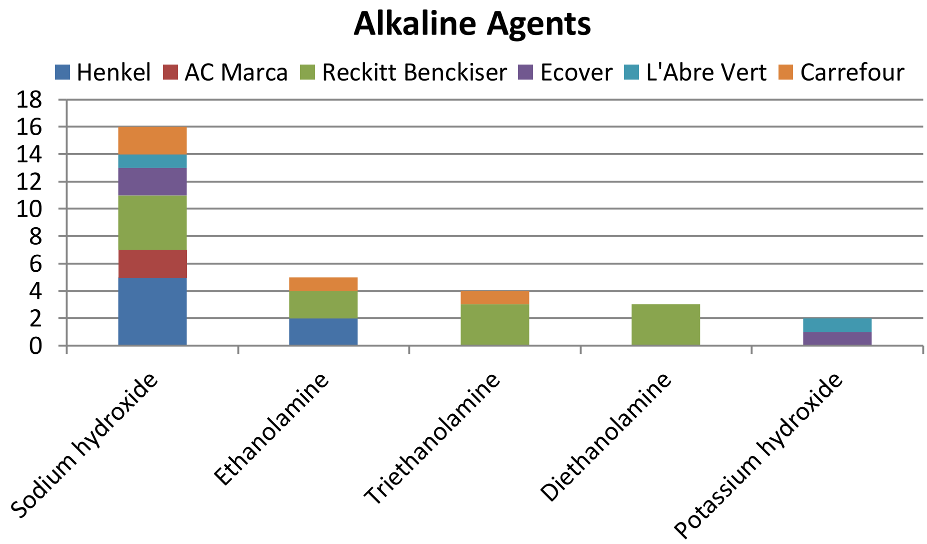

Figure 12. Common alkaline agents used in some representative special agents from the European market

4.6.Laundry Aids

4.6.1.General Composition

In order to get a global vision about the general composition of the different types of laundry aids in Europe, some tables from the web site *Clean Right* are shown in this report. There, the ingredients of this type of products are classified according their family and their type. Besides, the common range in which they appear is also described.

The laundry aids chosen in this study are: fabric conditioners or softeners regular (Table 12), fabric conditioners concentrated (Table 13) powdered and liquid bleach additives (Table 14) and, finally, stain removers (Table 15).

Table 12. Typical composition of a fabric conditioner regular (softener)

Fabric Conditioner Regular		
Family	**Type**	**Range (%)**
Fragrances		0.2 – 1
Solvents	IPA	1 – 5
Surfactants	Anionic surfactants Cationic surfactants Nonionic surfactants	1 – 5
Water		Balance to 100

Table 13. Typical composition of a fabric conditioner concentrated (softener)

Fabric Conditioner Concentrated		
Family	**Type**	**Range (%)**
Fragrances		0.2 – 1.5
Solvents	IPA	1 – 5
Surfactants	Anionic surfactants Cationic surfactants Nonionic surfactants	5 – 15
Water		Balance to 100

Table 14. Typical composition of powdered bleach composition

Bleach Additives - Powder		
Family	**Type**	**Range (%)**
Alkalinity Source	Sodium carbonate	0 - 50
Bleach Precursors	TAED	0 - 15
Bulking Agents		Balance to 100
Enzymes		0 – 1
Optical Brighteners		0 – 1
Oxidising Agents*	Oxygen-based bleaching agents	20 – 60
Surfactants	Anionic surfactants Nonionic surfactants	0 – 5 0 – 5

* Commonly sodium percarbonate

Table 15. Typical composition of stain removers

Stain Removers		
Family	**Type**	**Range (%)**
Builders	Citric acid/Citrates	0 – 5
Colourants		0 – 0.1
Enzymes		< 0.5
Fragrances		< 0.5
Oxidising Agents*	Oxygen-based bleaching agents	0 – 5
Preservatives		< 0.5
Sequestrants	Phosphonates	< 0.5
Solvents		0 – 10
Surfactants	Anionic surfactants Nonionic surfactants Soaps	0 – 5 0 – 5 0 – 5
Viscosity Controlling Agents		0 – 1
Water		Balance to 100

* Hydrogen peroxide

4.6.2.European Market

The representative laundry aids of the European market selected to be analyzed regarding their composition are the following:

Softeners

- From Henkel
  - German Market:
    - Vernel Hautsensitiv
    - Vernel Wild-rose
    - Vernel Crystals Frische Zauber

- From Unilever
  - French Market:
    - Cajolin Liquide Tentation Rose Jasmin
    - Cajolin Concentré Brin de Folie Lavande & Amande Deuce
    - Cajolin Capsules Fleur de la Passion & Bergamote

- From Reckitt Benckiser
  - Spanish Market:
    - Flor Frescor Oceánico Regular
    - Flor Azul 24h con Micro-cápsulas Concentrado

- From Eco Brands
  - Ecover:
    - Ecover Softener Amongst the Flowers

  - L'Abre Vert (Ecolabel):
    - L'Abre Vert Assouplissant Souffle de Purete Ecologique

- From Generics Brands:
  - Carrefour:
    - Hypoallergenic Fabric Conditioner

Bleach

- From Henkel
  - Spanish Market:
    - Neutrex Futura

- From Procter & Gamble
  - Italian Market:
    - Candeggina Classica Ace con Nuovo Salvafibra
    - Ace Candeggina Densa-Igienizzante

- From Generic Brands
  - Carrefour:
    - Carrefour Classic Bleach

Oxy Products

- From Henkel
  - German Market:
    - Sil Oxi Perfect 2 (liquid)
    - Sil Oxi Flecken-spray (liquid)

- From Procter & Gamble
  - Spanish Market:
    - Ariel Quitamanchas Blanqueador Polvo (powder)

- From Reckitt Benckiser
  - Italian Market:
    - Vanish Oxy Action Gel
    - Vanish Oxy Action Crystal White Polvere (powder)

- From AC Marca
  - Spanish Market:
    - Iberia Blanco Nuclear (liquid)

- From Delta Pronatura
  - Dr. Beckmann:
    - Oxy Magic Quitamanchas Multiusos (foam)

- From KH Lloreda
  - Spanish Market:
    - KH-7 Sin Manchas Oxy Effect (liquid)
- From Eco Brands
  - Ecover:
    - Ecover Laundry Bleach (powder)

- From Generic Brands
  - Carrefour:
    - Oxy Power Stain Remover (liquid)

Stain Removers

- From Procter & Gamble
  - Spanish Market:
    - Ariel Quitamanchas Ropa Color y Blanca (liquid)

- From Reckitt Benckiser
  - Italian Market:
    - Vanish Power Shots (liquid)

- AC Marca
  - Spanish Market:
    - Iberia Quitamanchas Diarias Roll-On (liquid)

- Delta Pronatura
  - Dr. Beckmann:
    - Prewash Quitamanchas con Cepillo Aplicador (liquid)
    - Stain Removal Roll-On (liquid)

- KH Lloreda
  - Spanish Market:
    - KH-7 Sin Manchas (liquid)

- From Eco Brands
  - Ecover:
    - Ecover Stain Remover (liquid)

  - L'Abre Vert (Ecolabel):
    - L'Abre Vert Stain Remover (gel)
    - L'Abre Vert Stain Remover (spray)

- From Generic Brands
  - Carrefour:
    - Carrefour Detachant Avant Lavage (spray)

4.6.3.Results

In this case, the evaluation of the ingredients has been carried out according to the type of laundry aid in the European market. Regarding softeners, the ingredients analyzed are cationic and non ionic surfactants and polymers; in bleach, anionic, active and alkaline agents have been studied. In both oxy products and stain removers anionic and non ionic surfactants, polymers and alkaline agents have been evaluated, besides the actives in oxy products.
The results are shown in the following graphs, where the X axis corresponds to the various types of the ingredient analyzed and the Y axis is the frequency they are used in the products selected. A complete description of the composition of each one can be found in ANNEX 4.

Softeners

In softeners is usual the use of cationic surfactants as active substance. In the case of the European market the most common cationic surfactants are derivates from fatty alkylammonium methosulfates. In Figure 13 the different types of cationic surfactants used in the softeners studied are shown.

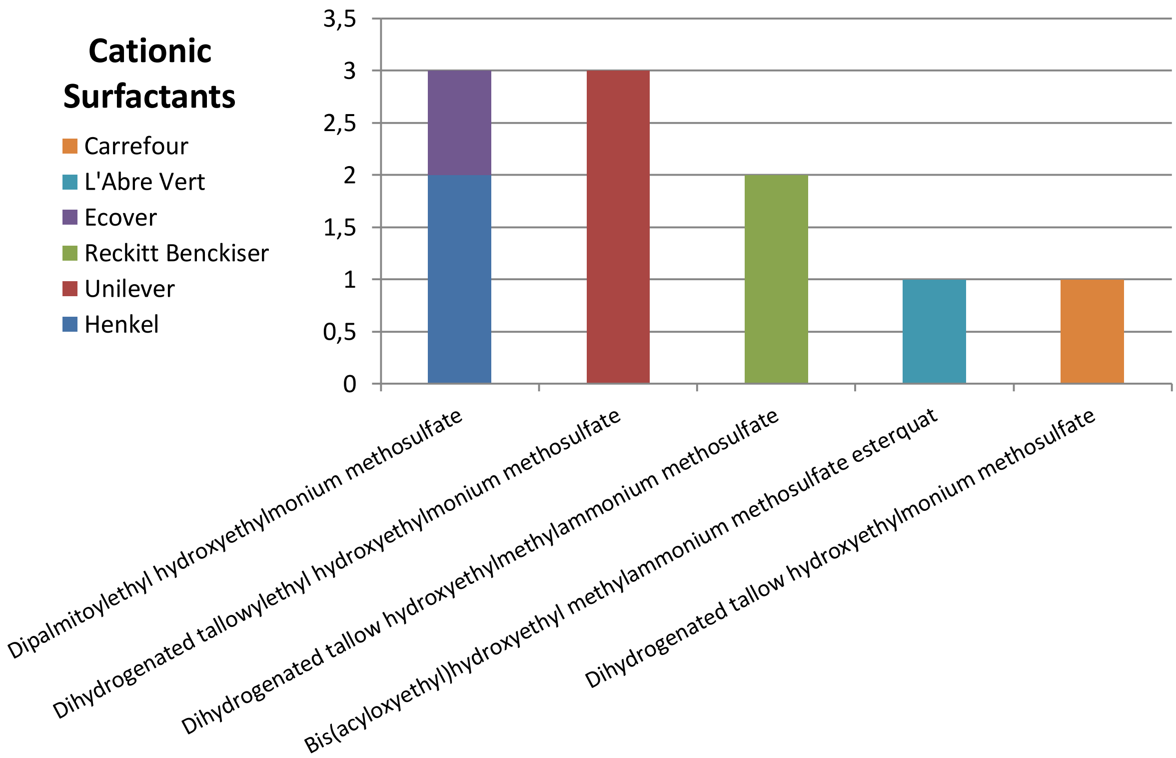

Figure 13. Common cationic surfactants used in some representative softeners from the European market

Only two of the six companies analyzed use non ionic surfactants in their softeners: Unilever and Henkel. These surfactants are varied, proceeding all from vegetable sources. In Figure 14 the non ionic surfactants used in the products studied are shown.

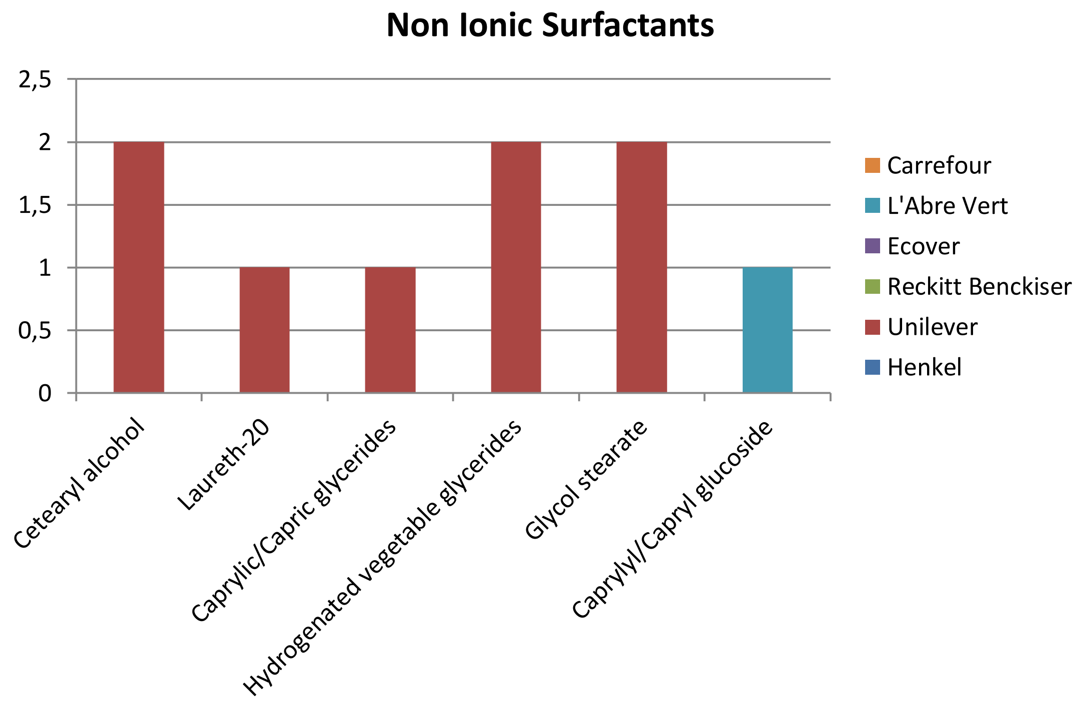

Figure 14. Common non ionic surfactants used in some representative softeners from the European market

Unilever and Reckitt Benckiser are the only two companies that use polymers in their formulations. They are shown in Figure 15.

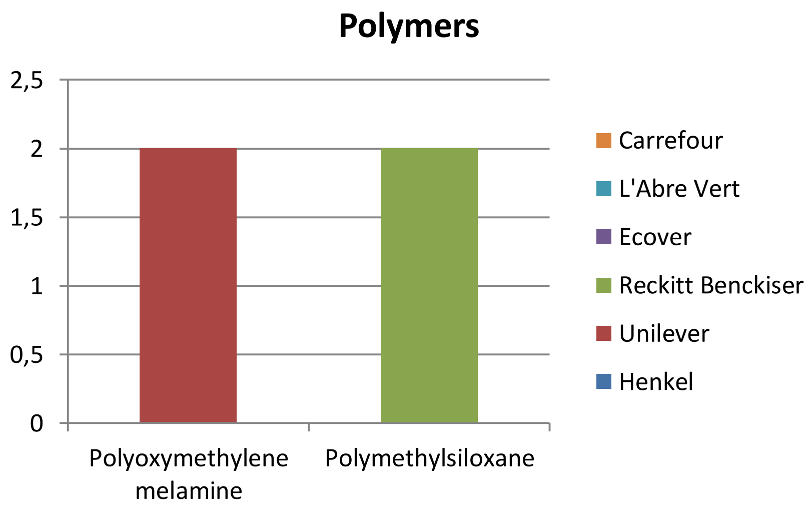

Figure 15. Common polymers used in some representative softeners from the European market

There is a new type of softeners which are offered in the European market which are presented as crystals, in solid state. An example of these is *Vernel Crystals Frische Zauber* from Henkel. It does not contain cationic surfactants, instead it uses as actives sucrose and bentonite.

Bleach

Bleach is a liquid product which active substance is sodium hypochlorite. Besides, it can contain other ingredients such as surfactants and alkaline agents, being sodium hydroxide the alkali present in all the bleaches from this study. Carrefour does not contain any anionic surfactant in its formulation. Procter & Gamble uses anionic surfactants from vegetable sources, while Henkel does not specify it. In Figure 16 the anionic surfactants used in these companies are shown.

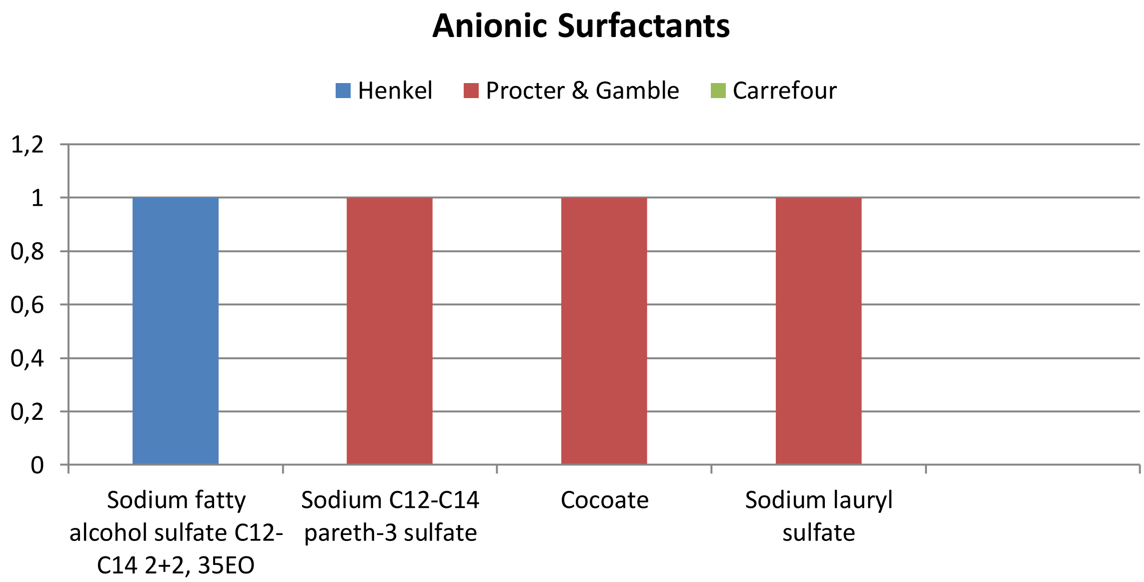

Figure 16. Common anionic surfactants used in some bleaches from the European market

Oxy Products

Oxy products are manufactured both in powder and liquid form. The actives used in oxy products from the European market are, basically, the four ones shown in Figure 17. Hydrogen peroxide is the most common in liquid formulations, while sodium percarbonate is typical in powdered formulations. TAED appears in the powdered products *Ariel Quitamanchas Blanqueador* from Procter & Gamble and *Vanish Oxy Action Crystal White* from Reckitt Benckiser. On the other hand, sodium hydrosulfite only appears in the liquid formulation *Iberia Blanco Nuclear* from AC Marca.

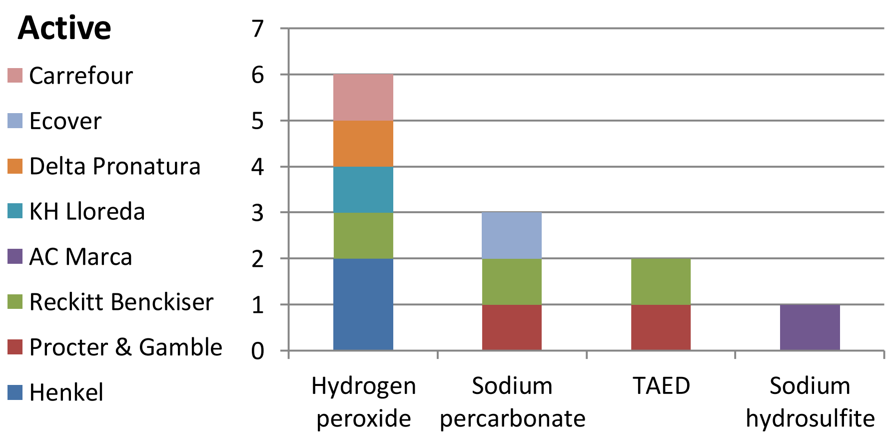

Figure 17. Common actives used in some representative oxy products from the European market

Oxy products usually include other ingredients such as surfactants and polymers. Carrefour is the only brand that does not include anionic surfactants in its oxy product formulation. The rest does, using a different one for each product, except Procter & Gamble which uses four anionic surfactants in the powdered formula of *Ariel Quitamanchas Blanqueador* (Figure 18).

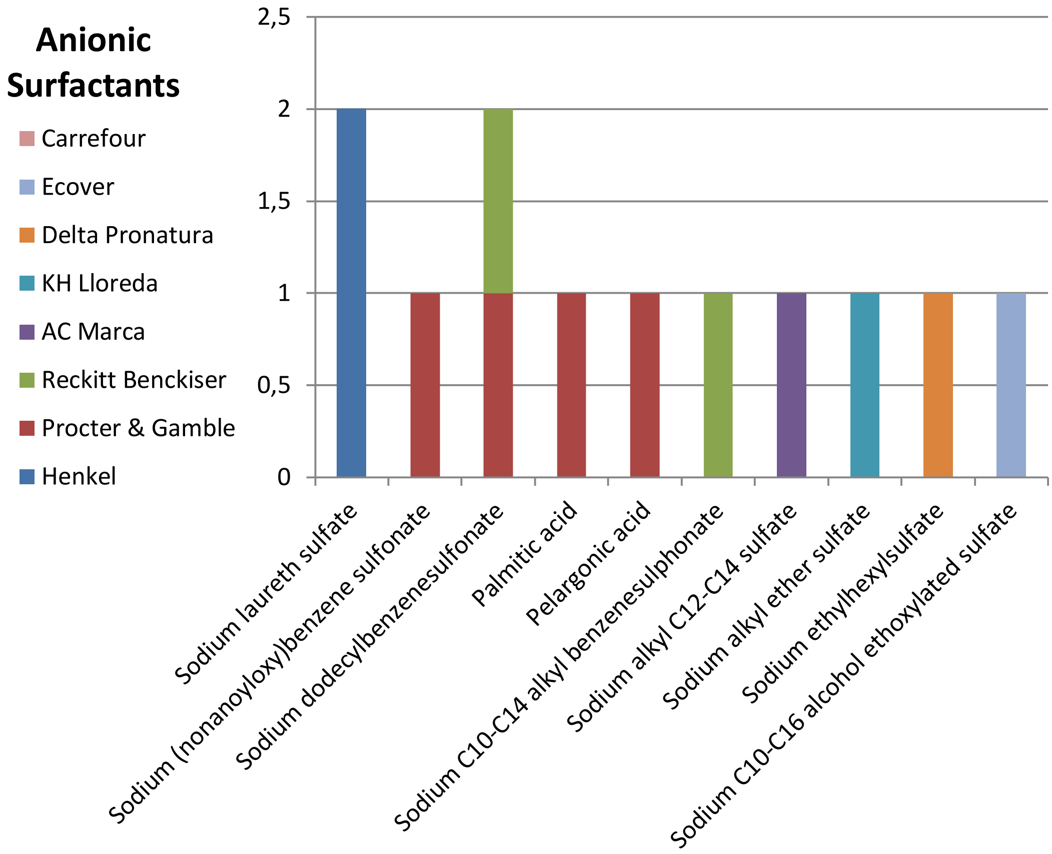

Figure 18. Common anionic surfactants used in some representative oxy products from the European market

In powdered formulations the anionic surfactants used are sodium (nonaoyloxy)benzene sulfonate, sodium dodecylbenzenesulfonate, palmitic acid and pelargonic acid; the rest appear in liquid products.

The same occurs with non ionic surfactants, a different type is used in each product of each company, except for Reckitt Benckiser which includes seven non ionic surfactants in the formulation of *Vanish Oxy Action Gel* (Figure 19). The oxy products from Carrefour and AC Marca do not contain non ionic surfactants.

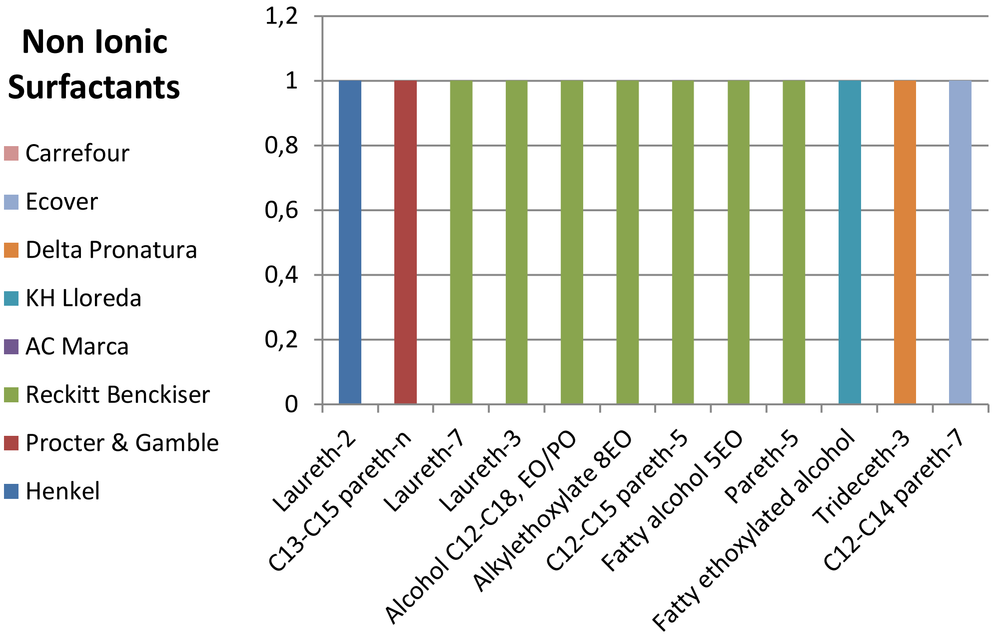

Figure 19. Common non ionic surfactants used in some oxy products from the European market

The non ionic surfactants used in powdered formulations are C13-C15 pareth-n and pareth-5, the rest ones appears in liquid formulations.

Polymers are only used by two companies: Procter & Gamble and KH Lloreda. The powdered product *Ariel Quitamanchas Blanqueador* (powder) from Procter & Gamble contains three polymers, while KH-7 Sin Manchas Oxy Effect (liquid) from KH Lloreda only uses polycarboxylate (Figure 20).

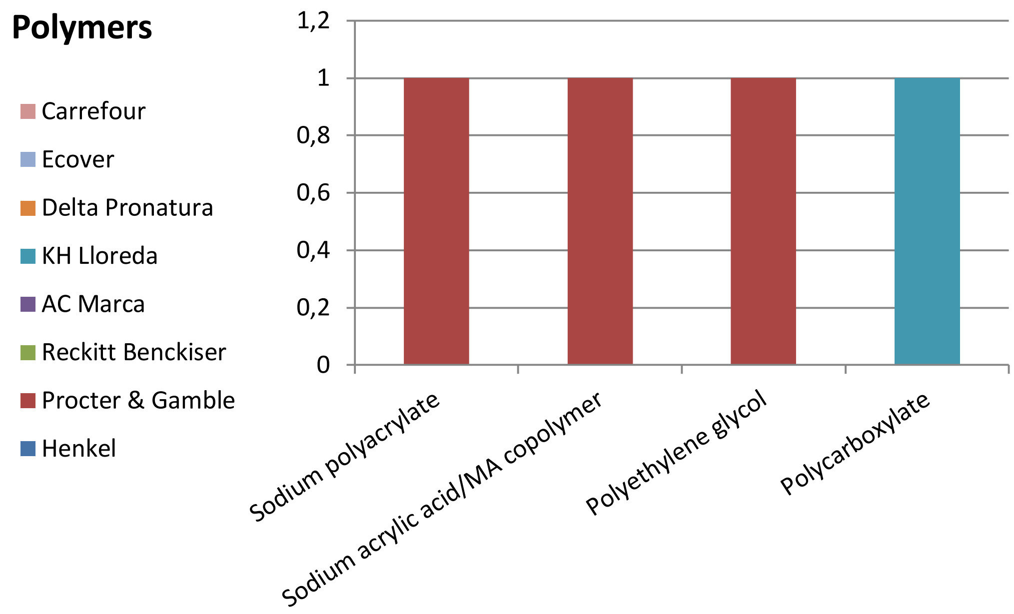

Figure 20. Common polymers used in some repersentative oxy products from the European market

Stain Removers

The base composition of stain removers is quite similar to regular detergents for laundry. The active substances are anionic and/or non ionic surfactants and can also contain polymers and alkaline agents. Most of the selected in this study are presented in liquid form saving the stain remover from Carrefour, which was launched as aerosol. Except AC Marca, all companies include anionic surfactants in their products. The most used is sodium laureth sulfate, which appears in three products; although the general trend is that each company uses their own surfactants (Figure 21). The product with more anionic surfactants (four) is the Stain Removal Roll-On from Delta Pronature – Dr. Beckmann.

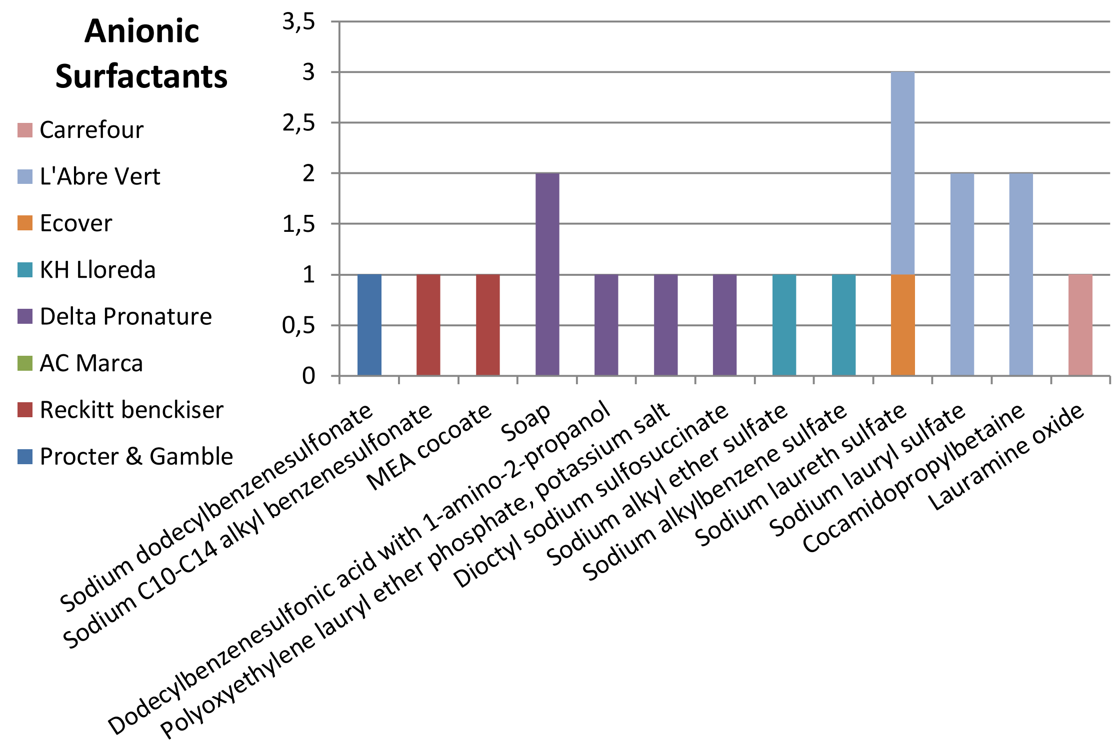

Figure 21. Common anionic surfactants used in some representative stain removers from the European market

In the case of non ionic surfactants, the general trend is also the use of specific types in each company. L'Abre Vert shares the kind of non ionic surfactant with AC Marca on the one hand and Ecover on the other hand (Figure 22). Stain Remover Roll-On from Dr. Beckmann is again the product with more non ionic surfactants (five).

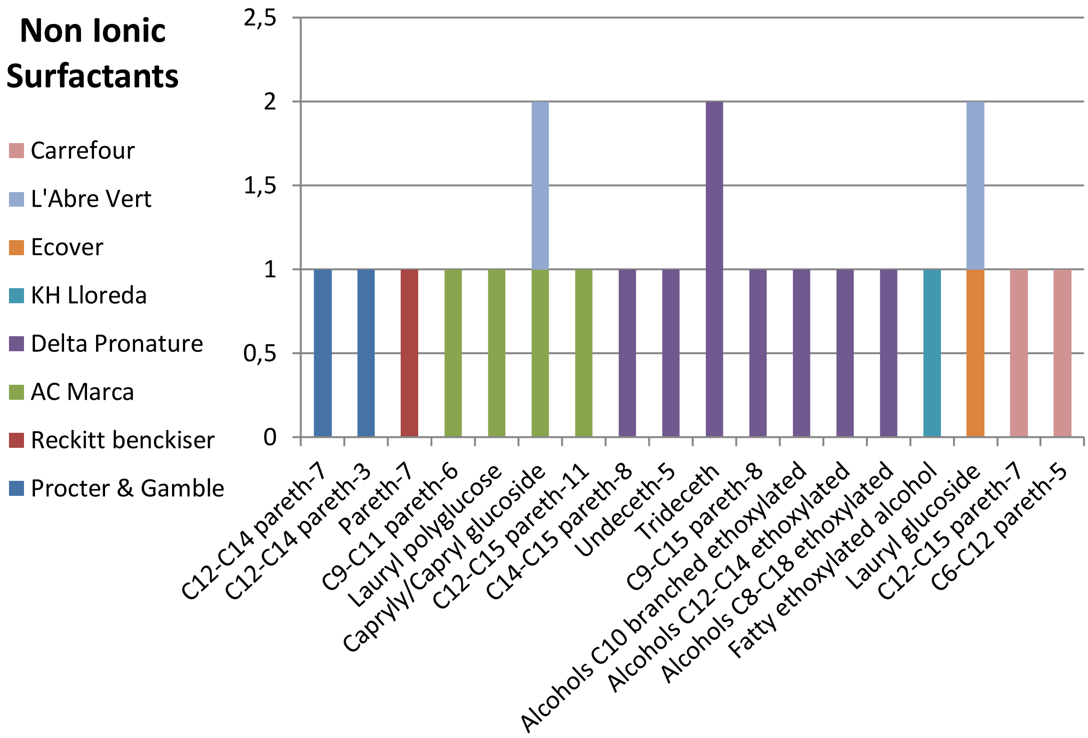

Figure 22. Common non ionic surfactants used in some representative stain removers from the European market

In the stain removers studied only two polymers are used, one in *Vanish Power Shots* from Reckitt Benckiser and the other in *Prewash Quitamanchas con Cepillo Aplicador* from Delta Pronature (Figure 23).

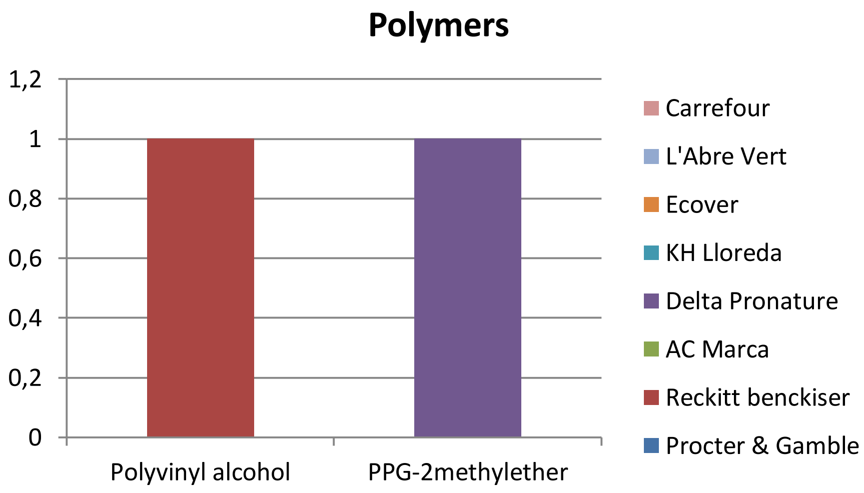

Figure 23. Common polymers used in some representative stain removers from the European market

The most widely used alkaline agent is sodium hydroxide, which appears in all companies except for Carrefour and AC Marca that do not use any alkaline agent. Ethanolamine is commonly used in concentrated formulations (Figure 24).

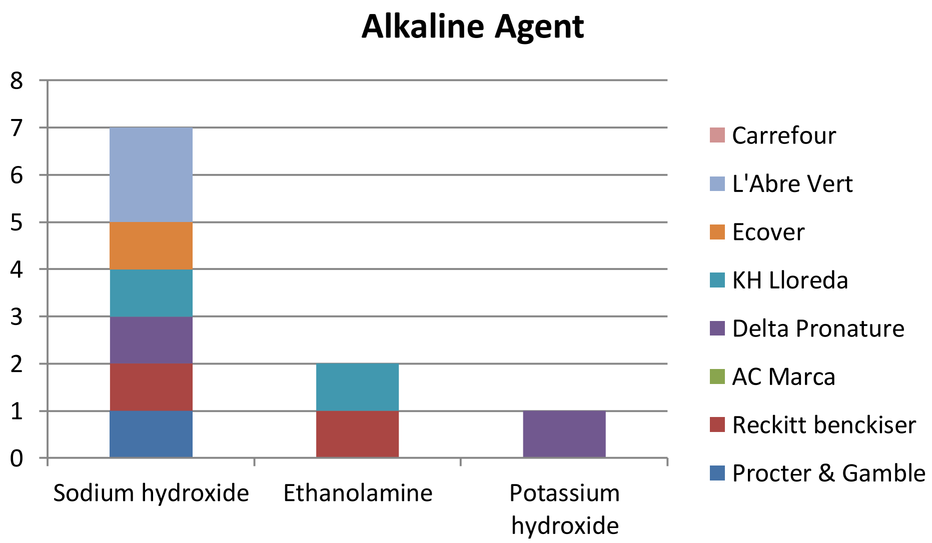

Figure 24. Common alkaline agents used in some representative stain removers from the European market

5. Conclusions

A big variety of laundry products with a very different composition exists in Europe, but always following a similar pattern. All contain surfactants and most of them contain polymers and alkaline agents, among other ingredients.
From anionic surfactants the most common in the products studied are laureth or lauryl sulfates, derivates of benzenesulfonates (above all dodecylbenzenesulfonate), derivates of fatty acid sulfates and various fatty acids from vegetable sources such as cocoate salts.

Regarding non ionic surfactants they are ethoxylated fatty alcohols, of which pareth and laureth derivates are very common. Derivates of glucosides are typical from special detergents and eco brands.

The variety of polymers used is very extensive. Some of the most used are derivates of polypropylene, polyacrylates, polyethyleneglycol and copolymers of styrene/acrylate and acrylate/MA, among others.

About alkaline agents, the most important is sodium hydroxide followed by ethanolamine, which is typically used in concentrated products.

Finally, softeners contain cationic surfactants instead of anionic, of which the most used are derivates of alkylmonium methosulfates. On the other hand bleaches and oxy products contain actives such as sodium hypochlorite and hydrogen peroxide (liquid formulations) or sodium percarbonate (powdered formulations), respectfully.

Therefore, the next steps are to study how some of the products analyzed in this report can increase or decrease the release of micro-fibers in the water before a number of machine washings. Thereby, the influence of some ingredients in the fiber care is expected to be determined, as well as its improvement adding new compounds in the detergent formulation.

6. References

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[3] Laundry Detergents. Eduard Smulders, 2002.

[4] *Revision of Ecolabel Criteria for Laundry Detergents 2008 – 2012, Background Report.* European Ecolabel, ENV. G. 2/SER 2007/007rl. Commission Decision of 28 April 2011. Ecolabeling Denmark. Available at: http://ec.europa.eu/environment/ecolabel/documents/Laundry%20Detergents%20technical%20report.pdf

PRODUCT INFORMATION:

www.gnpd.com

http://uk.cleanright.eu/index.php?option=com_product&task=section&Itemid=195

http://www.henkelconsumerinfo.com/products/Ctrl?_h=henkel.products.ChangeLanguage&lang=en

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http://www.khlloreda.com/es/productos/index.shtml?p=2

7. ANNEX 1 – Composition of Representative Liquid Heavy-Duty Detergents in the European Market

HENKEL – German Market

BRAND	SURFACTANTS – ANIONIC	SURFACTANTS – NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Persil Universal Gel**	Sodium Laureth Sulfate Benzenesulfonic acid, C10-13-alkyl derivates, sodium salts Fatty acids, C12-18, sodium salt	Fatty alcohol ethoxylate, C12-C18, 7EO	Polypropylene terephtalate	Sodium hydroxide	Propylene glycol Sodium Citrate Alcohol Parfum Boric Acid DTPMP heptasaodium salt Sorbitol Disodium distrylbiphenyl disulfonated Buthylphenyl methylpropional Limonene Hexyl cinnamal Benzyl salycilate Protease Geraniol Alpha-isomethyl ionone Amyl cinnamal Linalool Eugenol Amylase Lipase, triacylglycerin- Cellulase Mannanase, endo-1,4-betta Lyase, pectate Colorant Benzisothiazolinone

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Persil Duo-Caps Universal**	Benzenesulfonic acid, mono C10-13-alkyl derivates, compounds with ethanolamine Fatty acids, C12-18, compounds with ethanolamine	Fatty alcohol ethoxylate C13-C15, 8EO	Polypropylene terephtalate	Ethanolamine	Glycerine Propylenglycol Aqua Alcohol DTPMP heptasaodium salt Parfum Disodium distrylbiphenyl disulfonated Protease Citronellol Linalool Benzyl salycilate Buthylphenyl methylpropional Colorant Amylase Cellulase Denatonium benzoate Mannanase, endo-1,4-betta

<table>
<tr><th rowspan="2">BRAND</th><th colspan="2">SURFACTANTS</th><th rowspan="2">POLYMERS</th><th rowspan="2">ALKALINE AGENTS</th><th rowspan="2">OTHERS</th></tr>
<tr><th>ANIONIC</th><th>NON ONIC</th></tr>
<tr><td>
Persil Mega-Caps Universal</td><td>Fatty acid, coco, ethanolamine compounds (MEA-cocoate)
Dodecylbenzenesulfonic acid, 2-aminoethanol (1:1)</td><td>Alcohol, C10-C16, 7EO</td><td>Polyvinylalcohol</td><td>Ethanolamine</td><td>Propyleneglycol
1,3-propandiol-2-methyl-alcohol
Alcohol
Aqua
DTPMP heptasaodium salt
Optical brightener
Parfum
Subtilisin
Denatonium benzoate
Amylase, alpha
Mannanase, endo-1,4-betta
Cellulase
Buthylphenyl methylpropional
Geraniol
Citronellol
Colorant</td></tr>
</table>

HENKEL – Spanish Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Dixan Gel Eco**	Sodium Laureth sulfate Benzensulfonic acid, C10-C13, sodium salts	Fatty alcohol ethoxylate C12-C18 7EO	-	Sodium hydroxide	Aqua Glycerine Sodium Chloride Citric acid Alcohol Parfum DTPMP heptasaodium salt Sorbitol Hexyl cinnamal Buthylphenyl methylpropional Benzoisothiazolinone Disodium distrylbiphenyl disulfonate Methylisothiazolinone

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Wipp Express Gel Activo**	Sodium laureth sulfate benzenesulfonic acid, C10-C13-alkyl derivates, sodium salts Fatty acids, C12-18, sodium salts	Fatty alcohol ethoxylate C12-C18 7EO	-	Sodium hydroxide	Aqua Glycerin Sodium citrate Alcohol Boric acid Parfum Propyleneglycol Heptasodium DTMP DTPMP heptasaodium salt Sorbitol Hexyl cinnamal Protease Disodium distrylbiphenyl disulfonate Limonene Buthylphenyl methylpropional Benzyl salicylate Benzoisothiazolinone Lipasa Triacylglycerol Methylisothiazolinone Amylase Cellulase Colorant Mannanase, endo-1,4-betta-

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Wipp Express Duo-Caps**	MEA Benzensulfonic acid, mono-C10-13-alkyl derivates Fatty acids, C12-C18, compounds with ethanolamine	Fatty alcohol ethoxylate, C13-C15, 8EO	Aziridine, homopolymer, 20EO Polypropylene terephtalate	Ethanolamine	Aqua Glycerin Propylene glycol Alcohol DTPMP heptasaodium salt Parfum Disodium distrylbiphenyl disulfonate Sorbitol Protease Limonene Hexyl cinnamal Buthylphenyl methylpropional Benzyl salicylate Colorant Cellulase Mannanase, endo-1,4-betta-

PROCTER & GAMBLE – Spanish Market

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Ariel Líquido Básico**	Sodium dodecylbenzensulfonate Sodium palm kernelate MEA-dodecylbenzensulfonate Sodium C12-C15 pareth sulfate Sulfated ethoxylated hexamethylenediamine quaternized Sodium cumenesulfonate Hydrogenated castor oil	C12-C14 pareth-7 C14-C15 pareth-7	Co-polymer of PEG/vinyl acetate Styrene/Acrylates copolymer PEG/PGG-10/2 propylheptyl ether Polyethylen glycol	Ethanolamine Sodium hydroxide	Aqua Sodium citrate Propylenglycol Parfum Sodium formate Heptasodium DTMP Benzyl salicylate Hexyl cinnamal Citronellol Protease Linalool Coumarin Sorbitol Benzisothiazolinone Calcium chloride Ethoxylated m-toluidine Methylisothiazolinone Dimethicone Colorant Amylase Glyceryl stearate Hydroxyethylcellulose Silica

BRAND	SURFACTANTS ANIONIC	 NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Ariel Excel Gel Actilift**	MEA-dodecylbenzensulfonate MEA- laureth sulfate MEA-palm kernelate Palm kernel fatty acid Sulfated ethoxylated hexamethylenediamine quaternized Hydrogenated castor oil	C14-C15 pareth-7 C12-C14 pareth-7	Co-polymer of PEG/vinyl acetate PEG/PGG-10/2 propylheptyl ether	Ethanolamine	Aqua Propylenglycol MEA-citrate Diethyleneglycol Trimonoethanolamine etidronate Parfum Sodium formate Fluorescent brightener 9 Sorbitol Glycerin Protease Citronellol Calcium chloride Benzyl salicylate Geraniol Linalool Sodium chloride Amylase Eugenol Mannanase Sodium acetate Cellulase Colorant Benzisothiazolinone

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Ariel Excel Tabs Actilift**	MEA-dodecylbenzenesulfonate MEA-palm kernelate MEA-laureth sulfate Palm kernel acid Dodecylbenzesulfonic acid Hydrogenated castor oil	C12-C14 pareth-7 C14-C15 pareth-7	PEI ethoxylate	Ethanolamine	Propylene glycol Aqua Glycerin Trimonoethanolamine etidronate MEA salt or HEDP Parfum MEA citrate Magnesium chloride Disodium distyrylbiphenil disulfonate Potassim sulfite Benzyl salicylate Sodium formate Sorbitol Citronellol Protease Geraniol Amyl cinnamal Alpha-isomethyl ionone Linalool Hexyl cinnamal Isoeugenol Sulfuric acid Amylase Mannanase Sodium acetate Colorant Cellulase Colorant

RECKITT BENCKISER – Spanish Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Colon Intelligence Plus Gel**	Sodium C10-14 alkyl benzenesulfonate (Sodium + TEA) cocoate Sodium laureth sulfate	Pareth-7	-	Triethanolamine Sodium hydroxide	Aqua Sodium citrate Parfum Heptasodium DTMP Linalool Disodium distyrylbiphenil disulfonate Amylase Geraniol Pectate lyase Bezisothiazolinone Methylisothiazolinone Calcium chloride Colorant Mannanase

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Colon Power Gel**	Sodium C10-14 alkyl benzenesulfonate (Sodium + TEA) cocoate Sodium laureth sulfate	Pareth-7	Acrylic esther polymer Styrene/acrylates copolymer	Triethanolamine Sodium hydroxide	Aqua Sodium citrate Parfum Sodium formate Heptasodium DTMP Disodium distyrylbiphenil disulfonate Sodium sulphite Protease Citronellol Amylase Limonene Bezisothiazolinone Calcium chloride Butylphenyl methyl propional Hexyl cinnamal Alpha-isomethyl ionone Colorant

RECKITT BENCKISER – Italian Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Sole Bianco Solare Liquido**	Sodium C10-14 Alkyl Benzenesulfonate (Sodium + TEA) Cocoate	Pareth-7	Styrene/Acrylates Copolymer	Triethanolamine Sodium hydroxide	Aqua Heptasodium DTMP Parfum Sodium chloride Sodium bicarbonate powder Disodium distyrylbiphenyl disulphonate Amylase Limonene Alpha-iso-methylionone Benzisothiazolinone Methylisothiazolinone Propylene glycol

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Sole Perle di Pulito (Gel Caps)**	Benzenesulfonic acid, mono-C10-13-alkyl derivs., compds. with ethanolamine MEA Cocoate	Pareth-7	PVOH co-polymer film	Ethanolamine Sodium hydroxide	Propylene glycol Glycerol Aqua Parfum Protease Citric acid Heptasodium DTMP Disodium distyrylbiphenyl disulphonate BHT d-Limonene Alpha-iso-methylionone Benzisothiazolinone Linalool Cl 61585

UNILEVER – French Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Skip Liquide Active Clean**	TEA-dodecylbenzensulfonate Sodium dodecylbenzensulfonate Sodium laureth sulfate TEA-cocoate Sodium hydrogenated cocoate	C11-C15 pareth-7	Polypropylene terephthalate/polyoxyethylene terephtalate Polyvinyl Alcohol	Sodium hydroxide Triethanolamine	Aqua Sodium citrate Glycerin Parfum Heptasodium DTMP Propylene glycol Sorbitol Sodium sulfate Subtilisin ButylphenylMethylpropional Citronellol Disodium distyrylbiphenyl disulphonate Benzisothiazolinone Boronic acid, (4-formylphenyl) Amylase CI50325 Cellulose Gum CI45100

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Skip Petit & Puissant Active Clean**	MEA-Dodecylbenzenesulfonate Sodium Laureth Sulfate TEA-Hydrogenated Cocoate Sodium Lauryl Sulfate	C11-C15 pareth-7	Aziridine homopolymer ethoxylated Acrylates Copolymer Styrene/Acrylates Copolymer	Triethanolamine Sodium hydroxide Ethanolamine	Aqua Propylene glycol MEA-Citrate Etidronic acid Parfum Sorbitol MEA-Sulfate Sodium sulfite Disodium distyrylbiphenyl disulphonate ButylphenylMethylpropional Citronellol Sodium sulfate Subtilisin Glycerin Geraniol Boronic acid, (4-formylphenyl) Pectate Lyase Amylase Mannanase Benzisothiazolinone Cl 42051

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Skip Capsules Active Clean**	MEA-dodecylbenzenesulfonate MEA-hydrogenated cocoate	C12-C15 pareth-7	Polyvinyl alcohol Aziridine homopolymer ethoxylated	Ethanolamine	Dipropylene glycol Parfum Heptasodium DTMP MEA-sulfate Sorbitol Subtilisin Glycol ButylphenylMethylpropional Hexyl Cinnamal Starch Limonene Boronic acid, (4-formylphenyl) Linalool Disodium distyrylbiphenyl disulphonate Alpha-iso-methylionone Geraniol Polymeric Blue Colourant Amylase Talc Sodium chloride Mannanase Polymeric Pink Colourant Polymeric Red Colourant Denatonium benzoate

ECO BRANDS - ECOVER

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Ecover Laundry Liquid**	Sodium lauryl sulfate Sodium cocoate & Sodium oleate	Coco-glucoside Fatty acid methyl ester ethoxylate	Polypropylene terephtalate	Sodium hydroxide	Aqua Benzyl Alcohol Sodium chloride Sodium citrate Parfum Citric acid Linalool Limonene Citronellol Trisodium ethylenediamine disuccinate

ECOLABEL BRANDS – L'ABRE VERT

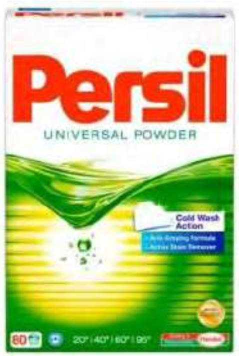
BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **L'Abre Vert Lessive Liquide au Savon Vegetal Ecologique**	Sodium Laureth Sulfate Potassium cocoate Sodium lauryl sulfate	Fatty alcohols C12-C18 ethoxylated Lauryl glucoside	Sodium polycarboxylate	Sodium hydroxide	Aqua Alcohol Sodium citrate Sodium chloride Parfum Calcium chloride Proteases Amylases Dimethicone

GENERIC BRANDS - CARREFOUR

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Carrefour Ultra Concentrated Laundry Detergent Liquid with Marseille Soap**	(Na +TEA) Savon LAS Na	Trideceth-2	Polystyrene	Triethanolamine	Aqua Sodium citrate Sodium borate Parfum Phosphonate Protease Amylase Sodium sulfite Disodium distyrylbiphenyl disulphonate Silicone Benzisothiazolinone Calcium chloride

8. ANNEX 2 – Composition of Representative Powdered Heavy-Duty Detergents in the European Market

HENKEL – Belgian Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Persil Universal Powder**	Benzenesulfonic acid, C10-13-alkyl derivates, sodium salts Fatty acids, C16-18, sodium salt	Alcohol ethoxylate, C13-C15, 7EO	Carboxymethylcellulose-Na Sodium polyacrylate Methyl alkyl polysiloxane	Sodium hydroxide	Sodium sulfate Sodium carbonate Sodium carbonate peroxide Silicic acid, sodium salt TAED Aqua Tetrasodium etidronate Parfum Starch CP anion. Mod. Alkylene glycolphtalate Optical brightener Zea mays (corn) seed flour Linalool Colorant Benzyl salicylate Hexyl cinnamal Protease Lipase type Lipex Amylase Mannanase type mannaway Cellulase

HENKEL – German Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Persil Tabs**	Sodium dodecylbenzenesulfonate Sodium lauryl sulfate Sodium soap C16-18	C12-C18 fatty alcohol 7EO Lauryl glucoside C12 - C18 fatty alcohol 5EO	Sodium acrylic acid/MA copolymer Methyl alkyl polysiloxane PEG-14M Sodium polyacrylate PEG-240	Sodium hydroxide	Zeolite Sodium bicarbonate Sodium carbonate peroxide Aqua Cellulose TAED Sodium sulfate Cellulose gum Tetrasodium etidronate Parfum Sodium silicate Starch Heptasodium DTPMP Zea mays (corn) starch Sodium chloride Sodium carbonate Sodium glycolate Optical brightener Corn flour Glyceryl stearate Colorant Hexyl cinnamal Linalool Benzyl salicylate Protease Sucrose Amylase Cellulase Mannanase

HENKEL – Spanish Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Dixan Business Line**	Benzenesulfonic acid, C10-C13 alkyl derivates, sodium salts	C13-C15 fatty alcohol 7EO	Sodium polyacrylate	Sodium hydroxide	Sodium sulfate Sodium carbonate Silicic acid, sodium salt Sodium carbonate peroxide Aqua Tetrasodium etidronate Carboxymethyl cellulose, sodium salt TAED Parfum Starch Fluorescent brightener 71 Colorant Hexyl cinnamal Protease Amylase Lipase, triacylglycerol Cellulose Mannanase, endo-1,4-beta

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Wipp Express Polvo Business Line**	Benzensulfonic acid, C10-C13, sodium salts	C12-C18 fatty alcohol 7EO	Sodium polyacrylate	Sodium hydroxide	Sodium sulfate Sodium carbonate Silicic acid, sodium salts Sodium carbonate peroxide Aqua Carboxymethyl cellulose, sodium salt TAED Tetrasodium etidronate Starch Parfum Fluorescent brightener 71 Colorant Protease Linalool Amylase Mannanase, endo-1,4-beta Cellulase

PROCTER & GAMBLE – Spanish Market

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Ariel Básico**	Sodium dodecylbenzenesulfonate Sodium C12-C15 pareth sulfate Dodecylbenzenesulfonic acid Sodium coceth-30 sulfate	C12-C14 pareth-7	Sodium acrylic acid/MA copolymer Sulfonated polyethylene/polyethylene terephtalate Sodium polyacrylate Polyethylene glycol	Sodium hydroxide	Sodium sulfate, Sodium carbonate, Sodium carbonate peroxide, Sodium silicate, Aqua, Sodium silicoaluminate, Citric acid, TAED, Sodium chloride, Parfum, Cellulose gum, Tetrasodium etidronate, Sulfuric acid, Brightener 15, Silicone compound, Hexyl cinnamal, Sodium starch octenylsuccinate, Microcrystalline cellulose, Limonene, Kaolin, Dextrin, Titanium dioxide, Citronellol, Protease, Calcium carbonate, Sorbitol, Mannanase, Zinc Phthalocyanine sulphonate, Colorant, Lipase, Glycerin, Magnesium sulfate, Amylase, Hydroxypropyl methylcellulose, Sucrose

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Ariel Actilift**	Sodium dodecylbenzenesulfonate Sodium C12-C15 alkyl sulfate Sodium tallowate Dodecylbenzene sulfonic acid Sodium coceth-30 sulfate	Ceteareth-15	Sodium acrylic acid/MA copolymer Calcium divinylbenzene styrene copolymer sulfonate Polyethylene glycol PEI-2500 Sodium polyacrylate	Sodium hydroxide	Sodium carbonate, Sodium sulfate, Bentonite, Sodium carbonate peroxide, Sodium silicoaluminate, Adipic acid, TAED, Sodium metasilicate, Aqua, Acetic acid, sodium salt, trihydrate, Sorbitol, Tetrasodium etidronate, Parfumm, Cellulose gum, Sodium silicate, Fluorescent brightener 260, Butylphenyl methylpropional, Calcium carbonate, Microcrystalline cellulose, Dextrin, Protease, Titanium dioxide, Silicone compound, Hexyl cinnamal, Kaolin, Coumarin, Limonene, Glycosidase, Etidronic acid, Glycosidase, Sodium chloride, Colorant, Zinc phthalocyanine sulphonate, Hydroxypropyl methylcellulose, Sucrose, Colorant, Glycerin

RECKITT BENCKISER – Spanish Market

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
**Colon Intelligence Plus Polvo**	Sodium dodecylbenzenesulfonate C14-C22 fatty acids, sodium salts	Trideceth-7	Sodium maleate-acrylate copolymer	Sodium hydroxide	Sodium sulphate Zeolite Sodium carbonate Sodiumpercarbonate Sodium silicate Tetraacetylethyleendiamine Parfum Carboxymethylcellulose Tetrasodium etidronate Silicone compound Fluorescent brightener 71 DTPMP sodium salt Protease Linalool Mannanase CI 74160 Amylase

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Colon Easy Clean Polvo**	Sodium dodecylbenzenesulphonate C14-C22 fatty acids, sodium salts	C12-C15 Pareth-7	Sodium maleate-acrylate copolymer	Sodium hydroxide	Sodium sulphate Zeolite Sodium carbonate Sodium carbonate peroxide Sodium silicate Aqua Sodium bicarbonate TAED Cellulose gum Parfum Tetrasodium etidronate Fluorescent brightener 71 Hexyl cinnamal Silicone compound DTPMP sodium salt Limonene Cl 74160 Subtilisin Amylase

RECKITT BENCKISER – Italian Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Sole Pulito e Igiene Polvere**	Sodium dodecylbenzenesulphonate C14-C22 fatty acids, sodium salts	C12-C15 pareth-7	Sodium maleate-acrylate copolymer	Sodium hydroxide	Sodium sulphate Sodium carbonate Zeolite Sodium carbonate peroxide Sodium silicate Aqua Sodium bicarbonate TAED Cellulose gum Parfum Tetrasodium etidronate Hexyl cinnamal Fluorescent brightener 71 DTPMP sodium salt Limonene Cl 74160 Subtilisin Amylase

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Sole Bianco Solare Polvere**	Sodium C10-C13 alkyl benzenesulfonate C14-C22 fatty acids, sodium salts	Trideceth-7	Sodium maleate-acrylate copolymer	Sodium hydroxide	Sodium sulphate Zeolite Sodium carbonate Sodium percarbonate Sodium silicate Aqua Sodium bicarbonate Tetraacetylethyleendiamine Parfum Carboxymethylcellulose Tetrasodium etidronate Fluorescent brightener 71 Hexyl cinnamal Silicone compound DTPMP sodium salt Limonene Cl 74160 Protease Amylase

UNILEVER – French Market

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Skip Poudre Active Clean**	Sodium dodecylbenzenesulfonate Stearic acid Sodium stearate Sodium cocoate	C12-15 pareth-7	Sodium acrylic acid/MA copolymer Polyethilene terephthalate PEG-75 Sodium polyacrylate Polyoxymethylene melamine Sodium polyaryl sulphonate	Sodium hydroxide	Sodium sulphate, Sodium carbonate ,Sodium carbonate peroxide, Sodium silicate, Aqua, Zeolite, TAED, Bentonite, Citric acid, Parfum, Corn starcho modified, Cellulose gum, Sodium chloride, Tetrasodium etidronate, Disodium anilinomorpholinotriazinyl aminostilbenesulfonate, Calcium sodium EDTMP, Parfum, Phenylpropyl dimethicone, Sodium bicarbonate, Cellulose, Calcium carbonate, Glyceryl stearates, Kaolin, Titanium dioxide, Dextrin, Alpha-isomethyl ionone, Subtilisin, Geraniol, Imidazolidinone, Sucrose, Sorbitol, Aluminium silicate, Cl 61585, Cl 45100, Lipase, Amylase, Cl 12490, Hydroxypropyl methyl cellulose, Xanthan gum, Disodium, Distrylbiphenyl disulfonate, Cl 42090, Sodium thiosulfate, Mannanase, Cl 11680, Etidronic acid, Tetrasodium EDTA

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Skip Tablettes Active Clean**	Sodium lauryl sulfate Sodium dodecylbenzenesulfonate Dodecylbenzenesulfonic acid	Lauryl glucoside	Sodium acrylic acid/MA copolymer Sodium polyacrylate PEG-75 Sodium polyaryl sulphonate	Sodium hydroxide	Sodium carbonate, Sodium carbonate peroxide, Sodium bicarbonate, Zeolite, Aqua, Sodium silicate, Cellulose, TAED, Hemicellulose, Lignin, Bentonite, Sodium chloride, Parfum, Tetrasodium etidronate, Sodium sulfate, Dimethicone, Disodium anilinomorpholinotriazinyl Aminostilbenesulfonate, Trimethylsiloxysilicate, Calcium carbonate, Cellulose, Titanium dioxide, Dextrin, Protease, Corn starch modified, Sucrose, CI 12490, Sodium thiosulfate, Amylase, Kaolin

ECOLABEL BRANDS – L'ABRE VERT

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **L'Abre Vert Lessive Pouder au Savon Vegetal Ecologique**	Sodium palmate Sodium alkyl C12-C18 sulfate Sodium cocoate Glycereth-17 cocoate	Ceteareth-25	Polycarboxylate sodium salt	Sodium hydroxide	Sodium bicarbonate Sodium carbonate Sodium percarbonate Sodium sulfate Sodium disilicate Sodium citrate N,N'-ethylenebis[N-acetylacetamide] Amylases Proteases Combination of silicone with silice Parfum

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **L'Abre Vert Doses Lessives Hydrosolubles Fraicheur Vegetale (powder)**	-	Ceteareth-25	Polyvinyl alcohol resin Polycarboxylate sodium salt	Sodium hydroxide	Sodium bicarbonate Sodium carbonate Sodium percarbonate Sodium citrate Sodium disilicate Combination of silicone with silice Amylases Parfum Manganese complex Proteases

ECO BRANDS - ECOVER

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Ecover Universal Washing Powder**	Sodium C12-C18 alkyl sulfate Sodium oleate	Laureth-3 laureth-9	Sodium polyaspartate Polyitaconate	Sodium hydroxide	Sodium sulfate Zeolite Sodium carbonate peroxide Sodium carbonate TAED Sodium disilicate Aqua Sodium carboxymethyl starch Parfum Limonene Cellulose gum Linalool Subtilisin Lipase Amylase Cellulase Mannanase

GENERIC BRANDS - CARREFOUR

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Oxy White Fabric Brightening Tablets**	-	Alkoxylated fatty alcohol	PEG-2M	Sodium hydroxide	Citric acid Sodium carbonate peroxide Tetraacetyl ethylene diamine Solium silicate Sodium bicarbonate Tetrasodium etidronate Cellulose Protease Subtilisin Bentonite Disodium bis triazinylamino stilbene disulfonate CI 73015 CI 42090 Parfum

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Carrefour Detergente en Tabletas**	Sodium C12-C18 alkyl sulfate Sodium dodecylbenzenesulfonate	Coco-glucoside	Sodium acrylate/maleate copolymer Modified terephtalate/ethyleneglycol copolymer	Sodium hydroxide	Sodium carbonate Sodium carbonate peroxide Sodium bicarbonate Aqua Zeolite Cellulose Sodium silicate TAED Bentonite Dimethicone Tetrasodium etidronate Parfum Alpha-amylase Subtilisin Sodium triazinylaminostilbenesulfonate Mannanohydrolase Limonene Linalool Benzyl salicylate CI 12085

9. ANNEX 3 – Composition of Representative Special Detergents in the European Market

HENKEL – German Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 Persil Color Gel	Sodium laureth sulfate Sodium C10-C13-alkyl benzenesulfonate Fatty acids, C12-18, sodium salt	Alcohol ethoxylate, C12-C18, 7EO	Polyprolylene terephtalate Vinylpyrrolidone/vinylimidazole copolymer	Sodium hydroxide	Aqua, Propylene glycol, Sodium citrate, Alcohol, Parfum, Boric acid, DTMP sodium salt, Sorbitol, Butylphenyl methylpropional, Limonene, Hexyl cinnamal, Benzyl salicylate, Protease, Geraniol, Alpha-isomethyl ionone, Amyl cinnamal, Linalool, Eugenol, Amylase, Liapse, triacylglycerin-, Cellulase, Mannanase, endo-1,4-beta-, Lyase, pectate, Colorant, Benzisothiazolinone, Methylisothiazolinone

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Persil Color Mega-Caps**	MEA-cocoate Dodecylbenzenesulfonic acid with 2-aminoethanol (1:1)	C10-C16 fatty alcohol 7EO	Polyvinylalcohol Polyquaternium-16	Ethanolamine	Propylene glycol 2-methyl-1,3-propandiol Alcohol Aqua DTMP sodium salt Parfum Subtilisin Denatonium benzoate Amylase, alpha- Mannanase, endo-1,4-beta- Cellulase Butylphenyl methylpropional Geraniol Citronellol Colorant

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Persil Duo-Caps Color**	MEA-Benzenesulfonate, C10-C13 alkyl derivates MEA-fatty acid, C12-C18 alkyl derivates	C13-C15 fatty alcohol 8EO	Polypropylene terephtalate	Ethanolamine	Glycerin Propylene glycol Aqua Alcohol Heptasodium DTMP Parfum Protease Citronellol Linalool Benzyl salicylate Butylphenyl methylpropional Colorant Amylase Cellulase Denatonium benzoate Mannanase, endo-1,4-beta

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Micolor Gel**	Sodium laureth sulfate Benzensulfonic acid, C10-C13, sodium salts Fatty acids, C12-C18, sodium salts	C12-C18 fatty alcohol 7EO	-	Sodium hydroxide	Aqua Glycerin Sodium citrate Sodium chloride Alcohol Boric acid Parfum DTPMP sodium salt Citronellol Butylphenyl methylpropional Hexyl cinnamal Limonene Benzisothiazolinone Methylisothiazolinone Colorant Cellulase Amylase Glutaral

HENKEL – Spanish Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Perlan Máquina Pétalos de Tiaré**	Sodium laureth sulfate Sodium dodecylbenzenesulfonate Sodium C12-C18 soap	C12-C18 fatty alcohol 7EO	Vinylpyrrolidone/vinylimidazole copolymer Styrene/acrylates copolymer	Sodium hydroxide	Aqua Sodium chloride Sodium metaborate Glycerin Parfum Sodium citrate Heptasodium DTPMP Citronellol Butylphenyl methylpropional Tetramethylolglycoluril Methylisothiazolinone Octylisothiazolinone Colorant

HENKEL – Finnish Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Perlana Color & Delicates**	Sodium laureth sulfate Alkylbenzene sulfonate C10-C13 sodium salt Fatty acid C12-C18 sodium salt	Fatty alcohol ethoxylate C12-C18 7EO	Styrene/acrylates copolymer Vinylpyrrolidone/vinylimidazole copolymer Polypropylene glycol Ethylene oxide/propylene oxide copolymer	Sodium hydroxide	Aqua Sodium chloride Sodium metaborate Glycerin Parfum Sodium citrate DTPMP sodium salt Sorbitol Propylene glycol Citronellol Butylphenyl methylpropional Hexyl cinnamal Limonene Benzisothiazolinone Methylisothiazolinone Cellulase Dimethicone Lipase Phenoxyethanol Amylase Mannanase type mannaway Calcium chloride Xylenes (individual or mixed isomers) Ethyl benzene

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Perlana Wool & Delicates**	Sodium laureth sulfate Alkylbenzene sulfonate C10-C13 sodium salt Fatty acid C12-C18 sodium salt	Fatty alcohol ethoxylate C12-C18 7EO	Styrene/acrylates copolymer Vinylpyrrolidone/vinylimidazole copolymer Polypropylene glycol Ethylene oxide/propylene oxide copolymer	Sodium hydroxide	Aqua Sodium chloride Parfum Glycerin Boric acid Sodium citrate DTPMP sodium salt Butylphenyl methylpropional Amyl cinnamal Sorbitol Citronellol Geraniol Propylene glycol Alpha-isomethyl ionone Benzisothiazolinone Methylisothiazolinone Dimethicone Lipase type lipex Phenoxyethanol Amylase Mannanase type mannaway Calcium chloride Colorant Ethyl benzene Ksyleeni – isomeerien seos Nerol

AC MARCA– Spanish Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Norit Cuidado Delicado Máquina**	Sodium dodecylbenzenesulphonate Sodium C14-16 olefin sulfonate Oleic acid	Fatty alcohol ethoxylated	-	Sodium hydroxide	Aqua Sodium chloride Parfum Benzisothiazolinone 2-bromo-2-nitropropane-1,3-diol Mixture of 5-Chloro-2-methyl-isothiazol-3(2H)-one and 2-Methylisothiazol-3(2H)-one with magnesium chloride and magnesium nitrate 2,2’-[(9, 10-dihydro-9,10-dioxo-1,4-anthrylene)diimino]bis[5-tert-butylbenzenesul]

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Norit Cuidado Color**	Sodium dodecylbenzenesulphonate Sodium C14-16 olefin sulfonate Fatty acid, palmkernel-oil, sodium salt	Fatty alcohol ethoxylated	-	Sodium hydroxide	Aqua Sodium chloride Parfum Benzisothiazolinone 2-bromo-2-nitropropane-1,3-diol Colorant Mixture of 5-Chloro-2-methyl-isothiazol-3(2H)-one and 2-Methylisothiazol-3(2H)-one with magnesium chloride and magnesium nitrate

RECKITT BENCKISER – Italian Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Sole Lana e Delicati Detersivo Liquido**	Sodium C10-C14 alkyl benzenesulfonate TEA cocoate Sodium laureth sulfate	Trideceth-7 Cocamide DEA	Polyamine stearate Styrene/acryaltes copolymer	Sodium hydroxide Triethanolamine Diethanolamine	Aqua Sodium chloride Parfum Butylphenyl methylpropional Hexyl cinnamal Benzyl salicylate DTPMP sodium salt Sodium sulfite BHT Methylisothiazolinone Benzisothiazolinone Colorant

With the contribution of the LIFE financial instrument of the European Comunity:

mermaids. OCEAN CLEAN WASH

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Sole Perle Proteggi Colore (gel caps)**	MEA benzenesulfonic acid, alkyl C10-C13 Sodium C10-C14 alkyl benzenesulfonate MEA cocoate	Pareth-7	PVOH copolymer film	Sodium hydroxide Ethanolamine	Glycerol Aqua Parfum Protease Citric acid DTPMP sodium salt Disodium distyrylbiphenil disulphonate BHT d-Limonene Alpha-iso-methylionone Benzisothiazolinone Linalool CI 61585 CI 45100

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Lip Woolite Mix Noir**	Sodium dodecylbenzenesulfonate Sodium laureth sulfate (Sodium + TEA) Cocoate	Pareth-7 Cocamide DEA	Polyvinylpirrolydone/vinylimidazole copolymer	Sodium hydroxide Triethanolamine Diethanolamine	Aqua Sodium chloride Parfum DTPMP sodium salt BHT Benzisothiazolinone Methylisothiazolinone Cellulase CI 42051 Acid Red 52 (CI 45100)

BRAND	SURFACTANTS ANIONIC	NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Lip Woolite Mix Color**	Sodium dodecylbenzenesulfonate Sodium laureth sulfate (Sodium + TEA) Cocoate	Pareth-7 Cocamide DEA	Polyvinylpirrolydone/vinylimidazole copolymer	Sodium hydroxide Triethanolamine Diethanolamine	Aqua Sodium chloride Parfum DTPMP sodium salt Linalool Limonene BHT Geraniol Citronellol Butylphenyl methylpropional 1,2-benzisothiazolin-3-one 2-methyl-2H-isothiazolin-3-one Cellulase CI 42051

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Lip Woolite Lana e Delicati (Gel Caps)**	MEA dodecylbenzenesulfonate MEA cocoate	Pareth-7	Polyvinyl alcohol Styrene/acrylates copolymer	Ethanolamine	Propylene glycol Glycerol Aqua MEA citrate Parfum DTPMP sodium salt Sodium sulfite Butylphenyl methylpropional BHT Citronellol Linalool Hexyl cinnamal Benzisothiazolinone Geraniol Benzyl salicylate Colorant

ECO BRANDS - ECOVER

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Ecover Delicate Laundry Liquid**	Potassium soap of coco and olein fatty acids Sodium lauryl ether sulfate Sodium octyl sulfate	Alkyl polyglycoside C10-16	-	Sodium hydroxide Potassium hydroxide	Water Sodium chloride Citric acid Parfum Limonene 2-bromo-2-nitropropane-1,3-diol

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Ecover Color Washing Powder**	Sodium C12-C18 alkyl sulfate Sodium oleate	Laureth-3 Laureth-9	Sodium polyaspartate Polytaconate	Sodium hydroxide	Sodium carbonate Sodium sulfate Zeolite Sodium bicarbonate Sodium disilicate Aqua Sodium carboxymethyl starch Parfum Limonene Subtilisin Lipase Cellulose gum Linalool Amylase Cellulase Mannanase

ECOLABEL BRANDS – L'ABRE VERT

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **L'Abre Vert Lessive Lana et Textiles Delicats Ecologique**	Sodium laureth sulfate Potassium cocoate Sodium lauryl sulfate	Fatty alcohols C12-C18 ethoxylated Lauryl glucoside	-	Sodium hydroxide Potassium hydroxide	Aqua Alcohol Sodium citrate Parfum Dimethicone

GENERIC BRANDS – CARREFOUR

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON ONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Carrefour Lana e Capi Delicati**	Sodium laureth/pareth sulfate MEA-dodecylbenzenesulfonate MEA-cocoate	C12-C15 pareth-7	Sodium styrene/acrylates copolymer Polyquaternium-7	Sodium hydroxide Ethanolamine	Aqua DTPMP sodium salt Sodium chloride Parfum Dimethicone Methylchloroisothiazolinone Methylisothiazolinone Octylisothiazolinone CI 45170 CI 42090

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON ONIC			
 **Detergente Lana Carrefour**	Sodium laureth 2-sulfate C10-C14 alkyl benzenesulfonic acid Potassium cocoate Sodium cumenesulfonate	C12-C18 pareth-7	-	Sodium hydroxide Triethanolamine	Aqua Sodium chloride Parfum Tetrasodium EDTA Simethicone Methylchlorotiazolinone Methyisothiazolinone Colorant

10.ANNEX 4 – Composition of Representative Laundry Aids in the European Market

10.1.SOFTENERS

HENKEL – German Market

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Vernel Hautsensitiv**	-	-	Dipalmitoylethyl hydroxyethylmonium methosulfate	-	-	Aqua Isopropyl alcohol Parfum 3-amino propyl Me, di-Me siloxane Benzothiazolinone

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Vernel Wild-Rose**	-	-	Dipalmitoylethyl hydroxyethylmonium methosulfate	-	-	Aqua Isopropyl alcohol Parfum 3-aminopropyl Me, di-Me siloxane Butylphenyl methylpropional Citronellol Coumarin Benzithiazolinone Colorant

BRAND	SURFACTANTS		ACTIVES	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Vernel Crystals Frische Zauber**	-	-	Sucrose Bentonite	PEG-150	-	Parfum Aqua Alpha-isomethyl ionone Colorant

UNILEVER – French Market

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Cajoline Liquide Tentation Rose Jasmin**	-	Cetearyl alcohol	Dihydrogenated tallowoylethyl hydroxyethylmonium methosulfate	-	-	Aqua Benzisothiazolinone Butylphenyl methylpropional Cetyl hydroxyethil cellulose Dimethicone Hexyl cinnamal Isopropyl alcohol Parfum Polymeric pink colourant

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Cajoline Concentré Brin de Folie Lavande & Amande Douce**	-	Cetearyl alcohol Laureth-20 Caprylic/Capric glycerides Hydrogenated vegetable glycerides Glycol stearate	Dihydrogenated tallowoylethyl hydroxyethylmonium methosulfate	Polyoxymethylene melamine	Sodium hydroxide	Aqua Isopropyl alcohol Parfum Benzyl salicylate Coumarin Hydrochloric acid Imidazolidinone Limonene Benzisothiazolinone Dimethicone Polymeric pink colourant Trimethylsiloxysilicate Cellulose gum Polymeric blue colorant Cetyl hydroxyethil cellulose

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Cajoline Capsules Fleur de la Passion & Bergamote**	-	Hydrogenated vegetable glycerides Glycol stearate	Ditallowoylethyl hydroxyethylmonium methosulfate	Polyoxymethylene melamine	-	Aqua Isopropyl alcohol Parfum Butylphenyl methylpropional Limonene Hexyl cinnamal Amyl cinnamal Linalool Alpha-isomethyl ionone Citronellol Dimethicone Imidazolidinone Benzisothiazolinone EtidrIONIC acid Trimethylsiloxysilicate Calcium chloride Cellulose gum Xanthan gum

RECKITT BENCKISER – Spanish Market

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Flor Frescor Oceánico Regular**	-	-	Dihydrogenated tallow hydroxylethylmethylammonium methosulfate	Polymethylsiloxane	-	Aqua Parfum Isopropyl alcohol Hexyl cinnamal Butylphenyl methylpropional Geraniol Coumarin Benzisothiazolinone Methylisothiazolinone Colorant

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Flor Azul 24h con Micro-Cápsulas Concentrado**	-	-	Dihydrogenated tallow hydroxylethylmethylammonium methosulfate	Polymethylsiloxane	-	Aqua Parfum Isopropyl alcohol Microencapsulated Parfum Hexyl salycylate Hexyl cinnamal Butylphenyl methylpropional Coumarin Geraniol Benzisothiazolinone Alpha-isomethyl ionone Colorant

ECO BRANDS - ECOVER

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Ecover Softener Amongst the Flowers**	-	-	Dipalmitoyethyl hydroxylethylmonium methosulfate	-	-	Aqua Dipropylene glycol Parfum Sorbic acid Linalool Magnesium chloride Citronellol

ECOLABEL BRANDS – L'Abre Vert

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **L'Abre Vert Assouplissant Souffle de Purete Ecologique**	-	Caprylyl/Capryl glucoside	Bis(acyloxyethyl)hydroxyethyl methylammonium methosulfate esterquat	-	-	Aqua Isopropyl alcohol Alcohol Parfum Calcium chloride

GENERIC BRANDS – CARREFOUR

BRAND	SURFACTANTS			POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC	CATIONIC			
 **Hypoallergenic Fabric Conditioner**	-	-	Dihydrogenated tallow hydroxyPEG-ethylmonium methosulfate	-	-	Aqua Isopropyl alcohol Parfum Phenoxyethanol

10.2.BLEACH

HENKEL – Spanish Market

BRAND	SURFACTANTS ANIONIC	SURFACTANTS NON IONIC	ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
 **Neutrex Futura (bleach)**	Sodium fatty alcohol sulfate C12-C14 2+2, 35EO	-	Sodium hypochlorite	-	Sodium hydroxide	Aqua Pentapotassium Hydrogen[nitriletris(methylene)]trisphosphonate Colorant

PROCTER & GAMBLE – Italian Market

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Ace Candeggina Classica con Nuovo Salvafibre (bleach)**	-	-	Sodium hypochlorite	-	Sodium hydroxide	Aqua Sodium chloride Sodium carbonate Potassium triphosphate

BRAND	SURFACTANTS ANIONIC	NON IONIC	ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
 **Ace Candeggina Densa-Igienizzante**	Sodium C12-C14 pareth-3 sulfate Cocoate Sodium lauryl sulfate	-	Sodium hypochlorite	-	Sodium hydroxide	Aqua Sodium chloride Sodium carbonate Sodium silicate Trimethoxybenzoic acid Parfum Pentasodium triphosphate Colorant Phenyl methicone Silica

GENERIC BRANDS – CARREFOUR

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Carrefour Classic Bleach**	-	-	Sodium hypochlorite	-	Sodium hydroxide	Aqua

10.3.OXY PRODUCTS

HENKEL – German Market

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 Sil Oxi Perfect 2	Sodium laureth sulfate	Laureth-2	Hydrogen peroxide	-	Sodium hydroxide	Aqua Sodium carbonate Trisodium dicarboxymethyl alanine Parfum Sodium hydroxymethylglycinate Dye

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Sil Oxi Flecken-Spray**	Sodium lauryl sulfate	-	Hydrogen peroxide	-	Sodium hydroxide	Aqua Tetrasodium etidronate Sodium benzoate Perfum

PROCTER & GAMBLE – Spanish Market

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Ariel Quitamanchas Blanqueador (powder)**	Sodium (nonanoyloxy)benzene sulphonate Sodium dodecylbenzenesulfonate Palmitic acid Pelargonic acid Dodecylbenzenesulfonic acid	C13-C15 pareth-n	Sodium carbonate peroxide TAED	Sodium acrylic acid/MA copolymer Polyethylene glycol Sodium polyacrylate	Sodium hydroxide	Sodium sulfate Sodium silicoaluminate Aqua Sodium carbonate Sodium chloride Cellulose gum Sodium metaborate Parfum Sodium silicate Disodium dityrylbiphenyl disulfonate Croscarmellose Magnesium sulfate Heteropolycycle [[(alkyl)oxy]-substitued alkyl] salt Calcium carbonate Sodium starch octenylsuccinate Microcrystalline cellulose Dextrin Hexyl cinnamal Sulfuric acid Cellulase Titanium dioxide Citric acid Colorant

RECKITT BENCKISER – Italian Market

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Vanish Oxy Action Gel**	Sodium C10-C14 alkyl benzenesulphonate	Laureth-7 Laureth-3 Alcohol C12-C18, EO/PO Alkylethoxylate 8EO C12-C15 pareth-5 Fatty alcohol 5EO	Hydrogen peroxide	-	Sodium hydroxide	Aqua Etidronic acid BHT Hexyl cinnamal Colorant

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Vanish Oxy Action Crystal White (powder)**	Sodium dodecylbenzenesulfonate	Pareth-5	Sodium percarbonate Tetraacetylethylendiamine	-	Sodium hydroxide	Sodium carbonate Sodium sulphate Sodium silicate Aqua Zeolite Carboxymethylcellulose Disodium distyrylbiphenyl disulphoante Protease Amylase

AC MARCA – Spanish Market

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Iberia Blanco Nuclear (gel)**	Sodium alkyl C12-C14 sulfate	-	Sodium hydrosulfite	-	Sodium hydroxide	Sodium sulfate Tetrasodium EDTA Sodium carbonate Cellulose gum Disodium dityrylbiphenyl disulfonate Sodium chloride Parfum Solum diatomeae Sodium glycolate Hexyl cinnamal Linalool Butylphenyl methylpropional

KH LLOREDA – KH-7

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **KH-7 Sin Manchas Oxy Effect (liquid)**	Sodium alkyl ether sulfate	Fatty ethoxylated alcohol	Hydrogen Peroxide	Polycarboxilate	Sodium hydroxide	Aqua Betaine Etidronic acid Sodium citrate BHT Fragrance

DELTA PRONATURA – DR. BECKMANN

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Dr. Beckmann Oxy Magic Quitamanchas Multiusos (foam)**	Sodium ethylhexylsulfate	Trideceth-3	Hydrogen peroxide	-	Sodium hydroxide	Aqua ethyl hexanol Parfum DTPMP Magnesium chloride Sodium gluconate Benzisothiazolinone Methylisothiazolinone Colorant

ECO BRANDS – ECOVER

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Ecover Laundry Bleach (powder)**	-	-	Sodium carbonate peroxide	-	-	Sodium carbonate

GENERIC BRANDS – CARREFOUR

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Carrefour Oxy Power Stain Remover (liquid)**	Sodium C10-C16 Alcohol ethoxylated sulfate	C12-C14 pareth-7	Hydrogene peroxide	-	Sodium hydroxide	Aqua 1-hydroxy ethylene-1,1-diphosphonic acid Parfum

10.4.STAIN REMOVERS

PROCTER & GAMBLE – Spanish Market

BRAND	SURFACTANTS		ACTIVE	POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC				
 **Ariel Quitamanchas Ropa Color y Blanca (liquid)**	Sodium dodecylbenzenesulfonate Dodecylbenzene sulfonic acid	C12-C14 pareth-7 C12-C14 pareth-3	Hydrogen peroxide	-	Sodium hydroxide	Aqua Sulfated ethoxylated hexamethylenediamine quaternized Disodium etidronate Parfum Disodium distyrylbiphenyl disulfonate Tetrabutyl ethylidinebisphenol Sulfuric acid Colorant

RECKITT BENCKISER – Italian Market

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **Vanish Power Shots (gel)**	Sodium C10-C14 alkyl benzenesulfonate MEA cocoate	Pareth-7	Polyvinyl alcohol	Sodium hydroxide Ethanolamine	Propylene glycol Glycerol Aqua MEA citrate DTPMP sodium salt Parfum Sodium sulphite Protease Amylase Hexyl cinnamal Benzisothiazolinone Citronellol CI 45100 Colorant

AC MARCA - IBERIA

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **Iberia Quitamanchas Diarias Roll-On (liquid)**	-	C9-C11 pareth-6 Lauryl polyglucose Capryly/capryl glucoside C12-C15 pareth-11	-	-	Aqua Dipropylene glycol Sodium citrate DTPMP sodium salt Benzisothiazolinone 2-bromo-2-nitropropane-1,3-diol Methylchloroisothiazolinone Methylisothiazolinone

DELTA PRONATURE – DR. BECKMANN

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **Prewash Quitamanchas con Cepillo Aplicador (liquid)**	Soap	C14-C15 pareth-8 Undeceth-5 Trideceth	PPG-2methylether	-	Aqua Alcohol Parfum Protease Limonene Ox Bile Citral MEK Methylchloroisothiazolinone Methylisothiazolinone

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **Stain Removal Roll-On (liquid)**	Soap Dodecylbenzenesulfonic acid with 1-amino-2-propanol Polyoxyethylene, lauryl ether, phosphate, potassium salt Dioctyl sodium sulfosuccinate	Trideceth C9-C15 pareth-8 Alcohols C10 branched, ethoxylated Alcohols, C12-C14 ethoxylated Alcohols, C8-C18 ethoxylated	-	Sodium hydroxide Potassium hydroxide	Aqua Propylene glycol Alcohol Dipropylene glycol Tetrasodium iminodisuccinate Parfum Citral Limonene Ox Bile MEK Methylchloroisothiazolinone Methylisothiazolinone

KH LLOREDA – KH-7

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **KH-7 Sin Manchas (liquid)**	Sodium alkyl ether sulfate Sodium alkylbenzene sulfate	Fattty ethoxylated alcohols	-	Sodium hydroxide Ethanolamine	Aqua Propylenglycol Phosphonate Calcium chloride Fragrance Subtilisin Alpha-amylase Lilial Amyl cinnamal Methylchloroisothiazolinone Methylisothiazolinone

ECO BRANDS – ECOVER

BRAND	SURFACTANTS – ANIONIC	SURFACTANTS – NON IONIC	POLYMERS	ALKALINE AGENTS	OTHERS
 **Ecover Stain Remover (liquid)**	Sodium laureth sulfate	Lauryl glucoside	-	Sodium hydroxide	Aqua Sodium chloride Alcohol Parfum Citric acid Subtilisin Linalool Methylisothiazolinone Benzisothiazolinone Cellulase

ECOLABEL BRANDS – L'ABRE VERT

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **L'Abre Vert Stain Remover (gel)**	Sodium laureth sulfate Sodium lauryl sulfate Cocamidopropylbetaine	Lauryl glucoside	-	Sodium hydroxide	Aqua Alcohol Sodium citrate Citric acid Amylases Proteases Sodium chloride

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **L'Abre Vert Stain Remover (spray)**	Sodium laureth sulfate Sodium lauryl sulfate Cocamidopropylbetaine	Caprylyl/capryl glucoside	-	Sodium hydroxide	Aqua Alcohol Sodium citrate Citric acid

GENERIC BRANDS – CARREFOUR

BRAND	SURFACTANTS		POLYMERS	ALKALINE AGENTS	OTHERS
	ANIONIC	NON IONIC			
 **Carrefour Detachant Avant Lavage (spray)**	Lauramine oxide	C12-C15 pareth-7 C6-C12 pareth-5	-	-	Aqua N-butane, isobutene, propane Isopropanol Isododecane Butoxypropanol Sodium benzoate Sodium nitrite Parfum Morpholine Subtilisin

TASK A.2.2.2 - Study of the different types of additives used in commercial detergents with the aim of improve the care of textiles during laundry process

1. Introduction

The aim of this report is to make a detailed analysis of the laundry additives directed to improve the fibre conservation.

To this end different type of additives are commonly used in laundry formulations. These new additives, most of them polymers, presumably should prevent the damage of fabrics and fibres, so providing protection to garments.

Repeating washing and drying of clothes puts strain on the fabric resulting in fibrillation and surface fibre damage, reducing the life cycle of a garment. This can often be identified as pilling of the fabric, "whitening" and fuzziness to the surface of our clothes and/or fibrillation of individual fibres when viewed under microscope. Damage to garments can also be identified as "cracking" of printed patters, resulting in garments looking worn and old.

It is important to mention that the following results and discussion was structured in two different parts. The first one contains the analysis of the properties of additives which are already used in commercially available detergents. The second part describes the characteristics of recently launched additives. It was divided according to the supplier.

2. Results and discussion

2.1. Laundry additives contained in commercial available detergents

Once the complete theoretical study of commercial laundry detergents was finished (deliverable A.2.2.1), the next step is further analyze the ingredients which are added in the detergent to enhance the care of textiles during the laundry process. Thus, it well worth to focus mainly on Light duty detergent category specifically designed to contribute to the reduction of fibre breakage and so, major fibre conservation.

The composition of each type of special detergent in Europe is very similar. In order to get a global vision about this composition it is well worth to include the following tables (Table 1: Liquid; Table 2: Powder, where the ingredients are classified according their family and type. Besides, the common range in which they appear is also described.

Table 16. Typical composition of liquid special detergents

Wool/Silk Laundry Detergent - Liquid		
Family	**Type**	**Range (%)**
Alkalinity sources		Low
Bleach precursors	Bleach activators	
Builders	Soap	1 – 5
Fragrances		0.2 – 0.5
Oxidising agents	Oxygen-based bleaching agents	
Sequestrants	Phosphonates	0.2 – 0.5
Surfactants	Anionic surfactants Non ionic surfactants	5 – 15 1 – 15

Table 2. Typical composition of powdered special detergents

Wool/Silk Laundry Detergent - Powder		
Family	**Type**	**Range (%)**
Alkalinity sources	Carbonate	Low
Builders	Zeolites Soap	15 – 30 1 – 5
Fragrances		0.2 – 0.5
Sequestrants	Phosphonates	0.2 – 0.5
Surfactants	Anionic surfactants Cationic surfactants Non ionic surfactants	5 – 15 0 – 5 1 – 15

In order to make a detailed analysis of ingredients contained in light duty detergents some European products have been selected. They are specified in the following table:

Table 3. Detergents selected to study its composition in detail.

Product	Name	Brand	Market
A	Perlan	Henkel	Spanish
B	Perlan Efecto Bálsamo	Henkel	Spanish
C	Micolor Fresh Gel	Henkel	Spanish
D	Micolor Colores Puros Gel	Henkel	Spanish
E	Norit Prendas Sintéticas	AC Marca	Spanish
F	Norit Cuidado Delicado Máquina	AC Marca	Spanish
G	Sole Lana e Delicati Detersivo Liquido	Reckitt Benckiser	Italian
H	Sole Lana e Delicati Tutticolori	Reckitt Benckiser	Italian
I	Lip Woolite Lana e Delicati (Gel caps)	Reckitt Benckiser	Italian
J	Woolite Classic	Reckitt Benckiser	Europe
K	Ecover Lana	Ecover	Europe
L	L'Abre Vert Lessive Lana et Textiles Delicats Ecologique	L'abre vert (Ecolabel)	Europe
M	Detergente Lana Carrefour	Carrefour	Europe
N	Bosque Verde Pieles Sensibles	Mercadona	Spanish
O	Bosque Verde Crema Prendas Delicadas	Mercadona	Spanish

To a better assessment of the selected detergents, their ingredients were divided in three groups:

- Surfactants
- Polymers
- Alkaline agents

The results are shown in the following graphics, where the different types of each ingredient used in the brands selected can be appreciated, as well as the frequency in which they are used. The y axis of all the graphics in this document represents the number of products in which appears each ingredient.

- Surfactants

To proceed with surfactants analysis they are classified in two groups: anionics and non ionics. In reference to the anionic surfactants (Figure 1) can be observed that all the selected light duty detergents comprises three different anionics in their composition. Related to the ones coming from industrial sources there is a trend in using ethoxylated sulphates mixed with benzensulfonates.

It is also noticed that in most of the products, with the exemption of Henkel products (A, B, C and D), fatty acids from natural sources such as oleate and cocoate were added (highlighted in blue).

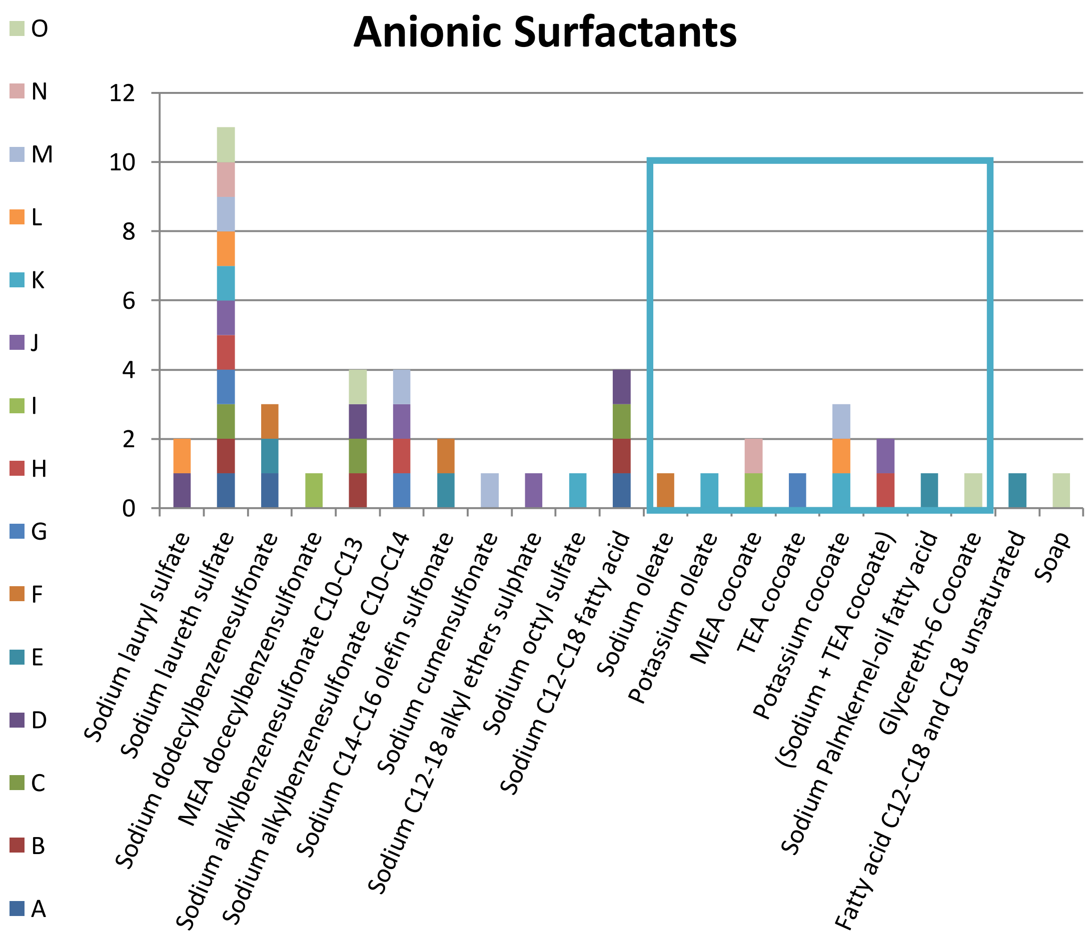


Figure 1. Anionic surfactants present in studied detergents.

In regard to non ionic surfactants (Figure 2) there aren't significant differences in the checked products, using in most cases one surfactant like a fatty alcohol among C12-C19 chains with 3-9 EO. Products K and L (Ecover and l'Abre vert, respectively), which are claimed as ecologic, include glucoside type surfactants, as well as product O (Bosque Verde) while some products from Reckitt Benckiser have cocoamide from natural resources.

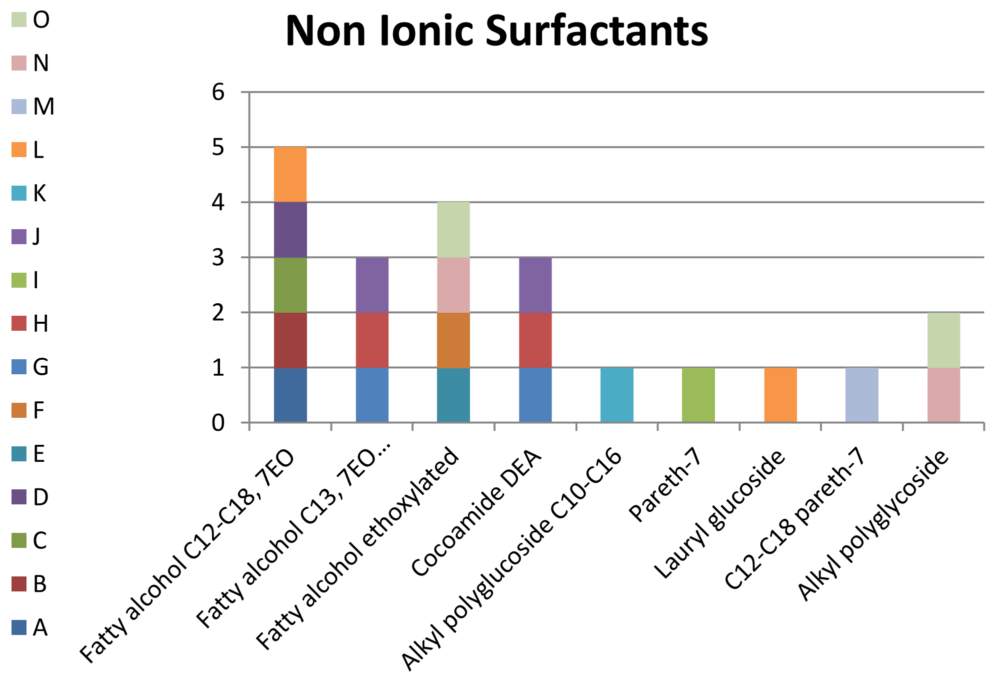


Figure 2. Non ionic surfactants present in studied detergents.

Polymers

In the case of polymers (Figure 3) it is easily appreciated that the most used is styrene/acrylates copolymer. As it was previously mentioned, in general the addition of polymers in laundry processes pursues the obtaining of extra benefits such as enhancement of the fabric care, dye transfer inhibition, minimize deposition...

Related to the selected detergents, the role of each one of the polymers showed in the graphic are specified below.

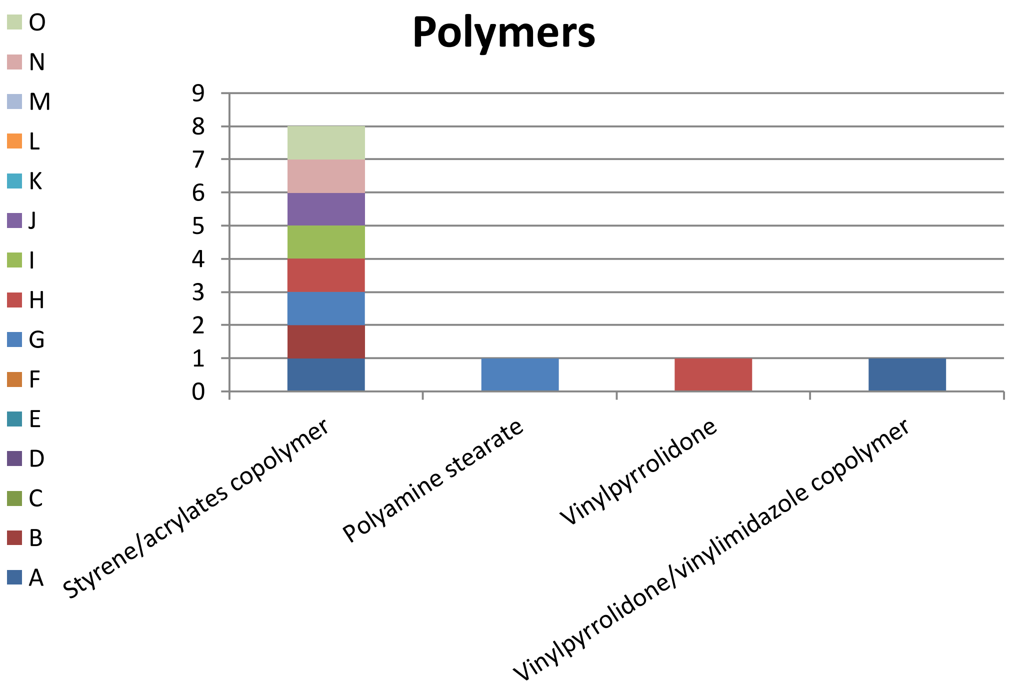


Figure 3. Polymers present in studied detergents.

- Styrene/acrylates copolymer

  It is widely used both in fabric care and in personal care. The main uses claimed for this polymer is opacifying and film forming.[3,4,5]

  The increasing number of products which contains styrene/acrylate copolymer is reflected in Mintel's data base (Figure 4). As it is shown in the graphic the number of launched products that have incorporate this polymer has increased from 1.8 to 2.6%.

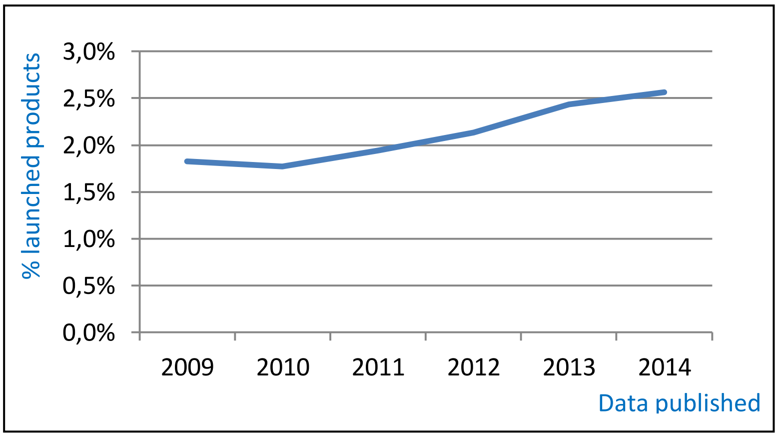


Figure 4. Percentage of launched products in fabric and personal care area containing styrene/acrylates copolymer

- Polyamine stereate

- Vinylpyrrolidone
  It acts as loose-color scavenger during laundering, improves emollient action and detergent storage stability, stabilizes foam, reduces irritation and in enzyme containing products it is used to coat and bind the enzyme thereby minimizing dusting[13].

- Vinylpyrrolidone/vinylimidazole copolymer
  The main characteristic of this copolymer is its ability to inhibit the transfer of dye from coloured to white textiles during the wash.[1,2]

To summarize, it can be concluded that an important improvement in the conservation of the fibre is due to the action of the polymers, some of which are described above. Thus, it could be interesting to further analyse this topic making a market study in order to look for new polymers/additives launched from the leading industries of raw materials with the "fabric care" claim.

2.2. New laundry additives

In this section it has been analyzed the portfolio of some of the principle industries of raw materials directed to laundry and care field applications. It has been selected among 1-3 products of each brand taking into account their properties in terms of protection of garment. Thus, the chosen products are indicated bellow and briefly described.

2.2.1.Lubrizol

The most appropriate compounds from Lubrizol correspond to a series named Merquat. Merquat family are polycationic polymers widely used in the personal care industry. Nowadays their incorporation in the laundry area has been extensively studied.

One of their components is Merquat 281, which is also called polyquaternium-22. Specifically, it is a copolymer of acrylic acid and diallyldimethylammonium chloride with antistatic and film forming properties. It is stable in a broad range of pH, from 2 to 11, and compatible with anionic components in pH values higher than 5.

2.2.2.Croda

Croda has a great number of products directed to the topic of fabric care. The presumably most interesting ones are described below.

- Cirrasol Expel
  It is a patent-pending additive designed to significantly boost stain removal in laundry products. Is really versatile, cold –processable and is fully compatible with anionic, nonionic and cationic detergent systems.
  Its effect on fabric has been evaluated using a solution of 15% of Cirrasol Expel with surfactant in water and compared to a benchmark commercial stain remover (Figure 6). It is important to mention that the recommended usage level in stain removers is 5-10%.

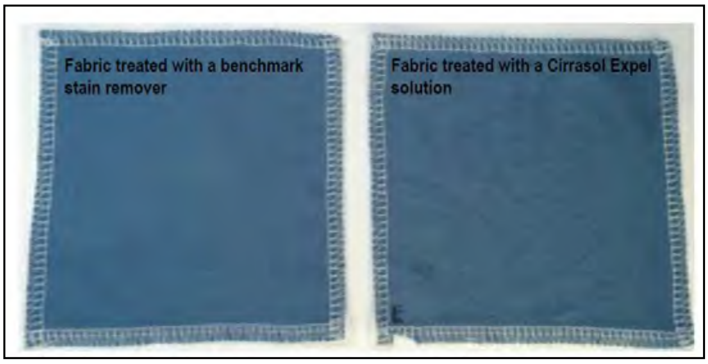


Figure 6. Fabric treated with a benchmark stain remover compared with fabric treated with a Cirrasol Expel solution. *(Source: Croda)*

- Coltilde Radiance
  It is a quaternised hydrolyzed wheat protein/silicone copolymer. It is mainly described as a new additive for fabric conditioners offering garments protection against colour fade, greying of whites and damage to fibres allowing fabrics to look radiant for longer. Specifically, it is interesting in our project for its properties of preservation and protection of fabrics and garments, reducing damage through the wash and reducing pilling. Therefore, it helps to reduce the visual signs of stress and results in a garment looking newer.
  By the way of example, Croda have made some interesting experiments in the laboratory, some of them are showed bellow.

- Visual inspection:
  As it can be observed in figure 7, the damage of the garments can be identified by consumers at a macroscopic level as it is shown in the printed pattern looking "cracked" and uneven. It is showed that after being washed 10 times the t-shirt where Coltide was used looks newer than the one washed without Coltide.

Figure 7. Printed t-shirts washed 10 times with and without Coltide Radiance.

(Source: Croda)

- Optical microscopy
  Samples of denim jeans have been observed until optical microscope to demonstrate the protective benefits of Coltide Radiance. As it is shown in Figure 8, a greater damage and whitening of the fabric is apparent from samples washed without Coltide Radiance compared to samples washed using 1% of this additive.

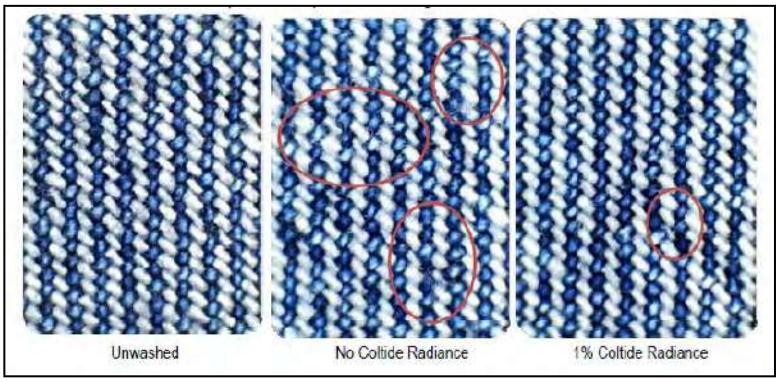


Figure 8. Fibre damage of cotton denim jeans after 10 washes with and without the use of Coltide Radiance. Fibre damage is highlighted and is compared to an unwashed garment. *(Source: Croda)*

- Scanning Electron Microscopy

  Fabric and fibre damage are highlighted further at a microscopic level when using Scanning Electron Microscopy (SEM). At this level of magnification, damage to the fabric is often identifies as uneven, rough and twisted fibre formations and frayed and loose fibre strands.

  Therefore, using this technique it has been analyzed some pillow protectors after they were washed 10 times with and without Coltide Radiance (Figure 9). The difference in their SEM images is clearly observed. The fibres pillow protectors washed with Coltide Radiance are similar to the unwashed samples. The fibres are still keeping their straight and uniform woven pattern, even after 10 washes. The fibres of pillow protectors washed without Coltide Radiance are on the other hand very uneven, frayed and loose indicating severe damage.

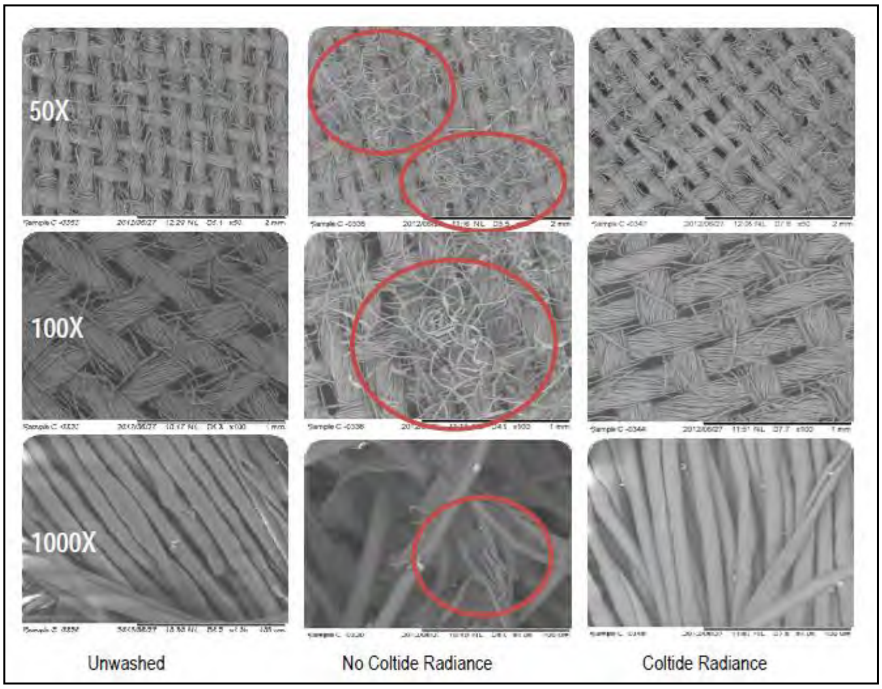


Figure 9. SEM images of pillow protectors washed 10 times with and without Coltide Radiance, compared to unwashed samples. *(Source: Croda)*

2.2.3.Basf

One the interesting candidates from BASF is Plantatex HCC, it is a wax crystal which builds a thin wax layer on the fibre, thereby protecting the fabric surface against wear and tear resulting from washing, tumble drying, and wearing. As it can shown in the following figure the textile shows a rejuvenated appearance over time compared with untreated textiles.

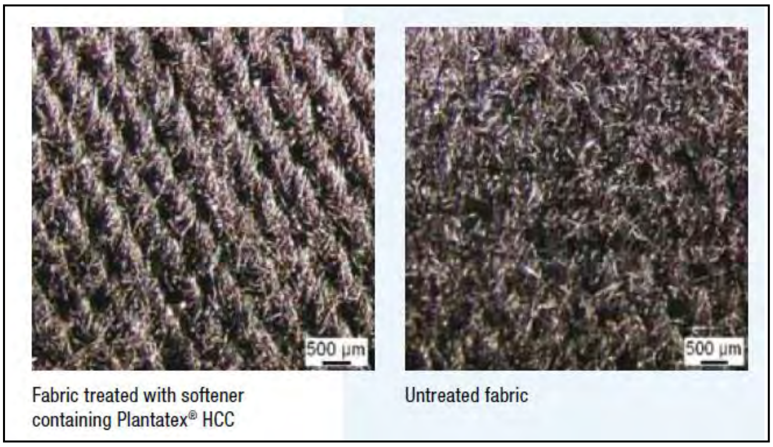


Figure 11. Test results after rubbing (200 cycles). *(Source: Basf)*

2.2.4.Wacker

Related to Wacker industries there is a family of special silicone additives named Wacker FC which could be interesting to our objective.

The active ingredients in a fabric softener reduce friction between the individual fibres and between fibres and metal in order to counteract undesirable consequences of the washing process; silicones can improve this effect thanks to their known significant lubricating and softening effects on textile fibres. The high flexibility of the Si-O-Si backbone of the silicone polymers and its functionalization results in their anchorage on the fibres (Figure 12). The resulting silicone loops has a high mobility which means that the treated fibres can easily sliced past each other reducing fibre/metal friction, too. In general, silicones can be anchored more efficiently to fibres when they are used together with quaternary ammonium compounds, which decrease production costs.

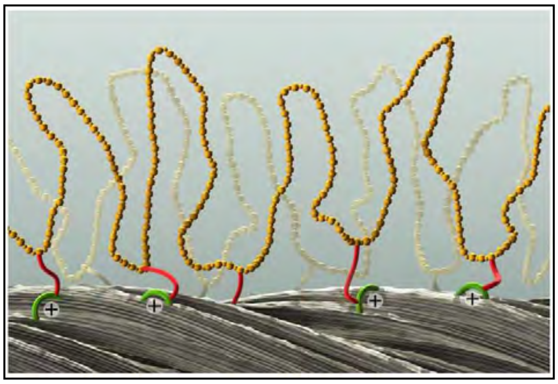


Figure 12. Silicone polymer anchored to fabric fibres forming loops. *(Source: Wacker)*

2.2.5.Clariant

The additive that could be interesting from Clariant is Nylostab S-EED. It is a new aromatic hindered amines light stabilizers which is claimed to lead a less filament breakage during spinning processes.

2.2.6.Rhodia/Solvay

Rhodia/Solvay has a family of compounds named Repel-O-Tex to boost cleaning and keep your fabric colours bright.
By increasing surface hydrophilicity, Repelotex reduces the interaction between synthetic fabric and greasily oil (Figure 13), so it allow a cold wash performance leading less fibre breakage.

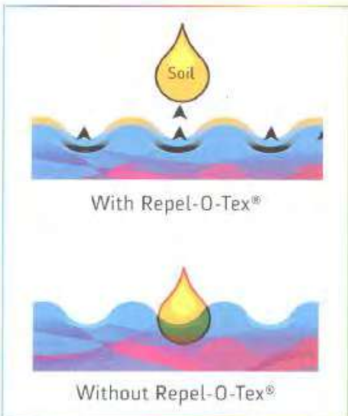


Figure 13. Repulsion effect between soil and fabrics produced by Repel-O-Tex. *(Source: Solvay)*

This family includes Repel-O-Tex crystal[14], which is designed for liquid detergents and both Repel-O-Tex SF2[15] and SRP6[16] for powder detergents.

3. Conclusions

This report is aimed to analyze how some laundry products in the market could increase or decrease the release of micro-fibres in water after a number of machine washings. Thereby, the influence of some ingredients directed to the fibre care is expected to be essential. Therefore, an extensively study of different type of additives has carried out considering both additives which are already in the commercially available detergents of the market and other novel launched ones.

The main properties of each one of the additives are basically directed to minimize friction between the individual fibres and between fibres and other materials. Therefore, the incorporation of the additives in laundry products should lead to a reduction of the breakage of the fibres and so, to a lower release of them in oceans.

Taking into account this theoretical information, different laundry washes will be carried out to verify that the properties of the additives leads into a lower plastic concentration in the waste water of the washing machine. The use of better additives should contribute to the mitigation of the impact caused by this micro and /or nanoplastics.

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12. http://www.rhodia.com/en/markets_and_products/product_finder/product_details.tcm?productCode=90022381&productName=REPEL-O-TEX+SRP+6
13. http://www.brenntagspecialties.com/en/downloads/Products/Multi_Market_Principals/Ashland/PVP_-_PVP_VA/PVP_Brochure.pdf

Product Information:

http://www.persan.es/informacion-de-productos/

http://www.henkelconsumerinfo.com/products/Ctrl?_h=henkel.products.ChangeLanguage&lang=en

http://www.rbeuroinfo.com/

http://www.arbrevert.fr/index.php/produits-ecologiques-pour-la-maison-l-arbre-vert/lessives-ecologique-l-arbre-vert.html

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TASK A.2.2.3 - Influence of several washing conditions on laundry effluent microplastics release. (Theoretical study)

1. Objectives

The aim of this study is to analyze the mean conditions that can influence in the release of textile micro-fibers to water during a machine washing. The conditions that can be evaluated (speed of washing machine, washing liquor, pH, temperature, time and water hardness) are related to Sinner Circle explained below.

2. Introduction[1]

According to Dr. Sinner (former R&D head Henkel) the cleaning effectiveness depends on four independent factors, which are represented in the so-called Sinner Model or Sinner Circle. Those factors are:

Chemical action (chemical energy): This represents the action of the dissolved detergent. The action is increased or decreased by the concentration of the detergent ingredients in the solution (water + product).

Mechanical action (mechanical energy): This is the mechanical action of the washing machine, which generates friction and pressure. For manual washing, when no equipment or devices are used, the person doing the cleaning is considered to provide mechanical action by rubbing, sometimes with the help of auxiliary devices.

Temperature effect (thermal energy): Heat is often used in cleaning activities. The elevated temperature enhances the chemical reactions, solubilises greasy soils and weakens the binding forces of the soil on the fabric.

Time: The duration of the cleaning operations determines how long the product is allowed to act. Combined with the mechanical, thermal and chemical action, the duration affects the cleaning power. Longer cleaning times will typically improve the cleaning performance.

Water was introduced by Stamminger as a fifth factor to show the importance of it as essential element in wet cleaning, since it acts as the agent for dissolving the detergents, for transportation of the detergent ingredients and heat to the laundry, for providing mechanical force on the soil particles via hydrodynamic flow resistance, for transporting the released soils away from the fibers and for diluting the detergent solution during the rising process.

The Sinner circle is represented graphically by a circle divided in four pies (five if the water is included), each representing one of the four independent factors. The size of these pieces represents the contribution of each factor to the overall cleaning result. The figure shows when one factor is reduced, at least one of the rest ones has to increase in order to maintain the surface of the circle and, therefore, the cleaning result. For instance, using short wash cycles at a low temperature requires a biggest input of chemical action, decreasing the mechanical action (Figure 25).

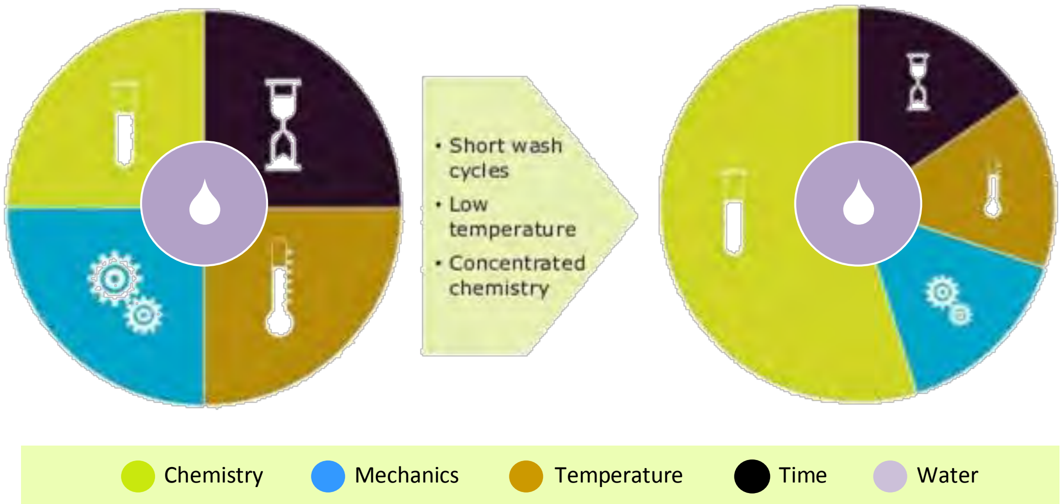


Figure 25. The Sinner Circle and its modification when specific conditions are chosen (Source: *Novozymes*)

3. Influence of Machine Washing Conditions in Fabric Care

All the conditions commented above can influence in the release of textile micro-fibers during the machine washing.

The **chemical action** is related to the detergent usage, so that the ingredients used in its elaboration could favor the loss of micro-fibers in fabrics. However, it is also possible to introduce additives such as polymers into the formula with the aim of prevent that effect or, at least, decrease it. In fact, there are already several polymers used in detergency which improve the whiteness maintenance of cotton and synthetic fibers, as well as specific classes of polymers that contribute to the overall laundry cleaning performance, for example by modifying the fabric surface by keeping soil suspended in solution to prevent redepositing on the fabrics. There are also certain enzymes also contribute to fabric care, for example by making fabrics softer[1]. Not only detergents have to be taken into account, but also fabric softeners can contribute to fabric care. The principal active ingredients in commercially available rinse-cycle softeners are usually cationic surfactants of the quaternary ammonium type. When applied in appropriate concentrations, cationic surfactants are adsorbed nearly quantitatively onto natural fibers, in contrast to their behavior with synthetic fibers. The additional benefits of fabric softeners are that they provide good antistatic properties on fabrics, make them easier to iron, reduce drying times and impart a pleasant fragrance to the laundry[2]. The addition of polymers into the softener formulation is a possibility to decrease fiber tearing; in fact, there are already biopolymers that can protect fibers from its damage during the washing cycle. The damage to garments can easily be identified at macroscopic level through the printed pattern looking “cracked” and uneven from the top of the Figure 26 , where any additive has been used. Less damage can be observed at the bottom of the image, where a biopolymer has been used as additive in a fabric conditioner[3].

No biopolymer

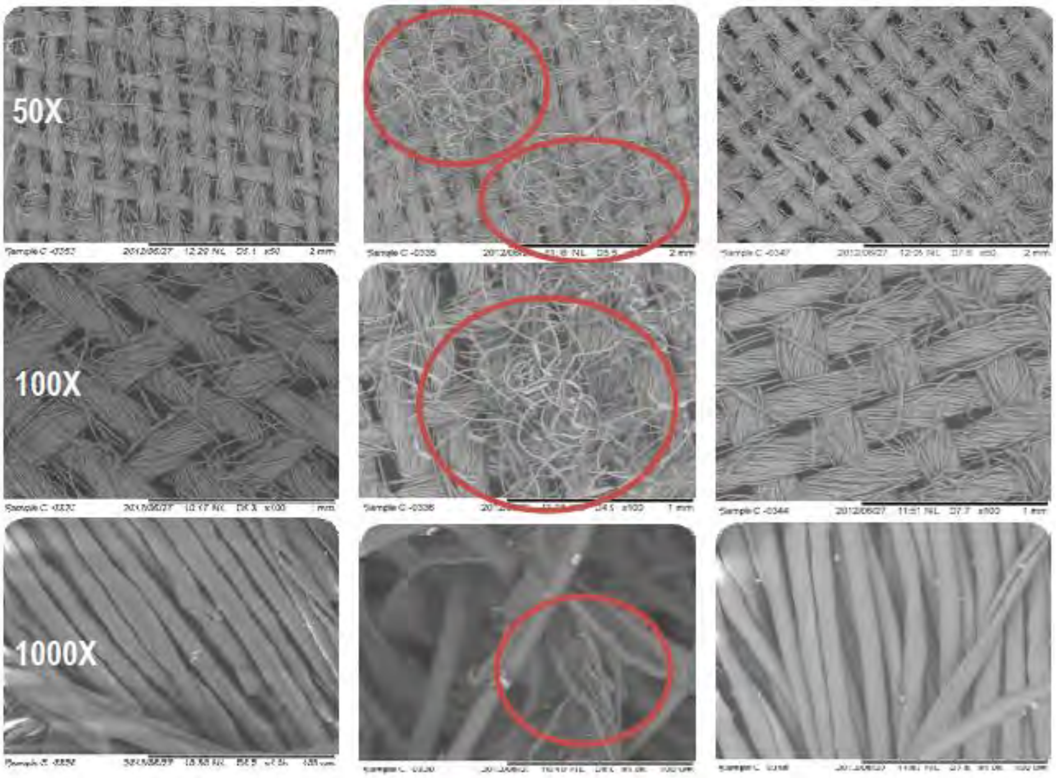
1% biopolymer

No biopolymer

1% biopolymer

Figure 26. Printed t-shirts washed 10 times with and without using a biopolymer as additive (*Source: Croda*)

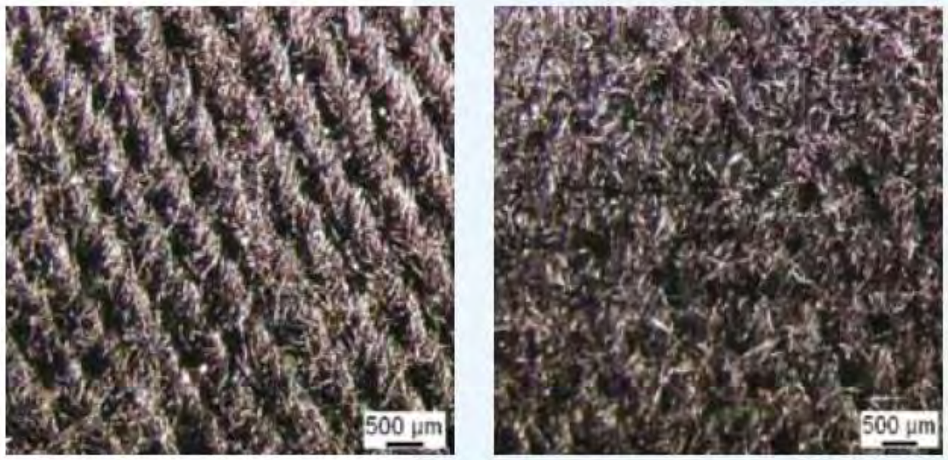
At microscopic level, the fiber tearing is appreciated in SEM images of pillow protectors from Figure 27, where fabric damage is more accentuated when no biopolymer is used during the washing cycle[3].



Unwashed | No biopolymer | Biopolymer

Figure 27. SEM images of pillow protectors washed 10 times with and without using a biopolymer as additive (*Source: Croda*)

Not only polymers are used as additives, but also wax crystals, which builds a thin wax layer on the fiber, thereby protecting the fabric surface against wear and tear resulting from washing, tumble drying and wearing. The textile shows a rejuvenated appearance over time compared with untreated textiles (Figure 28)[4].



With wax crystal Without wax crystal

Figure 28. Test results after rubbing (200 cycles) with an without wax crystal (Source: *BASF*)

Silicones can be also used as additives in fabric softeners. The active ingredients in a fabric softener reduce friction between the individual fibers and between fibers and metal in order to counteract undesirable consequences of the washing process; silicones can improve this effect thanks to their known significant lubricating and softening effects on textile fibers. Silicone polymers are anchored to the fibers forming loops with a high mobility, which means that treated fibers can easily slide past each other reducing fiber/metal friction, too. In general, silicones can be anchored more efficiently to fibers when they are used together with quaternary ammonium compounds, which decrease production costs[5].

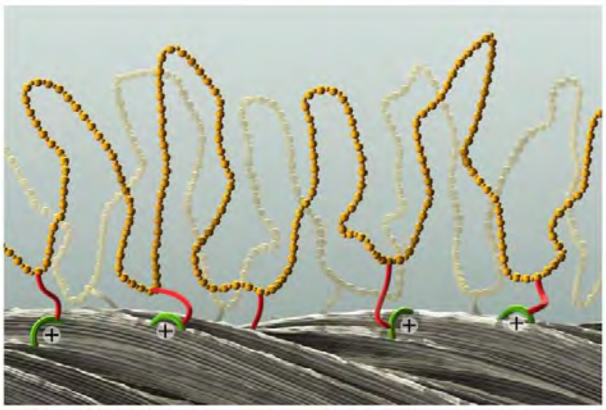


Figure 29. Silicone polymer anchored to fabric fibers forming loops (Source: *Wacker*)

Other factor to be taken into account is the **pH** of the detergents, which can also influence the release of micro-fibers, since overly acidic or alkaline water can damage elastics and delicate fabrics, and make clothes seem dingy[6].

It is often believed that high **mechanical action** is required for good cleaning result; however, high mechanical action also means a high likelihood of fabric damage, which can result in product loss[7]. There are two main types of washing machine according to their rotation mechanics: one type is top-loading with a lid on top and an upright basket and the other type is front-loading, with a door on the front and sideways-mounted basket. Top-loading washing machines use an agitator to move clothes around the basket. The **agitator** is a vertical device in the center of the basket with ridges that help push the clothes. The agitator alternates directions on that vertical axis. This movement creates the friction the top-loading machine needs to loosen dirt from fabrics. Front-loading washing machines use paddles that extend a short ways from the sides of the basket inward toward the center. The paddles help move the clothes and stir the water while the basket turns. Like the agitator in top-loading machines, the paddles loosen dirt from fabrics by creating friction[8].

Mechanical action factor is dependent on **time**, since the longer the washing time, the greater the amount of mechanical energy delivered to the laundry load, consequence of the contact between fabric items[9].

Better cleaning results are obtained at higher **temperature**; however, every day lower temperature detergents are used. This increasing tendency is due to the fact that washing at lower temperatures entails a substantial energy saving, which results in money saving. Also, there are some campaigns such as “I prefer 30º” from A.I.S.E. which encourage carrying out laundry at a temperature of 30 ºC as maximum because of environmental reasons. These initiatives are favorable to fabric care, since it has been demonstrated that washing at low temperature improves color appearance from fabrics[1]. Higher temperatures encourage the loss of dye and fade black and bright clothes by opening up the fibers[10]. However, according to Sinner Circle, a lower temperature supposes a longer washing time, resulting in bigger redeposition of the pigment soil causing bigger greying and the mechanical damage of textiles[11].

Water hardness is an important factor to be taken into account, since it is responsible of the reduction of cleaning effectives from the detergent. Calcium and magnesium ions cause water to be hard, which tend to bone to the anionic extreme of the anionic surfactants making them precipitate on fabrics making them stiff and harsh, besides giving them a faded color appearance. There is a linear relationship between concentration of detergent and water hardness at maximum soil removal efficiency, being necessary to use more detergent in order to obtain the same cleaning efficiency in hard water than in soft water[12]. This means that if the detergent contain any damaging ingredient for fabrics, the damage will be worse in hard water than in soft water, because more detergent will be use and, therefore, a higher amount of this ingredient will be in contact with fabrics. However, most of detergents contain sequestering agents, which capture the calcium and magnesium ions avoiding its binding with anionic surfactants. On the other hand, the usage of water softeners is also a solution to avoid using a bigger amount of detergent.

4. Conclusions

There are some factors to be taken into account when the loss of textile micro-fibers during machine laundry occurs. A high pH of the detergent can be aggressive to fibers, as well as some kind of ingredients of the formulation; however, some additives can be included in order to decrease the release of those fibers. The water hardness can be involved in this effect, since hard water requires a bigger amount of detergent to obtain the same cleaning result versus soft water. On the other hand, long periods of washing cause more friction between fabrics, what supposes more fiber tearing. Temperature is also another another factor to be observed, because worst damage in fabrics color has been demonstrated when clothes are washed at high temperatures in comparison with lower temperatures.
Therefore, the next steps to be followed are the experimental assays to analyze the real effect of these factors over the loss of micro-fibers during the machine washing, as well as to find additives that can fix fibers to clothes in order to minimize its release to water.

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Experimental Study

1. Description of the washing process

The washing process employed simulates the domestic washing, in which one cycle of the test simulates five domestic washing cycles. This test is based on the standard ISO 105 C06. The washing has been performed in a Lini-test equipment. The machine is composed of a water bath containing a routable shaft which supports steel containers. The shaft/container assembly is rotating at an established frequency and at a defined temperature. The machine is presented in the next picture:

Figure 30. Lini-test machine

2. Fabrics and laundry products used

In order to study the influence of different types of laundry products for washing machine in the micro-fibres release, representative commercial brands of heavy-duty detergents (HDD), and special detergents as light-duty detergents (LDD) have been tested. Softeners and Laundry additives as bleaches and oxy-products are studied, too. Products have been tested on three different types of fabrics.

The fabrics used to carry out the tests were:

- Woven Polyester 100% (ref. 777)
- Woven Poplypropylene 100% (ref. 983)
- Knitted Polyester, double knit jersey, disperse dyeable (ref. 720 WOB)

Pieces of each type of fabric were prepared taking into account the bath ratio employed for the test. Each piece of fabric was sewed on the sides with cotton thread in order to avoid interferences in the results caused by the release of microfibers from the thread.

The laundry products used are specified in the table below:

Table 17. Laundry products tested

Type of Product	Code	Dose* (ml/15L water)	Washing pH
HDD (powder)	Detergent Powder 1 (DP1)	115 (73 g)	10.7
HDD (liquid)	Detergent Liquid 1 (DL1)	37	7.7
HDD (liquid)	Detergent Liquid 2 (DL2)	100	8.1
Bleach (liquid)	Bleach Liquid 1 (BL1)	100	9.7
Bleach (liquid)	Bleach Liquid 2 (BL2)	100	10.8
Oxy-product (powder)	Oxy Powder 1 (OP1)	60 (65.78 g)	10.7
Oxy-product (liquid)	Oxy Liquid 1 (OL1)	85	5.2
Softener (liquid)	Softener Liquid 1 (SL1)	40	4.6
LDD (liquid)	Detergent Liquid 3 (DL3)	60	7.4
LDD (liquid)	Detergent Liquid 4 (DL4)	66	8.7

*Dose recommended by the manufacturer

3. Washing conditions

Some pre-trials were made in order to check and set the definitive domestic conditions in lini-test based on ISO 105-C06:2010 and to check all equipment and filtration tests.

After all this work the actual conditions have been:

- Temperature: 40 ºC
- Time: 45 minutes
- Number of steel balls: 10
- Medium: distilled water

Some assays have been carried out changing the washing conditions in order to observe changes in the microfibers release. The detergent used has been a commercial liquid detergent with a dose of 65 ml/15L water, which washing pH is 8.1. These conditions are specified in the table below.

Table 18. Changes in the washing conditions

Condition	Temperature (ºC)	Time (minutes)	Number Steel Balls	Medium
C0 (Standard)	40	45	10	Distilled water
C1	60	45	10	Distilled water
C2	40	45	0	Distilled water
C3	40	90	10	Distilled water
C4	40	45	10	Hard water (27 ºd)

4. Filtration

After the washing in the Lini-test, the samples were filtrated using Durapore® membrane filters from Merck Millipore with a pore diameter of 5 µm (ref. SVLP04700). Once the filtration was finished, the filters were exposed to 85 ºC during 30 minutes and, finally, analyzed with a Scanning Electron Microscope (SEM).

TASK A2.2.4. Characterization and quantification of micro and nanoplastic following washing in several conditions with different types of commercial laundry detergents, softeners, additives

5. Counting method

The counting procedure developed is based on the acquisition of several electron micrographs of the filter surfaces using a scanning electron microscope, SEM, FEI Quanta 200 FEG.

SEM observations were performed in low vacuum mode (PH_2O = 0.7 torr), using a large field detector (LFD) and an acceleration voltage of 5-20 kV. The observations were performed on the whole filter without applying metal coating.

Taking into account the geometry and the dimensions of the used filters, 21 SEM micrographs were acquired for each filter. Every micrograph represents a squared area (A_S) of the filter surface, equal to 7.8 mm^2 (see ***Figure 31***).

SEM micrographs were acquired along two orthogonal diameters of the circular filter (see ***Figure 32***).

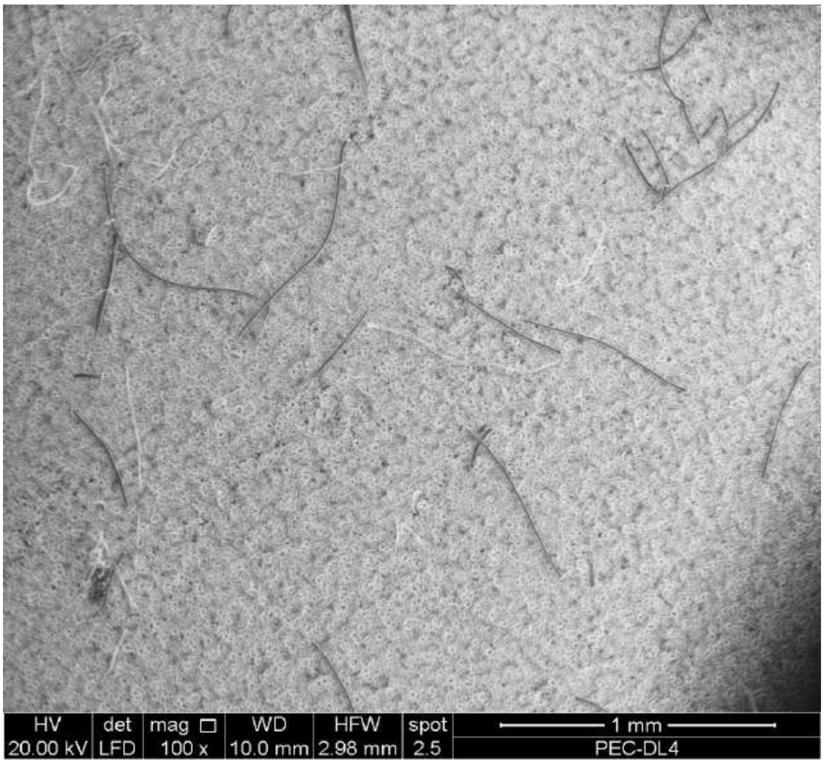


Figure 31. SEM micrograph of a squared area (A_S = 7.8 mm^2) of a filter containing PEC fibres

It was hypothesised that the fibre distribution is equal along all the traceable diagonals, thus 6 concentric circles were taken into account (see ***Figure 33***):

- The first circle is right in the centre of the filter and it circumscribes the central square;
- The other 20 squares are included, four by four, in 5 annuli.

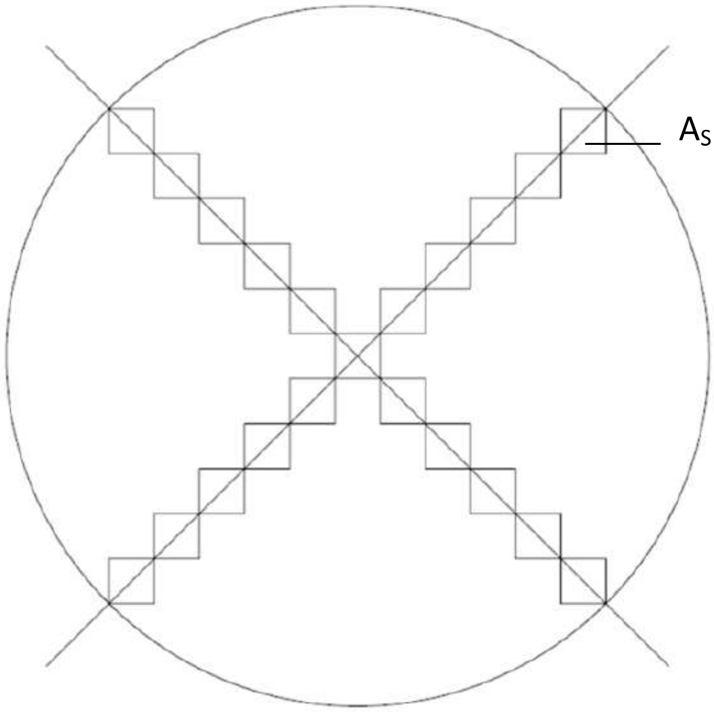


Figure 32. Position of the images along the filter

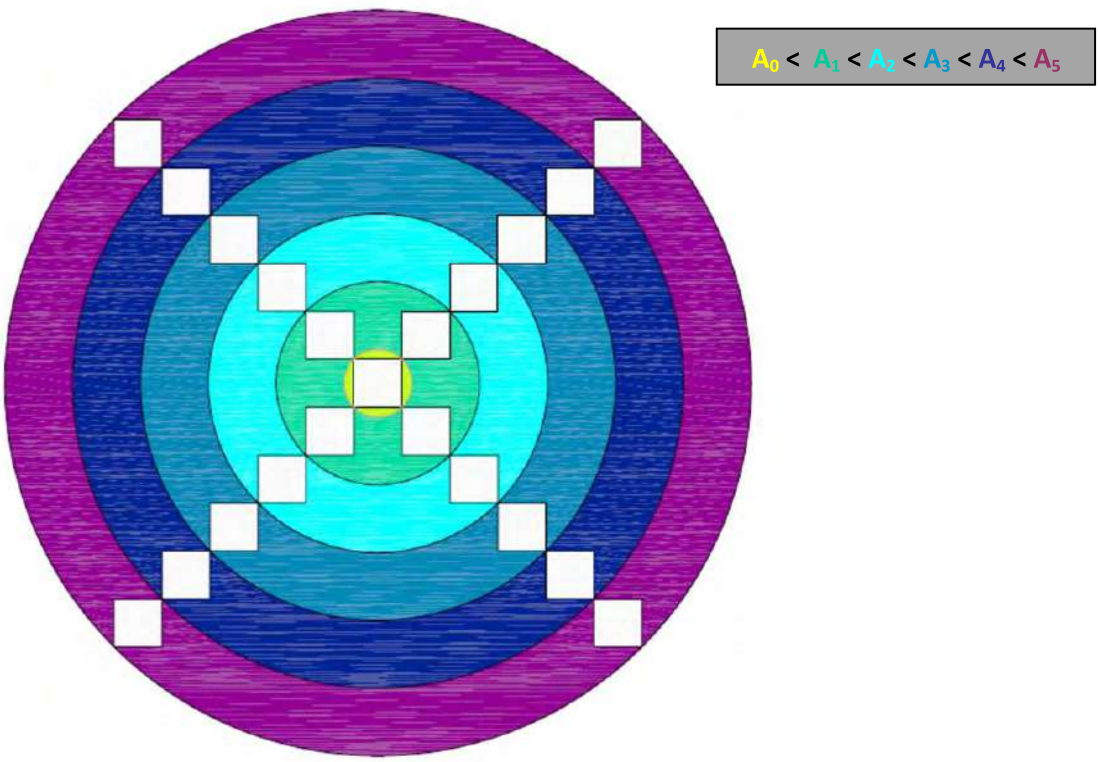


Figure 33. Concentric circles

The counting develops in 4 steps explained below:

1. Counting of the number of fibres in the first image in the centre of the filter (n_0) and division of this number for the area of the single square (A_S = 7.8 mm^2). In this way it is possible to obtain the number of fibres per area ($n_0/A_s = C_0$). Considering the area of the circle (A_0) that circumscribes the central square and multiplying this value for C_0, the number of fibres contained in the first circle is easily obtained ($N_0 = C_0 \times A_0$).

2. In the first annulus, the number of fibres is counted in each of the four squares (n_1, n_2, n_3, n_4) and an average value is calculated $\left(\frac{n_1+n_2+n_3+n_4}{4} = n_A\right)$. As done in the previous step, this value is divided for A_S ($n_A/A_S = C_1$) and multiplied for the area of the considered annulus (A_1), obtaining the number of fibres contained in the first annulus ($C_1 \times A_1 = N_1$).

3. The previous step is repeated for the other 5 annuli, obtaining N_2, N_3, N_4, N_5.

4. Finally N_0, N_1, N_2, N_3, N_4 and N_5 are added together giving the total number of fibres on the filter surface.

6. Results

The results of this method are reported in ***Table 19***. In the collected micrographs, besides the synthetic fibres, additional cotton fibres were detected (see ***Figure 34***). Such fibres belong to the cotton thread used to sew the sides of the piece of fabric washed in the Lini-test machine. Of course these fibres were excluded during the counting procedure.
The numbers of fibres *per* filter in red (see ***Table 19***) are affected by a high error rate due to the thick layer of the detergent powder visible to the naked eye on the filter. Such layer creates an unclear background that makes difficult to recognise the fibres and moreover it can trap them partially, as depicted in ***Figure 35*** (red circle), or maybe completely.

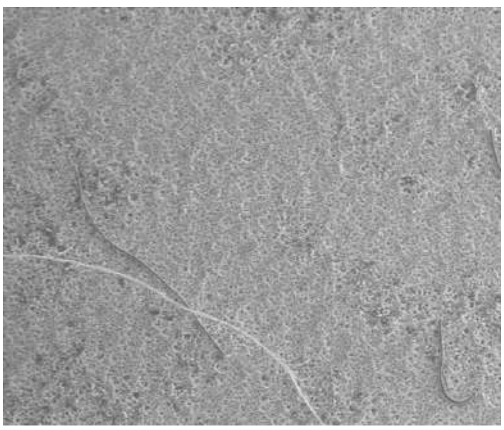


Figure 34. Magnification of a SEM micrograph showing a cotton fibre (white)

Table 19. Counting results

Sample	N° fibres/filter	Sample	N° fibres/filter	Sample	N° fibres/filter	TOTAL (by product)
PEC R1	123	**PEP R1**	100	**PP R1**	187	**410**
PEC DL1	1833	**PEP DL1**	2429	**PP DL1**	1293	**5555**
PEC DL2	1304	**PEP DL2**	199	**PP DL2**	2040	**3543**
PEC DL3	692	**PEP DL3**	658	**PP DL3**	610	**1960**
PEC DL4	1279	**PEP DL4**	216	**PP DL4**	446	**1941**
PEC BL1	513	**PEP BL1**	152	**PP BL1**	994	**1659**
PEC BL2	1520	**PEP BL2**	388	**PC BL2**	891	**2799**
PEC OL1	1319	**PEP OL1**	174	**PP OL1**	688	**2181**
PEC SL1	674	**PEP SL1**	177	**PP SL1**	217	**1068**
PEC OP1	3239	**PEP OP1**	4281	**PP OP1**	2485	**10005**
PEC DP1	2350	**PEP DP1**	2197	**PP DP1**	1340	**5887**
TOTAL (by textile)	**14846**		**10971**		**11191**	
PEC C0	1033					
PEC C1	1389					
PEC C2	1183					
PEC C3	1018					
PEC C4	1516					

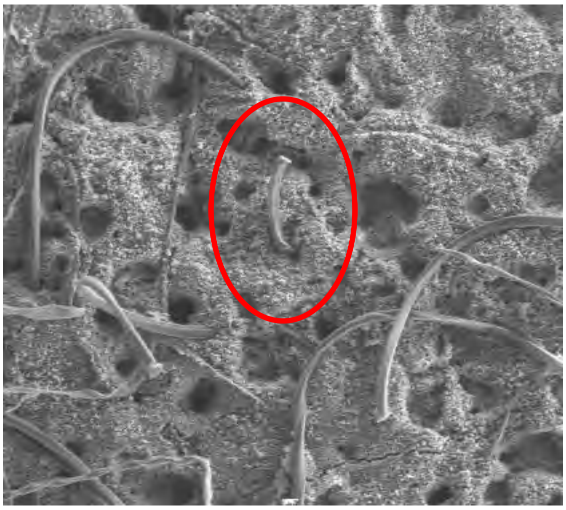


Figure 35. SEM micrograph of a magnification of a filter containing fibres coming from a knitted polyester textile (PEP) washed with Detergent Powder 1 (DP1)

In order to corroborate some of the results obtained, a second replicate of the samples PEC_DL3, PEC_BL1, PEC_DP1, PEC_C0 was analyzed.

The results are shown in the table below along with the results obtained in the previous counting of the corresponding replicates.

Table 20. Counting results

	N° of fibers/filter		
Sample	**Replicate 1**	**Replicate 2**	**Average**
PEC_DL3	1299	692	995
PEC_BL1	513	812	662
PEC_DP1	2350	3215	2782
PEC_C0	1147	1033	1090

The number of fibers highlighted in red is very approximate since, as clearly visible in *Figure 36*, there is an area of the filter with a great amount of fibers that are quite difficult to count properly. This cluster of fibers is also visible to the naked eye.

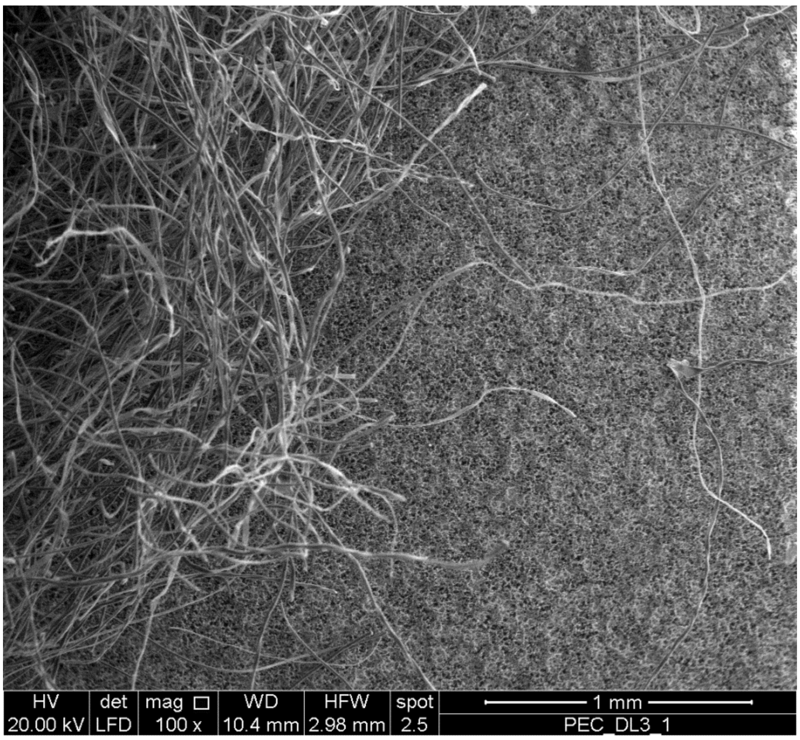


Figure 36. SEM micrograph of part of the area of the filter PEC_DL3 Replicate 1

Concerning the sample PEC_DP1, as already mentioned in the report of the Task A2.2.4 also for the Replicate 2, the counting result is affected by an high error rate due to the thick layer of the detergent powder visible to the naked eye on the filter (see *Figure 37*). Such layer creates an unclear background that makes difficult to recognise the fibres and moreover it can trap them partially or completely.

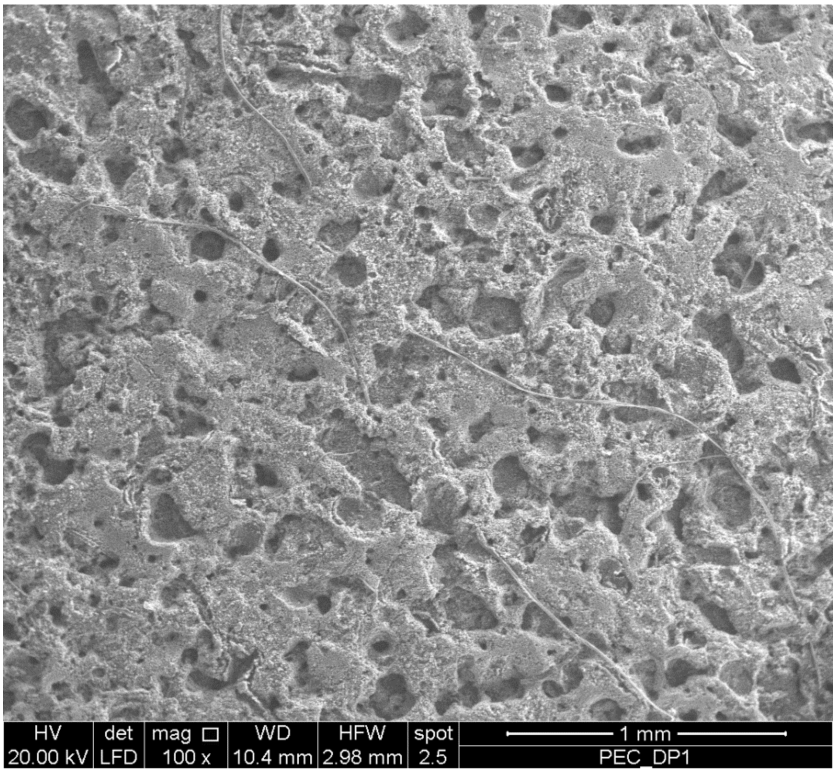


Figure 37. SEM micrograph of part of the area of the filter PEC_DP1 Replicate 2

Legend:

TEXTILES

PEC: Woven polyester

PEP: Knitted polyester

PP: Woven polypropylene

DETERGENTS

R: Reference (only distilled water)

DL1: Detergent Liquid 1

DL2: Detergent Liquid 2

DL3: Special detergent Liquid 3 -> product for wool

DL4: Special detergent Liquid 4 -> product for synthetic textiles

BL1: Bleach Liquid 1

BL2: Bleach Liquid 2

OL1: Oxy Liquid 1

SL1: Softener Liquid 1

OP1: Oxy Powder 1 (percarbonate powder standard laundry aid product)

DP1: Detergent Powder 1 (standard powder detergent)

WASHING CONDITIONS

C0: liquid detergent product, 40 ºC, 10 balls, program 45 minutes, soft water (distilled water)

C1: (change in temperature) liquid detergent product, 60 ºC, 10 balls, program 45 minutes, soft water (distilled water)

C2: (change in mechanical action) liquid detergent product, 45 ºC, 0 balls, program 45 minutes, soft water (distilled water)

C3: (change in time) liquid detergent product 40 ºC, 10 balls, program 90 minutes, soft water (distilled water)

C4: (change in water hardness) liquid detergent product 40 ºC, 10 balls, program 45 minutes, hard water (27ºd)

After obtaining these first results, few more trials were carried out using, in the one hand, extreme conditions of temperature (90 ºC, PEC_C5) and an increase in mechanical action (20 steel balls, PEC_C6) in order to confirm their influence on the release of microfibers, and on the other hand, a detergent with a neutral pH (DL3) with an increase of pH to 10 (DL3-pH10) in order to check if it produces a higher microfiber rupture. The results of counting the microfibers of these samples are shown in the table below.

Table 21. Counting results

	N° of fibers/filter		
Sample	**Replicate 1**	**Replicate 2**	**Average**
PEC_C5	783	951	867
PEC_C6	1449	1734	1591
PEC_DL3-pH10	471	947	709

The numbers of fibers highlighted in red are very approximate since, as visible in *Figure 38*, probably as effect of the harder mechanical action, there are a lot of little fiber fragments whose nature is not of straightforward attribution: actually, it is not possible to distinguish between plastic and cotton fibers from the threat used to sew the sides of each piece of fabrics. The reported numbers refer to the only distinguishable ones.

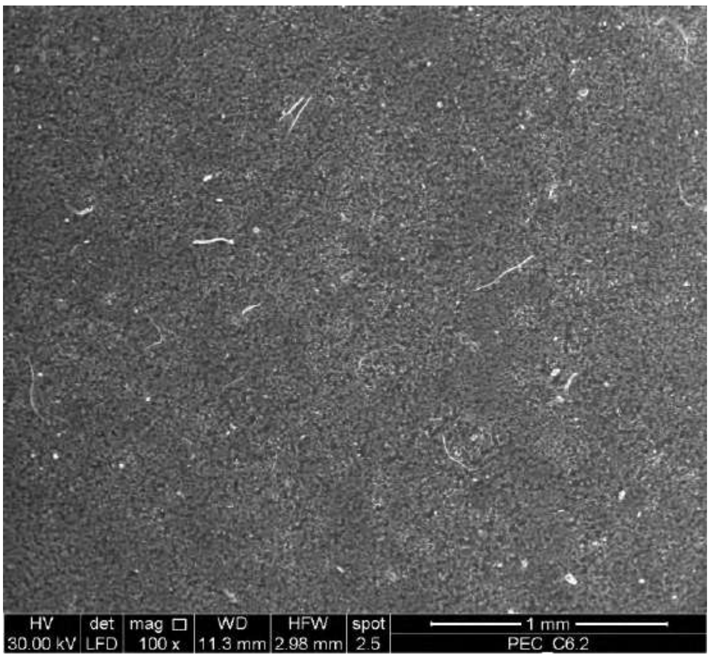


Figure 38. SEM micrograph of the filter PEC_C6 (Replicate 2)

Legend:

TEXTILES

PEC: Woven polyester

PEP: Knitted polyester

PP: Woven polypropylene

DETERGENTS

DL3-pH 10: Special detergent Liquid 3 (product for wool) which pH has been increased from 7.5 to 10.5

WASHING CONDITIONS

C5: (change in temperature) liquid detergent product, 90 ºC, 10 balls, program 45 minutes, soft water (distilled water)

C6: (change in mechanical action) liquid detergent product, 40 ºC, 20 balls, program 45 minutes, soft water (distilled water)

7. Conclusions

Several trends in the micro-fibers release have been observed after analysing the filters through the SEM. Once the results have been obtained and studied, some tendencies can be drawn:

- Powder products seem to favor the micro-fiber release, which can be caused by the friction of the solid particles with the fabrics. Indeed, the small particles of powder detergents would penetrate into the fabric's structure and could be responsible of increasing the friction between the fibres and then their propensity to protrude, increase their height and form pilling balls or knots on the fabric surface. It has been demonstrated in the deliverable "Report on the influence of commercial textile finishing, fabrics geometry and washing conditions on microplastics release" that those knots contains microfibers which could be eliminated during washing.
- A higher pH of the washing liquor can be aggressive to fibers in several cases, a trend shown by the results.
- Products containing powder oxidizing agents (percarbonate) present a higher fiber release, but we can´t conclude whether the effect is due only to the oxidizing agent or because the combination of powder product and alkaline pH in the same formulation.
- The lower fiber release results have been obtained in the samples where a softener was used. This result confirms the hypothesis of the project related to the positive action of softening agents (from textile finishing or detergents) on the fabric. Softening agent reduce the friction between the fibres and decrease their probability to break.

- The special detergents (for delicates and synthetic fabrics) studied, presented lower fiber release than other commercial liquid detergents.
- Regarding laundry aids, the oxy liquid product analysed, based on hydrogen peroxide releases fewer fibers than the oxy powder product studied with a percarbonate formulation and higher pH value.
- In general, the knitted polyester fabric (referenced here as PEP) releases fewer fibers than woven polyester fabric (referenced here as PEC). This observation confirms the results obtained in the deliverable "Report on the influence of commercial textile finishing, fabrics geometry and washing conditions on microplastics release" from preparatory action A2. Indeed, it has been seen in this report that the knitted polyester fabric (referenced here as PEP) is made of continuous fibres, with low probability to form microfibers. Moreover, woven polyester fabric (referenced here as PEC) were in many case the most prone to form microfibers according to its surface hairiness analysis (before and after washing in water only) and due to its low twist of weft yarns, high linear density and relatively elevated weight (mass per unit area).
- Woven Polypropylene seems to be less critical than woven polyester. That is due to the fact that PP is made of continuous fibers while PEC yarn is made of short staple fibers.
- Related to washing conditions, higher temperature and higher water hardness seem to have a negative impact on fiber breakage, as expected.

All trends identified in the preparatory action A2 will be the basis for studying the best possibilities related to additives and washing conditions in actions B2 and B3.

Name of the deliverable:

Report on the influence of commercial textile finishing, fabrics geometry and washing conditions on microplastics release

Number of the associated action: A2

Involved Partners: LEITAT - POLYSISTEC

(20/03/2015)

LIFE13 ENV/IT/001069

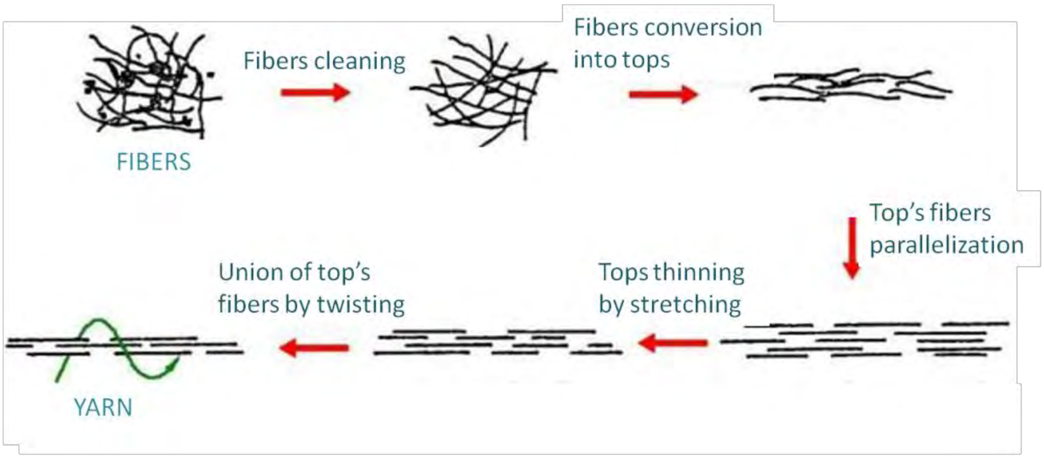
Mitigation of microplastics impact caused by textile washing processes

Contents

TASK A.2.1.1. Study on the influence of fibres and fabrics properties in relation with fibres loss.

1. Introduction

The spinning industrial process during which the fibres are transformed into a yarn is a complex process due to the various steps which are involved from the selection of the raw material to the obtaining of the final product. The industrial spinning processes can vary depending on the type of fibres used. It can be divided into cotton spinning process and wool spinning process, as different techniques are employed for each. However, there are common and general steps for each spinning processes which include the cleaning of the material, conversion into tops, fibres parallelisation, fibres stretching and fibres twisting.



The fibres breakage capacity of textile products depends on various factors which can be their composition, their spinning and weaving/knitting process and the different mechanical treatments which have been applied on the fabric during and after his production.

The aim of this deliverable is to determine the different characteristics of the fabrics which could enable the microplastics formation and the different textile process which could be responsible of creating microfibers breakage.

As a result, this report will give an overview of the fabrics properties and production process conditions, responsible of the formation of microplastics.

2. Description of the physical and mechanical properties of fibers influencing the fibers breakage performance

In order to identify the cause of the formation of microfibers on ready made garments, all the processes from the textile industry have to be studied. The tendency of a fabric to form microfibers is conditioned by the production steps involved in the textile processing, from the spinning process to the weaving and the mechanical finishing process.

The influence of the spinning process

Different spinning processes can be used to produce a yarn (conventional spinning processes such as rotor spinning, ring spinning and compaction spinning among others non conventional spinning methods). The spinning parameters can influence the final appearance of the yarn and so its final properties. The yarn twist is one of the main process conditions which can affect the formation of protruding microfibers in the fabric surface after a certain period of use. Indeed, the higher the twist value the tighter the fibres are catch into the yarn structure. With low yarn twist, the fibres are free and can easily untie themselves from the rest of the yarn structure. As it can be seen on the next picture, the stretch of the yarn is also conditioned by its twist value.

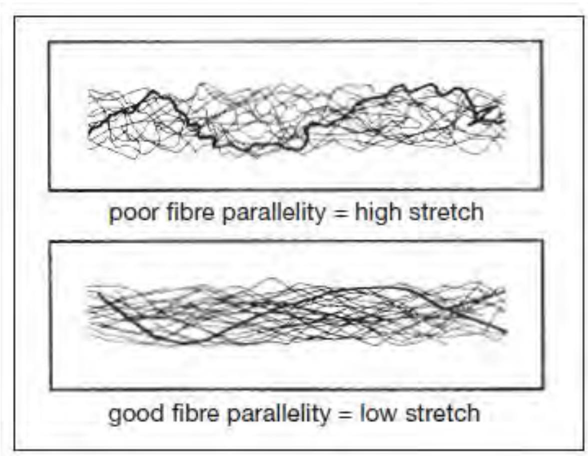


Figure 1. Effect of yarn twist on the fibres layout inside a yarn. [1]

The spinning processes which can be used in the textile industry and on which will depend the formation of protruding microfibers on the fabric surface are various. However, the study of the principal spinning processes like the rotor, ring and compaction spinning (a type of ring spinning) will give us an idea of how their operating conditions could increase the microfibers formation.

[1] Prof. Dr. rer. nat. Hans-Karl Rouette, Encyclopedia of Textile Finishing, Chapter R, page 115.

Description of the ring spinning process

The ring spinning process is the most widely used spinning process in the textile industry. It can be employed for a wide range a fibres type, for short fibres such as cotton, long fibres such as wool and of any composition. The type of yarn obtained from this spinning process is uniform, of good quality and of different yarn count. The ring spinning process is known to produce the strongest and finest yarns. The highest tensile strength of the yarn is due to the roving and winding steps during which high twisting value and cohesion between the fibres is obtained. The following picture presents a possible structure of yarn obtained from ring spinning. Others tightest structures are possible as the twisting value can be adjusted.[2, 3, 4]

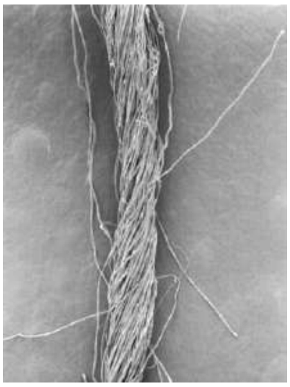


Figure 2. Representation of a yarn obtained from the ring spinning process[3].

The following image describes a conventional ring spinning process:

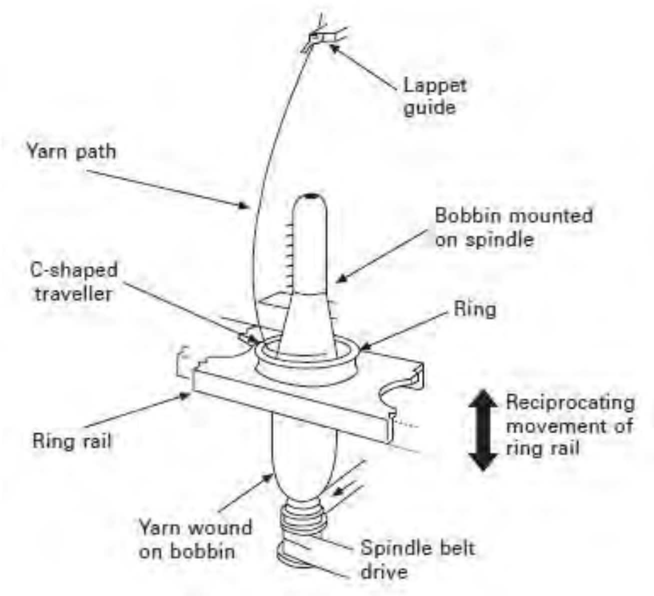


Figure 3. Representation of the ring spinning process[4]

[2] F. E. Lockuán Lavado, La industria textil y su control de calidad, Chapter III Hilandería.

[3] Cotton Guide, Chapter 2, Textile Processing, Yarn formation,

Description of the rotor spinning process

The rotor spinning is the second widely used spinning process in the textile industry. It's a shorter process than the ring one and for which the roving frame and the winder has been eliminated. Indeed, during this process the top of fibres is introduced into the comb wheel and lead to the rotor, where the combed fibres will be converted directly into a yarn. This spinning process is composed of less machine's element but will produce yarns of medium to high linear density. A wide range of fibres type can be employed with this technique, such as short fibres (cotton type) and long fibres (woolen type) and also synthetic and artificial fibres. The advantages of this spinning process resulting from the simplification of the machine resides in the lower cost of the yarn obtained. However, thin yarn can't be obtained and their resulting structure present fibres oriented in different directions (see next picture). [2, 3, 4]

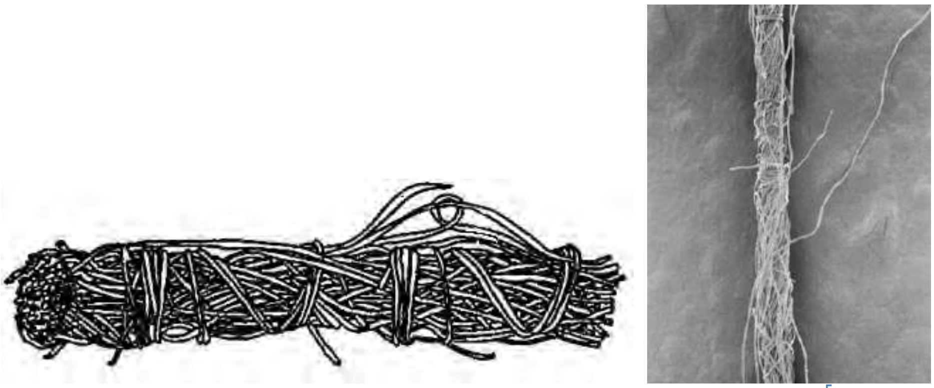


Figure 4. Resulting irregular structure of a yarn obtained by rotor spinning[5].

As it can be seen on this previous picture, the yarn obtained from the rotor spinning process is made of fibres which are tightly catch into the yarn structure and could be lost after mechanical action (during use) and after washing of the resulting garment.

The following picture describes the rotor spinning process:

[4] An Overview of Developments in Yarn Spinning Technology, textilelearner.blogspot.com.es.

[5] F. E. Lockuán Lavado, La industria textil y su control de calidad, Chapter III Hilandería, p. 40 of 272, 2012.

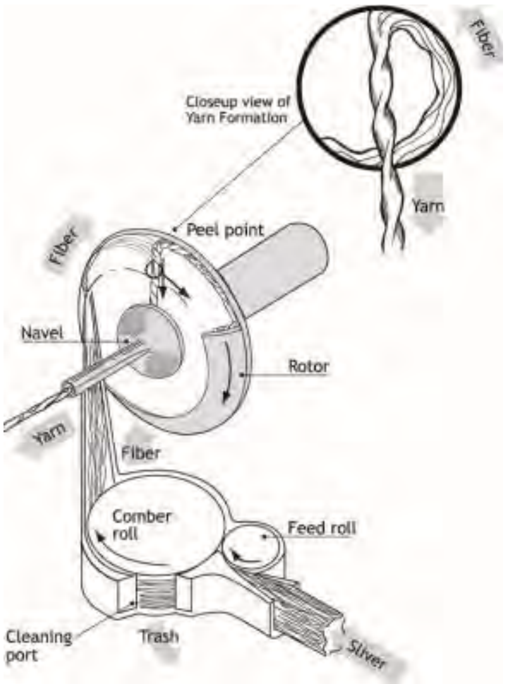


Figure 5. Representation of the rotor spinning process3.

Description of the compaction spinning process

The compaction spinning is a type a ring spinning in which a compaction step of the yarn has been added. The compaction is employed to reduce the size of the triangle which is formed when the top is converted into the yarn. The resulting yarn will have less hairiness and will be more resistant.

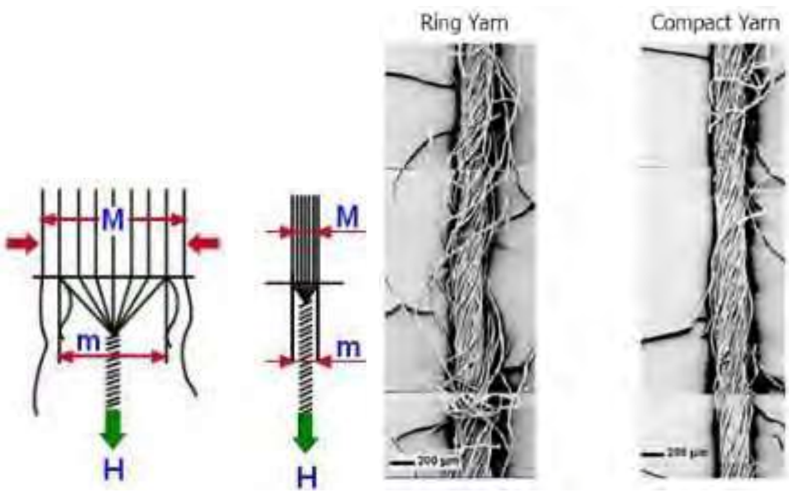


Figure 6. Description of a yarn obtained from a ring process modified with a compaction unit[6].

[6] Zinse Saurer Group.

The next picture presents a yarn obtained from the ring process (top) and from a ring process modified with a compaction unit (below). As it can be seen on this picture, the hairiness is considerably reduced after compaction. The compaction process is then a good option for the reduction of microfibers formation.

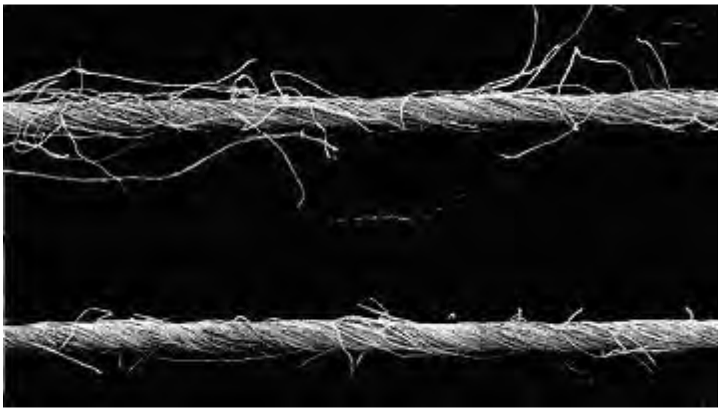


Figure 7. Top: yarn obtained from a ring spinning process. Below: yarn obtained from a ring spinning process with compaction unit.[7]

Description of the extrusion process

The extrusion process is the technique used to produce filaments from polymeric materials. For the polymeric process, the term filaments refers to the polymeric fibres from which the yarn is prepared. The process consists in melting a polymeric material (polyester, polypropylene, polyamide, etc.) available in the form of chips into a continuous filament which is extruded through various spinnerets. The number of spinnerets will defined the number of filaments which will compose the yarn. After cooling, continuous manufactured filaments are obtained and can be then cut into small pieces for the rotor or ring or other spinning process, or can be maintained continuous to produce a yarn. The filaments are then cut into small pieces of determined size defined by the fibres length required for each type of spinning process. Those cut fibres will be then employed alone or blended with others synthetic or natural fibres to form a short fibres yarn. A twisting unit is present at the end of the extrusion process to give torsion to the final yarn.

The following picture is a simplified scheme of the extrusion process:

[7] F. E. Lockuán Lavado, La industria textil y su control de calidad, Chapter III Hilandería, p. 39 of 272, 2012.

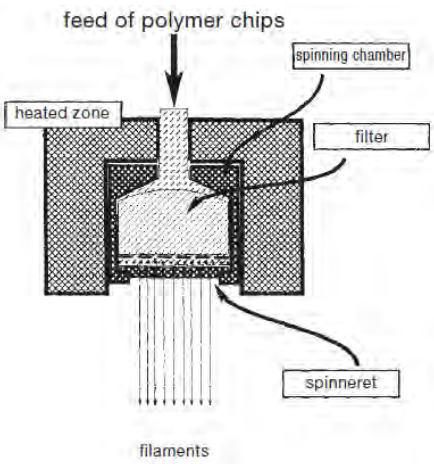


Figure 8. Simplified representation of the spinning process for yarn production[8].

Comparison between the rotor and the ring spinning process

The fabrics made from the rotor and ring spinning processes will present different characteristics. It has been observed that the rotor spinning process will present less **yarn irregularities** than the ring spinning process. This will induce fewer irregularities on the fabric made from these yarns as well and so less probability to observe microfibers formation and liberation. Moreover, the rotor spinning process will give a higher **extension coefficient** to the yarn and a more favourable **winding tension**. This means that the final yarn will have a **good parallelism of its fibres** and so less probability of protruding fibres. The pilling behaviour of a fabric is determined by the capacity of a rubbing material to extract fibres ends from its surface. The **pilling resistance** of rotor spun yarns is usually better than the pilling resistance of ring spun yarns. **Hairiness** is the main characteristic of fabric responsible of the loss of microfibers during domestic washings. Hairiness is defined as the projection of free floating fibres ends or loops from the surface of the fabric (see the next figure). Hairiness occurs during the spinning process when fibres migrate outward and when low twist is applied, during the weaving and the knitting processes, during finishing and during handling.

[8] Prof. Dr. rer. nat. Hans-Karl Rouette, Encyclopedia of Textile Finishing, Chapter H, page 51.

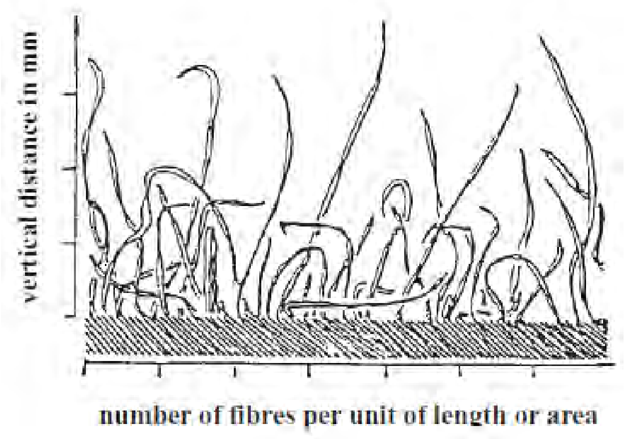


Figure 9. Illustration of fabric hairiness. [9]

It has been demonstrated that the rotor spinning process produces less hairiness on the fabric surface than the ring spinning process (between 40 and 80% less). Indeed, high **spindle speed** of the ring during spinning, low twist, high **spindle roughness** and **traveler surface** are responsible of increasing the hairiness in the ring and rotor spinning processes. Others factors influencing the hairiness and independent from the spinning process are the **fibres length** and the **yarn count**. A high quantity of short fibres inside a yarn will increase its probability to form protruding fibres on the fabric surface. The number of loosen microfibers will increase with the yarn count [10] as the yarn count will be increased when the quantity of fibres per cross section of the yarn will increase as well. Hairiness is responsible of fibres breakage during the weaving and knitting process (see part *the influence of the weaving and knitting processes*). At high speed, the protruding fibres and fibres loops are caught into machine elements and needles causing breakage in the fabric structure.

It seems that the best spinning process (from the conventional processes studied) to avoid the formation of microfibers on fabrics is the rotor spinning process.

As it has been specified in the preparatory action A1 of the MERMAIDS project, the main sources of microplastics' pollution are plastic microfibers coming from manufactured yarns such as polyester, polyamide, polyacrylic or polypropylene. Rotor and ring spinning processes can be employed for the production of yarns made of different compositions fibres (natural, synthetic or artificial). Regarding the extrusion process, the formation of microfibers will be induced by the same factors than the conventional ring or rotor spinning processes. Those factors are reviewed in the following part.

[9] Prof. Dr. rer. nat. Hans-Karl Rouette, Encyclopedia of Textile Finishing, Chapter H, page 1.

[10] A. Basu, Assessement of yarn hairiness, Indian Journal of Fibre & Textile Research. Vol 24. June 1999. Page 86-92.

Review of the hairiness and microfibers formation factors

The study of the different existing spinning processes has demonstrated that the formation of microfibers on the fabric surface can be due to different factors which are detailed next.

Twist value:

It has been observed that the twist of the yarn will condition the number of microfibers present on its surface. Indeed, a high yarn twist is employed to produce compact yarns. Moreover, the yarn resistance and elasticity increase with the twist. The twist is an important factor to avoid the formation of microfibers but it can be less desired by the textile manufacturing companies as it will reduce the softness of the fabric. On the other hand, a high twist can be valuable for less garment shrinkage and wrinkle formation.

Parallelism of the fibres:

The parallelism of the fibres is also an important factor as at less parallelism for ring spinning process, more protruding fibres will be present on the yarn surface. In the rotor process, the non parallel fibres will wrap the yarn reducing the protrusion of the others fibres.

Yarn irregularities:

As explained before, the yarn irregularities is also responsible of increase the formation of microfibers on the fabric. Less yarn irregularities have been observed by using the rotor spinning process.

Fibres per yarn or yarn count:

The yarn count may also be an important factor as the more fibres quantity into the yarn cross section, the more probability for them to protrude to its surface.

Yarn tenacity which depends on yarn twist (*for degradation under use*):

The yarn tenacity will define the resistance of the fabric during its use. At low mechanical resistance, the yarn can break during usage forming shorter fibres inside the yarn and so pilling on the garment surface. Then, the broken fibres can be discharged to domestic waste waters from laundry machines.

Fibres length:

The microfibers formation will increase when the fibres' length is reduced. The short fibres present in the structure of the yarn won't be easily wrapped into the yarn and will be lost after washing.

Conclusions

Hairiness is mostly present on fabrics composed yarns made of staple fibres. Indeed, staple fibres have short size which will increase their probability to stick out from the yarn body. Hairiness effect is sometimes desired during the fabric production in order to obtain a soft hand or touch of the fabric and warm effect in inner layers. Synthetic fibres such as polyester or polyamide are continuous fibres obtained from extrusion process and which can be cut or not into small pieces. From this process, monofilament, short fibres multifilament or continuous multifilament yarns can be produced. The hairiness of the resulting fabric will depend on the length or twist value of the cut fibres.

In conclusion the most important factors influencing the formation of protruding fibres on the fabric surface are the fibres length and parallelism and the yarn twist and irregularities for yarns made of staple fibres. For multifilament yarns the probability to form microfibers is lower.

The influence of the weaving and knitting processes

The weaving machine is composed of different elements which principal parts are the loom supporting the warp yarns and the weft yarns. The warp threads are unrolled from the loom and directed to a comb where the threads are separated forming an opening angle where the weft is inserted horizontally. During this coming and going movement, the weft is creating friction onto the warp yarns damaging them. The modern machine speed can reach 450 weft insertions into the warp opening angle per minute.

In the case of knitting machines, a unique thread is used to constitute the final two-dimensional fabric. This thread is inserting into various needles placed side by side in a 2 to 3 metres length knitted machine.

The hairiness of the yarn will also affect the weaving and the knitting process. Indeed, the short fibres protruding from the yarn surface (see next picture) may hook into the textile machines parts and break. This will create an excessive formation of fibres dust which could also be woven inside the fabric structure. After several domestic washing, those fibres dust will be discharged in the domestic waste waters.

Figure 10. Example of a yarn with protruding fibres capable of hooking into the textile machine parts[2].

The influence of the mechanical finishing

Mechanical treatments in the textile industry are employed to confer new properties to the fabrics such as a different touch, density or even width. The different mechanical textile finishing and their relation with the microfibers formation is detailed next. The “raising” process is responsible of the formation of microfibers whereas the “singeing” or the “calendaring” processes have the property to eliminate those protruding fibers.

The raising process:

This mechanical finishing is done onto textiles to give them a special insulating effect by lifting the fibers up to the surface and forming a dense and high fibers layer on the textile.

The mechanical treatments which can be employed against the microfibers formation are:

Singeing:

During this mechanical finishing, the protruding fibres of the fabric are eliminated by burning the fabric’s surface. More precisely, the fabric is preliminary moisten and then is put in contact with a flame or gas or electric system to eliminate the superficial fibres.

Calendering:

The calendaring process aims at modifying the final touch of the fabric and reaching a shiny and smooth touch. This effect is obtained by putting pressure on the fabric between two hot rolls.

The influence of the chemical finishing

The chemical finishing is not responsible of the microfibers formation but can be employed to reduce this phenomenon. Indeed, the chemical finishing will cover the fabric surface with a thin protective layer and by different techniques (padding, exhaustion, spraying and coating). The ability of the treatment to fix the protruding fibers and avoid their lost will depend on the chemical agent applied on the fabric surface. The different chemical agents capable of avoiding the microfibers lost during domestic washing are detailed in the next part *A.2.1.2. Study of textile auxiliaries*.

3. Description of the selected fabrics

In order to study the influence of fibres and fabric properties in relation with fibres loss, fabrics of different composition (polyester, blended polyester/cotton, polyamide and polyacrylic), structure (woven and knitted) and weave or spinning type (plain weave, single or doubled yarn, staple fibres yarn or continuous yarn, etc.) have been studied.

The fabrics selected for this Action A2 are described in the next table and characterized in the next parts of this deliverable. Some of those fabrics will be characterized as well in the implementation action B1:

Fabric's reference	Description
777	Polyester 100 %
361	Polyamide (6.6) 100%
983	Polypropylene 100 %
864	Polyacrylic 100%
7409	Polyester/Cotton 65/35 style Bleached without optical brightener.
867	Acrylic. Acrylic Knit (tubular).
720	Polyester. Double Knit Jersey (Disperse Dyeable).
314 WOB	Polyamide. Nylon 6.6 Stretch fabric, Double Knit.

Table 1. Description of the fabrics of the preparatory actions

4. Fabrics mechanical and physical performances characterization

As it has been studied in the previous part 2 (*Description of the mechanical properties of fibres influencing the fibres breakage performance*), many factors from the spinning, weaving or knitting processes may increase the microfibers loss on the fabric. In order to confirm the hypothesis made in the previous part, different standard fabrics described previously have been analyzed. The microfibers presence on these fabrics and their propensity to form microfibers has been determined. Those characterization tests are described next.

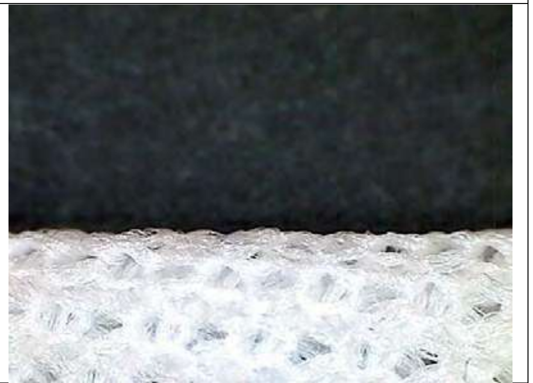
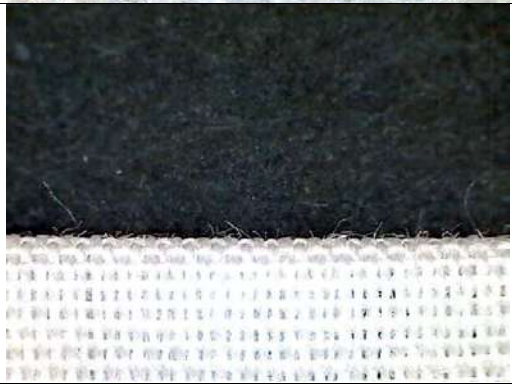
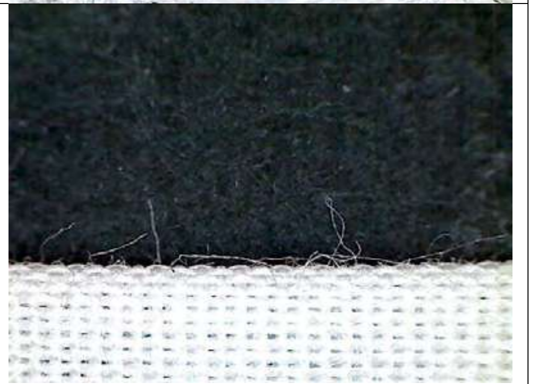
Characterization of the fabrics and their propensity to form microfibers

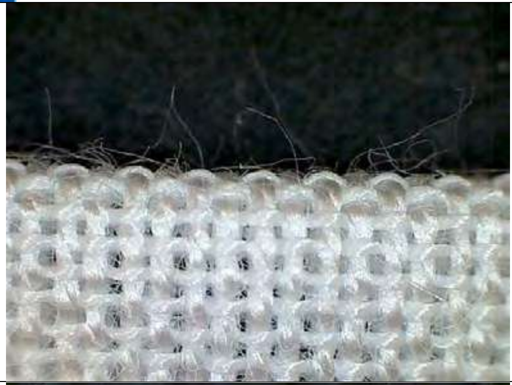
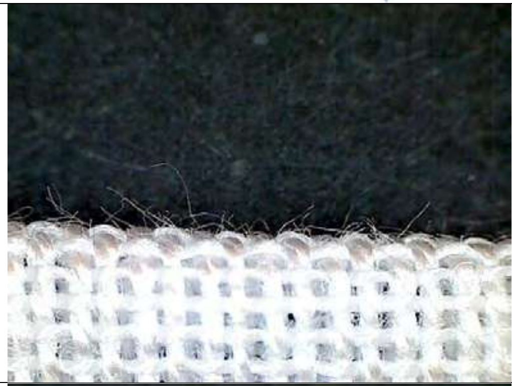
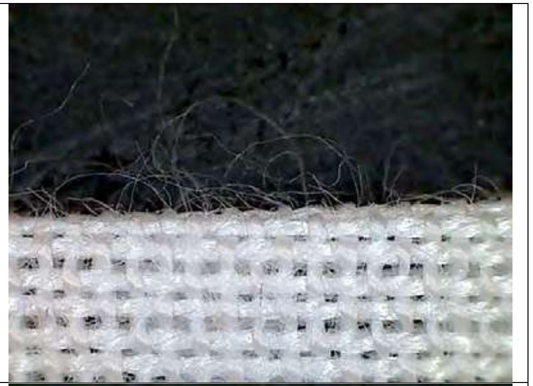
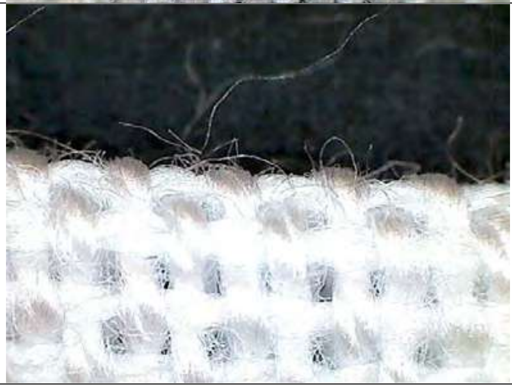
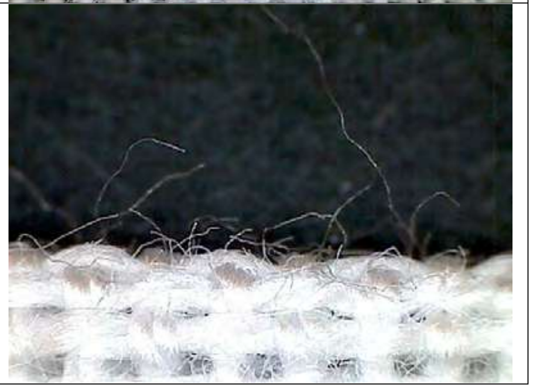
The evaluation of the physical and mechanical characteristics of the fabrics have been done on the fabrics referenced as 720 (100% knitted polyester), 7409 (65% polyester, 35% cotton), 777 (100% woven polyester) and 983 (100% polypropylene). The rest of the fabrics described in the *Table 1. Description of the fabrics of the preparatory actions* will be characterized during the implementation actions. For this preparatory action, the microfibers formation factors have been studied theoretically and this study has been complemented with the following physical and mechanical analysis.

Determination of the hairiness of the standard fabrics

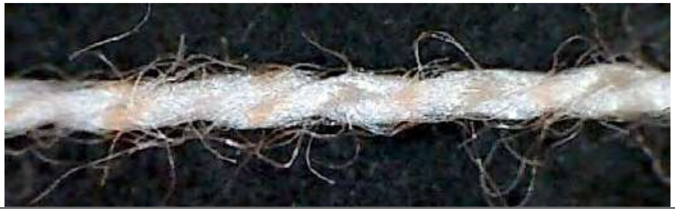
As it has been studied in the previous part 2, the hairiness of the fabrics is the source of the microfibers presence and loss. Few techniques exist to determine and quantify the hairiness of a fabric (Hairiness Module for Uster Tester 3, hairiness Tester G566…). The test employed in this part to analyze the hairiness of the fabric is a visual technique consisting of photographing a yarn from the fabric structure and a section of this fabric.

The hairiness pictures are presented in the next table:

Fabric description	Fabric cross section		
720: 100% knitted PES			
7409: 65PES 35CO			

777: 100% woven PES			
983: 100% PP			

The next table presents the yarn structure of the previous fabrics. A photography of the weft and the warp yarns (for woven fabrics) has been made to find out if there is differences between both yarns.

Fabric description	Weft yarn	Warp yarn
720: 100% knitted PES		
7409: 65PES 35CO		
777: 100% woven PES		
983: 100% PP		

Conclusions:

- 720 (100% knitted polyester):

As it can be seen on in the table, this sample doesn't present any hairiness on its surface. Looking at the yarn we can see that it's a continuous yarn, so we can deduce that this is the reason why any hairiness is formed on this kind of fabric. On the other hand, we can observe that the yarn have a very low twist but this will not influence the formation of microfibers since the yarn is continuous.

- 7409 (65% polyester, 35% cotton):

We can observe on the pictures this fabric present more hairiness than the knitted polyester fabric. However, the height of the microfibers protruding from the fabric surface is relatively low. The yarn observation shows the presence of short protruding staple fibres from its structure and low twist value.

- 777 (100% woven polyester):

The pictures of this fabric show that the microfibers formation is not occurring homogeneously on the fabric surface. Some zones of the sample present more hairiness than others. The height of the microfibers is relatively high. The images of the yarn show that the weft and the warp yarns don't present the same structure: the warp is doubled yarn and seems to produce less microfiber than the weft, which is single yarn.

- 983 (100% woven polypropylene):

The pictures show that the fabric has a high percentage of hairiness. The height of the microfibers is relatively high. As it can be seen, the weft and the warp are doubled yarns. The weft yarn seems to have the same hairiness than the warp yarn.

Those pictures permit to conclude that each fabric have different hairiness depending on their twist value, if they are simple or doubled yarn, and if the yarn is made of short staple fibres or continuous fibres. It seems that fabrics 777 and 983 have the highest hairiness, the hairiness of 777 being a little bit more pronounced.

Determination of the yarns' twist and retwist (ISO 17202)

As studied previously in the part 3, the fibres parallelism increases with the yarn twist. This parallelism will induce that the fibres are well bonded into the yarn structure with less probability of protrusion. Indeed, the highest the yarn twist the lowest the hairiness.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Standard: UNE 40600-4:1996- ISO 17202:2002

Scope: This test is intended to determine the retwist of yarns extracted from a fabric and twist.

Equipment used: JBA twist tester

Conditioning of the specimens: 24 hours to 20ºC ± 2ºC - 65 % ± 4 % h.r.

h.r.: relative humidity

Test conditions:

Test tensile strength: 0.5 cN/tex.
Number of yarns extracted: 20
Test length: 50 cm.
Previous treatment: Null

Results obtained:

Ref: 777 (woven PES 100%)	Warp	Weft
Whole:		
Retwist direction	S	--
Retwist (v/m)	671.6	--
C.V %(1)	1.5	--
Individual		
Twist direction	Z	Z
Twist (v/m)	739.3	665.3
C.V %(1)	3.1	2.9
Ref: 983 (woven PP 100%)	**Warp**	**Weft**
Whole:		
Retwist direction	S	S
Retwist (v/m)	283.6	266.6
C.V %(1)	3.6	3.8
Individual		
Twist direction	Z	Z

Twist (v/m)	501.7	526.0
C.V %(1)	5.7	3.8
Ref: 7409 (woven 65% polyester, 35% cotton)	**Warp**	**Weft**
<u>Whole:</u>		
Retwist direction	---	---
Retwist (v/m)	---	---
C.V %(1)	---	---
<u>Individual</u>		
Twist direction	Z	Z
Twist (v/m)	968	996.5
C.V %(1)	3.3	3.4

(1) The coefficient of variation (CV%) is defined as the ratio of standard deviation and the absolute value of the arithmetic mean.

$$CV = \frac{s}{|\bar{x}|}$$

A lower coefficient of variation we consider that the distribution of the measured variable is more homogeneous.

Conclusions:

- 777 (100% woven PES):

The warp of the fabric is doubled yarn whereas the weft is single yarn.

- 983 (100% woven polypropylene):

The warp and the weft are both doubled yarns but their twist and retwist value are relatively low.

- 7409 (woven 65% polyester, 35% cotton):

The weft and the warp of this fabric are single yarns but their twist value is very high.

The previous studies of part 2 permit to do the hypothesis that a fabric made of double yarns will have less hairiness than a fabric made of single yarn. Indeed, the retwist of two yarns will help to hold the short fibres inside the yarn structure. Moreover, the fabric with higher twist will have less hairiness formation. So, according to these hypothesis, the fabric with less propensity to form microfibers, taking into account his twist value, is the fabric referenced as 7409 (woven polyester/cotton). The pictures of the cross section of this fabric in the previous part (*Determination of the hairiness of the standard fabrics*) confirm this theory. The twist direction (S or Z) has any influence on the hairiness formation, in principle.

Determination of the linear density of the yarns (UNE 40600-5:1996. Method A)

The hairiness potential of a fabric increase with its yarn count as it will contain more fibres in its cross section.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Standard: UNE 40600-5:1996. Method A

Scope: This test is intended to determine the linear density of the yarns removed from a fabric.

Equipment used: Shirley Crimp Tester and Sartotius Balance.

Conditioning of the specimens: *24 hours to 20ºC ± 2ºC - 65 % ± 4 % h.r.*

Test conditions:

Test atmosphere: 20ºC ± 2ºC - 65% ± 4% h.r.
Number of yarns extracted: 100
Previous treatment: Null

Results obtained:

	Ref: 777 PES	Ref: 983 pp	Ref: 7409
Warp (Tex)	19.91 / 2	36.43 / 2	12.56 / 1
Weft (Tex)	40.18 / 1	36.64 / 2	12.79 / 1

Conclusions:

- 777 (100% woven polyester):

The weft and the warp linear density is quite different. Those results show that a medium and a thick yarn have been employed to produce the fabric. The weft yarn is thicker and is just a single yarn. This yarn could be responsible of producing more hairiness on the fabric surface.

- 983 (100% woven polypropylene):

The weft and the warp linear densities of the fabric are very similar. The yarn count's value is relatively high but since each yarn is doubled the microfibers protrusion won't be so important.

- 7409 (woven 65% polyester, 35% cotton):

The warp and the weft of this fabric are very similar. This fabric is composed of the thinnest yarns of the three fabrics studied. This means that the yarn cross section contains less fibres than the others fabric's yarns. This could imply less hairiness formation on the fabric surface (which has also been observed in the previous pictures).

Determination of the fabrics density (UNE-EN 1049-2:1995. Method A)

The fabric density or the number of yarns per unit length is also related to the microfibers loss of a fabric. Indeed, a fabric with low density has an opened structure where the distance between the yarns is higher. This opening structure will promote the interstitial movements between the yarns and their friction. This friction will be responsible of forming protruding microfibers.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Standard : UNE-EN 1049-2:1995. Method A.

Scope: This test is intended to determine the density of a fabric.

Equipment used: Counting glass.

Conditioning of the specimens: 24 hours to 20ºC ± 2ºC - 65 % ± 4 % h.r.

Test conditions:

Test atmosphere: 20ºC ± 2ºC - 65% ± 4% h.r.
Average length:

- Warp: Ref 777: 5 cm. ; Ref 983: 5 cm; Ref 7409: 2 cm
- Weft: Ref 777: 5 cm.; Ref 983: 10 cm; Ref 7409: 3 cm

Number of measurements: 5

Previous treatment: Null

Results obtained:

	Ref: 777	Ref: 983	Ref: 7409
Warp	20 yarns/cm	12.5 yarns/cm	44 yarns/cm
Weft	20 courses/cm	9 courses/cm	29.5 courses/cm

Conclusions:

- 777 (100% woven polyester):

The density of the fabric for both warp and weft yarns is medium compared to the others two fabrics.

- 983 (100% woven polypropylene):

The density of this fabric is the lowest. Looking at the fabric it can be seen that it presents an opened structure. The gaps between the fibers may induce more formation of microfibers than the others two fabrics.

- 7409 (woven 65% polyester, 35% cotton):

The density of this fabric is very high which means that the yarns are woven tight together. This will reduce the friction between the yarns making this fabric the less prone to form microfibers, taking into account the fabric density parameter only.

Determination of the fibres length

As commented before, the shortest the fibres' length the highest the probability to obtain microfibers on the fabric surface. Indeed, short fibres have more possibility to go outward the yarn body and not being embedded into it.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Scope: This test aims to determine the fibres length by means of a handmade diagram.

Conditioning of the specimens: 24 horas a 20ºC ± 2ºC y 65 % ± 5 % h.r.

Test conditions:

Previous treatment:

- REF. 983-PP Y REF. 777-PES: Null
- REF. 7409 CO/PES:

Polyester length: Removed cotton with sulfuric acid according to UNE-EN ISO 1833-11:2011.

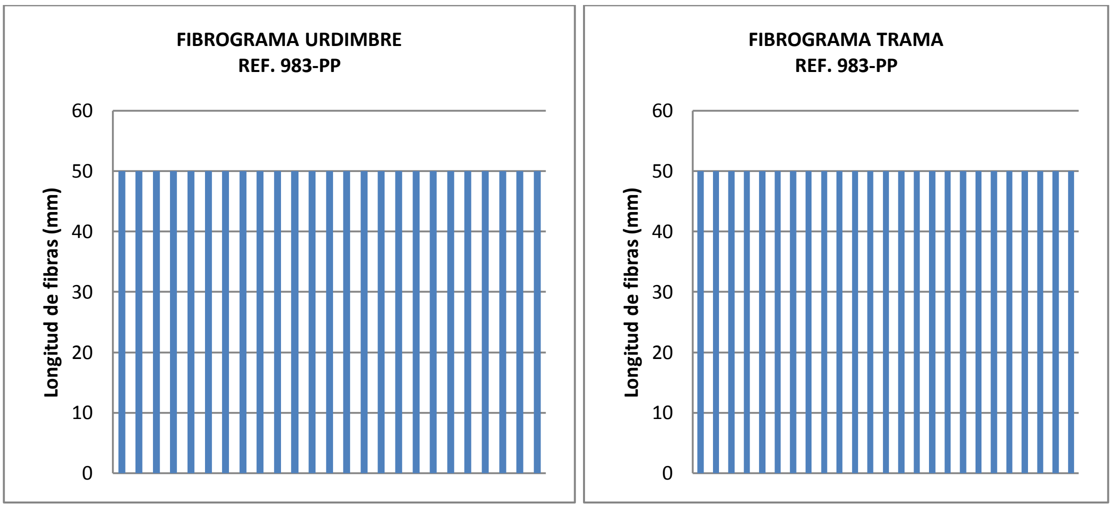
Cotton length: Removed polyester with tricloroacetic acid / cloroform according to UNE-EN ISO 1833-25:2014.

Test results:

REF. 983

Fibres length	
Type of fibre: PP	
Maximum length(mm)	50
Minimum length (mm)	50
Average length (mm)	50

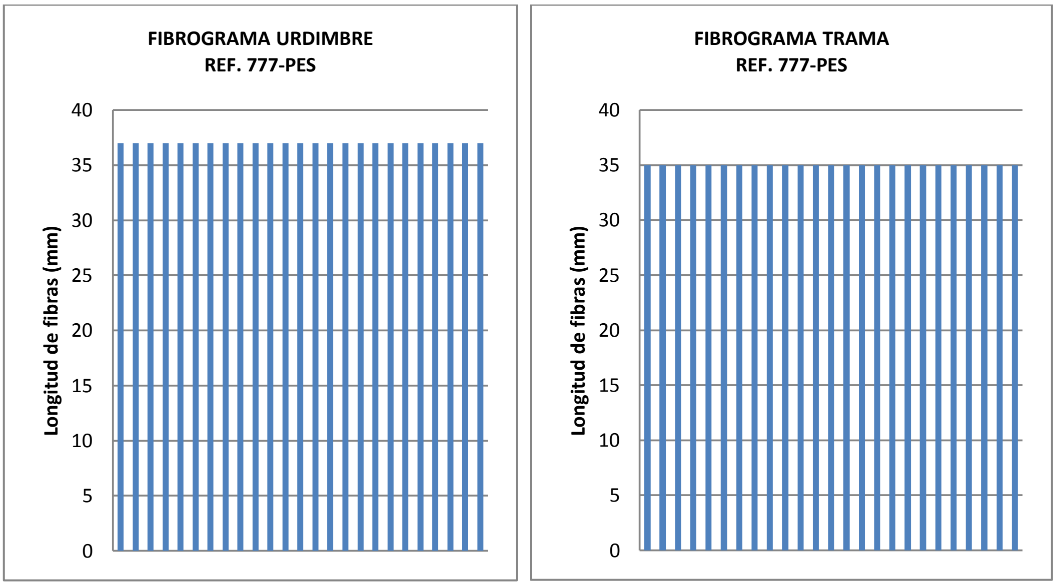
Fibre diagram:



REF. 777

Fibre length	
Type of fibre: PES	
Maximum length (mm)	35-37
Minimum length (mm)	35-37
Average length (mm)	35-37

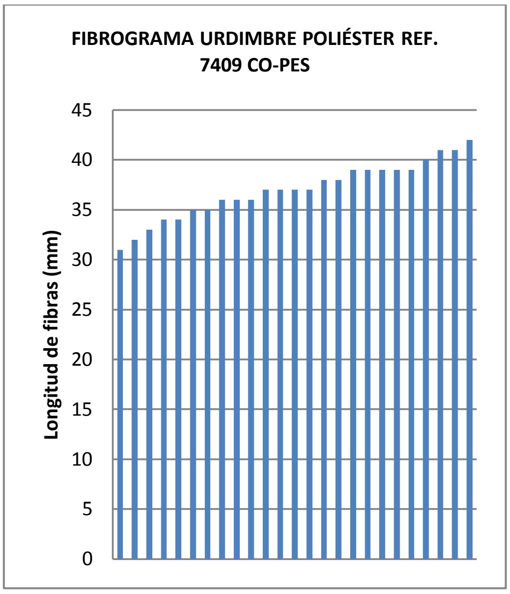
Fibre diagram:

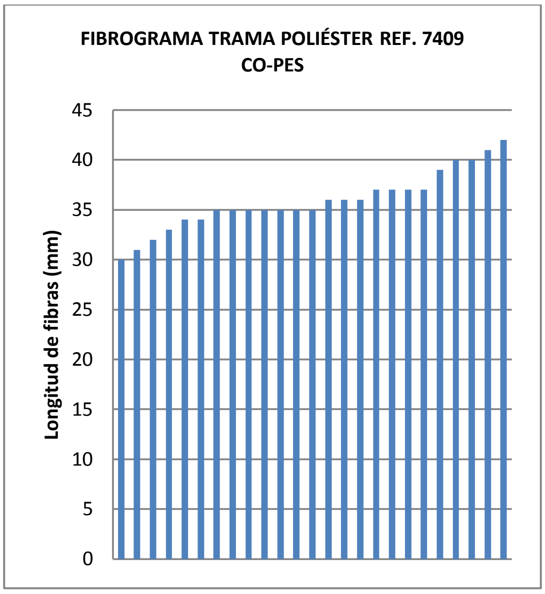


REF. 7409

Fiber length	
Type of fibre: PES	
Maximum length (mm)	42
Minimum length (mm)	30-31
Average length (mm)	36-37

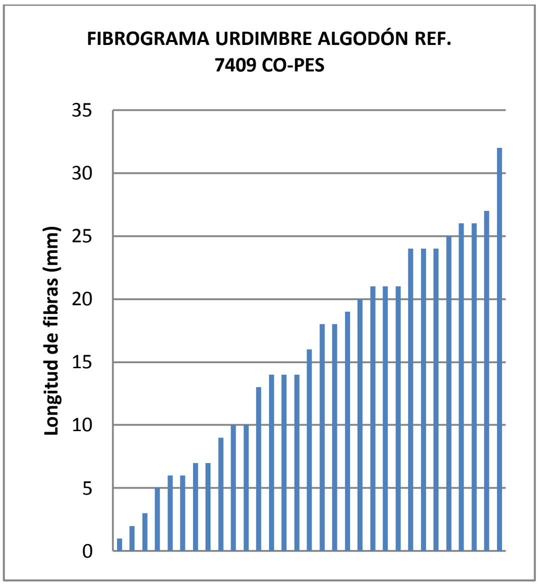
Fibre diagram:

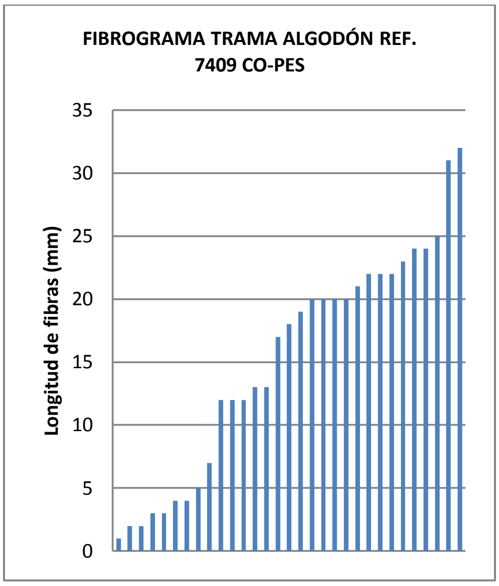




Fiber length	
Tipo de fibra: CO	
Maximum length (mm)	*32*
Minimum length (mm)	*1*
Average length (mm)	*15-16*

Fibre diagram





Conclusions:

- 983 (100% woven polypropylene):

The average length is identical for each fibres as the fabric is made of synthetic fibres cut at the same length after the extrusion process. This fabric has the highest fibres length so taking into account only this parameter, his hairiness should be less than others fabrics. But taking into account others parameters studied previously, its high fibres length is not enough to reduce the microfibers loss.

- 777 (100% woven polyester):

This fabric is also made of synthetic and regular fibres. Their length is medium compared to the others two fabrics.

- 7409 (woven 65% polyester, 35% cotton):

This fabric is composed of two different fibres: polyester and cotton. This type of blend is called "close blend" as one yarn is composed of two different composition fibres. Chemical tests have been performed to dissolve a fibre and isolate another, to perform the analysis. As it can be seen, the length of the polyester fibres is not homogeneous like the others synthetic fibres. This may be due to the fact that those fibres have been obtained from a different process: the staple polyester fibres are blended to the staple cotton fibres into a conventional spinning process (ring or rotor spinning). The cotton fibres length is less homogeneous due to the fact that they are natural fibres. This fabric has the shortest fibres composition but due to their nature and the possible conventional spinning process employed to produce the yarn, the formation of hairiness might be lower.

Determination of the fabric's weight (UNE-EN 12127)

The weight of a fabric will give an indication of the material mass per unit area. The highest is this value; the highest numbers of fibres will be present per unit area. However, as it has been seen before, the fibres lost is not only related to the number of fibres present per unit are but how these fibres are spun together and the physical properties of these fibres.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Standard: UNE-EN 12127:1998

Scope: This test is intended to determine the mass per unit area of small conditioned samples of fabric.

Equipment used: Manual die of 100 cm²

Conditioning of the specimens: 24 hours to 20ºC ± 2ºC y 65% ± 4% h.r.

Test conditions:

Test atmosphere: 20ºC ± 2ºC - 65% ± 4% h.r.
Dimensions of the specimens: 10 cm x 10 cm
Number of specimens tested: 5
Previous treatment: Null

Results obtained:

	Ref: 777 PES	Ref. 983 PP	Ref:7409
Mass per unit area (g/m²)	128.5	165.1	97.6
C.V. (%)	0.7	0.2	0.2
Uncertainty (k=2) (1)	1.1	0.4	0.3

(1) This value corresponds with the uncertainty expanded of obtained measure multiplying the typical uncertainty of measurement by the factor of coverage k = 2 that for one normal distribution, corresponds to a probability of coverage of approximately 95 %.

Conclusions:

The results of the table show that the fabric with the highest weight is the polypropylene fabric (983) followed by the woven polyester one (777). The blended fabric polyester/cotton has the lowest weight. This result can also be appreciated visually. These results are coherent with the analysis of the number of fibres linear density. As it has been seen in this test, the linear density of the polyester/cotton yarns is the lowest whereas the polypropylene yarns have the highest yarn count.

Characterization of the fabrics degradation under use conditions

Pilling description

The pilling effect is characterized by the formation of small fibres balls or knots on the fabric surface. These knots are formed during the rubbing of the fabric with another fabric or another surface. The pilling formation is higher on knitted fabrics than on woven fabrics due to the stretch structure and the loops formed by the yarn. Some fibers have more propensity than others to form microfibers such as rayon fibres. Synthetic fibres are also known to produce pilling on garments. The length and the fineness of the fibres will also influence the microfibers formation. As detailed before, the yarn spinning process is also a determining factor the microfibers formation, and so the pilling formation.

The pilling formation is a process which starts with the union of the protruding microfibers of the fabric's surface. Then, some short fibers will break and get inside the ball structure. This ball will be maintained to the textile surface thanks to the longest fibers present there. The next pictures describe the pilling formation process:

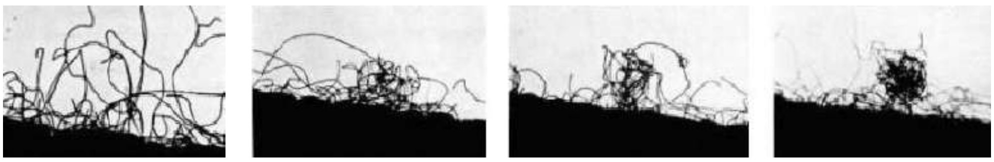


Figure 11. The pilling formation process. From left to right: the protruding microfibers will join together forming a ball made of short fibers in the inside and kinked to the fabric's surface by the longest fibers.

Determination of the pilling by pilling Random (ASTM D 3512)

This test is employed to simulate the degradation of a fabric under use but also it could simulate the degradation occurring inside a washing machine. More precisely, the test consists of inserting three samples of fabric inside a closed cabin composed of rotating metal blades and where air is circulating. The fabric will be in a rotating movement simulating the washing centrifugation. The contact of the fabrics with the metal blades will be responsible of degrading the fabric during the essay. An example of pilling random equipment is presented in the next picture:

Figure 12. Description of a Pilling random equipment.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Standard: ASTM D 3512:2010

Scope: This test is intended to determine fabric propensity to surface fuzzing and to pilling with the method Random.

Equipment used: Pilling Random

Conditioning of the specimens: 24 h / 20ºC ± 2ºC / 65 ± 4 % h.r.

Test conditions:

Test atmosphere: 20ºC ± 2ºC - 65% ± 4% h.r.
Test time: 5 min – 15 min - 30 min.
Samples size: 105 x105 mm
Number of specimens tested: 3
Number of observers: 3
Previous treatment: Null

Results obtained:

	Pilling index		
	5 min	**15 min**	**30 min**
Ref.777	4	3-4	3
Ref.983	4	4	3-4
Ref.7409	4	4	3-4

Interpretation of index or pilling according ASTM D 3512	
1	Thick fuzzing and/or pilling on the surface. Pills of several sizes and densities covering the surface of the specimen completely.
2	Evident fuzzing and/or pilling on the surface. Pills of several sizes and densities covering the surface of the specimen largely.
3	Moderated fuzzing and/or pilling on the surface. Pills of several sizes and densities covering the surface of the specimen partially.
4	Slight fuzzing and/or pilling on the surface
5	No change.

The next pictures present two standardized templates of the degradation of fabrics after the pilling process. The value 5 is considered to be the best result, whereas the value 1 is the worst one.

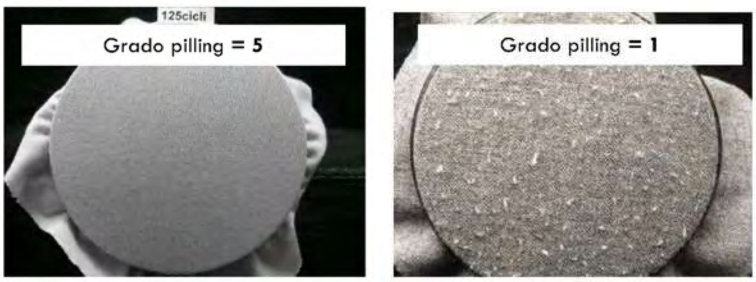


Figure 13. Presentation of two degradation templates for the pilling resistance test (left: pilling index number 5; right: pilling index number 1).

Conclusions:

The analysis of the results is done by comparing the sample degradation with standardized fabric degradation templates. The results table shows that the three samples studied have similar degradation, but the sample 777 (100% woven polyester) present a slightly superior degradation.

Determination of the pilling by pilling Box (ISO 12945-1)

The pilling box test will be used to simulate the degradation of the fabric under use. More precisely, it will simulate the friction occurring in parts in contact when wearing a garment (underarms, for example). During this test, four fabric samples fixed on a cylindrical support are in movement and collide together. The fabric will be degraded by the contact fabric/fabric, fabric/box material (rubber), fabric/cylindrical support material (plastic rubber). The next picture present the pilling box equipment employed to perform the tests:

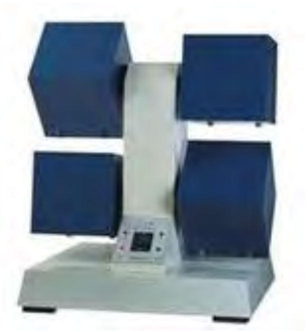


Figure 14. Description of the pilling box equipment.

The test conditions and the results obtained are detailed next (the results of the fabric reference 720 - 100% knitted PES- will be included in the implementation action):

Standard: UNE-EN ISO 12945-1:2001

Scope: This test is intended to determine fabric propensity to surface fuzzing and to pilling.

Conditioning of the specimens: 24 hours to 20ºC ± 2ºC - 65 % ± 4 % h.r.

Equipment used: ICI Pilling-Box

Test conditions:

Test atmosphere: 20ºC ± 2ºC - 65% ± 4% h.r.
Number of cycles: 7.000 (requested by the customer)
Number of specimens tested: 4

Number of observers: 3	
Previous treatment: Null	

Results obtained:

Reference	**Pilling grade**	**Aspect of the surface**
777	2	Fuzzing or pilling
7409	3	Fuzzing
983	3	Fuzzing

Interpretation of index or pilling according to the standard UNE EN ISO 12945/1:2001	
1	Thick fuzzing and/or pilling on the surface. Pills of several sizes and densities covering the surface of the specimen completely.
2	Evident fuzzing and/or pilling on the surface. Pills of several sizes and densities covering the surface of the specimen largely.
3	Moderated fuzzing and/or pilling on the surface. Pills of several sizes and densities covering the surface of the specimen partially.
4	Slight fuzzing and/or pilling on the surface
5	No change.

Conclusions:

The table shows that the degradation of the fabrics is similar but the 100% woven polyester fabric seems to have a slightly superior degradation value. The tendency of the fabric 777 to have a higher degradation has been observed as well during the pilling random test. The previous physical analysis of the fabrics will tend to suggest that the polypropylene fabric (referenced as 983) has the highest propensity to form microfibers on its surface. However, the mechanical analysis show that, under use, the woven polyester fabric (referenced as 777) is more prone to form hairiness. This could be due to the fact that the mechanical essays take into account the behaviour of the fabric structure under mechanical action.

5. Fabrics physical characterization after washing

This part aims to study the degradation of the selected fabric after different cycles of a domestic washing process. All the physical tests employed to evaluate the propensity of the fabric to form microfibers won't be repeated as we suppose that these properties won't be affected after domestic washing. Indeed, after washing, the yarn twist, yarn count, fibres length, fabric density and fabric weight won't be influenced by the washing process. However, the hairiness and the fabric's weight will be subjected to changes after washing. The weight of microfibers lost after washing small samples won't be appreciable by measuring the fabric weight per unit area. So, this test hasn't been performed after washing in this preparatory action.

This part describes the hairiness results obtained after washing the fabrics. The mechanical resistance of the fabric (Pilling Random and Pilling Box tests) will be determined in the implementation actions. The hairiness will give us a preliminary perception of the influence of the washing process on the formation of microfibers. The mechanical tests which will be done in the implementation actions will indicate the resistance of the fabrics to pilling after domestic washings.

Description of the washing process

This part is intended to describe the washing process employed to simulate the domestic washing. This test is based on the standard ISO 105 C06. One cycle of the test can simulate five domestic washing cycles. The washing has been performed in a Lini-test equipment. The machine is composed of a water bath containing a routable shaft which supports steel containers. The shaft/container assembly is rotating at an established frequency and at a defined temperature. The machine is presented in the next picture:

Figure 15. Presentation of a Lini-Test machine

Material preparation

Small dimension samples have been prepared (around 15 cm x 15 cm) taking into account the bath ratio employed for the test and the required dimensions for the next mechanical essays to be performed. The samples have been sewed with a cotton thread as the objective is to observe the fabric surface and its mechanical properties, but not to filter the residual bath.

Washing conditions

The washing conditions are based on the ISO 105-C06 standard and have been optimized according to the objective of our essay. These conditions are described in the following table:

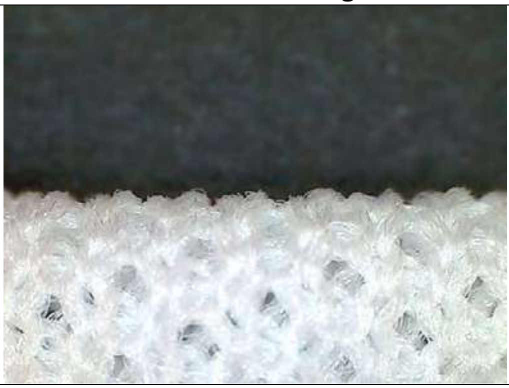
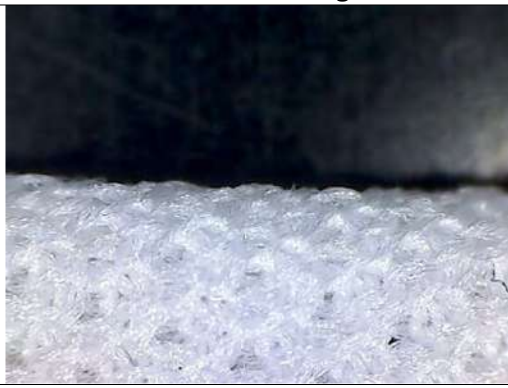
Number of washing cycles	2 (simulating 10 at domestic level)
Water type	Distilled water
Detergent	No
Temperature	40ºC
Time	45 minutes
Bath ratio	1:15
pH adjustment	No
Presence of chlorine, sodium perborate	No
Presence of steel balls	Yes, 10 balls

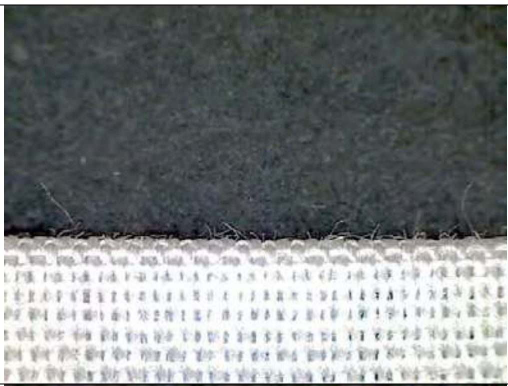
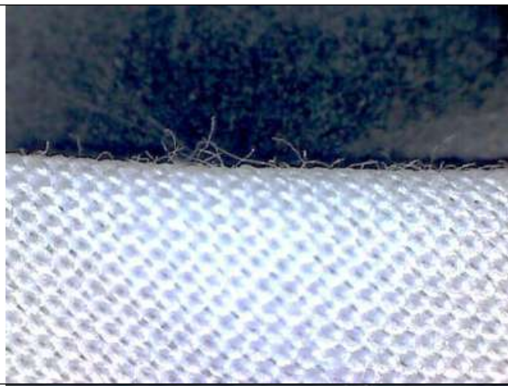
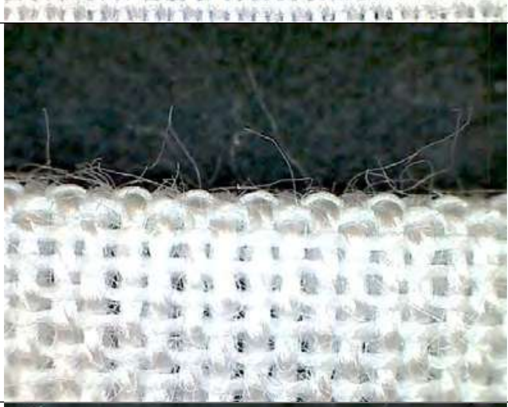
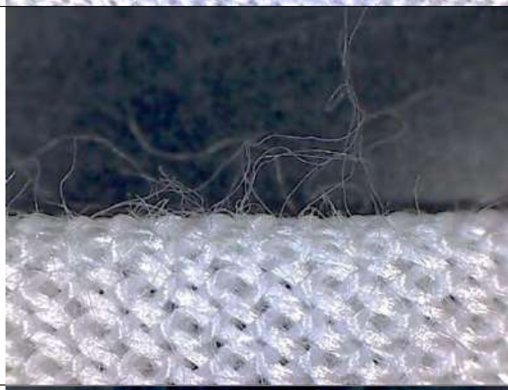
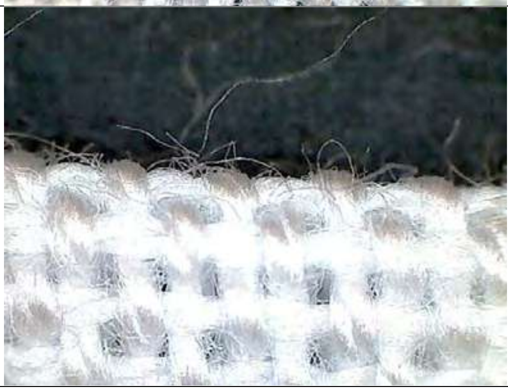
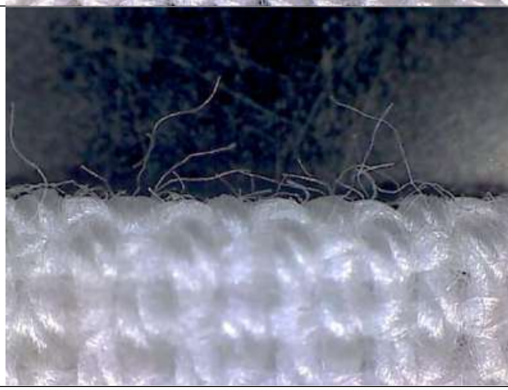
Table 2. Description of the washing conditions in Lini-Test

In this test, two washing cycles have been done, which is equivalent to 10 domestic washing cycles. The temperature employed is the washing temperature most used in Europe (40ºC), according to the questionnaire and preliminary study made in the preparatory action A1.

Results after domestic washing

The washed fabrics have been characterized by observing the hairiness on their surface. The pictures of the fabrics section are included in the following table:

Fabric description	Fabric cross section BEFORE washing	Fabric cross section AFTER washing
720: 100% knitted PES		

7409: 65PES 35CO		
777: 100% woven PES		
983: 100% PP		

Conclusions:

The sample 720 (knitted 100% polyester fabric) still presents good results with no hairiness formation on the fabric surface.

It’s difficult to estimate the changes in the hairiness of the others fabrics as the microfibers presence is not homogeneous onto the whole surface. However, we can deduce from these pictures that the protruding fibres quantity hasn’t increased excessively. However, we can observe from these images that the height of the protruding fibres is higher. This could imply, as it has been studied in the pilling description part (into the part 2.2) a higher propensity to form pilling knots or balls after mechanical degradation (normal use). The height increase is more evident on the pictures of the samples 7409 (woven cotton/polyester fabric) and 777.

6. Conclusions

The theoretical study and the preliminary tests done on the fabrics permit to confirm different hypothesis on the microfibers formation and lost during domestic washing. It has been demonstrated that this microfibers formation can due to the spinning, weaving or mechanical process used in the textile industry. The spinning process seems to be the initial process where the reduction of microfibers actions should be considered, in order to avoid the increase of the hairiness in the next textile processes.

As it has been studied and confirmed, this hairiness can be due to physical properties of the yarns and the fabrics and can be increased during mechanical degradation actions (friction occurring during use, friction occurring during domestic washing).

The main physical properties responsible of increasing the hairiness formation are:

a. The fibres length.
b. The yarns twist and retwist.
c. The yarn count.
d. The fabric warp and weft densities.
e. The fabric's weight.

The main mechanical actions responsible of producing microfibers loss during use and of increasing this effect after washing are:

a. The pilling (which can be measured by pilling box, random or Martindale methods).
b. The domestic washing.

The domestic washing of fabrics has demonstrated to increase the height of the microfibers yarns. The use of garments made of these fabrics and then washed domestically will produce mechanical degradation which will end in the pilling formation (this hypothesis will be confirmed in the implementation actions). Indeed, the microfibers with increased height will start to link together and form a ball on the fabric surface. This ball will contain short fibres and could be able to be washed off during laundry.

TASK A.2.1.2. Study of textile auxiliaries.

1. Introduction

During fabric manufacturing, the process or condition parts of fibre can get semi-cut or leaving on the surfaces. These parts of fibre could be separated from the fabric during the domestic washing.

The synthetic fibres (polyester, polypropylene, polyamide,...) are more subjected to compounding the pollution of soils, rivers and the sea by microfibers due their low biodegradability an are more subjected to than natural fibres (cotton, flax,…) due to their low capacity to store water.

Existing chemical textile auxiliaries with the potential to reduce the fibre’s breakage and to impede the loss of microfibers will be compiled.

The aim of this report is to study and define different types of textile auxiliaries with the potential to reduce the fibre’s breakage and to impede the loss of micro/nanofibres in the domestic washing.

2. Textile auxiliaries

All textile auxiliaries selected for Mermaids project must meet minimum requirements:

1. Should not change the touch of the fibre.
2. Must comply whit current environmental regulations and standard textiles (REACH, OEKO-TEX, Clear to wear…).
3. Have to have fastness to the washing.
4. Be soluble in water.

Considering these parameters, we have selected seven textile auxiliaries of different types:

- Polyurethane
- Acrylic resin
- Blend of polyurethane and acrylic resin
- Macro-emulsion silicon
- Enzyme

Polyurethane

Polyurethane (PUR and PU) is a polymer composed of a chain of organic units joined by urethane links.

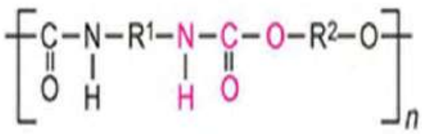


Figure 16. Chemical formula of polyurethanes

Polyurethane creates a protective film on the surface, thereby fixing the fibre parts that could lose in domestic washing.

The application conditions of polyurethanes are:

- Polymerization temperature 160°C
- Application by roll-padding
- Amount 10-50 g/l

PURLASTIC 8139

PURLASTIC 8139 is an aqueous dispersion of aliphatic polyurethane completely polymerised. It is anionic polyurethane soluble in water. Its touch is very soft and elastic. This polyurethane creates a bright coating with good fastness to temperature and water.

PURLASTIC 8139 physical properties:

- pH (20°C): 6.00 – 8.00
- Viscosity (25°C): <160 mPa·s
- Dry matter: 38.00 – 42.00%

Acrylic resin

Acrylic resins are a group of related thermoplastic or thermosetting plastic substances derived from acrylic acid, methacrylic acid or other related compounds.

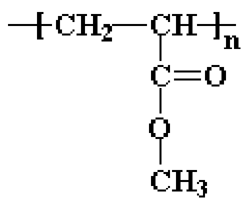


Figure 17. Chemical formula of acrylic resin

Acrylic resin creates a protective film on the surface, thereby fixing the fibre parts that could lose in domestic washing.

The application conditions of acrylic resin are:

- Dry temperature 130°C
- Application by roll-padding
- Amount 10-50 g/l

POLYACRIL 97

POLYACRIL 97 is a self-cross-linking acrylic polymer in aqueous emulsion. It is APEO free and soluble in water. If this acrylic resin is dried over the film formation minimum temperature (26°C), it creates a film with high hardness and with enough elasticity that avoids the cracking of the film.

POLYACRIL 97 is anionic and it has a good fastness to UV, rub hot and chlorine.

POLYACRIL 97 physical properties:

- pH (20°C): 4.00 – 6.00
- Viscosity (25°C): <350 mPa·s
- Dry matter: 42.00 – 46.00%

POLYACRIL 73

POLYACRIL 73 is an auto-crosslinkable acrylic polymer soluble in water. It is nonilphenols free and non-ionic. This acrylic resin has been specially developed as a fixing agent.

POLYACRIL 73 makes an elastic and soft film which has got high fastness to rub and water.

POLYACRIL 73 physical properties:

- pH (20°C): 5.00 – 7.00
- Viscosity (25°C): <266 mPa·s
- Dry matter: 42.00 – 46.00%

POLYACRIL 56 ECO

POLYACRIL 56 ECO is a self-cross-linking acrylic polymer in aqueous emulsion. It is APEO and formaldehyde free and soluble in water. If this acrylic resin is dried over the film formation minimum temperature (O°C), it creates a transparent film smooth and free to tack.

POLYACRIL 97 is anionic and it has a good fastness to temperature and water.

POLYACRIL 97 physical properties:

- pH (20°C): 6.50 – 7.50
- Viscosity (25°C): < 160 mPa·s
- Dry matter: 55.00 – 57.00%

Blend of polyurethane and acrylic resin

Blend of polyurethane and acrylic resin is a product where we have the proprieties of polyurethane and acrylic resin combined.

PURLASTIC 8189

PURLASTIC 8189 is an acrylic-urethane hybrid dispersion. It is anionic and soluble en water. It has excellent gloss and adhesion on a wide variety of substrates including wood. This acrylic-urethane creates a bright coating with good fastness to temperature and water.

PURLASTIC 8189 physical properties:

- pH (20°C): 7.00 – 9.00
- Viscosity (25°C): <200 mPa·s
- Dry matter: 40.00– 44.00%

Macro-emulsion silicone

Silicones are polymers that include any inert, synthetic compound made up of repeating units of siloxane, which is a functional group of two silicon atoms and one oxygen atom frequently combined with carbon and/ or hydrogen. They are typically heat-resistant and rubber-like.

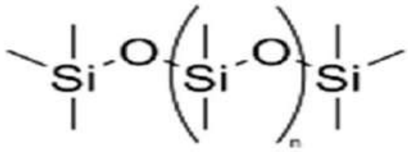


Figure 18. Description of silicone molecular structure

Macro-emulsion silicone is an emulsion silicone with particles sizes between 0.1 – 0.01 mm.

Macro-emulsion silicone creates a layer on the surface, thereby fixing the fibre parts that could lose in domestic washing.

The application conditions of macro-emulsion silicone are:

- Application by roll-padding or exhaustion
- Amount by roll-padding 10 - 40 g/l
- Dry temperature by roll-padding 120°C
- Amount by exhaustion 1.5 – 3% o.w.f.
- Temperature by exhaustion 40 - 60°C

POLYSILK CTE

POLYSILK CTE is an amino-funtional elastomeric silicone macro-emulsion. It is cationic whit non-ionic emulsifiers. This macro-emulsion silicone has a very good elasticity and good recovering strength, besides it has not yellowish effect at high temperatures.

POLYSILK CTE is excellent performance to avoid the formation of pilling and has good fastness to water.

POLYSILK CTE physical properties:

- pH (20°C): 9.00 – 11.00
- Density (20°C): 1.00 – 1.10 g/ml
- Dry matter: 44.00 – 48.00%

Enzyme

Enzymes are molecules that accelerate, or catalyse, chemical reactions. In this case, enzymes accelerate the decomposition process of the fibres.

Enzymes transform the products into biodegradable substances and they have a clear application.

The next picture present the effect of fabric hairiness treated with enzymes (bio-polishing):

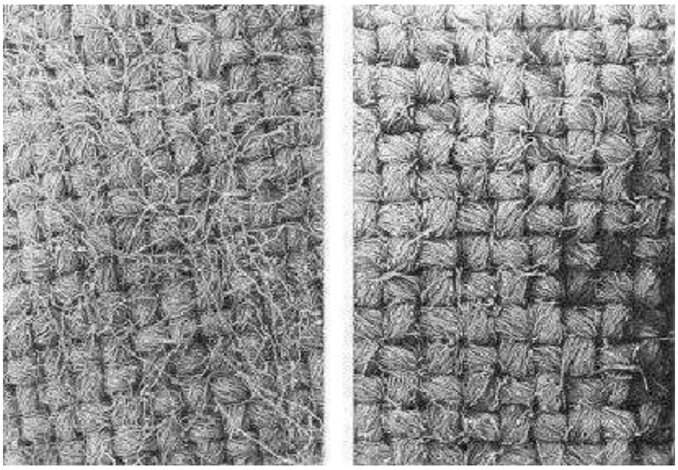


Figure 19. Effects of the bio-polishing[11]

The application conditions of enzyme are:

- Application by exhaustion
- Amount 0,5 – 2% o.w.f to pH 4-5
- Temperature 55 - 60°C

POLZYME – EA 50

POLZYME – EA 50 is an acid cellulase for finishing treatment of articles including cellulose fibres. It is soluble in water. This enzyme reduces the tendency to pilling formation.

POLZYME – EA 50 increase the softness effect in the fabric.

POLZYME – EA 50 physical properties:

- pH (20°C): 4.00 – 6.00
- Density (20°C): 1.00 – 1.10 g/ml

[11] F. E. Lockuán Lavado, La industria textil y su control de calidad, Chapter VI Ennoblecimiento textile, page 38 of 82.

3. First laboratory scale finishing with textile additives (preliminary trials from action B1)

Finishing trials have been performed on blended cotton/polyester fabric with the textile auxiliary products detailed previously. The process parameters such as the application method and the auxiliary concentration have been studied. Then, the touch of the fabric has been evaluated. These trials aim to define the best process parameters for the application of the textile additives on the fabrics. The hairiness of these treated fabrics will be analyzed in the implementation action B1 to determine which finishing is more prone to reduce the microfibers formation.
Two different application techniques have been employed in this study and are detailed next:

Padding
The padding process consists of impregnating the fabric into a solution composed of the textile auxiliary agent. Then, the fabric is squeezed between two rolls with a controlled pressure, defining the absorption or pick up of the textile. Finally, the fabric is dried and then cured at a controlled temperature and during a determined period of time.

Figure 20. Laboratory padding machine (Source: LEITAT)

Exhaustion
The exhaustion process consists of immersing the fabric into a solution containing the textile auxiliary during a determined period of time and under temperature. The concentration of textile auxiliary employed is usually lower than for the padding method as the fabric is immersed in the solution during a longer period of time and under heat. The quantity of product employed is measured according to the weight of the fabric. This process usually doesn't require a final curing process. The laboratory machine employed to realize the exhaustion process is presented next:

Figure 21. Laboratory exhaustion machine (Source: Ugolini)

The next table presents the different finishing trials done at laboratory scale:

Products/Formula	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Units	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l	*%	*%	g/l	g/l	g/l	g/l
PURLASTIC 8139	10	20	10	---	10	---	---	---	---	---	---	---	---	---	---	---	---	---
POLYACRIL 73	---	---	---	---	---	---	---	---	10	20	10	10	---	---	---	---	---	---
POLYACRIL 97	---	---	---	---	---	---	---	---	---	---	---	---	---	---	10	20	10	10
POLYCROSS MLC	---	---	---	---	---	---	5	10	---	---	---	---	---	---	---	---	---	---
POLYSILK CTE	---	---	2	5	---	---	---	---	---	---	2	---	---	---	---	---	2	---
POLYSILK MC	---	---	---		2	5	---	---	---	---	---	2	---	---	---	---	---	2
POLZYME EA 50	---	---	---		---	---	---	---	---	---	---	---	1	2	---	---	---	---
Absorption (%)	60	68	63	58	60	55	63	42	63	68	61	61	---	---	61	67	65	61
Touch	✓	✓	✓	✓	✓	✓	✗	✗	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

Table 3. Description of the first finishing trials. Preliminary studies for the action B1.

The last two rows of the table represent the fabric absorption or pick-up value (in %) and the final touch of the fabric. As it can be seen in this table two formulations (7 and 8) gave an unacceptable touch. The pick up formula is described next:

$$Pick-up\ (\%) = \frac{Pfinal - Pinitial}{Pinitial} \times 100$$

Where:

Pfinal is the weight of the wet fabric after padding immersion and *Pinitial* is the weight of the dry fabric before immersing the fabric in the solution.

The application conditions have been the following:

Samples 1 to 12 and 15 to 18:

Application technique	Padding

Additive concentration value	Measured according the bath volume
Drying temperature	110ºC
Curing temperature	160ºC
Curing time	1 minute 30 seconds

Table 4. Application condition of samples 1 to 12 and 15 to 18

Samples 14 and 15:

Application technique	Exhaustion
Additive concentration value	Measured according the weight of the fabric
Process temperature	55-60ºC
Process time	30 – 45 minutes
pH	4 – 5

Table 5. Application condition of samples 14 and 15

Conclusions

As it can be observed, the application parameters have to be studied in detail in order to do not affect the touch of the fabric. Indeed, these products have the advantage to create a protective layer on the fabric, but due to their polymeric nature their can reduce the softness of the fabric once they are cured. The time and curing temperatures can be adjusted in the implementation action in order to maintain a good fabric hand and softness. The textiles treated by exhaustion have a better touch as the treatment temperature and the auxiliary concentration are lower.

TASK A.2.1.3. Influence of the washing conditions.

1. Introduction

One of the objectives of the project is to take advantage of textile auxiliaries products to apply them on fabrics' surface and reduce the formation of microfibers. The textile auxiliaries (studied in the previous Task A.2.1.2) will be applied on the selected fabrics for the study, during the implementation actions. The application method will consist of impregnating the fabric by padding into the textile finishing by padding.

Another objective of this project is to obtain a synergistic effect between the textile finishing of the fabrics with auxiliaries and the washing of the treated textiles with detergent additives susceptible to reduce the microfibers formation. To obtain a synergistic effect between both treatments (textile finishing and washing with specialized detergent additives) we have to ensure the compatibility of the textile auxiliaries with the washing conditions.

So, the aim of this part of the report is to study the washing conditions used in domestic washing machines and confirm that they will not affect the effectiveness or durability of the textile finishing previously applied on the fabrics by padding.

2. Definition of the textile finishing conditions

The finishing of the textiles can be made by using different application methods and each method will have different process parameters. The application methods evaluated in this part are the padding and the exhaustion technique.

The washing conditions varying according to each washing program and which should be considered for the study of their compatibility with the textile finishing process are detailed in the following table. These conditions are an example from MIELE washing machines.

Composition	Temperature	Loaded garment weight	Water consumption (in Litres)	Program information	
				Short program	Normal program
Cotton	95 ºC	7.0 kg	55	-	2 h 19 min
	60 ºC	7.0 kg	55		2 h 09 min
	60 ºC	3.5 kg	45	1 h 12 min	
	40 ºC	7.0 kg	68		2 h 09 min
	40 ºC	3.5 kg	45	59 min	
Synthetic / blends	40ºC	3.5 kg	50		1 h 29 min
Synthetic	30ºC	2.0 kg	69	49 min	59 min
Wool	30ºC	2.0 kg	39	-	39 min
Express 20	40ºC	3.5 kg	30	20 min	-
Automatic	40ºC	5.0 kg	62	-	1 h 27 min

Plus					
Jeans	40ºC	3.0 kg	52	1 h 02 min	1 h 12 min
Intensive Plus	60ºC	7.0 kg	72	-	2 h 47 min

Table 6. Example of washing machine program (MIELE, automatic washing machine W 1914)

The process parameters which are considered for the finishing of the fabrics are the auxiliary concentration, the bath conditions (ratio, pH and temperature), the process time and temperature and the fabric absorption (pick-up). The parameters which may be considered during laundry to avoid the degradation of the finishing are detailed next:

Washing temperature

The auxiliaries applied during the finishing of the textile are effective in different conditions, such as the temperature. At the end of the finishing process a relatively high temperature is applied to fix the product on the fabric surface. This temperature can reach 170ºC. Highest temperature could degrade the fabric or the auxiliary applied on the fabric. Most of the textile auxiliaries studied in the previous *Task A2.1.2 Study of textile auxiliaries* (polyurethane, acrylic resin, blend of polyurethane with acrylic resin, macro-emulsions of silicone) is stable until 170ºC. The washing temperature will never exceed 170ºC, so the washing temperature is not a problem for these auxiliaries. However, the enzymes used for bio-polishing of the fabrics are stable at a specific temperature, generally not exceeding 60ºC. Most of the washing program will not exceed the washing temperature of 60ºC. Indeed, the most common laundry temperatures are 30ºC and 40ºC, and sometimes 60ºC. Washing programs of cotton at 95ºC exist but should not present any risk for the enzymatic treatment of the fabric since the enzymes are deactivated after bio-finishing.

Water pH

The water pH employed during laundry could be considered as well as it could affect the finishing applied on the fabric. However, most of the textile auxiliary selected for the treatment of the fabric are compatible with water pH between 6 and 8.

Washing time

The washing is not decisive parameters for the durability and effectiveness of the previously applied finishing treatment. However, it should be taken into account that combined with temperature; the washing fastness of the textile finishing applied will be reduced. At the highest temperature and highest washing time the finishing degradation will be faster. Indeed, few washing cycles will be necessary to eliminate the auxiliary finishing layer from the fabric surface at 95ºC during 2 hours and 19 minutes, for example (cotton washing program).

3. Compatibility study of the textile finishing behaviour at the domestic washing conditions

Another solution which will be considered in this project is to realize the finishing process with textile auxiliaries directly inside the washing machine, during laundry. Then, the textile additives and the detergent additives will be applied together, in one step. In this case, the compatibility of the textile finishing agent with the washing process will be more restricted. Then, the washing conditions to be taken into account to ensure the fixation of the textile additives to the fabric are described next:

Textile auxiliary concentration and bath ratio

In washing machine, the concentration of the textile finishing agent should be adjusted to the concentration specified for padding or exhaustion application. According to the preliminary finishing application realised in this action (see previous task), the concentration of the polymeric additives should be between 10 and 20 g/L to produce a protective layer on the fabric (this concentration will be optimized in action B1 after characterization of the hairiness before and after laundry and pilling tests). According to the different washing program available in MIELE washing machine (see previous table), the maximum water consumption is 72 litres, which will correspond to a quantity of 720 g to 1440 g of textile auxiliary.

Bath temperature

The bath temperature is required for the exhaustion process but not for the padding finishing process. The finishing of the fabric using the polymeric additives and with temperature will not affect the effectiveness of the treatment. So, the finishing of the textile additives different from the enzymes can be made at the indicated temperature for each washing program. However, the washing program temperature for the treatments with enzymes should not exceed 60ºC and should be more than 40ºC. According to the previous washing conditions example from MIELE, only cotton program could be employed for the bio-finishing of textiles during laundry.

Bath pH

As indicated in the preliminary application, the bio-polishing of the textiles required a specified pH (usually pH 4-5). This will complicate the bio-polishing of the fabric during laundry. However, to avoid this problem, enzymes actuating at higher pH (at tap water pH) could be identified. Moreover, the activity of the enzymes at higher pH may be reduced but still active.

Process time

The laundry time should be at least higher than the finishing time (only for exhaustion process). For finishing done by padding the impregnation time is short (few seconds in the bath) so highest process time in washing machine will not reduce the effectiveness of the finishing. Moreover, the additive concentration could be reduced. The laundry time as to be higher than of 30 minutes for finishing processes done by exhaustion (in this case bio-finishing). Most of the washing programs answer this requirement except the Express 20 program, done in 20 minutes.

Drying and polymerization of the fabric

The padding process required a post treatment which is the drying and the curing of the fabric. The curing process is the most problematic as it should be done at least at 160ºC for some polymeric auxiliaries. To solve this problem, the washed garment could be dried in a drying machine and then "cured" by ironing it at the highest temperature suitable for each garment composition during at least 1 minute and 30 seconds. The minimum ironing temperature for each textile auxiliaries will be studied in the implementation action B1.

4. Conclusions

The analysis made in this task has permitted to determine 1) the laundry conditions which could affect the effectiveness and the durability of the textile auxiliary finishing and 2) the washing machine programs most suitable for a finishing of the garment during the laundry process.

1) Laundry conditions:

The best laundry conditions to maintain the durability and effectiveness of the finished textiles with textile additives are low washing temperatures and short programs' time. High temperature and long washing time will eliminate the finishing layer of the fabric earlier.

2) Washing machine programs:

In order to realize the finishing of the garments in the washing machine, the laundry conditions have to be compatible with the auxiliary properties. The polymeric additives should be submitted to air drying o machine drying and the ironed at a specified temperature to ensure the polymerization of the additives on the fabric surface. Ironing up to 160ºC will permit to create a polymeric protective layer on the fabric.

For enzymatic finishing, most of the existing washing program could be used to realize the bio-polishing in washing machine, verifying that the temperature won't exceed 60ºC and the washing time will last at least 30 minutes. The most critical parameters is to identify enzymatic formulation operating or with constant activity at pH 6 to 8, equivalent to household water pH.

Name of the deliverable:

Analysis of the regulatory framework and voluntary schemes concerning microplastics release by laundry processes [1]

Number of the associated action: A3

Involved partners: LEITAT, PSF

(27/03/2015)

LIFE13 ENV/IT/001069

[1] The title of this Deliverable has been agreed to be modified from the project proposal, where appeared as "Stakeholders interviews". This change has been performed in order to be more in line with the content of the action A3.

Mitigation of microplastics impact caused by textile washing processes

Contents

1. Introduction

This deliverable is intended to provide an **analysis of the regulatory framework** (including a core list of policies and standards), together with a SWOT analysis, on which policy recommendations will be set in Action B6 of the MERMAIDS project. It involves the review of the regulatory framework concerning micro/nanoplastics pollution, detergents, additives, Ecolabels and washing machines, among other related issues, in order to detect what regulatory and non regulatory framework address microplastics (directly and indirectly), and in order to detect related strengths, weaknesses, opportunities and threats on which policy recommendations will be set in action B6.

2. Methodology

2.1. Literature review

The current regulatory framework - as well as some voluntary schemes- have been analyzed by taken into account the theoretical context on which the project is framed. The analysis has been based on the following principles: (a) What materials make up microplastic litter and (b) What key words should be selected?

a. Materials that make up microplastic litter[2]

Polymers:

The main component of most microplastic particles is **synthetic polymer**(s). Polymers are synthesized either by joining monomer units to form a polymer, e.g. nylon, or by creating a free radical monomer, which by a chain reaction quickly produces a long chain polymer, e.g.polyvinyl chloride (Bolgar et al. 2008). The plastics with the highest production volumes - polyethylene, polypropylene, polyvinylchloride, polystyrene and polyethylene terephthalate - together supply 75% of the demand for plastics in Europe (Anon. 2011).

List of **commonly produced plastic polymers** (Anon. 2011).

Polypropylene (PP)	Acrylonitrile butadiene styrene (ABS)	Polyamides (PA) (Nylons)
Polystyrene (PS)	Polyethylene terephthalate (PET)	Polyvinyl chloride (PVC)
High impact polystyrene (HIPS)	Polyester (PES)	Polyurethanes (PU)
Polycarbonate (PC)	Polyethylene (PE)	Polycarbonate/Acrylonitrile
Polyvinylidene chloride (PVDC) (Saran)		Butadiene Styrene (PC/ABS)

[2] Leslie, H.A., M.D van der Meulen, F.M. Kleissen A.D. Vethaak (2011) Microplastic Litter in the Dutch Marine Environment. Providing facts and analysis for Dutch policymakers concerned with marine microplastic litter. Deltares, report 1203772-000.

Additives:

The polymers in plastics are almost never pure. Plastics can be regarded as a cocktail of polymers combined with different additives. By way of a 'compounding' process, additives give the plastic product a variety of desirable properties. Additives include plasticizers that make plastics flexible and durable, flame retardants, surfactants, additives that enhance resistance to oxidation, UV radiation and high temperatures, modifiers to improve resistance to breakage, pigments, dispergents, lubricants, antistatics, nanoparticles or nanofibres, inert fillers, biocides, and even fragrances.

Besides additives, **other chemicals such as auxiliary substances** (catalysts of polymerization, initiators and accelerators) are used and may be emitted during the plastics production process (Mulder 1998).8

Additives need to be considered part of the potential ecological impact of microplastics due to their sheer production volumes and the known or suspected toxicity of many of these substances.

Sometimes additives are already added to preproduction pellets, but other additives may be added after that stage, when the plastic is being processed into the end product. The additives in polymers can leach out of plastics at various points during the life cycle of the product (e.g. Sajiki & Yonekubo 2003). This can amount to large emissions of chemical additive leachates downstream in the plastic use chain, which may cause toxicity to aquatic life (Lithner et al. 2009).

b. Selection of key words

Taking into consideration all the information mentioned above, the following key words have been considered for the analysis of the regulatory framework carried out:

- *Microplastics*
- *Microparticles*
- *Plastics*
- *Microfibers*
- *Textile fibers / release / fiber loss / fragmentation*
- *Synthetic polymer*
- *Monomer*
- *Polypropylene (PP)*
- *Polystyrene (PS)*
- *High impact polystyrene (HIPS)*
- *Polycarbonate (PC)*
- *Plyvinylidene chloride (PVDC) (Saran)*
- *Acrylonitrile butadiene styrene (ABS)*
- *Polyethylene terephthalate (PET)*

- *Polyester (PES)*
- *Polyethylene (PE)*
- *Polyamides (PA) (Nylons),*
- *Polyvinyl chloride (PVC)*
- *Plyurethanes (PU)*
- *Polycarbonate/Acrylonitrile*
- *Butadiene Styrene (PC/ABS)*
- *Additives*
- *Other chemicals such as auxiliary substances (catalysts of polymerization, initiators and accelerators)*
- *Water*

The following template has been used for the analysis of each piece of regulation/policy:

PIECE OF REGULATION / POLICY	
Link	
General objective	
TOPIC*	
Does it address "microplastics"?	
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed.	

* TOPIC: Detergents, Textile, Water, Wastewaters, marine environment.

** Plastics; microparticles; microfibers /textile fibers / release / fiber loss / fragmentation; laundry; washing; synthetic polymer; monomer; polypropylene (PP); polystyrene (PS); High impact polystyrene (HIPS); Polycarbonate (PC); Plyvinylidene chloride (PVDC) (Saran); Acrylonitrile butadiene styrene (ABS); polyethylene terephthalate (PET); Polyester (PES); Polyethylene (PE); Polyamides (PA) (Nylons); Polyvinyl chloride (PVC); Plyurethanes (PU); Polycarbonate/Acrylonitrile; Butadiene Styrene (PC/ABS); additives; other chemicals such as auxiliary substances (catalysts of polymerization, initiators and accelerators).

The analysis of the voluntary schemes regarding BREF and Ecolabel has been carried out with a separate approach. The review has focused on BREF and Ecolabel related to textile and laundry detergents and on the identification of those criteria on which the potential updating from the project results will be directed. The template used for Ecolabel has been the following:

Type of Ecolabel **Validity date:** ***Title***
Criteria related to the principles explained above **…**
…
…

2.2. Stakeholders interviews/consultations

In order to complete the information obtained from the analysisand in order not to disregard any relevant matters, several interviews and specific consultations to public and private relevant bodies, stakeholders, organisms, SMEs, manufacturers, environmental agents and experts on micro plastic and on industrial laundry issues have been carried out. Firstly, a careful identification has been done of the most relevant agents of the scientific/industrial/legislative scenario. These have been classified in three different profiles:

- Maritime
- Textile
- Laundry products

The outlined list of stakeholders is the following, taking into account that it remains open to be extended along the project:

RELEVANT STAKEHOLDERS
KIMO
Plymouth University
CEFAS
IFREMER
Plastics Europe Nederland industry
The North Sea Foundation (Noordzee)

DELTARES
VU UNIVERSITY
Mark Browne (University of California)
IVM research
Ministry of Environment the Netherlands
Rijkswaterstaat Zee en Delta (OSPAR delegation NL, Chair EIHA)
OCEANA
PTEPA (Spanish Technological platform for fisheries and aquaculture)
PROTECMA (Spanish Technological platform for the coastal and marine protection)
Euratex
The European Technology Platform for the Future of Textiles and Clothing
AEI – Agrupació d'Empreses Innovadores Tèxtils
TEXFOR - Confederación de la Industria Textil
Consejo Intertextil Español (CIE)
Agrupación española del género de punto (AEGP)
Association Internationale de la Savonnerie, de la Détergence et des Produits d'Entretien (AISE)
Comité Européen des Agents de Surface et leur Intermédiaires Organiques - European Committee of Organic Surfactants and their Intermediates (CESIO)
European Centre of Studies on LAB/LAS (ECOSOL)
ADELMA (Asociación de Empresas de Detergentes y Productos de Limpieza)

Three types of questionnaires have been elaborated and distributed among the stakeholders identified. Specific questions have been addressed by type of expert taking into account the differences in knowledge and expertise. They address the following issues, among others:

- Textile and Clothing manufacturing and management.
- Microplastic marine environment effects knowledge.
- Regulatory framework and policy recommendations.
- Prevention and mitigation technologies and methodologies in order to avoid plastic marine pollution.
- Socioeconomic impacts of plastic garments.
- Environmental impacts on marine biota.
- Human risk once the micro plastic particles are introduced into the food chain.

The templates of these questionnaires are enclosed in Annex I of this deliverable.

A reasonable number of questionnaires have been received so far, and more of them are expected to be obtained in the future. For that reason, it has been decided that the final conclusions from questionnaires will be gathered further on during the project.

3. Analysis of the regulatory framework and voluntary schemes

3.1. Regulatory framework

This chapter includes the main findings per policy/program/normative analyzed.

PIECE OF REGULATION / POLICY	**MARINE STRATEGY FRAMEWORK DIRECTIVE (MSFD) DIRECTIVE 2008/56/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 17 June 2008 establishing a framework for community action in the field of marine Environmental policy**
Link	http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32008L0056&from=EN
General objective	The Marine Strategy Framework Directive aims to achieve Good Environmental Status (GES) of the EU's marine waters by 2020 and to protect the resource base upon which marine-related economic and social activities depend.
TOPIC*	Marine environment
Does it address "microplastics"?	YES (even though without mentioning the word "microplastics").
How does it address microplastics?	It doesn't mention "microplastics" throughout the document but addressing "marine litter" the Directive includes "microplastics" (as shown in *COMMISSION DECISION of 1 September 2010 on criteria and methodological standards on good environmental status of marine waters*, addressed below).
Does it address other key issues related to the release of microplastics caused by textile washing processes?*	No, even though at a general level it fits into the objectives of the MSFD.
How does it address other issues?	The MSFD establishes a framework within which Member States shall take the necessary measures to achieve or maintain good environmental status in the marine environment by the year 2020 at the latest.
Indicate the most relevant sections in which "microplastics" could be addressed	According to some policy-experts, there is already a discussion at EU level related to the inclusion of microplastics in MSFD article 15.

PIECE OF REGULATION / POLICY	MARINE STRATEGY FRAMEWORK DIRECTIVE (MSFD) COMMISSION DECISION of 1 September 2010 on criteria and methodological standards on good environmental status of marine waters
Link	http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32010D0477%2801%29&from=EN
General objective	The Marine Strategy Framework Directive aims to achieve Good Environmental Status (GES) of the EU's marine waters by 2020 and to protect the resource base upon which marine-related economic and social activities depend.
TOPIC*	Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	Part B. Criteria for good environmental status relevant to the descriptors of Annex I to Directive 2008/56/EC: Descriptor 10. Trends in the amount, distribution and, where possible, composition of micro-particles (in particular micro- plastics) (10.1.3)
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	10.1.3 MSFD could address specific sources of microplastic pollution.

PIECE OF REGULATION / POLICY	WATER FRAMEWORK DIRECTIVE DIRECTIVE 2000/60/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 23 October 2000 establishing a framework for Community action in the field of water policy
Link	http://eur-lex.europa.eu/resource.html?uri=cellar:5c835afb-2ec6-4577-bdf8-756d3d694eeb.0004.02/DOC_1&format=PDF List of priority substances: http://ec.europa.eu/environment/water/water-framework/priority_substances.htm
General objective	The WFD aims to establish a framework for the protection of inland surface waters, transitional waters, coastal waters and groundwater which, among others (a) prevents further deterioration and protects and enhances the status of aquatic ecosystems, (b) aims at enhanced protection and improvement of the aquatic environment, inter alia, through specific measures for the progressive reduction of discharges, emissions and losses of priority substances and the cessation of phasing out of discharges, emissions and losses of the priority hazardous substances, (c)the protection of territorial and marine waters and (d) to cease or phase out discharges, emissions and losses of priority hazardous substances, with the ultimate aim of achieving concentrations in the marine environment near background values for naturally occurring substances and close to zero for man-made synthetic substances.

TOPIC*	Water
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO, even though at a general level it fits into the objectives of the WFD.
How does it address other issues?	The WFD indicates to be based on the precautionary principle and on the principles that preventive action should be taken, environmental damage should, as a priority, be rectified at source and that the polluter should pay.
Indicate the most relevant sections in which "microplastics" could be addressed	In general throughout the WFD & in the *list of priority substances*. Depending on the evolution of the research carried out involving microplastics, the inclusion in the *list of priority hazardous substances* should also be taken into consideration.

PIECE OF REGULATION / POLICY	**International Convention for the Prevention of Pollution from Ships (MARPOL)**
Link	http://www.imo.org/About/Conventions/ListOfConventions/Pages/International-Convention-for-the-Prevention-of-Pollution-from-Ships-%28MARPOL%29.aspx
General objective	The International Convention for the Prevention of Pollution from Ships (MARPOL) is the main international convention covering prevention of pollution of the marine environment by ships from operational or accidental causes.
TOPIC*	Marine environment
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	YES
How does it address other issues?	Annex IV mentions laundry and shower drainage as part of "grey water" (both vectors for microplastic pollution). Annex V prohibits the discharge of all garbage into the sea which are related to food waste, cargo residues, cleaning agents and additives and animal carcasses. It also includes other garbage including plastics and domestic waste.
Indicate the most relevant sections in which "microplastics" could be addressed	Annex IV - Prevention of Pollution by Sewage from Ships. Annex V - Prevention of Pollution by Garbage from Ships.

PIECE OF REGULATION / POLICY	Global Programme of Action for the Protection of the Marine Environment from Land-based Activities
Link	GPA: http://www.gpa.unep.org/ Global Partnership on Marine Litter (GPML): http://unep.org/gpa/gpml/issue.asp
General objective	The **Global Programme of Action for the Protection from the Marine Environment from Land-based Activities (GPA)** is the only global intergovernmental mechanism directly addressing the connectivity between terrestrial, freshwater, coastal and marine ecosystems. It aims to be a source of conceptual and practical guidance to be drawn upon by national and/or regional authorities for devising and implementing sustained action to prevent, reduce, control and/or eliminate marine degradation from land-based activities. The **Global Partnership on Marine Litter (GPML)** is a voluntary multistakeholder coordination mechanism in which all partners agree to work together to further reduce and better manage marine litter. Among its objectives are, to enhance international cooperation and coordination through the promotion and implementation of the Honolulu Strategy - a global framework for the prevention and management of marine debris, as well as the Honolulu Commitment – a multi-stakeholder pledge.
TOPIC*	Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	In June 2014, governments attending the first UN Environment Assembly noted with concern the impacts of plastics and microplastics on the marine environment, fisheries, tourism and development calling for strengthened action, in particular by addressing such materials at the source. A resolution (addressed below in this document) was adopted calling for the strengthening of information exchange mechanisms, requesting UNEP to present scientific assessments on microplastics for consideration by the next session of the Assembly.
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	

PIECE OF REGULATION / POLICY	Honolulu commitment & strategy A Global Framework for Prevention and Management of Marine Debris
Link	Honolulu commitment: http://www.marinelitternetwork.org/honolulu-commitment Honolulu strategy: http://www.marinelitternetwork.org/page/honolulu-strategy-global-framework-prevention-and-management-marine-debris

	During this review, the Honolulu strategy was not available for consultation thus, the sections that follow refer to the Honolulu commitment.
General objective	The Honolulu Commitment (HC) invites international organizations, governments at national and sub-national levels, industry, non-governmental organizations, citizens and other stakeholders, to commit to: 1. Make choices that reduce waste in order to halt and reverse the occurrence of marine debris. 2. Encourage all citizens, industry and governments to take responsibility for their contribution and find solutions to the marine debris problem; 3. Share openly and freely technical, legal, policy, community-based and economic / market-based solutions that will help prevent, reduce and manage marine debris; 4. Advocate mechanisms that emphasise the prevention or minimisation of waste; 5. Facilitate initiatives that turn waste into a resource in an environmentally sustainable manner; 6. Develop global, regional, national and local targets to reduce marine debris; 7. Improve global knowledge, understanding and monitoring of the scale, nature, source and impact of marine debris, and raise awareness of its impact on public health, biodiversity and economic development; 8. Collaborate with global, regional and sub-regional organisations, to enhance the effectiveness of multi-lateral initiatives aimed at preventing, reducing and managing marine debris; 9. Encourage financial support for global, regional, national and local actions that contribute to the implementation of the Honolulu Strategy; 10. Encourage relevant intergovernmental fora, including those at global and regional scales, to express support for the HC and encourage governments to take action consistent with the objectives and strategic activities outlined in the Honolulu Strategy; and 11. Participate in a global network of stakeholders committed to understanding, preventing, reducing and managing marine debris in an environmentally sustainable manner; 12. Contribute to the development and successful implementation of the Honolulu Strategy – a framework for the prevention, reduction and management of marine debris – and its periodic review.
TOPIC*	Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	The HC considered marine debris to include any anthropogenic, manufactured or processed solid material, irrespective of its size, discarded, disposed of or abandoned in the environment, including all materials discarded into the sea, on the shore, or brought indirectly to the sea by rivers, sewage, storm water or winds;

	The HC also welcomed the ongoing work of scientists, research organisations and other citizens to better and more accurately understand the sources, nature and extent of marine debris, including the effects of micro-plastics, heavy metals, persistent organic pollutants, endocrine disruptors and other chemicals on marine biodiversity and public health;
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO, even though at a general level it fits into the objectives of the Honolulu Commitment above-mentioned.
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	

PIECE OF REGULATION / POLICY	**UNEA resolution on plastics and microplastics**
Link	http://www.unep.org/unea/UNEA_Resolutions.asp
General objective	This resolution was adopted calling for the strengthening of information exchange mechanisms, requesting UNEP to present scientific assessments on microplastics for consideration by the next session of the Assembly.
TOPIC*	Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	Resolution 1/6. Marine plastic debris and microplastics
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	YES
How does it address other issues?	Recognizes that microplastics in the marine environment originate from a wide range of sources, including the breakdown of plastic debris in the oceans, industrial emissions and sewage and run-off from the use of products containing microplastics;
Indicate the most relevant sections in which "microplastics" could be addressed	

PIECE OF REGULATION / POLICY	**United Nations Convention on the Law of the Sea (UNCLOS)**
Link	http://www.un.org/depts/los/convention_agreements/convention_overview_convention.htm
General objective	The United Nations Convention on the Law of the Sea lays down a comprehensive regime of law and order in the world's oceans and seas establishing rules governing all uses of the oceans and their resources. It enshrines the notion that all problems of

	ocean space are closely interrelated and need to be addressed as a whole.
TOPIC*	Marine environment
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO, even though at a general level it fits into the objectives of UNCLOS.
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	Section 5, International rules and national legislation to prevent, reduce and control pollution of the marine environment (Article 207. Pollution from land-based sources and Article 211. Pollution from vessels).

PIECE OF REGULATION / POLICY	**Convention on the Prevention of Marine Pollution by Dumping of Wastes and Other Matter (London Convention and London Protocol)**
Link	http://www.imo.org/About/Conventions/ListOfConventions/Pages/Convention-on-the-Prevention-of-Marine-Pollution-by-Dumping-of-Wastes-and-Other-Matter.aspx
General objective	To protect the marine environment from human activities. Its objective is to promote the effective control of all sources of marine pollution and to take all practicable steps to prevent pollution of the sea by dumping of wastes and other matter. The **London Convention** contributes to the international control and prevention of marine pollution by prohibiting the dumping of certain hazardous materials. The **London Protocol** represents a major change of approach to the question of how to regulate the use of the sea as a depository for waste materials. Rather than stating which materials may not be dumped, it prohibits all dumping, except for possibly acceptable wastes on the so-called "reverse list", contained in an annex to the Protocol.
TOPIC*	Marine environment
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO, even though at a general level it fits into the objectives of London Convention and London Protocol.
How does it address other issues?	

Indicate the most relevant sections in which "microplastics" could be addressed	The London Protocol stresses the "precautionary approach", which requires that "appropriate preventative measures are taken when there is reason to believe that wastes or other matter introduced into the marine environment are likely to cause harm even when there is no conclusive evidence to prove a causal relation between inputs and their effects".

PIECE OF REGULATION / POLICY	**DIRECTIVE 2000/59/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 27 November 2000 on port reception facilities for ship-generated waste and cargo residues**
Link	http://eur-lex.europa.eu/resource.html?uri=cellar:15945efb-a7e8-4840-ab4d-0535f12692a8.0004.02/DOC_1&format=PDF
General objective	To reduce the discharges of ship-generated waste and cargo residues into the sea, especially illegal discharges, from ships using ports in the Community, by improving the availability and use of port reception facilities for ship-generated waste and cargo residues, thereby enhancing the protection of the marine environment.
TOPIC*	Marine environment
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	No, even though at a general level it fits into the objectives of the Directive.
How does it address other issues?	'ship-generated waste' shall mean all waste, including sewage, and residues other than cargo residues, which are generated during the service of a ship and fall under the scope of Annexes I, IV and V to Marpol 73/78 and cargo-associated waste as defined in the Guidelines for the implementation of Annex V to Marpol 73/78;
Indicate the most relevant sections in which "microplastics" could be addressed	Address "Microplastics from laundry wastewater" un der section "type and amount of waste and residues to be delivered and/or remaining on board, and percentage of maximum storage capacity.

PIECE OF REGULATION / POLICY	**Directive 2010/75/EU of The European Parliament and of The Council on industrial emissions**
Link	http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2010:334:0017:0119:en:PDF
General objective	Integrated prevention and control of pollution arising from industrial activities
TOPIC*	Wastewaters, textile, detergents
Does it address "microplastics"?	NO
How does it address microplastics?	NO directly, but it adresses general water pollution
Does it address other key issues related to the release of	YES

microplastics caused by textile washing processes?**	
How does it address other issues?	Chemicals and textile industry are listed in Annex I; microplastics are not addressed as 'micro' in Annex II (water)
Indicate the most relevant sections in which "microplastics" could be addressed	Chapter II (art.10, 15); Chapter V. Annex I, Annex II.

PIECE OF REGULATION / POLICY	**Regulation (EU) No 1007/2011 of the European Parliament and of the Council of 27 September 2011 on textile fibre names and related labelling and marking of the fibre composition of textile products and repealing Council Directive 73/44/EEC and Directives 96/73/EC and 2008/121/EC of the European Parliament and of the Council Text with EEA relevance**
Link	http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32011R1007&from=EN
General objective	This Regulation lays down rules concerning the use of textile fibre names and related labelling and marking of fibre composition of textile products, rules concerning the labelling or marking of textile products containing non-textile parts of animal origin and rules concerning the determination of the fibre composition of textile products by quantitative analysis of binary and ternary textile fibre mixtures, with a view to improving the functioning of the internal market and to providing accurate information to consumers.
TOPIC*	Textile
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	

PIECE OF REGULATION / POLICY	**COUNCIL DIRECTIVE of 21 May 1991 concerning urban waste water treatment (9 1 /271 /EEC)**
Link	http://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:31991L0271&from=EN
General objective	Its objective is to protect the environment from the adverse effects of urban waste water discharges and discharges from certain industrial sectors (see Annex III of the Directive) and concerns the collection, treatment and discharge of: Domestic waste water; Mixture of waste water; Waste water from certain industrial sectors (see Annex III of the Directive)

TOPIC*	Wastewaters
Does it address "microplastics"?	NO
How does it address microplastics?	
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO. It addresses other industrial sectors different than textile, but this could be included
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	Art. 4, 5. Annex I, Annex III Microplastics can be addressed as a hazardous substance.

PIECE OF REGULATION / POLICY	**DIRECTIVE 2008/1/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 15 January 2008 concerning integrated pollution prevention and control**
Link	http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2008:024:0008:0029:en:PDF
General objective	The purpose of this Directive is to achieve integrated prevention and control of pollution arising from the activities listed in Annex I. It lays down measures designed to prevent or, where that is not practicable, to reduce emissions in the air, water and land from the abovementioned activities, including measures concerning waste, in order to achieve a high level of protection of the environment taken as a whole, without prejudice to Directive 85/337/EEC and other relevant Community provisions.
TOPIC*	Wastewaters, Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	The Directive highlights the importance of the use of BAT and environmental quality standards. When mentioning the main polluting substances in water environment, it includes 'Persistent hydrocarbons and persistent and bioaccumulable organic toxic substances' (Annex III on the main polluting substances, water section)
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	Annex III

A part from the above mentioned pieces of legislation and/or policies, it is also important to consider that **Regional Sea Conventions** in the Baltic (HELCOM), Mediterranean (UNEP/MAP) and North (OSPAR) Seas also address microplastics:

REGIONAL SEA CONVENTION	HELCOM
Link	http://helcom.fi/
General objective	HELCOM (Baltic Marine Environment Protection Commission - Helsinki Commission) is the governing body of the Convention on the Protection of the Marine Environment of the Baltic Sea Area, known as the Helsinki Convention .
TOPIC*	Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	Marine litter, which are not currently covered in the COMBINE manual, is included in the revision of the strategy.
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	NO
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	Could be included as "mandatory" under HELCOM monitoring activities (COMBINE).

REGIONAL SEA CONVENTION	Mediterranean Action Plan (MAP) – Barcelona Convention
Link	http://www.unepmap.org/index.php?module=content2&catid=001001004
General objective	The Barcelona Convention's main objectives are: (1) to assess and control marine pollution; (2) to ensure sustainable management of natural marine and coastal resources; (3) to integrate the environment in social and economic development; (4) to protect the marine environment and coastal zones through prevention and reduction of pollution, and as far as possible, elimination of pollution, whether land or sea-based; (5) to protect the natural and cultural heritage; (6) to strengthen solidarity among Mediterranean coastal States; and (7) to contribute to improvement of the quality of life.
TOPIC*	Marine environment
Does it address "microplastics"?	YES

How does it address microplastics?	Microplastics are included in the draft Monitoring and assessment methodological Guidance Document
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	Could be included as "mandatory" under BARCELONA CONVENTION monitoring activities.

REGIONAL SEA CONVENTION	**OSPAR**
Link	http://www.ospar.org/
General objective	OSPAR is the mechanism by which fifteen Governments of the western coasts and catchments of Europe, together with the European Union, cooperate to protect the marine environment of the North-East Atlantic.
TOPIC*	Marine environment
Does it address "microplastics"?	YES
How does it address microplastics?	OSPAR Regional Action Plan (RAP). OSPAR has identified microplastics as a non-prioritized candidate indicator.
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	
How does it address other issues?	
Indicate the most relevant sections in which "microplastics" could be addressed	Could be included as "mandatory" under OSPAR monitoring activities.

In addition to this, European Commission published, in 2013, the ***Green Paper on a Strategy on Plastic Waste in the Environment***[3]. Microplastics were highlighted since *"they are ubiquitous and reach even the most remote areas with a concentration in water sometimes higher than that of plankton. These micro-plastics, and the chemical additives they contain, if*

[3] Green Paper on a Strategy on Plastic Waste
http://ec.europa.eu/environment/waste/pdf/green_paper/green_paper_en.pdf

ingested in large quantities by marine fauna may have a high potential for contaminating the food chain through predator-prey interaction". (Green Paper, p. 6 and 14).

3.2. Other voluntary schemes: BREF and Ecolabel

PIECE OF REGULATION / POLICY	Reference Document on Best Available Techniques for the Textiles Industry July 2003 (BREF Textiles Industry)
Link	http://eippcb.jrc.ec.europa.eu/reference/BREF/txt_bref_0703.pdf
General objective	This document should act as valuable tools to drive environmental performance by providing relevant information concerning best available techniques in Textiles Industry.
TOPIC*	Textile
Does it address "microplastics"?	NO
How does it address microplastics?	No microplastics, but it addresses the loose of fibres (loose fibre drying)
Does it address other key issues related to the release of microplastics caused by textile washing processes?**	YES
How does it address other issues?	It addresses the discharge of poorly or nonbiodegradable substances, as well as the loose of fibre
Indicate the most related sections	All but more specifically: 2.2 Fibre manufacturing: chemical (man-made) fibres; 2.4 Yarn manufacturing; 2.6.4 Pretreatment of synthetic material; 2.7 Dyeing; 2.8 Printing; 2.9 Finishing (functional finishing); 2.10 Coating and laminating; 2.12 Washing; 2.13 Drying; 3.6 General issues concerning solid & liquid wastes generated in the textile industry; 4.2 Quality management of incoming fibre, 4.9 Washing; 5 BAT; 10 Annex III

Every four years on average, the EU Ecolabel criteria are revised to reflect technical innovation such as evolution of materials, production processes or emission reduction and changes in the market. Within this framework, the results of the MERMAIDS project are expected to reveal some evidences that could improve the environmental performance related to some of the product groups currently covered by EU Ecolabel, more specifically textile products and laundry detergents.

With the aim of supporting future EU Ecolabel criteria revision of these product groups, the project results will be offered to the European Union Ecolabelling Board (EUEB) in the form of recommendations designed to specific product groups and criteria. In particular, the project review will be focused on the following:

Textile Ecolabel - (2014/350/EU) **Validity date: 05/06/2018** ***COMMISSION DECISION of 5 June 2014 establishing the ecological criteria for the award of the EU Ecolabel for textile products***
Criterion 4. Acrylic Criterion 6. Polyamide (or nylon) Criterion 7. Polyester Criterion 8. Polypropylene Criterion 10. Fillings Criterion 11. Coatings, laminates and membranes Criterion 12. Accessories **Criterion 15. Washing, drying and curing energy efficiency** **Criterion 17. Dimensional changes during washing and drying** **Criterion 24. Fabric resistance to pilling and abrasion** **Appendix 3. Best available technique in the field of washing, drying and curing energy efficiency (2. Washing and rinsing process)**
Ecolabel for laundry detergents - (2011/264/EU) **Validity date: 28/04/2015 (revision ongoing) – last amended in May 2014** ***COMMISSION DECISION of 28 April 2011 on establishing the ecological criteria for the award of the EU Ecolabel for laundry detergents***
Criterion 2 — Toxicity to aquatic organisms: Critical Dilution Volume (CDV) Criterion 3 — Biodegradability of organics Criterion 4 — Excluded or limited substances and mixtures **Criterion 6 — Washing performance (fitness for use)** Criterion 7 — Points
Ecolabel for Industrial and Institutional Laundry Detergents - (2012/721/EU) **Validity date: 14/11/2016 – amended in May 2014** ***COMMISSION DECISION of 14 November 2012 establishing the ecological criteria for the award of the EU Ecolabel for Industrial and Institutional Laundry Detergents***
Criterion 2 — Toxicity to aquatic organisms: Critical Dilution Volume (CDV) Criterion 3 — Biodegradability Criterion 4 — Excluded or limited substances and mixtures **Criterion 6 — Washing performance (fitness for use)**

The criteria listed above have been identified as thematically related with the project. Criteria in bold are directly linked with washing processes. Potential updating from the project results will be assessed into all of them.

4. SWOT analysis (Strengths –Weaknesses – Opportunities – Threats)

SWOT analysis of the current regulatory framework on which policy recommendations will be set.

Strengths	Weaknesses
- The Marine Strategy Framework Directive (MSFD) aims to achieve Good Environmental Status (GES) of the EU's marine waters by 2020. This means that Member States have obligations and commitments related to microplastic pollution in marine waters. - According to some policy-experts, there is already a discussion at EU level related to the inclusion of microplastics in MSFD article 15. - The WFD indicates to be based on the precautionary principle and on the principles that preventive action should be taken, environmental damage should, as a priority, be rectified at source and that the polluter should pay. - MARPOL Convention mentions laundry drainage as part of “grey water” (vector for microplastic pollution). - A voluntary multistakeholder coordination mechanism in which all partners agree to work together to further reduce and better manage marine litter exists (Global Partnership on Marine Litter). - In June 2014, governments attending the first UN Environment Assembly noted with concern the impacts of plastics and microplastics on the marine environment, fisheries, tourism and development calling for strengthened action, in particular by addressing such materials at the source. A resolution (addressed below in this document) was adopted calling for the strengthening of information exchange mechanisms, requesting UNEP to present scientific assessments on microplastics for consideration by the next session of the Assembly. - The Honolulu Commitment (HC) also welcomed the ongoing work of scientists, research organisations and other citizens to better and more accurately understand the sources, nature and extent of marine debris, including the effects of micro-plastics. - Regional Sea Conventions (HELCOM, UNEP/MAP and OSPAR address microplastics). - Most of the analyzed Directives address microplastics directly or indirectly, and specific sections for potential updating have been identified. - The European Commission published, in 2013, the Green Paper on a Strategy on Plastic Waste in the Environment in which microplastics were highlighted, indicating as well	- There is no piece of regulation that specifically addresses the release of microplastics by textile washing processes. - The WFD doesn’t address microplastics directly. - MARPOL doesn’t specifically address microplastic pollution found in laundry waste waters in ships. - The Directive on urban waste water treatment does not address the textile sector but it could be addressed.

that these micro-plastics, and the chemical additives they contain, if ingested in large quantities by marine fauna may have a high potential for contaminating the food chain through predator-prey interaction".	
Opportunities	**Threats**

- Research is expanding in relation to the occurrence, fate and effects of microplastics in the marine environment.
 	
- Technologies to treat/remove textile fibers or microplastics from waste waters are known and available (such as adding a 3rd phase to water treatment plants).
 	
- There are successful experiences of waste water textile fibers removal/treatment and lessons can be learned from frontrunner countries in this area (e.g. Scandinavia).
 	
- There are several ways to address the problem of textile fibers loss (and thus microplastics release) into waste water:
  - Improving textile finishing.
  - New water treatments.
  - New technologies in washing machines.
  - Improving laundry products.
 	
Some experts prefer one in front of the others but other experts prefer to address a combination of all of the above.
 	
- The scientific community is providing key information related to the impacts of microplastics on marine biota / ecosystems.
 	
- Socio economic impacts of plastic garments' production/consumption exist:
  - SOCIAL IMPACTS:
Natural materials use a lot of water, Co2 emissions. Stop using synthetic chothes/textiles will create other negative environmental problems for society. In the future it could have an effect on food production and cause possible health problems.
  - ECONOMIC IMPACTS:
Pollution of water systems.
Fibers from clothes contributing to the plastic soup and fibers entering the food chain can damage the image of fish consumption.
It can also have a negative impact in the clothing and textile industry.
Problems could also arise with dredging material, possibly polluted with microplastics.
 	
- Several successful experiences exist concerning microplastic prevention and/or removal from waste waters:
  - Beat the microbead campaign addressing microplastics in cosmetics and achieving commitment from big and strong companies (such as Unilever who announced in December 2012 that all of its products
	- European citizens are not aware of the release of microplastics by textile washing processes. When citizens are aware, social pressure increases and product demand changes requiring for more sustainable and safe products. This is not the case at the moment in European society.
	
	- Technologies to treat/remove textile fibers or microplastics from waste waters are known and available but expensive.
	
	- Much more research is needed in relation to microplastics at a general level.
	
	- Much more research is needed in order to tell the effects of fibers / microplastics on the human body.
	
	Some experts think further research is needed in order to provide further legislation and/or regulatory instruments to address microplastics' pollution caused by laundry processes.
	
	- There are several ways to address the problem of textile fibers loss (and thus microplastics release) into waste water:
	  - Improving textile finishing.
	  - New water treatments.
	  - New technologies in washing machines.
	  - Improving laundry products.
	
	Some experts indicate that before indicating which of the above would be the best to address, more information is required.
	
	- Even though successful examples exist in the ban of microbeads in cosmetics for example, it is important to consider that textile and laundry sectors are strong and it will take time until this approach reaches these sectors. Currently the textile and the laundry industries are not receiving any pressures in relation to this matter.
	
	- High economic risk for companies investing in new laundry products in front of already commercially successful products.
	
	- Companies may be reluctant to change in their products.
	
	- No large-scale survey in Europe has been carried out in order to know if the textile sector in Europe is open to address the problem of microplastic fiber release by textile washing processes.
	
	- No large-scale survey in Europe has been carried out in order to know if the laundry products' sector in Europe is

worldwide would be plastic free by 2015, Procter & Gamble indicated their products would only be free from microbeads by 2017 at the earliest, or Johnson & Johnson who said it has already started phasing out microbeads and was no longer developing products containing microbeads. (Plastic Soup Foundation) - Clean sweep to avoid the loss of production pellets. (Plastics Europe). - Some experts new legislation and/or regulatory instruments are necessary to address microplastics' pollution caused by laundry processes. - Some experts think a regulatory system should be in place for water quality monitoring schemes. - According to some policy-experts, emissions of plastic to the environment should be tackled. A tool should be developed to measure the possible impact of different products, including synthetic clothing and textiles. - If the European textile sector addresses the problem of microplastic fiber release by textile washing processes this could bring an added value and a positive image to European textiles in front of non-European textiles. Nevertheless, more work is required in order to analyze advantages and disadvantages of this.	open to address the problem of microplastic fiber release by textile washing processes.

SWOT analysis of the three related EU Ecolabel concludes as follow:

Strengths	**Weaknesses**
- The EU Ecolabel covers a wide range of product groups, including textile products and laundry detergents. - Ecolabel addresses the main environmental impacts over the entire life cycle of the product. - All three addressed EU Ecolabel aim to guarantee reduced water pollution and reduced impact on aquatic ecosystems, among others. - EU Ecolabel of laundry detergents (also industrial and institutional laundry detergents) aims to guarantee the increased biodegradability of these products. - A high number of laboratory testing will be performed in MERMAIDS to support the future recommendations for updating EU Ecolabel criteria. Testing will include: Pilling-Box Method (EN ISO 12945/1), Random Method (EN ISO 12945/3), weight per unit area (EN 12127), density (EN 1049/2), yarns (ISO 7211/5) - (EN ISO 2061) - torsion constant "k", fiber length.	- Textile colour and shrink resistance during washing are highly encouraged in the EU textile Ecolabel, but there is no focus to the textile fibers loss (and thus microplastic release). - The latest version of the revised EU Ecolabel performance test for laundry detergents includes test criteria for soil and stain removal, basic degree of whiteness, colour maintenance and dye transfer inhibition; but it does not include criteria for the fistness/fastness of fibers.
Opportunities (expected to be addressed during the action B6)	**Threats**
- MERMAIDS will assess the inclusion of textile fibers loss/microplastic release into the EU textile Ecolabel. The assessment will be focused on the criteria related to plastics and washing processes (criterion 4, 6, 7, 8, 10, 11, 12, 15, 17 and 24; appendix 3). - MERMAIDS will assess potential insertion of criteria for the	- The action B6 will start in January 2016 and the recommendations will be delivered at the end of the project, in September 2016. The current criteria analyzed here regarding detergents will be expired, one along the project and the other one just the month after finishing the project. Then, at the beginning of the action B6 this SWOT need to

fitness/fastness of fibers into the EU Ecolabel performance test for laundry detergents. - The MERMAIDS results can be used for reviewing the criteria of EU Green Public Procurement (GPP) in the groups: “Cleaning products and services” and “Textiles”, both of them currently under revision.	be reviewed in order to adddress the analysis in line with the latest information.

5. Conclusions

Very little pieces of legislation address microplastics in a specific way. Nevertheless, there is existing regulation targeting microplastics directly such as the Marine Strategy Framework Directive and others indirectly. The problem of the release of microplastics by laundry washing processes is not specifically legislated in any of the reviewed pieces, nevertheless key areas have been identified in different pieces of legislation/others in which this problem could be addressed, in some cases the specific article, chapter, etc has been proposed for revision and/or update.

The analysis of the regulatory framework establishes the basis on which policy recommendations will be set in the future Action B6.

When developing recommendations under B6, possible updates and/or new regulatory pieces and non-regulatory pieces will be taken into account.

6. Annexes

Annex I. Questionnaires

- MARITIME MARINE POLICY QUESTIONNAIRE
- LAUNDRY PRODUCTS QUESTIONNAIRE
- TEXTILE QUESTIONNAIRE

Annex I. Questionnaires

With the contribution of the LIFE financial instrument of the European Comunity:

mermaids.
OCEAN CLEAN WASH

MARITIME MARINE POLICY QUESTIONNAIRE
(Annex I.1 Deliverable A3)

KNOWLEDGE OF EUROPEAN CITIZENS

1. Do you think European citizens are aware of the release of microplastics by textile washing processes?

YES or NO

PREVENTION AND MITIGATION TECHNOLOGIES AND METHODOLOGIES IN ORDER TO AVOID MICROPLASTIC MARINE POLLUTION

2. By what means/in what way do you think that textile fibers loss (and thus microplastic release) into waste waters should be addressed?

   a. Improving textile finishing (including textile care products/additives).
   b. New water treatments (in wastewater treatment plants).
   c. Including new technologies in the new washing machines that will hit the market soon (e.g. filtering technology).
   d. Improving laundry products (e.g. detergents including textile care products/additives).
   e. Others: ____

   Could you please specify which sectors and organizations should be involved and how:
   ____

3. Do you know of any technology to treat/remove textile fibers or microplastics from waste waters?

YES or NO

If your answer is YES, please specify the following:

Technology/system	Website/link/reference	Other relevant information
…	…	…

4. Do you know of any successful experience of waste water textile fibers removal/treatment?

YES or NO

If your answer is YES, please specify the following:

References	Website/link
…	…

IMPACTS OF MICROPLASTICS ON MARINE BIOTA / ECOSYSTEMS

5. Do you know of any examples related to environmental impacts of microplastics' on marine biota / ecosystems?

YES or NO

If your answer is YES, please specify and indicate any references supporting your answer:

….

SOCIO ECONOMIC IMPACTS OF PLASTIC GARMENTS' (POLYESTER, POLYAMIDE, POLYPROPYLENE, ETC) PRODUCTION/CONSUMPTION

6. What kind of **social impacts** do you think current synthetic garments' production/consumption causes today or could cause in the future?

Causes today: Could cause in the future:

7. What kind of **economic impacts** do you think current synthetic garments' production/consumption causes today or could cause in the future?

Causes today: Could cause in the future:

HUMAN RISK ONCE MICROPLASTICS ARE INTRODUCED IN THE FOOD CHAIN

8. Do you know about any information concerning human risk of textile fibers and microplastics in food chain? If your answer is YES, please specify below.

Reference	Website/link/etc.
…	…
…	…

REGULATORY FRAMEWORK

9. Do you know of any national/regional/local legislation/regulations concerning microplastics' pollution specially related to waste waters in Europe?

YES or NO

If your answer is YES, please specify below:

If related to waste waters indicate here: …

If not related to waste waters indicate here: …

10. Would you propose any modifications in the existing regulatory framework in order to reduce microplastics' release by means of waste waters into seas and oceans?

YES or NO

If your answer is YES, please specify below:

Law/Directive/Policy…	Modification
…	…
…	…

11. Do you think new legislation and/or regulatory instruments are necessary to address microplastics' pollution caused by laundry processes?

YES or NO

If your answer is YES, please specify briefly in what specific area new legislation and/or regulatory instruments are necessary:

…

12. Do you know any successful experience on the implementation of any legislation/policy concerning microplastic prevention and/or removal from waste waters?

YES or NO

If your answer is YES, please specify:

Law/Directive/Policy…	Modification
…	

13. Do you have any policy-related recommendations that you think could be considered by the European Commission and/or EU Member States?

YES or NO

If your answer is YES, please specify:

…

14. If you know of any other information of interest related to the release of microplastics by laundry waste waters, we would be happy to gather it.

Please specify below:

…

LAUNDRY PRODUCTS QUESTIONNAIRE
(Annex I.2 Deliverable A3)

LAUNDRY PRODUCTS INDUSTRIES

1. Which "textile care" laundry products do you produce for synthetic fibers textiles?

…

2. Do you have available data regarding their efficiency regarding fiber loss/retention?

YES or NO

If you have non confidential data, please provide below a reference/link/mail of contact/etc to access this data:

…

With the contribution of the LIFE financial instrument of the European Comunity:

TEXTILE QUESTIONNAIRE
(Annex I.3 Deliverable A3)

PREVENTION AND MITIGATION TECHNOLOGIES AND METHODOLOGIES IN ORDER TO AVOID MICROPLASTIC MARINE POLLUTION

1. By what means/in what way do you think that textile fibers loss (and thus microplastic release) into waste waters should be addressed?

    a. Improving textile finishing (including textile care products/additives).
    b. New water treatments (in wastewater treatment plants).
    c. Including new technologies in the new washing machines that will hit the market soon (e.g. filtering technology).
    d. Improving laundry products (e.g. detergents including textile care products/additives).
    e. Others: ____

    Could you please specify which sectors and organizations should be involved and how:
    ____

TEXTILE AND CLOTHING MANUFACTURING AND MANAGEMENT

2. Which of the processes you use to manufacture garments/clothes generate attrition and thus result in textile fibres loss?

    Where do these fibres go?

3. Which of the following textile finishing treatments do you use in your manufacturing process?

    a. Silicon macroemulsions
    b. Polyurethane binders
    c. Acrylic binders
    d. Others binders

4. Do you use any textile finishing at pH 7-8?

YES or NO

If your answer is YES, please specify.

…

5. Do you use any technology/system in order to remove textile fibers from waste water or to prevent textile fibers release into waste waters? If yes, could you please specify which technology/system and how does it work?

Technology/system	Operability	Textile fibers retained (typology and amount)
…		

6. Do you think the textile sector in Europe is open to address the problem of microplastic fiber release by textile washing processes?

YES or NO

7. Do you think that if the European textile sector addresses the problem of microplastic fiber release by textile washing processes this could bring an added value and a positive image to European textiles in front of non-European textiles?

YES or NO

REGULATORY FRAMEWORK

8. Do you know of any national/regional/local legislation/regulations concerning microplastics' pollution specially related to waste waters in Europe?

YES or NO

If your answer is YES, please specify below:

If related to waste waters indicate here: …

If not related to waste waters indicate here: …

9. Would you propose any modifications in the existing regulatory framework in order to reduce microplastics' release by means of waste waters into seas and oceans?

YES or NO

If your answer is YES, please specify below:

Law/Directive/Policy…	Modification
…	…
…	…

10. Do you think new legislation and/or regulatory instruments are necessary to address microplastics' pollution caused by laundry processes?

YES or NO

If your answer is YES, please specify briefly in what specific area new legislation and/or regulatory instruments are necessary:

…

Name of the deliverable:

Report of the reduction of fibres loss by the use of textiles auxiliaries

Number of the associated action: B1

Involved Partners: LEITAT - POLYSISTEC

(11/03/2016)

LIFE13 ENV/IT/001069

Mitigation of microplastics impact caused by textile washing processes

Contents

B1. Innovative textile auxiliaries

TASK B.1.1 Lab scale finishes with textile additives

B 1.1.1 Introduction

Chemical finishing includes processes that change the chemical composition of the fabrics that they are applied to. Typically chemical finishing takes place after coloration (dyeing or printing) and before fabrics are made into garments or other textile articles. However, many chemical finishes are made into garments or other textile articles.

In nearly all cases, the chemical finish is a solution or emulsion of the active chemical in water. Chemicals that have strong affinity for fiber surfaces can be applied in batch processes by exhaustion in dying machines, usually after the dying process has been completed. Chemicals that do not have an affinity for fibers are applied by a variety of continuous processes that involve either immersing the textile in the solution or applying the finishing solution by some mechanical means. After application of the chemical finish, the fabric must be dried and if necessary, the finish must be fixed to the fiber surface, usually by additional heating in a curing step[i].

Two main processes can be indentified to apply the textile finishing on the fabric:

- *Padding application process:*

The fabric is impregnated in a textile auxiliary bath and then squeezed between two squeeze rollers (see Figure 1), by applying a certain pressure and speed. The amount of finishing solution or emulsion applied is referred as the "*pick-up*" of the fabric and is usually expressed as a percentage on the weight of the dry untreated fabric:

$$\%pick\ up = \frac{wt\ \ solution\ applied}{wt\ dry\ fabric} \cdot 100 \qquad \textbf{(Eq.1)}$$

- *Exhaustion application process:*

The fabric is immersed in the bath solution by setting a certain temperature and time of cycle. The laboratory scale equipment used for this procedure is shown in Figure 2. The amount of chemical finish to be applied is usually expressed as a weight percentage based on the original fabric weight *(owf)*.

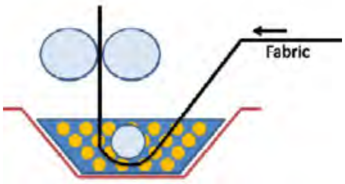


Figure 1. Scheme of padding machine

Figure 2. Exhaustion process (lab-scale) apparatus

- Curing chemical finishes:

The same heating equipment used to dry wet textiles can also be used to heat the fabric to the desired temperatures for optimal curing. It is important to mention that the temperature of the fabric do not exceed 100ºC until all of the water is removed. Once all the water has gone, the fabric temperature rises to the set temperature for curing.

B 1.1.2 Description of the selected textile auxiliaries

Several textile additives have been selected in order to prevent the pill formation and to minimize the fiber loss during washing.

Firstly, following the preliminary study done in Action A 2.1.2, different commercial products from POLYSISTEC S.L. have been acquired in order to be tested in this implementation action. The chemical and physical characteristics for each product as well as the recommended application process are summarized in Table 1.

Product name	PURLASTIC 8139	PURLASTIC 8189	POLYACRIL 73	POLYACRIL 97	POLYACRIL 56 ECO	POLYSILK CTE	POLZYME EA 50
Chemical description	PU resin	PU /Acrylic resin	Acrylic resin	Acrylic resin	Acrylic resin	Silicone emulsion	Acid cellulase
pH	6.0-8.0	7.0 – 9.0	5.0-7.0	4.0-6.0	6.5 – 7.5	9.0-11.0	4.0-6.0
Viscosity (25ºC) mPa·s	160	200	266	350	160	350	---
Dry matter (%)	38.0-42.0	40.0 – 44.0	42.0-46.0	42.0-46.0	55.0 – 57.0	44.0-48.0	---
Tg (ºC)	10	-50/-1	34	26	-1	---	---
Ionic character	Anionic	Anionic	No-Ionic	Anionic	Anionic	Cationic / Non-ionic	---
Application	Padding	Padding	Padding	Padding	Padding	Padding/ Exhaustion	Exhaustion

Table 1. Chemical and physical properties of the selected textile auxiliaries

The selected acrylic resins differ themselves in their stiffness. This property can be explained, in general, by the Tg. Glass transition temperature is one of the most important parameters in order to determine the minimum usage temperature for polymeric materials. Below this temperature, polymers become glassy and brittle, and develop a high elastic modulus. Above this temperature the polymer behaves as rubber-like elastic fluid. The Tg will also influence on the wetting properties of the auxiliary on the applied fabric. Therefore, Polyacril 97 will form a harder coating than Polyacril 56ECO, which is a much softer copolymer in aqueous emulsion. Polyacril 73 is a semicrystalline polymer and therefore it will result in a harder and more thermally stable coating. Purlastic 8139 is a polyurethane aqueous dispersion and Purlastic 8189 is an acrylic and polyurethane hybrid that combines the properties of such groups. Polysilk-CTE is an amino-functional elastomeric silicon macro-emulsion. The siloxane backbone provides flexibility and high surface smoothness that is important for the final touch of the fabric. Moreover it claims that it provides a good performance to avoid the formation of pilling. Polzyme EA50 is an enzymatic treatment based on acid cellulase used in fabrics that include cellulose fibers. This product has been discarded during the execution stage since it is not suitable for the problem that we are tackling: synthetic microfibers are not removed from the fabric using this enzymatic treatment.

For all these products, padding process is recommended. Exhaustion application has been tried in Polysilk-CTE in order to compare both methods. All the selected parameters for both processes are explained in the following section.

B 1.1.3 Selection and optimization of application process parameters

As explained in the preliminary action A 2.1.2 (Study of textile auxiliaries), two main processes are used to apply the auxiliaries on the fabric: padding and exhaustion. The selection and optimization of the process parameters is important to have a proper balance of the final properties in the fabric. In a first stage, padding has been used in order to pre-select the most interesting auxiliaries. A design of experiments has been planned to compare different parameters and their effect in the treated fabrics (Table 2). For padding, the variables are: auxiliary concentration, pressure and speed.

	Fabric: woven polyester (Ref. 777)																		
Experiment	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
Units										g/l									
POLYACRIL 97	10				20				20	30									
POLYACRIL 73		10				20					30								
PURLASTIC 8189			10				20					30							
POLYSILK CTE				10				20					30						
PURLASTIC 8139														10	20	30			
POLYACRIL ECO56																	10	20	30
Process parameters																			
Pick-up (%)	55	55	54	64	58	59	61	64	53	59	60	60	64	64	64	65	63	67	63
v (m/min)	3	3	3	3	3	3	3	3	6	3	3	3	3	3	3	3	3	3	3
P (bar)	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3

Table 2. Design of experiments for padding method

In most of these trials, *v* and *P* are set at 3 m/min and 3 bar respectively. For trial 9, the speed is increased up to 6 m/min to determine the influence in the pick up. The "*pick-up*" has been calculated in each case to have an idea of the amount of chemical that has been absorbed by the textile. Moreover, pick-up value will be useful in a future stage during the pilot plant implementation, where the adjustment of the speed and pressure will be carried out according to this value.

In general terms, when the auxiliary concentration is increased, slightly increases the pick-up value (excluding the Polysilk CTE). An increase of the speed, results in a decrease in the absorption (9 compared to 10) since the impregnation time is lower.

An additional trial is performed through exhaustion method (see Table 3) in order to compare the effect of the application process in the final performance. According to the pick-up calculated for a concentration of 30g/L of Polysilk-CTE, the concentration of this product in the bath has been set at 2% (grams auxiliary/grams fiber). The bath ratio has been set at 1:10 (grams fiber: grams solution), the temperature at 60ºC and the time of the experiment 35 minutes.

woven polyester (Ref. 777)	
Experiment	20
Units	%owf (g product/g fabric)
POLYSILK CTE	**2**
Process parameters	
T (ºC)	**60**
t (min)	**35**
Bath ratio	**1:10**

Table 3. Design of experiments for exhaustion method

For these initial samples, a complete characterization has been carried out to determine which final treatment is suitable for solving or minimizing the problem of the fiber release.

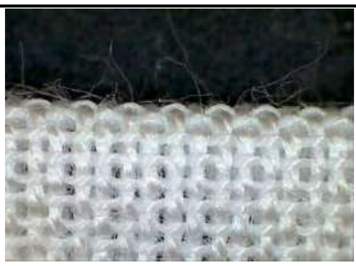
TASK B.1.2 Characterization of the lab scaled finishing textiles

B 1.2.1 Determination of morphological properties

The aim of this study is to identify, if any, the main differences of the fabrics before/after treatment. This study has been performed initially on woven Polyester fabric. Woven polyester (Ref.777) has been selected to be the worst quality textile in terms of pilling formation (see Preliminary action A.2.1). Moreover, in clothing textile production weight, the market is dominated by cotton, which accounts for more than 43% of all fibers, and followed by polyester (16%)[ii]. Thus, it will be an important improvement to tackle the problem of fiber release in this fabric.

HAIRINESS

Polyester fabric is shown in Table 4 before and after the treatment. When treated with the different textile auxiliaries (Polyacril 97 and Polyacril 73) there are no differences regarding the hairiness. This behavior is expected since the function of the auxiliaries would be to bind the microfibers to the fabrics and prevent them to break during its use but not to remove or cut these protruding fibers from the fabric. In this study it is also visible that the different auxiliary concentrations (e.g. Polyacril 97) are not showing any change in the hairiness.

HAIRINESS ANALYSIS		
BEFORE TREATMENT		
100% woven PES (Ref: 777)		

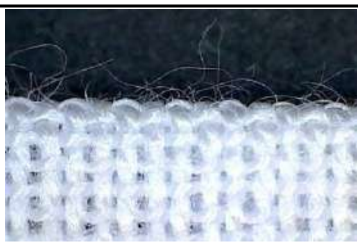
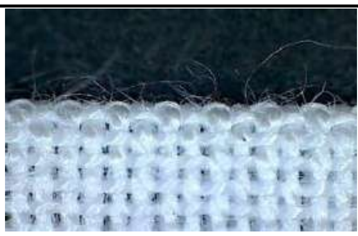
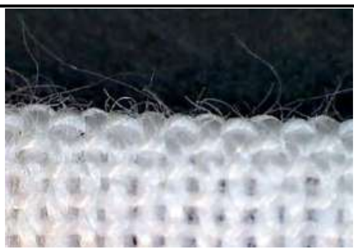
AFTER TREATMENT		
Polyacril 97 10g/L		
Polyacril 97 20g/L		
Polyacril 97 30g/L		
Polyacril 73 10g/L		

Table 4. Hairiness analysis of treated woven polyester.

SCANNING ELECTRON MICROSCOPE ANALYSIS

A complete characterization for all the selected fabrics and textile auxiliaries have been carried out. In this section, the morphological analysis of the woven polyester (Ref.777) is described. The other fabrics are detailed in the annex. Purlastic 8189 has been indentified as the best finishing product. Indeed, it has been possible to observe upon the treatment, the higher number of neighboring fibers linked by its film on the textile surface (Figure 3 left). Nevertheless, a limit in the efficiency of its coating ability was revealed: in some cases, it was observed an interruption of the product layer between two or more fibers (Figure 3 right).
With Purlastic 8139 (Figure 4) and Polysilk CTE (Figure 5 left) applied on woven polyester fabrics, auxiliary filaments of different thickness were identified among the fibers; moreover, an irregular distribution of product over the fabric is observed, in particular for Purlastic 8139.

Non-homogeneous coating was also revealed for woven polyester textile treated with Polyacril 56 ECO (Figure 5 right).

In case of the treatment with Polyacril 73 and 97, a discontinuous distribution of the product on the surface was observed, appearing in the form of a rough layer or agglomerates of different dimensions (Figure 6).

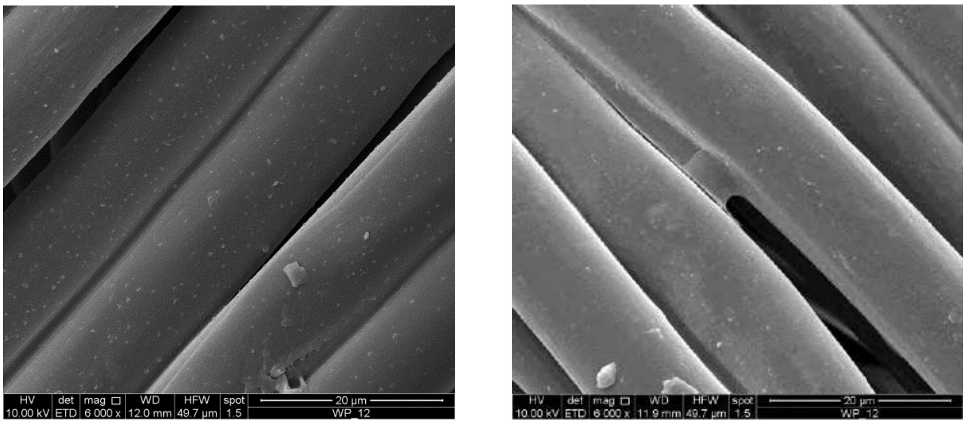


Figure 3. SEM micrographs of Woven Polyester treated with Purlastic 8189.

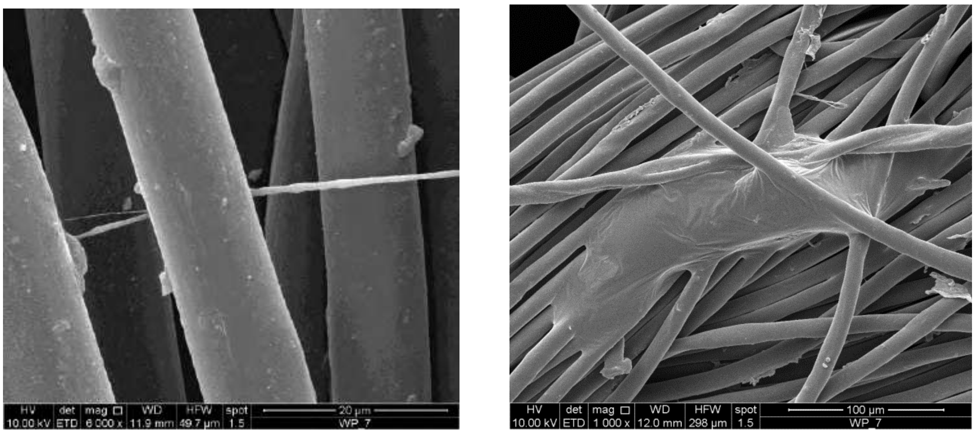


Figure 4. SEM micrographs of Woven Polyester treated with Purlastic 8139.

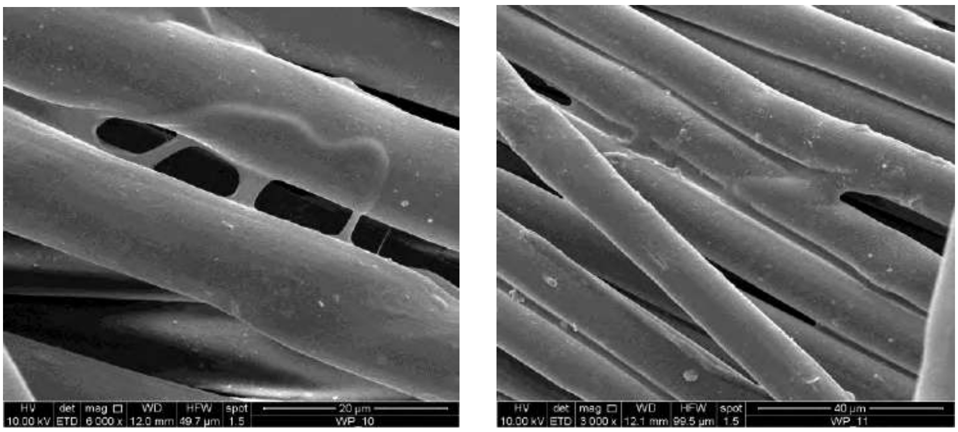


Figure 5. SEM micrographs of Woven Polyester treated with Polysilk CTE (left) and Polyacril 56 ECO (right).

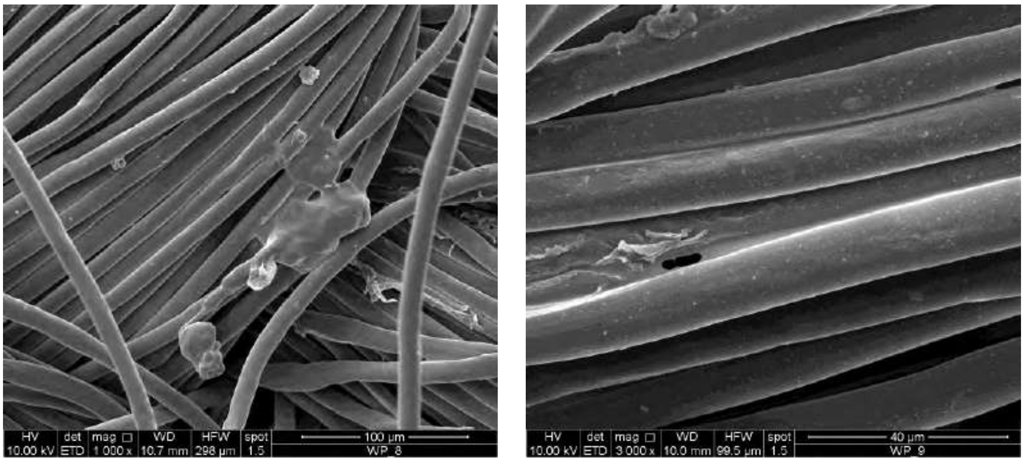


Figure 6. SEM micrographs of Woven Polyester treated with Polyacril 73 (left) and Polyacril 97 (right).

B 1.2.2 Determination of mechanical properties (pilling Martindale)

Pilling method is explained in detail in the preliminary action A.2.1.1. Table 5 includes the results of Martindale pilling for the untreated textiles. In pilling Martindale (UNE EN 12945-2), samples are rubbed against wool fabric (during 5000 cycles) which provides an extremely high abrasion to the textiles. A new textile has been added to the comparison: 100% woven polyester (Ref.TIS105) made of continuous filaments. This new fabric has been introduced since all woven fabrics are made of staple fibers and this characteristic (fiber length) may be significant in the consequent fiber loss.

	Woven PES (Ref.777)	Woven PES (Ref.TIS105)	Woven Polyamide	Woven PP	Woven acrylic	PES/CO 65/35	Knitted PES	Knitted polyamide
Martindale Method	1-2	1-2	2-3	2	2	2-3	5	4

Table 5. Determination of pilling by Pilling-Box Method, Martindale Method and Random Method

It is observed that in both woven polyester fabrics the pilling indexes are the lowest. In Ref.TIS105, the result has not been expected as continuous filaments should not be released easily from the yarn structure. This can be explained by the abrasion suffered during the spinning, the weaving/knitting and the textile desizing processes as they can be very aggressive and some fibers may break.

First studies have been performed on PES (Ref: 777). Table 6 shows the values for pilling Martindale on this fabric after applying the different auxiliaries. Differences between neat and treated fabric are not huge. However it is observed that with Polyacril 73 the values are around 2, which is slightly better pilling than the neat textile. Neither Polyacril 97 nor Purlastic 8139 do not show any improvement compared to the control whereas samples treated with

Polysilk-CTE show a more pronounced pilling than the neat polyester. Purlastic 8189 slightly enhances the pilling at highest concentration (30g/L) but it keeps the same value than the control at lower concentrations. Polyacril 56ECO provides the best results in terms of pilling. However, the fabric rigidity seem to be quite high by using this product. Fabric bending rigidity will be further evaluated for the best candidates.

Woven PES (Ref.777)		Conc. (g/L)			Pilling-Martindale Method	
Control (no treatment)	-	-	-		1/2	
Polyacril 97	10	20	30	1/2	1/2	1/2
Polyacril 73	10	20	30	2	2	2
Purplastic 8189	10	20	30	1/2	1/2	2
Polysilk-CTE	10	20	30	1	1	1
Purlastic 8139	10	20	30	2	1/2	1/2
Polyacril 56ECO	10	20	30	2	2	2/3
Polysilk-CTE (exhaustion)		2%			1/2	

Table 6. Determination of pilling Martindale for the different treated fabrics

Exhaustion method, although only compared in Polysilik-CTE, shows a small improvement in the pilling index. This result should be contrasted with more experiments, changing some of the variables such as temperature, time or concentration. By eye observation, it is also visible that the application of Polyacril 73 enhances the final performance in comparison to the control.

Samples after Martindale test are represented in Figure 7, where it is also evident by macroscopic observation that Polysilk-CTE presents much more pilling than untreated fabric. This could be explained since the introduction of softeners on the fabric would decrease the coefficient of friction fiber-fiber and as a consequence it would increase the fiber mobility and the pilling formation. Nonetheless this silicone macroemulsion presents the softest touch and should not be discarded yet. Polyacril 73 and Polyacril 56ECO present the best pilling results.

Control (woven PES 777)

Woven PES 777 + Polyacril 73 (20 g/L)

Woven PES 777 + Polysilk CTE (20 g/L)

Woven PES 777 + Polyacril 56 ECO (20 g/L)

Figure 7. samples of treated woven PES 777 after pilling Martindale test

Next step is to corroborate that the best candidates selected with the Matindale pilling method are also suitable for reducing the microplastics after domestic washings. For this, washing cycles in Linitest are going to be performed comparing the different textile additives.

B 1.2.3 Washing resistance: Characterization and quantification of micro and nanoplastics

The aim of this section is to study the washing durability of the treated fabrics, contrasting these results with the pilling values from Martindale test. The washing conditions in Lini-test (equivalent to 5 washing cycles), based on ISO 105-C06:2010, have been set according to the preliminary work in action A2.1:

- Temperature: 40 ºC
- Time: 45 minutes
- Number of steel balls: 10
- Medium: distilled H_2O (1)/distilled H_2O + liquid detergent (2)

For sample preparation, 1 gram of fabric has been cut. Before introducing the cut sample into the vessel of the Lini-test, it must be sewed from the edges with cotton thread to avoid the fiber release which is coming from the free ends of the fabric. The bath ratio has been set at 1:150 (g fabric: g solution). Durapore® membrane filters (from Merck Millipore) with a pore diameter of 5 µm have been used. Once the filtration is finished, the filters have been exposed to 85 ºC during 30 minutes and, finally, analyzed with a Scanning Electron Microscope (SEM). The quantification of micro and nanoplastics is explained in the action A.2 (*task A2.2.4).*

According to the results from the pilling Martindale, the following textile auxiliaries have been selected to be washed in the explained conditions:

- Purlastic 8189
- Polyacril 97
- Polyacril 73
- Polysilk-CTE
- Purlastic 8139
- Polyacril 56 ECO

A first batch of samples have been sent to CNR for fiber counting, with the aim of selecting the most interesting products. Three replicates have been prepared from each sample. Unfortunately some of them arrived damaged to CNR with no possibility to be analyzed. The results of fiber counting are shown in the table below:

Sample	Replicate	N° of fibers/filter	Replicate	N° of fibers/filter	Average
Purlastic 8189	B	732	-	-	-
Polyacril 97	A	1257	C	1016	1137
Polyacril 73	B	1163	C	1151	1157
Polysilk-CTE	A	819	B	1031	925
Purlastic 8139	A	1857	B	1043	1450
Polyacril 56 ECO	A	1360	B	1623	1492
Woven PES	A	123	-	-	

Table 7. Results for the fiber counting in the treated samples (1st batch)

It is observed that Purlastic 8189 and Polysilk-CTE show the lowest results of fiber release. However, if we compare them with the results for the reference fabric, non-treated woven PES, the release has been of 123 fibers/filter (result from the previous task A 2.2.3). Therefore, according to these results, none of these treatments is suitable for solving the problem of fiber breakage.

A second batch of samples has been prepared to corroborate the previous results. These new washings have been performed in two different mediums (*H_2O* and *H_2O+detergent*). The detergent used is a commercial liquid detergent with a dose of 65 ml/15L water and a pH of 8.1.
Purlastic 8139 has been discarded from the comparison since the process of filtration has been significantly long compared to the other treatments. Moreover, the pilling results were not successful for this treatment. Polyacril 97 has also been removed from the comparison to present lower performance in terms of pilling compared to the other two acrylic resins (polyacril 73 and polyacril 56ECO) while keeping similar results of fiber breakage after washing.

Woven polyester (Ref.777) has been washed again (with and without detergent) and added to the comparison. The results from the second batch of fiber counting are summarized in Table 8 and represented in Figure 8. The mean and the standard deviation are calculated for each one.

Sample	Average (released fibers/g fabric)			
Medium	*H_2O*		*H2O+detergent*	
	mean	SD	mean	SD
Purlastic 8189	158	28	1658	334
Polyacril 73	166	87	1251	128
Polysilk CTE	164	51	550	48
Polyacril 56 ECO	77	57	1258	132
Woven PES 777 (control)	240	100	1048	635
Woven PES .TIS105	-	-	1251	128

Table 8. Results for the fiber counting in the treated samples (2nd batch)

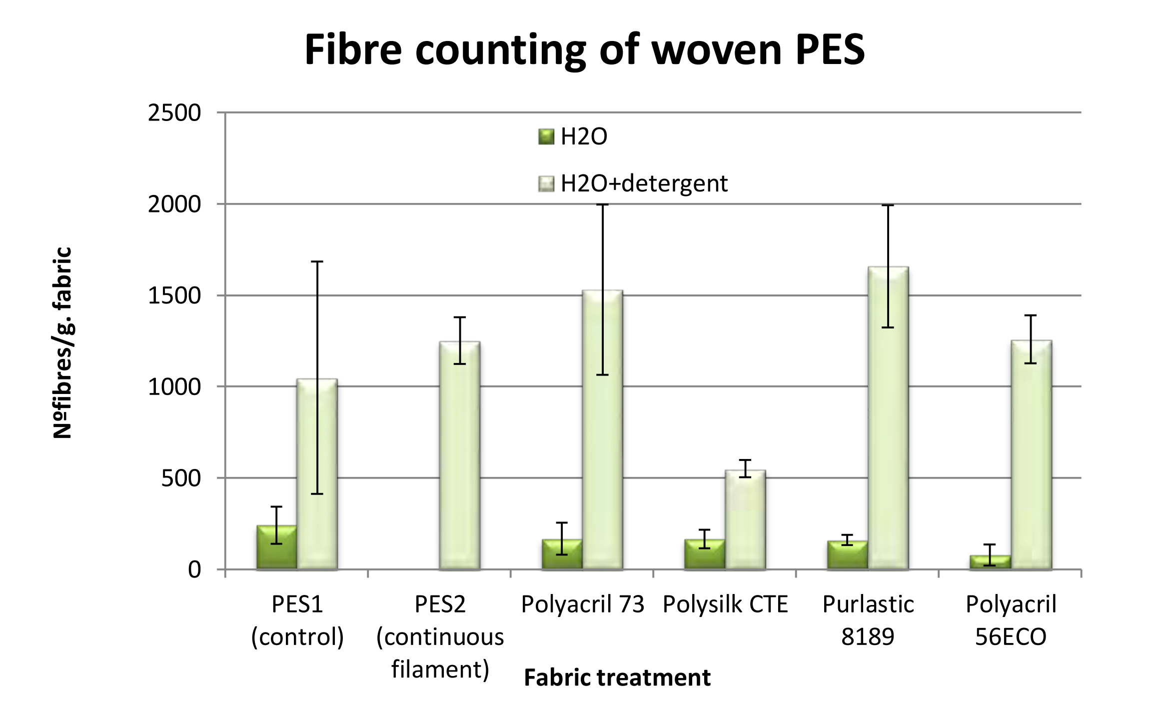


Figure 8. Representation of the results from fiber counting

First of all, the differences between washings with H_2O and washings with H_2O+detergent are visible. In the second case, the number of fibers released is much higher. This concludes that the effect of detergent is highly significant in the fiber loss during laundry.

The use of the different textile auxiliaries slightly decrease the fibers released during the washings with distilled water.

However, when the detergent is used, the effect is not the same for all the finishing treatments. The only effective treatment is Polysilk-CTE, which shows a clear improvement compared to the other treatments and the non-treated polyester (Ref. 777). This finishing is giving a better anchorage of the polyester fibers to the fabric and a higher washing resistance. Probably this explains the low pilling indexes obtained by applying this treatment since the pills that are formed are not easily released from the fabric and, as a consequence, they accumulate on its surface. Polysilk-CTE is an amino-functional polysiloxane emulsion. For this reason it may react with the ester groups of the fabric. Instead, Polyacril 73 and Polyacril 56ECO, which showed very good results in the pilling test, do not improve the anchorage of the fibers to the piece of cloth. This leads to high results in the fiber quantification as it can be observed in Figure 8.

An important point to highlight is the high deviation observed in these values. This indicates that this method present an important variability that can be either from the washing and filtering process or from the methodology of quantification by SEM.

B 1.2.4 Determination of the flexural rigidity: Cantilever Test

This test (ASTM D 1388:1996) has the objective to determine the rigidity at flexure of a fabric. With this system it is possible to measure the interaction between the laminar mass and the rigidity of the fabric as well as analyze the action of the gravity on a fabric strip in cantilever.

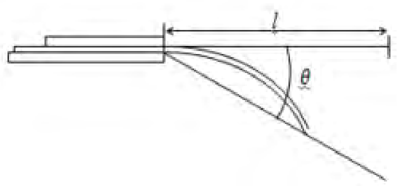


Figure 9 . Fabric stiffness, cantilever principle

A rectangular strip of fabric is mounted on a horizontal platform in such a way that it bends downwards as shown in Figure 9. The length l of the fabric that will bend under its own weight to a definite extent (and with an angle θ) is called bending length. The higher the bending length the stiffer the fabric.

The flexural rigidity is calculated with the equation below (Eq. 2), where W is the mass per unit area and l is the bending length.

$$\boldsymbol{G = W \cdot l^3} \qquad \text{(Eq. 2)}$$

Results for this test are summarized in Table 9. There is quite a significant difference between the treatments applied to the polyester fabric. Polyacril 65ECO provides the highest rigidity, and Polysilk-CTE, as it could be expected from a softener, presents the lowest flexural rigidity.

Woven PES (Ref:777)		Bending length (cm)	Flexural rigidity (mg·cm)	Flexural rigidity (total fabric)
Polyacril 73	Warp	6.1	403.1	191.6
	Weft	3.7	91.10	
Polisilk CTE	Warp	4.2	126.3	102.8
	Weft	3.7	83.6	
Polyacril 56ECO	Warp	6.9	3.47	252.2
	Weft	4.1	2.04	
Woven PES (Ref.777)	Warp	4.7	168.5	112.9
	Weft	3.6	75.8	

Table 9. Results of Cantilever test for the different treatments

B 1.2.5 Characterization of 100% woven polyamide and 100% woven acrylic fabrics

Two new textiles are added to the characterization: polyamide and acrylic (studied in detailed in action A 2.1). Among the synthetic textiles, these two are the most used in the industry after the polyester (see

Figure 10). For this reason, it is important to include them in the study.

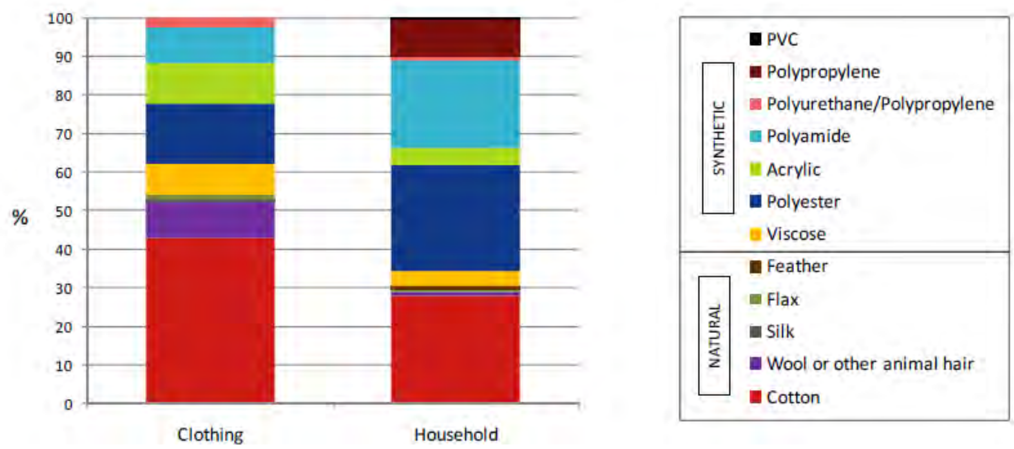


Figure 10. Percentage breakdown of consumption by fiber type for clothing and household textiles[ii]

These fabrics have been treated directly with Polysilk-CTE to provide the best results in terms of fiber loss and also with Polyacril 73 to enhance the pilling by keeping a moderate flexural rigidity and acceptable values of fiber release (in water). The characterization of the new fabrics has been based in the pilling (Martindale test) and washing performance (Lini-test). The results of pilling Martindale for woven polyamide 100% (Ref.361) and woven acrylic 100% (Ref. 864) have been summarized in Table 10 and Table 11.

Woven polyamide 100% (Ref.361)	Pilling- Martindale Method
Control (no treatment)	2/3
Polyacril 73	3
Polysilk-CTE	1/2

Table 10. Determination of pilling Martindale for treated woven polyamide100%

Woven Acrylic 100% (Ref. 864)	Pilling- Martindale Method
Control (no treatment)	2
Polyacril 73	3
Polysilk-CTE	2

Table 11. Determination of pilling Martindale for treated woven acrylic100%

The same trend detected in woven polyester has been now observed in polyamide and acrylic fabrics. Therefore, Polyacril 73 finish results in a decrease in the pilling effect while Polysilk-CTE shows the opposite effect and it increases the pilling compared to the non-treated fabric. The quantification of the fibers has also been analyzed to complement these results.

Table 12 and the graphic below (Figure 11) shows the fiber quantification for the woven polyamide. It is observed that by applying the finish Polysilk-CTE, the fiber loss is significantly reduced compared to the reference fabric. The other treatment, Polyacril 73, does not show an improvement in terms of fiber release but the opposite, as we have already observed in the woven polyester fabric.

Sample	Average (released fibers/g fabric)	
Medium	*H2O+detergent*	
	mean	*SD*
Polyacril 73	1456,29	239,79
Polysilk CTE	359,54	143,13
Woven PES 777 (control)	978,58	173,75

Table 12. Results for the fiber counting in the treated samples (100% woven polyamide)

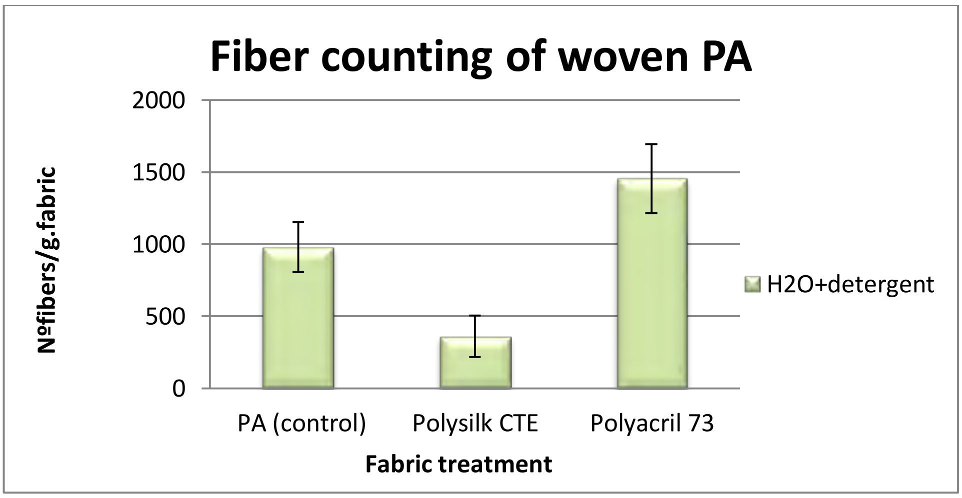


Figure 11. Representation of the results from fiber counting (woven PA)

B 1.2.6 Application and characterization of chitosan formulation

In addition to the finishing treatments with commercial product indentified by LEITAT, CNR-ISMAC performed lab scale experiments with innovative and eco sustainable solutions, based on chitosan, in order to reduce the microfibers release from textiles.

Chitosan (Figure 12) is a polymer deriving from a natural source, obtained from the deacetylation of the chitin contained in the exoskeleton of crustaceans, a waste product of the food industry and widely available in the market at a low price. Moreover, chitosan shows the advantages of being non-toxic, biocompatible and completely biodegradable, with a chemical structure similar to the cellulose; it is the polymer of the 2-amino-2-deoxy-β-D- glucopyranose, because the hydroxyls –OH situated in position 2 are substituted by amino groups –NH2.

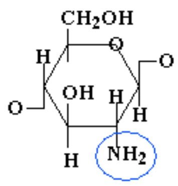


Figure 12. Chemical structure of the chitosan monomer

All the experiments performed in CNR use chitosan solubilized in acid solution, obtained with the addition of citric acid in the same concentration of chitosan (%w).

Antipilling and antifelting properties of chitosan are well-known. However chitosan treatments are established processes only for textiles based on natural fibers, especially cotton; the novelty of the study consist in the treatment of synthetic fabrics and the evaluation of their surface enhancements regarding the fibers retaining. Since in our previous assumptions the pilling tendency could be an important parameter affecting the fiber losses, the use of a natural and biodegradable product for finishing treatments, appeared to be a suitable solution to the microplastics problem.

The chitosan solution receipt was prepared as follows:

1. Prepare a 3%w/v citric acid solution (the quantity of citric acid, in grams, must be the same of the chitosan powder)
2. Add 3%w/v of chitosan powder
3. Stir at 50°C for 4 hours
4. Add hydroxyethyl methacrylate in the same quantity of chitosan.

After the deposition of the chitosan based solution, the fabrics were dried in oven at 150°C for 2 minutes, in order to ensure the chitosan reticulation.

APPLICATION OF CHITOSAN FORMULATION ON DIFFERENT FABRICS

1. Fabric impregnation with 3% chitosan solution:

During the first attempts of the study ISMAC set up a chitosan finishing treatment based on the impregnation of the textiles with a sufficient quantity of solution to wet all fabrics followed by the squeezing of the excess.

These tests have been performed on standard fabrics made of polyamide, polyester and the blend of polyester and cotton (20x20cm).

These treatments allowed to reach high percentages of chitosan load; the data related to the weight load of the fabrics are shown in Figure 13. The weight load means the dry weight loaded on the textiles (dried at 105°C).

woven polyester (Ref. 777)

Experiment	21	22	23
Fabric	woven PES (Ref.777)	Woven PA (Ref.361)	Woven CO/PES (Ref. 7409)
Weight load (%)	6,90	7,62	5,39

Table 13: Weight load% of the treated fabrics (solution containing chitosan 3% w/v)

2. Lab scale treatment by padding of chitosan solution:

Lab scale padding treatments have been performed on woven polyester fabric. The weight loads reached for each solution are shown in Table 14.

woven polyester (Ref. 777)

Experiment	24	25	26
Chitosan charge (% w/v)	1	2	3
Weight load (%)	0,33	2,58	4,06
Process parameters			
P (bar)	4		
v (m/min)	4		

Table 14. Design of experiments of chitosan formulation by padding method

The fabrics treated with the different chitosan formulations by lab-scale padding system were characterized in order to evaluate the fastness of the treatment to standard washings and the release of microplastics.

CHARACTERIZATION OF CHITOSAN SOLUTION

1. Fabric impregnation with 3% chitosan solution:

After the treatment, the presence of the finish was checked with a reactive dyeing method based on a staining with Red Remazol (Red Remazol 1.2% ofw, bath ratio 1:50, 60°C for 30 minutes).

A reactive dye could be an effective method to indicate the chitosan presence, because of the chemical composition (similar to cellulose), it shows a major affinity for reactive dyes compared to polyamide or other synthetic fibers. Thus, the treated polyamide appear darker than the untreated, as shown in Figure 13.

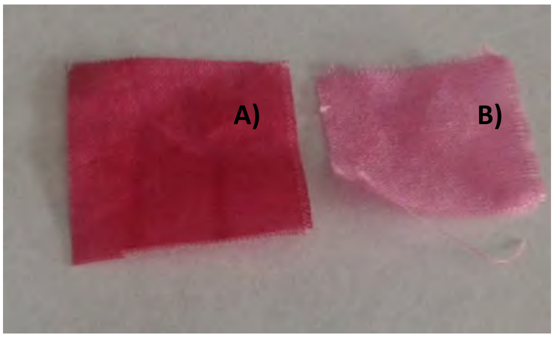


Figure 13. Image showing polyamide fabrics after the staining with Red Remazol. A) Polyamide treated with a 3%w/v chitosan solution, B) untreated polyamide.

The presence of chitosan was confirmed by SEM investigations. In Figure 14, the SEM images of the polyamide treated with the chitosan solution are shown; the presence of the treatment can be assessed by the formation of bridges between fibers constituted by a thin layer of polymer, visible also after the standard washing (red circles).

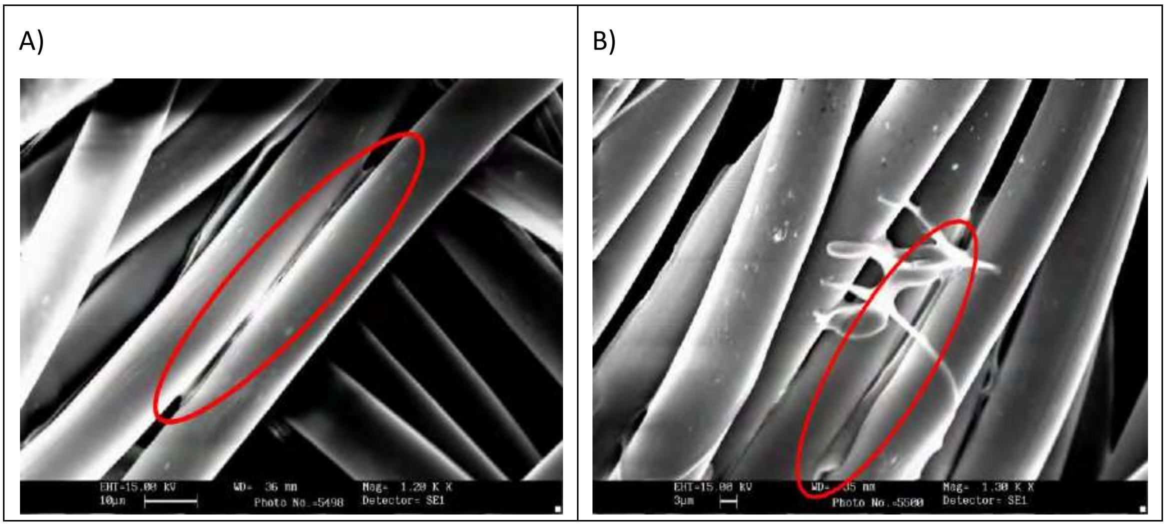


Figure 14. SEM images of polyamide fabrics treated with a chitosan solution with the addition of hydroxylethyl methacrylate. A) treated fabric B) treated fabric after washing (method ISO 105-C06).

In order to characterize the finishing, ISMAC performed also a spectroscopy analysis by FT-IR ATR. The spectra of the untreated and treated polyamide are shown in Figure 15 in comparison with HEMA and chitosan spectra.

The absorption bands, for chitosan, are assigned as follows:

Wavenumbers [cm^{-1}]	Signals
3358	-OH stretch
2918	-CH stretch
1650	-C=O stretch
1587	-NH bend
1150	-C-O-C- stretch
1062	-C-O- stretch

Table 15. Band absorption of chitosan powder.

The characteristic signals referrable to the chitosan are the bands at 1150 and 1062 cm^{-1}, related to the -C-O-C- bond. In the spectrum of the treated polyamide the presence of these signals isn't clear, due to the overlap with the characteristics peaks of the polyamide. Thus, FT-IR spectroscopy was not useful to determine the presence of chitosan on the textile substrate.

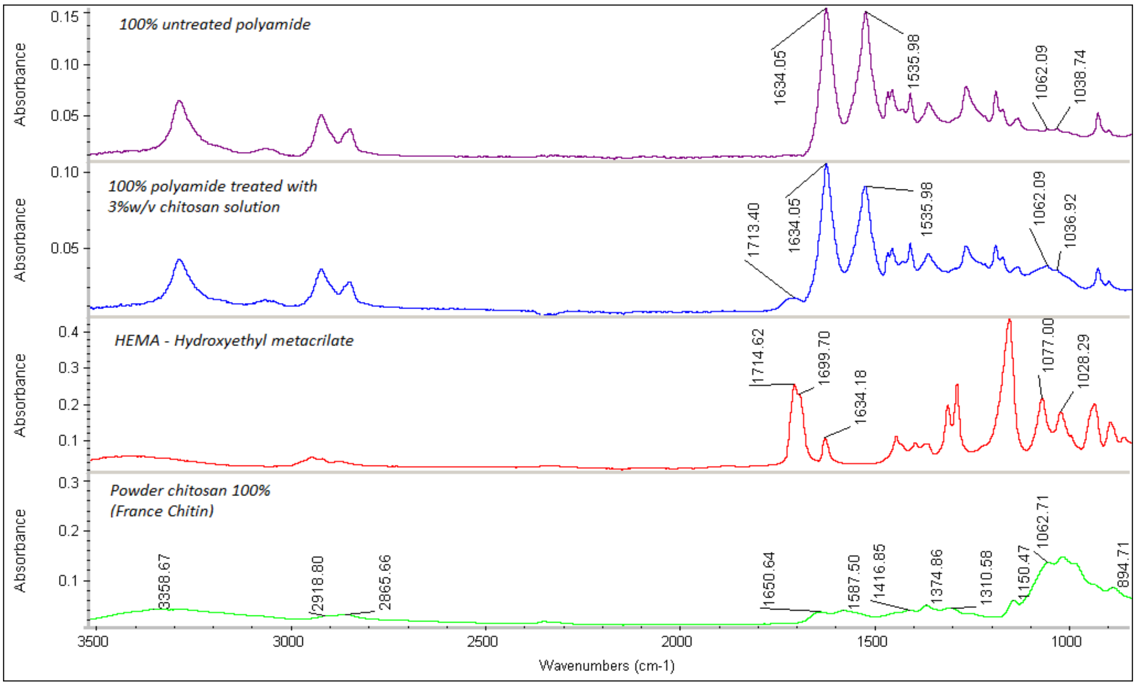


Figure 15. FT-IR ATR spectra of treated and untreated polyamide fabrics, in comparison with chitosan powder and HEMA absorptions.

The characterization of the textile treated with chitosan were also performed on the other standard textile treated, before and after the standard washings. The results obtained from the staining are shown in Figure 16 and Figure 17. These tests allow to confirm the adhesion of the treatment on the fabrics surface, as requested in the project DOW.

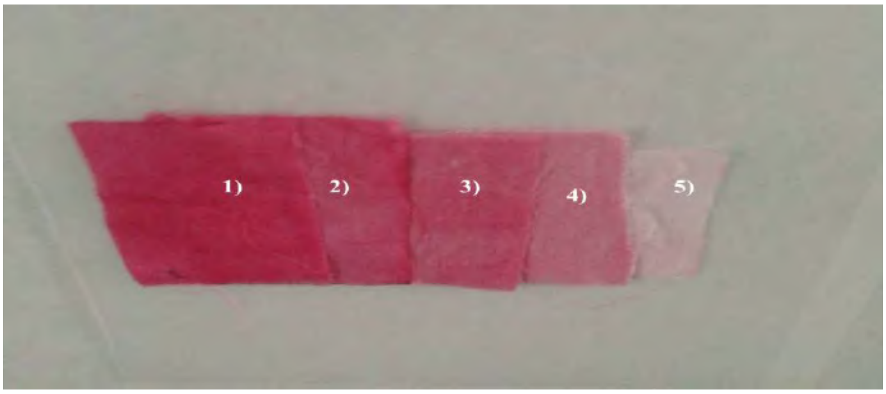


Figure 16. Images of 100% polyester fabrics treated with 3% of chitosan with a load+ squeezing procedure. 1) Polyester treated with 3% chitosan solution, 2) polyester treated and washed with cold water, 3) polyester treated and washed with Triton solution without stainless steel balls, 4) polyester treated and washed with Triton solution, with 10 stainless steel balls 5) Untreated polyester.

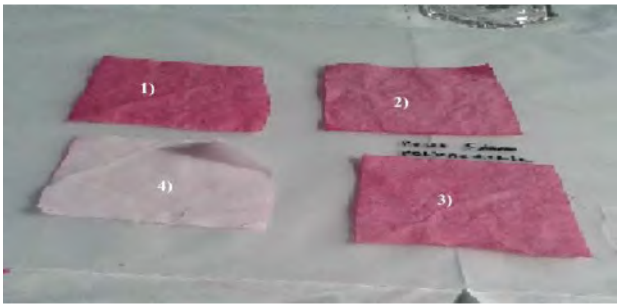


Figure 17. Images of the blend 65% polyester/35% cotton fabrics treated with 3% of chitosan with a load+squeezing procedure. 1) Polyester/Cotton treated with 3% chitosan solution, 2) Polyester/Cotton treated and washed with cold water, 3) Polyester treated and washed with Triton solution, 4) Untreated Polyester.

2. Lab scale treatment by padding of chitosan solution:

The fabrics treated with the lab-scale padding system were characterized, in order to evaluate the fastness of the treatment to standard washings and the release of microplastics.
In Figure 18 the flow chart followed for the characterization is shown. The standard method ISO 105-C06 was modified with the selection of a non-ionic surfactant (Triton), based on CNR experience; indeed strong washing conditions and anionic surfactants could affect the chitosan persistence on the textile surface. The standard washings were performed to characterize the fastness of the finishing treatment but also to quantify the microplastics release coming from the effluents collected by the Linitest.

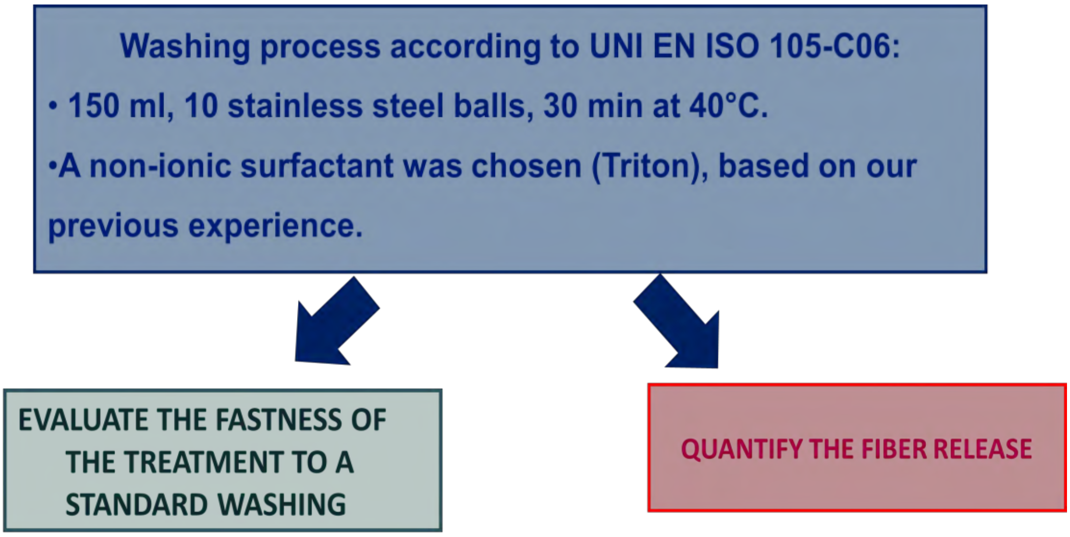


Figure 18. Flow chart of the characterization of the treated textiles.

The results of the fastness tests are shown in Figure 19. The staining method on polyester is particularly effective because untreated polyester can't be with reactive dyes. Indeed polyester can only be died with disperse pigments at high temperatures and pressures, without the formation of a chemical bond. With the staining test the untreated fabric remains raw-white, while the treated fabric becomes pink. Also the treated and washed fabric turns pink, thought the color seems to be less bright than the unwashed.

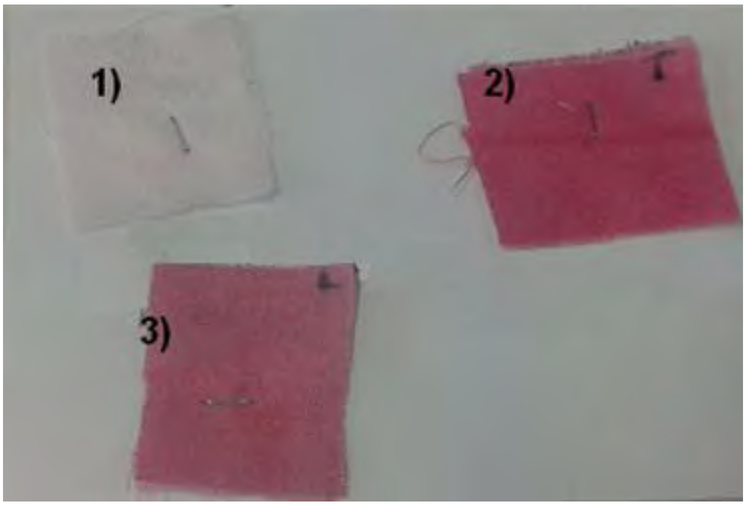


Figure 19: Staining test on polyester fabric treated with chitosan; 1) untreated 2) treated 3) treated and then washed.

The results of the fiber release quantification are shown In Figure 20, for both detergent and blank tests. In the diagram is also reported a red line, representing the maximum value achieved with the experiments performed with the untreated fabric (reference) washed with a detergent solution (1 g/l Triton). All the mean values achieved for the treated fabrics are below the maximum threshold, reaching a reduction of 27 and 18% for the polyester treated with 3% and 2% chitosan solution respectively. However, the data obtained from the repeatability tests performed on the treated fabrics, show a quite high dispersion, in comparison with the analysis on the untreated one, whose results are shown in Figure 21.

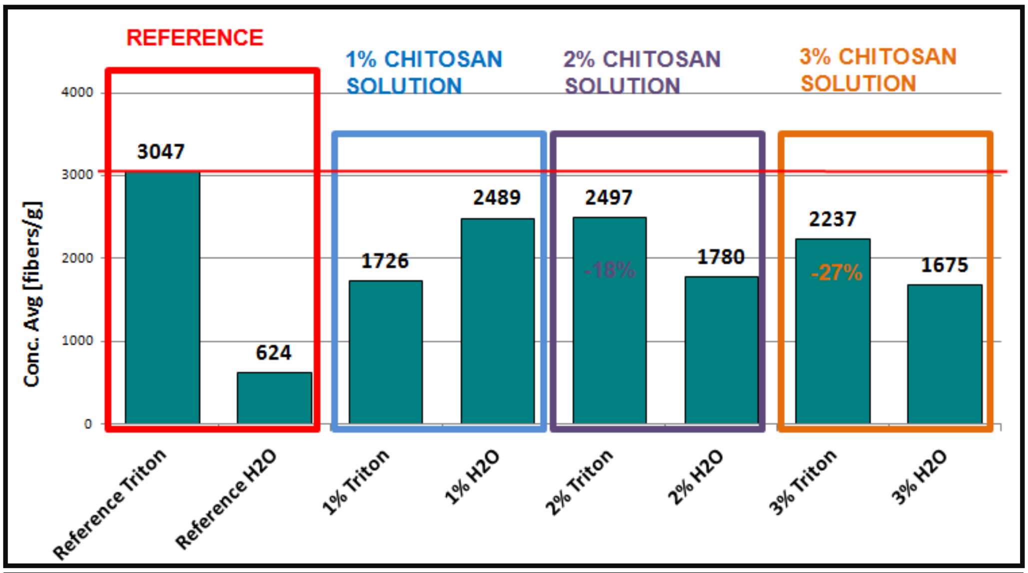


Figure 20. Quantification of microplastic release from standard woven fabrics made of 100% polyester, expressed as fibers for each gram of fabric tested. The results represented in the diagram are mean values, obtained by at least 2 experiments.

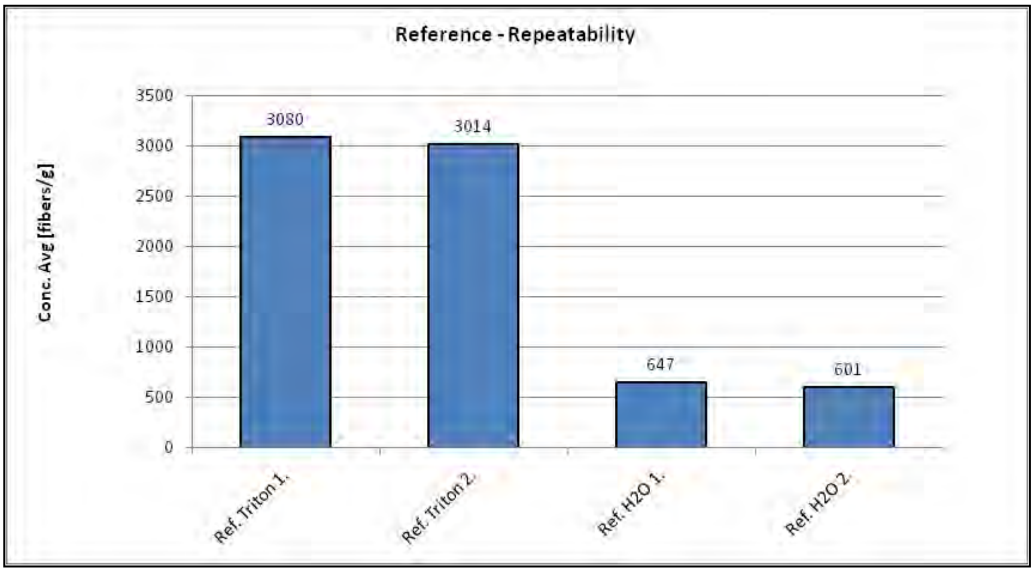


Figure 21. Quantification of microplastics release, espressed as fibers for each gram of fabric. Repetability tests performed on the untreated fabrics (reference).

B 1.2.7 Application and characterization of pectin formulation

Pectin is a mainly linear polysaccharide whose principle chemical unit is based on D-galacturonic acid monomer (Figure 22), occasionally alternated to L-rhaminose units or other monomers such as sugars as D-galactose, L-arabinose, D-xylose, L-rhamnose, L-fucose, and traces of 2-*O*-methylfucose. It can be mainly found in cell walls of several vegetal species, where it is responsible for maintaining plant structure and support.
It has high molecular weight, ranging from 50,000 to 180,000 g/mol. Depending on the degree of substitution of D-galacturonic acid carboxyl groups by methoxyl groups, pectin may be classified with high (over 50%) or low (below 50%) methoxylation/esterification grade (EG).
Such polysaccharide represents an interesting biomaterial because cheap and abundantly available, being a waste product of fruit juice, sunflower oil, and sugar manufacture. Commercially available pectin is mainly extracted from suitable agro-by-products like citrus peel and apple pomace and used in the food industry as natural ingredients for their gelling, thickening, and stabilizing properties. Thus, it can be supposed to be safe for human health.
What makes such material also chemically interesting is represented by its heterogeneous and complex chemical structure, rich in esteric, carboxyl and hydroxyl group, responsible for the peculiar pectin high reactivity, rendering it sensitive to plenty of common reagents.
The choice of pectin as suitable material for application in textile finishing treatments, was suggested, besides by its reactivity and biocompatibility, also by the similarity of its structure to other polymers, abundantly used for natural and synthetic textiles treatment, such as tragacanth, chitosan and alginate.
However, the greatest problem found to its use in this field is related to its solubility in aqueous medium, that would cause the loss of the functional treatment during the garments washing.
The best way to reduce the high solubility of polysaccharides is to chemically modify them, without affecting too much their natural structure, by masking the most polar groups, such as the carboxyl and hydroxyl, through their conversion into esteric units. One example is given by the use of glycidyl methacrylate (GMA) that makes the polymer more stable and less water soluble. Furthermore, such acrylic derivative displays also the advantage to introduce in the new cross-linking product, Pectin-GMA, another reactive functionality, useful for the next grafting on the polyamide fabrics surface. The objective of this modification reaction is the introduction of vinylic groups in the polysaccharide structure, that will later react through free radicals and generate a bridge between pectin and polyamide, with formation of a good film on the fabrics surface.

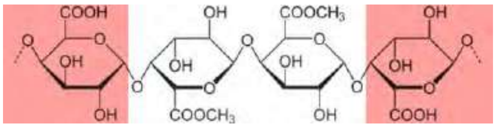


Figure 22. ***Chemical structure of the polysaccharide Pectin; in red the galacturonic acid residues.***

EXPERIMENTAL SECTION

1. Materials and Methods

The raw 100% polyamide-6,6 woven fabric (item code 361, weight of 130 g/m^2) was purchased by Testfabrics Inc. USA. Citrus Pectin Classic CU 701 low EG was supplied by Herbstreit & Fox, glycidyl methacrylate (GMA, 97%) by Sigma–Aldrich and Dibenzoyl Peroxide (BPO, ≥ 97%) by Fluka. Milli-Q deionized water was used as a solvent and reagent grade ethanol by Sigma-Aldrich.

2. Synthesis of Pectin-GMA

1g of Pectin (around 0.005 moles of monomer) was dissolved in 50ml of water under continuous magnetic stirring for 24 hours, resulting in a pale yellow solution. Then, 0.68 ml of GMA (0.005 moles) was added to the pectin solution and the mixture was kept under continuous magnetic stirring for 24 hours in nitrogen atmosphere at 50°C. The cross-linking product was precipitated with ethanol as a gel. The precipitate was dried under vacuum and weighted. The yield of the cross-linking reaction was of about the 60%.

3. Finishing treatment of Polyamide fabrics with Pectin-GMA

The Pectin-GMA gel was dissolved in water by stirring the mixture for 2 hours at 40°C and then, 0.5 g of polyamide fabrics, cut in square and previously wetted with MilliQ water, were dipped into the solution for the preliminary adhesion/adsorption of the pectin-GMA product and stirred for 1 hour at 50°C. Then, the fabrics were removed from the solution and 0.24 g of BPO was added, as initiator. In order to quantitatively solubilize this powder, the resulting mixture was sonicated for 4-6 minutes (power 25%).
Once the BPO was completely dissolved, the fabrics were dipped again and the mixture was kept under continuous magnetic stirring for 2 hours in nitrogen atmosphere at 70°C. The final reaction is activated by the relatively high temperature.
After 2 hours, the polyamide fabrics were removed from the mixture and the remaining solution was collected as reference. Both fabrics and reaction mixture were kept in the oven for 24 hours to dry at 40°C. After 24 hours, the polyamide square textiles were rinsed with distilled water to remove the unreacted BPO from the surface and dried in air.
Preliminary tests were performed in order to set the described conditions. In particular, the textiles were analysed upon 1, 2 and 24 hours of treatment in the Pectin-GMA/DBPO mixture. For such samples, besides the normal drying at 40°C overnight after the removal from the mixture, also a pre-treatment at 100°C for 3 minutes (to activate the radicalic reaction) was attempted (before the 40°C drying step). With such latter additional step,

the morphological analysis revealed worst results for the resulting treated samples and the same was revealed for the immersion of the textiles for 1 and 24 hours in the Pectin-GMA/DBPO mixture.

CHARACTERIZATION OF PECTIN TREATMENT

A. SEM analysis

The morphological characterization of the pectin-GMA-polyamide textiles was performed by using a scanning electron microscope, SEM, FEI Quanta 200 FEG, in low vacuum mode (PH_2O = 0.7 torr), using a large field detector (LFD) and an acceleration voltage of 5-20 kV, upon application of metal coating.

B. FTIR-ATR analysis

Attenuated Total Reflectance (ATR) Fourier Transform InfraRed (FT-IR) spectral analyses of neat Pectin, of Pectin-GMA film and pectin-GMA-polyamide textile samples were recorded by means of a Perkin Elmer Spectrum One FTIR spectrometer, equipped with the Universal ATR accessory, using 32 scans and a resolution of 1 cm^{-1}, over the range 4000–400cm^{-1}.

C. TGA analysis

Thermal stabilities of neat Pectin, of Pectin-GMA film, meat polyamide and pectin-GMA-polyamide textile samples were evaluated with a Perkin Elmer Pyris Diamond TG/DTA thermogravimetric analyser. A small piece of each sample was placed in a platinum open pan and heated from 30 to 850°C at 10° C/min. High purity nitrogen was fluxed through the furnace at a flow rate of 50 mL/min.

RESULTS AND DISCUSSION

The reaction responsible for the formation of the Pectin-GMA product is in charge of the epoxide group of GMA, whose three membered ring reacts with a carboxyl group of the pectin monomer through a nucleophilic substitution reaction (Figure 23, left). It induces the ring opening of GMA and the esterification of the pectin carboxyl group. Such reaction, hiding the highly polar carboxyl group, has the final aim to reduce the water solubility of pectin on the textiles surface in view of textile finishing applications.
GMA was chosen because it displays two useful reactive sites: the epoxy ring and acrylic double bond. While the former represents the attaching site for pectin in the first reaction, the latter is important for the link with polyamide. The grafting reaction of pectin-GMA cross-linking product on the textile surface involves actually the GMA double bond, and occurs through a free radical chemical polymerization method using BPO, as common redox initiator system. Such peroxide, under proper heating conditions, dissociates by means of an homolytic cleavage giving two reactive radicals, $BPO^{\bullet}$, working as starters for the next reaction.

The hypothesised mechanism for such step comprises the removal of an hydrogen atom of the polyamide backbone from the radical, with consecutive formation of free radical active centres on the textile surface. Such sites are responsible for the addition to the double bond of GMA with formation of a new bond among the nitrogen atom of the amide group of the fabric and the carbon of GMA involved in the broken double bond (Figure 23**,** right). The reaction ends when the collision of two radicals occurs.

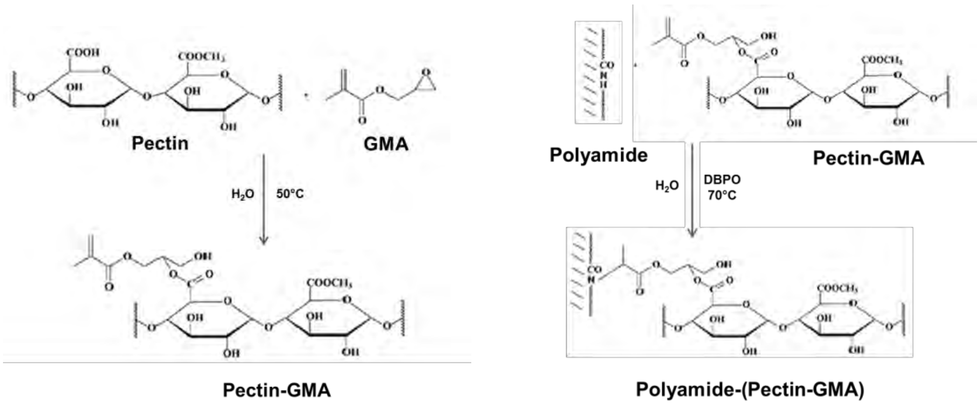


Figure 23. ***Reactions schemes: Pectin-GMA synthesis (left) and Woven Polyamide 6.6 grafting (right).***

The SEM investigation showed good perspectives for the pectin application in the textile finishing field, since it displayed the efficacy of the coating treatment. Actually, in some regions of the textile surface of the polyamide fabrics, a pectin based coating was observed among two or three neighbouring fibers (Figure 24). Anyway, the film did not appear perfectly homogeneous and continuous over the fabric surface and agglomerates of the products were encountered in some regions, suggesting both low adhesion tendency towards the polyamide and self-reticulation of the Pectin-GMA. This behaviour clearly introduces the need to improve the finishing procedure, since the filming ability of the used products is visible but its adhesion on the fabrics is still not optimal.

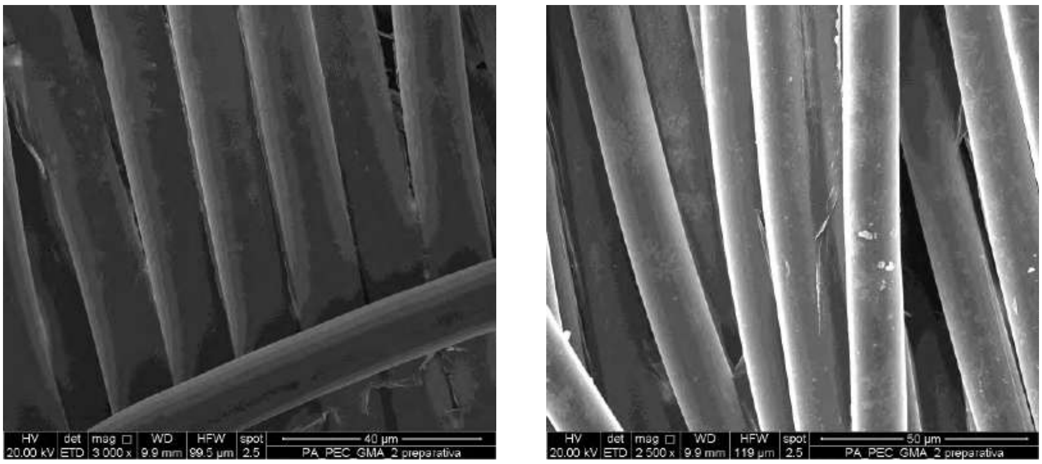


Figure 24. ***SEM micrographs of Woven Polyamide 6.6 treated with Pectin-GMA.***

FT-IR spectra were not particularly diagnostic since no evident and completely new signals appear as result of the polyamide grafting and actually the bands related to the main transitions in the neat textile corresponds to those observed in the treated one. Just in the regions 1160-850 cm^{-1} and 1700-1800 cm^{-1} , an increase of the intensity of absorption for the treated polyamide with respect to the neat one can be observed, ascribable to the presence of pectin-GMA product on the treated textile (Figure 25).

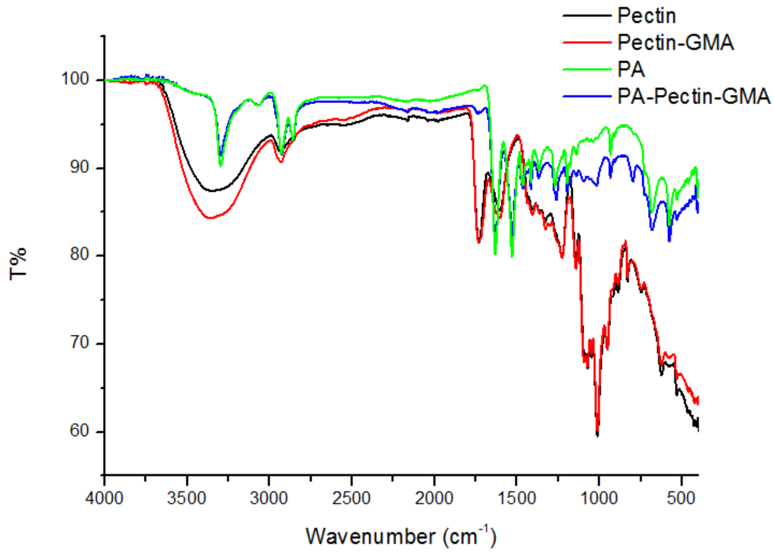


Figure 25. *FT-IR ATR spectra of neat Pectin (black), Pectin-GMA (red), neat Woven Polyamide 6.6 (green) and treated with Pectin-GMA (blue).*

Thermal stability of woven polyamide textile upon treatment with Pectin-GMA was evaluated by means of thermogravimetric analysis in comparison with Pectin, Pectin-GMA and neat Woven Polyamide**.**

As clearly visible from the degradation curves (Figure 26) reported for the neat textile with respect to the treated polyamide, under nitrogen flow, the temperature corresponding to the maximum weight loss does not appear significantly affected by the treatment and actually for the two sample T_d is around 450°C. The second step of degradation instead displays differences among the two samples: the neat PA degrades more slowly than the treated one. The faster degradation step in the treated sample maybe should be attributed to the Pectin fraction on the fabric surface but anyway it is not important for the field of application discussed since such high temperatures should be out of interest.

The TGA curves for unmodified and GMA modified Pectin display higher number of degradation steps. It can be observed an initial mass loss between 30 and 200°C for both samples ascribable to the desorption of humidity from the polysaccharide structure. The second decomposition step starts at approximately 210 ◦C and occurs for both samples with very similar degradation rate. The third one starting around 260°C appears clearly faster for neat pectin, as evidence of the stabilizing effect of the cross-linking reaction with GMA.

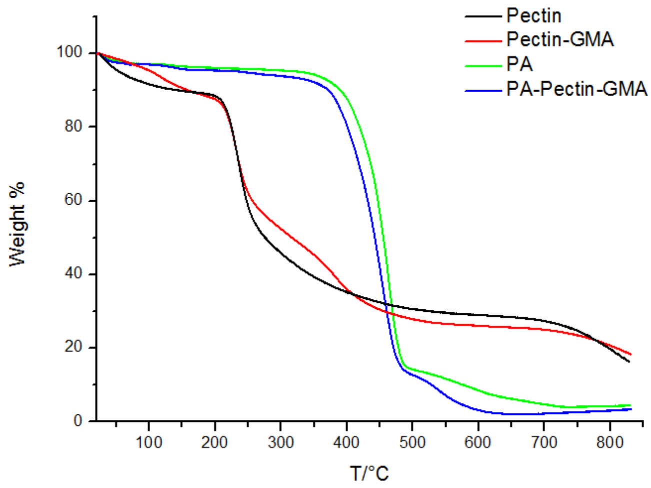


Figure 26. ***TGA curves of neat Pectin (black), Pectin-GMA (red), neat Woven Polyamide 6.6 (green) and treated with Pectin-GMA (blue) normalized for the weight.***

B 1.2.8 Conclusions

- All selected products in the preliminary action A.2.1 have been applied successfully in a laboratory scale. Some of the variables in the process, such as auxiliary concentration and padding speed have been modified to see the effect in the final performance. Three different auxiliary concentrations have been tried: 10, 20, 30 g/L. However no differences in the measured pilling indexes have been observed.

- Process parameters have been adjusted to obtain homogeneous and reproducible finishes. The modification in the padding speed, for instance, has resulted in a reduction of the absorption or *pick-up.* However this change is not enough to impact on the mechanical properties of the treated cloth.

- A complete characterization of the treated fabrics has been carried out including morphological, mechanical and washing resistance studies. Woven polyester fabric has shown the lowest properties against abrasion, determined by pilling Martindale, and as a consequence, it has been selected to start the study with the different textile auxiliaries. The applied treatments have shown some important effects against pilling and microfiber loss. The pilling formation can be slightly reduced by applying an acrylic treatment on the fabric. *Polyacril 73* and *Polyacril 56ECO* have presented the best pilling results. However, flexural rigidity tests demonstrated that the touch of these treatments is highly altered and they present a high rigidity, specially *Polyacril 56ECO*. This result is expected since the acrylic resins form a protective layer on the fabric, reducing significantly the pilling formation under abrasion. On the contrary, polyester treated with *Polysilk-CTE (polysiloxane)* results in a softer touch than the untreated fabric although it presents worse pilling values.

The washing resistance study in Lini-test has been critical to select an optimum fabric treatment to solve the problem of microfiber release. When a conventional liquid detergent is used in the Linitest study, the results of fiber release get considerably worse than using just distilled water to wash. This test has concluded that the most effective treatment to reduce the microplastics in the washing water is *Polysilk-CTE*. This may be explained by the softening effect of this auxiliary that reduces the friction between the detergent and the fabric. The acrylic treatment in contact with water swells and, in combination with the liquid detergent, it is easily eliminated from the surface of the fabric. The removal of this layer might cause the loss of more microfibers which results in worse values than the untreated fabrics. The same trend observed in the polyester fabric has also been detected in 100% polyamide and 100% acrylic woven fabric. As a consequence, it is assumed that the behaviour observed in these fabrics will be the same for others, as it is due to a physical effect more than a chemical interaction between fibers and auxiliaries.

- Regarding the bio-based textile treatments on woven polyester it is concluded that the chitosan solution presents good results in terms of fiber release compared to the untreated fabric. However, pectin treatment still needs optimization to achieve a higher adhesion to the fibers.

- Finally, for the next pilot plant implementation trials (Task B.1.3), the selected textile auxiliaries have been:

- *Polysilk-CTE* to decrease the microfiber loss after washing (in a lab-scale equipment) and to improve durability and effectiveness of the finish.
- *Polyacril 73* to avoid the pill formation without much damage to the fabric touch.

TASK B.1.3 Pilot plant implementation of new textile additives

The two best finishing treatments identified by LEITAT were applied at pilot scale, with the contribution of "Tintoria Finissaggio 2000", a company that performs finishing, printing and dyeing processes in outsourcing. Each of the two treatments chosen, with Polysilk CTE and Polyacril 73 ECO, were applied on three meters of standard polyester woven fabric bought by Ausiliari Tessili S.r.l. (Ref. 777). After the treatment a characterization of the textile properties was performed, regarding the pilling tendency, physical and microscopic properties and the adhesion of the product to the textile surface (fastness of the finish). The industrial scale treatment with Polysilk is shown in Figure 27.

Figure 27. ***Impregnation, padding, drying and curing process in the industry.***

Regarding the water and energy consumption, 60 litres of bath resulted to be necessary to guarantee the fabric impregnation. With this bath capacity, 50 meters of fabric can be treated. Therefore, it is estimated a water consumption of about 1,2 litres for meter of fabric.

The actual energy consumption for this process (padding, drying and curing) is about 70 KWh.

The amount of methane used from the company is about 800-900 m^3/d, distributed in 20 working hours, corresponding to 45 m^3/h; however, when it is necessary to perform a curing above 150°C, the thermal power of the exhaust gases is recovered from a two step heat exchange, with a first thermal exchange step exhaust/air and a second one exhaust/water. The line is able to process only one exhaust flow at once.

B 1.3.1 Pilot-scale implementation of the finishing treatment with Polysilk CTE

Polysilk CTE is an amino-functional elastomeric siliconic macro-emulsion. It is a commercial product, provided by Polysistec, that aims to give a soft and full handle effect and a surface smoothness. At lab scale, the application of this product showed the best performance for the retention of microplastics deriving from standard domestic washing processes.

For the pilot scale application, the parameters of the padding treatment were chosen in order to adapt the lab scale process to the industrial requirements and were set as follows:

- v = 4 m/min (average speed of the fabric in the padding system)
- P = 3 bar.

The pH of the bath was adjusted with acetic acid, reaching a value of 4,7.

A drying process was performed in a continuous oven at 110°C, according to the process specifications. The oven was composed by six modules of 3 meters and the drying velocity was set at 18 m/min in order to guarantee a residence time of 1 minute. The reticulation was then achieved in a continuous oven with a velocity set at 10 m/min as a precautionary measure, in order to ensure the curing.

SEM CHARACTERIZATION

After the padding treatment, the fabric was characterized and the presence of the finishing product and its adhesion to the fabric surface were confirmed with SEM investigations.

In Figure 28, the SEM images of the polyester fabric treated with Polysilk-CTE are shown.

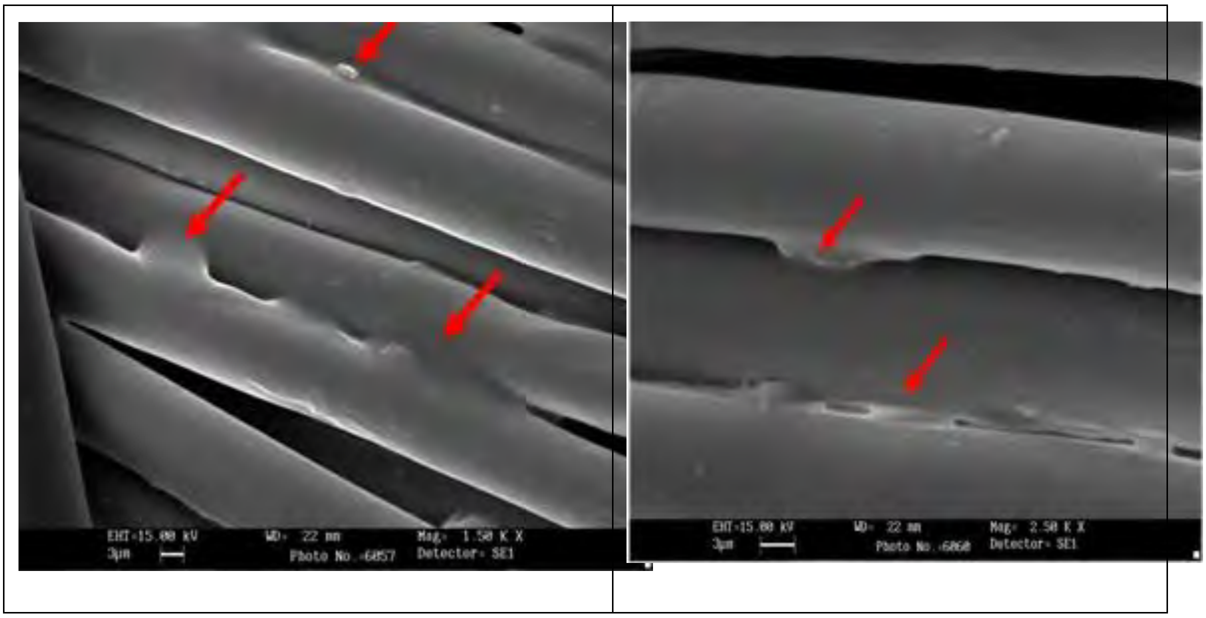


Figure 28. SEM images of the woven standard fabric treated with Polisilk CTE.

After the finishing, polyester fabrics were washed in order to assess the adhesion of the treatment and also to confirm the reduction of microplastics released.

The washings were performed in Linitest according to ISO 105-C06 modified, the same procedure explained in section B 1.2.3 (Washing resistance: Characterization and quantification of micro and nanoplastics).

The presence and the surface morphology of the finishing product were confirmed by SEM analysis (Figure 29) performed on the polyester samples after the standard washings.

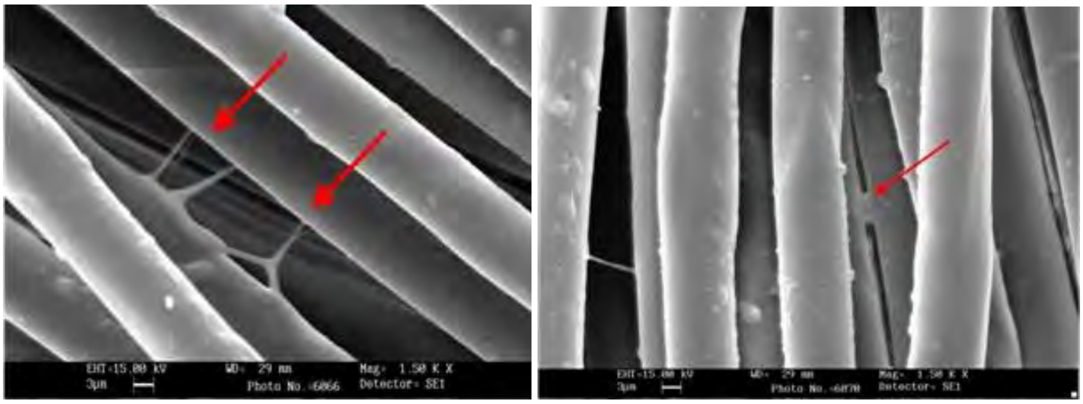


Figure 29. SEM Images of the woven standard polyester fabric treated with Polysilk after one washing cycle with (a) water and (b) washed with detergent solution, according to ISO 105-C06 modified.

<u>FT-IR CHARACTERIZATION</u>

The fabric treated was also characterized by FT-IR. Table 16 and Table 17 show the characteristic absorption bands of the untreated PES (Ref.777) and the auxiliary Polysilk-CTE respectively. The auxiliary used, Polysilk CTE, is an amino-functional elastomeric silicone macro-emulsion. The most relevant signals related to its presence are represented by the vibrations of the bonds formed by silicon. In particular, the absorption bands at 1098 and 1029 cm-1 , related to the Si-O-Si stretching vibration identify the nature of the product: when the two signals are separated and distinguishable, the chemical structure shows an organic character; on the contrary, silica presents a unique broad signal (Polysilk-CTE).
CNR analyzed the dry matter coming from the auxiliary product (Polysilk CTE, dried at 105°C); a comparison between the spectra of the treated, untreated polyester and the finishing product is shown in Figure 30.
On the treated fabric, the peaks related to the presence of the finishing treatment are not clearly visible, probably due to the low percentage of the product load and to the overlapping with the characteristic signals of polyester bond vibrations.

Wavenumbers [cm^{-1}]	Peak absorption
1713	C=O Bond
724	Aromatic ring vibration
1340-1177	–CH2 bending vibration
1242	C-O bond vibration (stretching)
1095	C-O bond vibration crystalline fraction (stretching)

Table 16. Absorption peaks of polyester fabric.

Wavenumbers [cm^{-1}]	Peak absorption
1258	-CH3 asymmetric deformation (-Si- CH3)
1098	Si-O-Si stretching
1029	Si-O-Si stretching
796	-Si-C- stretching and - CH3 rocking

Table 17. Absorption peaks of Polysilk-CTE (dry matter, dried at 105°C)

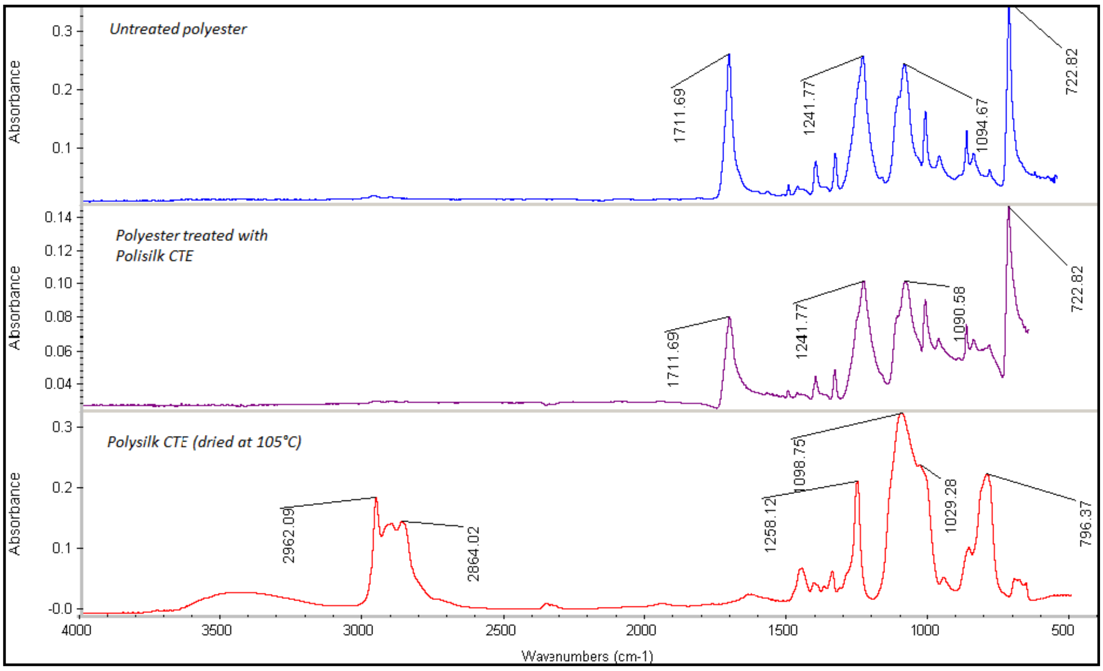


Figure 30. FT-IR spectra of the polyester reference, treated polyester and Polysilk-CTE.

PILLING CHARACTERIZATION

The textile characteristics of the treated fabric were also evaluated. CNR performed the pilling test with Martindale according to ISO 12945/2 standard; the results are shown in Table 18. This analysis allowed to confirm the data achieved at lab scale: the application of Polysilk-CTE led to a slight worsening of the pilling tendency. Related to these results we could suppose that the mechanical stress related to a single standard domestic cycle is not enough to lead to the pill formation while the daily use of the textiles can cause an higher mechanical stress that induces the phenomenon and affects the microplastic loss. This will be confirmed in the next *Action B4* (Pilot Scale Washing Experiences).

Material tested	Pilling Index (2000 cycles)	Pilling Index (5000 cycles)
Woven PES 777 (control)	2	1-2
Polysilk-CTE	1	1

Table 18. Pilling tendency with Martindale according to ISO 12945/2.

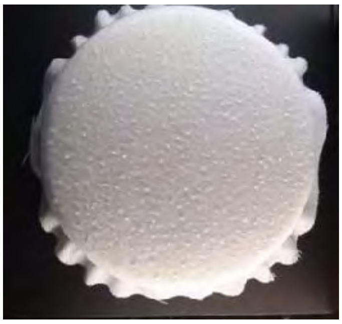


Figure 31. Image of standard woven polyester samples treated with Polysilk CTE after the pilling test with Martindale (ISO 12945/2) ; A) image of the sample after 2000

The quantity of product load was defined by the determination of the mass per unit area, according to ISO 12127:1999 (Table 19). The weight gain (at 20°C, 65% relative humidity) resulted to be about 7%.

Material tested (conditioned, 20°C, 65% RH)	Weight [g/m2]
Woven PES 777 (nominal value)	126
Woven PES 777 (measured value)	130,3
Polysilk-CTE	139,5

Table 19. Determination of the mass per unit area according to ISO 12127:1999.

B 1.3.2 Pilot-scale implementation of the finishing treatment with Polyacril 73 ECO

Polyacril 73 ECO is a self-crosslinkable acrylic resin. The same version of Polyacril 73, used in B.1.2, but nonylphenols and formaldehyde free. Useful for coating and padding processes on polyamide, polyester fabrics and their blends. At lab scale, the application of this product showed the best performance for the reduction of the pilling tendency of textiles, but led to a slight increase in the microplastic release.

The parameters of the treatment were chosen in order to adapt the lab scale process to the industrial requirements and were set as follows:

- v= 4 m/min (average speed of the fabric alimentation to the padding system)
- P= 3 bar.

The pH of the bath did not need to be adjusted.

After the padding, a drying process was performed in a continuous oven at 110°C according to the process specifications. The oven was composed by six modules of 3 meters, and the drying velocity was set at 18 m/min in order to guarantee a residence time of 1 minute. The reticulation of the polymer was then performed in a continuous oven, with a velocity set at 10 m/min as a precautionary measure, in order to ensure the occurrence of the curing.
The definition "acrylic resin" includes a wide group of polymers and co-polymers obtained by the polymerization of acrylic and methacrylic monomers. The general structure of the polymers belonging to this class is reported in **Errore. L'origine riferimento non è stata trovata.**Figure 32. In Table 20 the characteristic absorption peaks of this resin are reported.

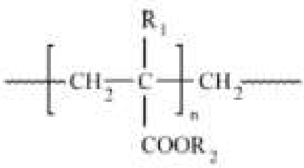


Figure 32. General structure of acrylic resins, where R1 and R2 can be:

R1= H, R2= CH3 (Polymethylacrylate)
R1=CH3, R2=CH3 (Polymethylmethacrylate)
R1=CH3, R2= CH2-CH3 (Polyethylmethacrylate)

FT-IR CHARACTERIZATION

Wavenumbers [cm^{-1}]	Peak absorption
3440	-OH stretch
2958-2929-2872	-CH stretch
1736	-C=O stretch

Table 20. Absoption peaks of Polyacril 73 ECO (dry matter, dried at 105°C)

In Table 20 the spectrum of Polyacril 73 ECO is reported. Figure 34 shows a comparison between the spectra of the treated and untreated polyester fabrics; it can be noticed that the signals related to the presence of the finishing product are not visible in the diagram, due to the overlapping with the polyester bond vibrations.

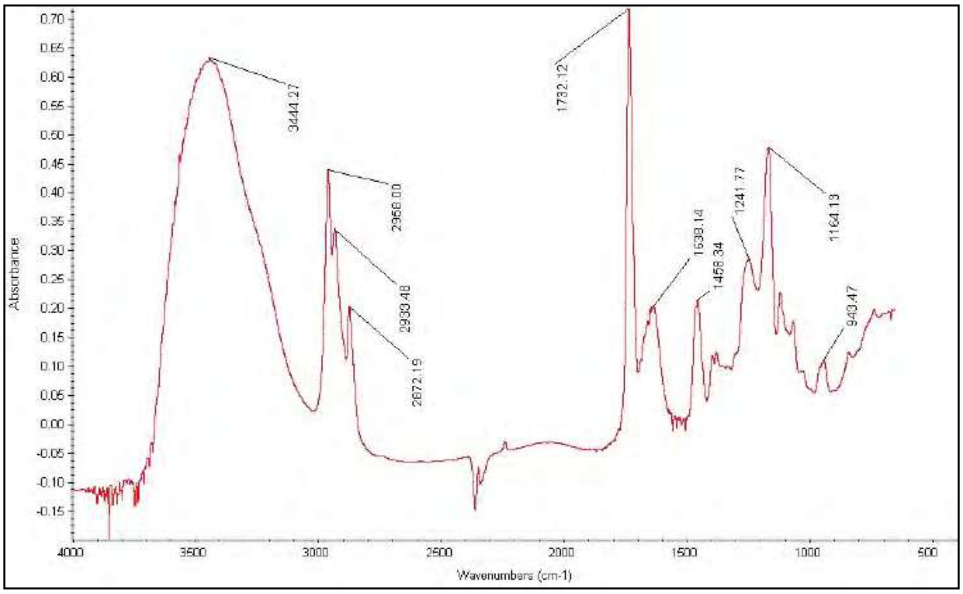


Figure 33. IR Spectrum of Polyacril73ECO.

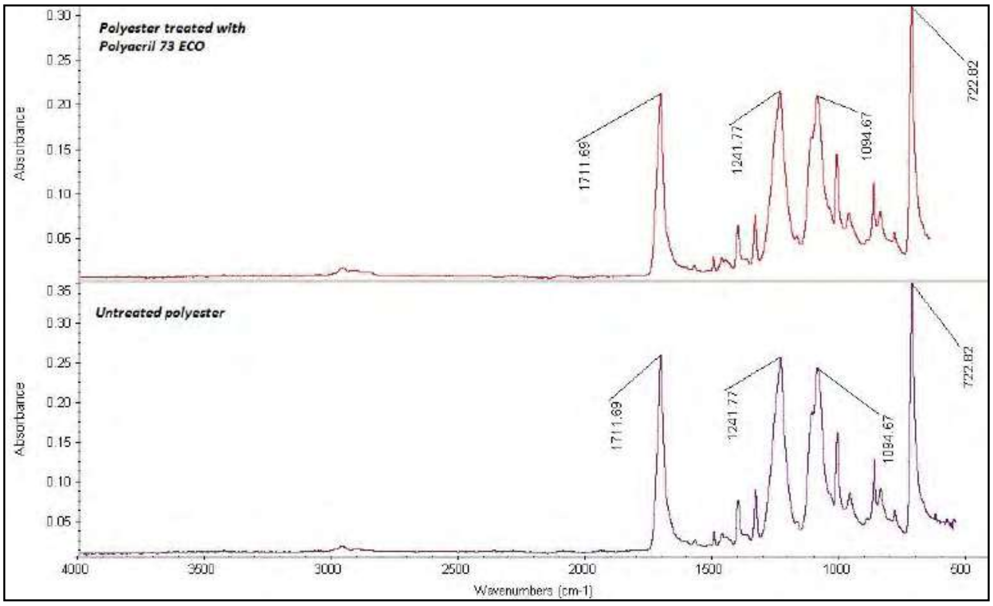


Figure 34. IR Spectra of polyester treated with Polyacril73ECO and untreated.

SEM CHARACTERIZATION

The presence of the coating was confirmed by SEM analysis. In Figure 35 the SEM images of the treated and untreated polyester are shown.

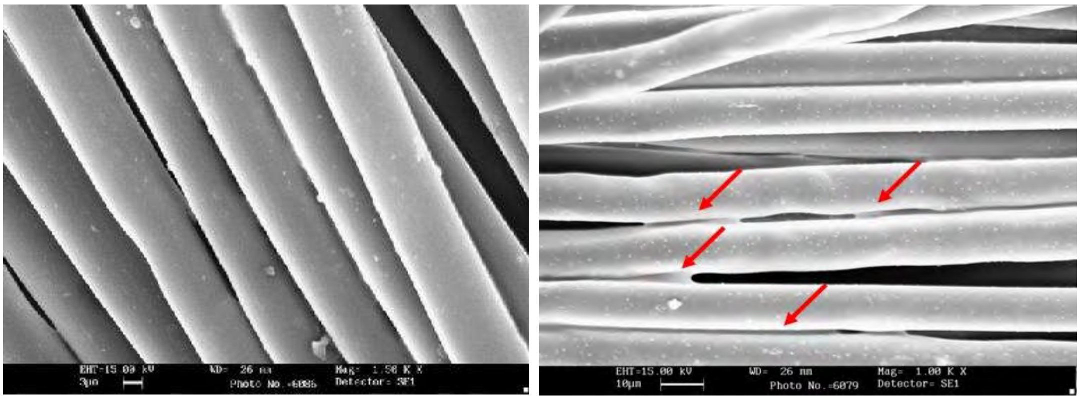


Figure 35. SEM images of a) untreated polyester woven fabric and b) polyester treated with Polyacril73ECO.

After the finishing, the treated polyester fabrics were washed in order to assess the adhesion of the treatment and also to confirm the reduction of microplastic release. The washing was performed in Linitest with the same procedure explained in section B 1.2.3 (Washing resistance: Characterization and quantification of micro and nanoplastics). Figure 36 shows the images of the polyester fabrics treated with Polyacril 73 ECO and then washed either with only water or a detergent solution; the presence and the adhesion of the finishing product are clearly visible and confirmed.

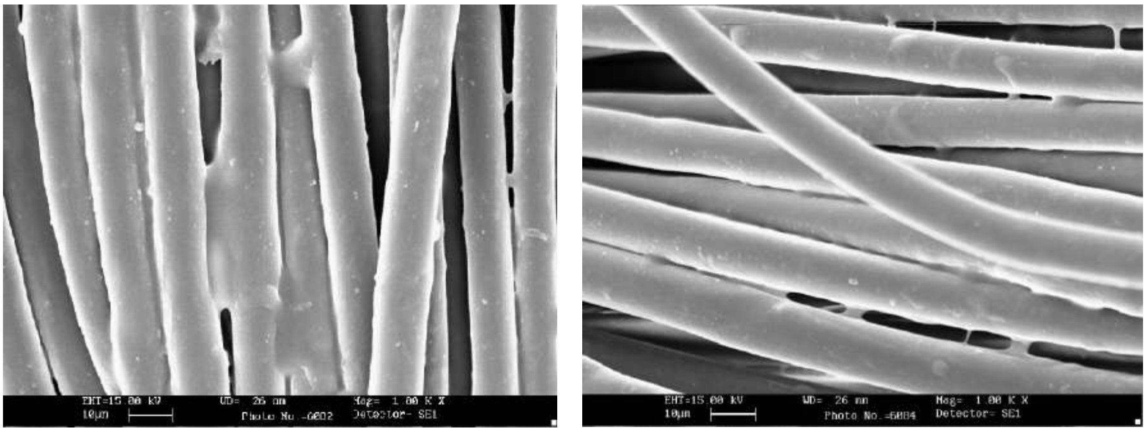


Figure 36. Polyester woven fabric treated with Polyacril73ECO and washed according to ISO 105-C06 modified with a) water and b) detergent solution.

PILLING CHARACTERIZATION

The textile characteristics of the treated fabric were also evaluated. CNR performed the pilling test with Martindale according to ISO 12945/2 standard; the results are shown in Table 21.

Material tested	Pilling Index (2000 cycles)	Pilling Index (5000 cycles)
Woven PES 777 (control)	2	1-2
Polyacril 73 ECO	1-2	1-2

Table 21. Pilling tendency with Martindale according to ISO 12945/2.

The quantity of finishing product loaded on the textile was defined by the determination of the mass per unit area, according to ISO 12127:1999 (Table 22 shows that the weight gain at 20°C, 65% relative humidity is about 2,76%).

Material tested (conditioned, 20°C, 65% RH)	Weight [g/m2]
Woven PES 777 (nominal value)	126
Woven PES 777 (measured value)	130,3
Polyacril 73 ECO	133,9

Table 22. Determination of the mass per unit area according to ISO 12127:1999.

B 1.3.2 Solid state NMR analysis

Two samples of polyester fabric treated with Polyacril 73 ECO and Polysilk CTE respectively were analyzed trough solid state nuclear magnetic resonance spectroscopy (NMR) in order to determine the quantity of grafted additive on the fiber surface. Solid-state 13C magic angle spinning (MAS) spectra were collected on a Bruker Avance II 400 spectrometer operating at a static field of 9.4 T, equipped with a 4 mm MAS probe. Finely ground samples were packed into 4 mm zirconia rotors sealed with Kel-F caps and spun at a spinning speed of 9 kHz. All spectra were referenced to external adamantane (CH signal at 38.48 ppm downfield of tetramethylsilane (TMS), set at 0.0 ppm).

On the sample treated with Polyacril 73 ECO, cross-polarization (CP) spectra were recorded with a variable spin-lock sequence (ramp CP-MAS), using a 1H π/2 pulse width of 3.6 μs, a contact time of 2 ms and a repetition time of 4 s. On the sample treated with Polysilk CTE only direct polarization (SP) spectra are reported, due to the low efficiency of cross polarization on the highly mobile siloxane chains. SP spectra were recorded using a 13C π/2 pulse width of 3.6 μs and a repetition time of 40s.

The spectrum recorded on the Polyacril-treated polyester shows a signal attributed to the methylene moieties of polyacrylic chains (≈ 25 ppm, red circled in Figure 37). An approximate quantitative analysis of the spectrum reveals the presence of 0.8 wt% of Polyacril on the fibers.

The spectrum of Polysilk-treated samples shows a narrow signal at ≈ 1 ppm (red circled in Figure 38), compatible with the CH3-Si groups of siloxane chains. An approximate quantitative analysis of the spectrum reveals the presence of 0.5 wt% of siloxane on the fibers. In both Figure 37 and Figure 38 the spinning sidebands are indicated by dots.

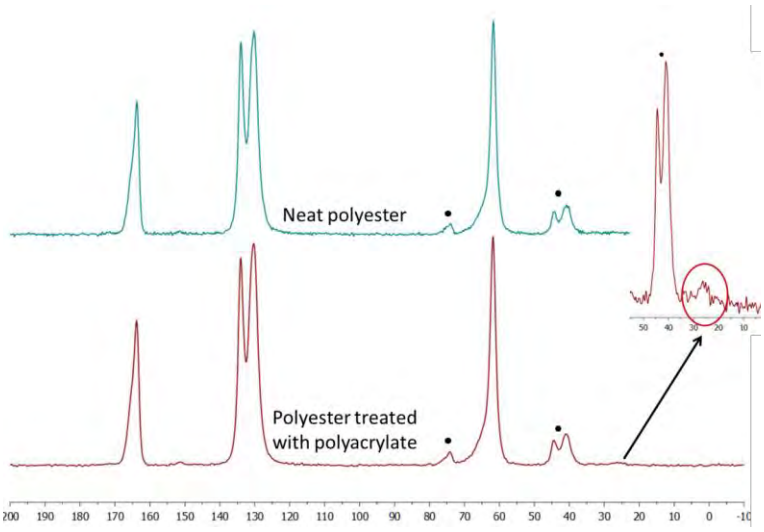


Figure 37. Cross-polarization spectrum of neat and polyacrylic-treated polyester fibers. In the insert, a magnification of the aliphatic region is reported highlighting the signal attributed to the acrylate modifier.

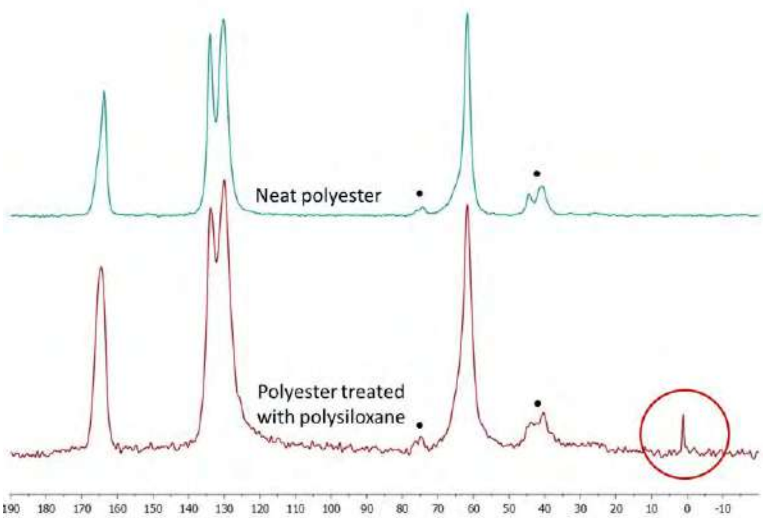


Figure 38. Direct excitation spectrum of neat and polysiloxane-treated polyester fibers. The signal attributed to the siloxane modifier is highlighted in red.

B 1.3.3 Calorimetric analysis

Differential scanning calorimetric analyses of the neat polyester fibers and of the corresponding treated samples were performed by using TA-Q2000 differential scanning calorimeter (DSC) equipped with a RCS-90 cooling unit (TA Instruments) in nitrogen atmosphere. For all measurements, Tzero® aluminum pans were used at a constant nitrogen flow rate of 20 mL/min. The sample weight was about 5 mg for all measurements. Samples were heated from 25 to 270 ◦C at a rate of 270 ◦C/min, kept at 270 ◦C for 1min to erase previous thermal history, then cooled to −70 ◦C at 20◦C/min. Finally, samples were heated again at 10 ◦C/min until 270°C for the complete melting.

The second heating step is reported for all samples. From the comparison between the specimen of fibers treated with the additives (Polysilk CTE , Figure 39, and Polyacril 73 ECO, Figure 40) no effects ascribable to the treatment can be observed. Just the thermal transitions of the Polyester fabric are visible for all the samples, probably due to the low amount (under the detection limits of the instrument) of additive on the fiber surface. In particular a glass (Tg=76.47°C) and a melting (Tm=242.09°C) transitions can be observed in all the profiles around the same temperature values.

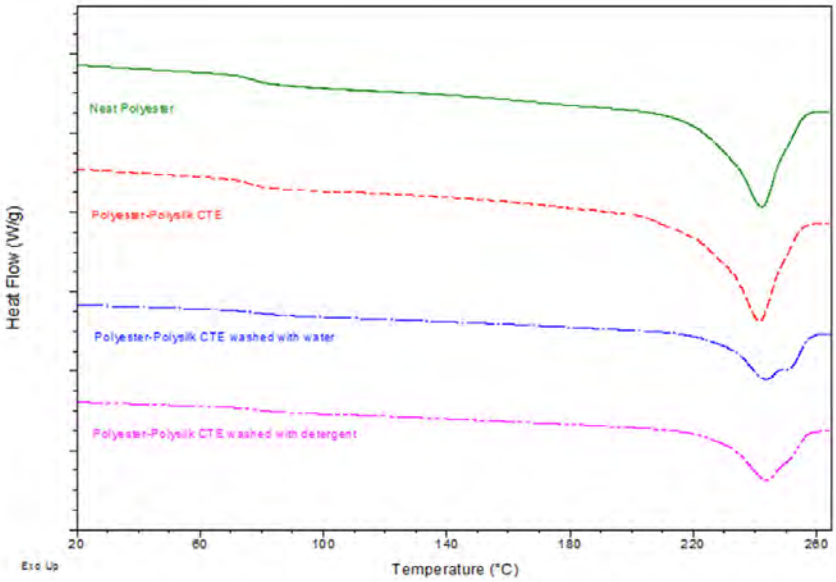


Figure 39. DSC heating curves of neat Polyester fibers and of Polysilk CTE treated ones, before and after the washing with water and detergent.

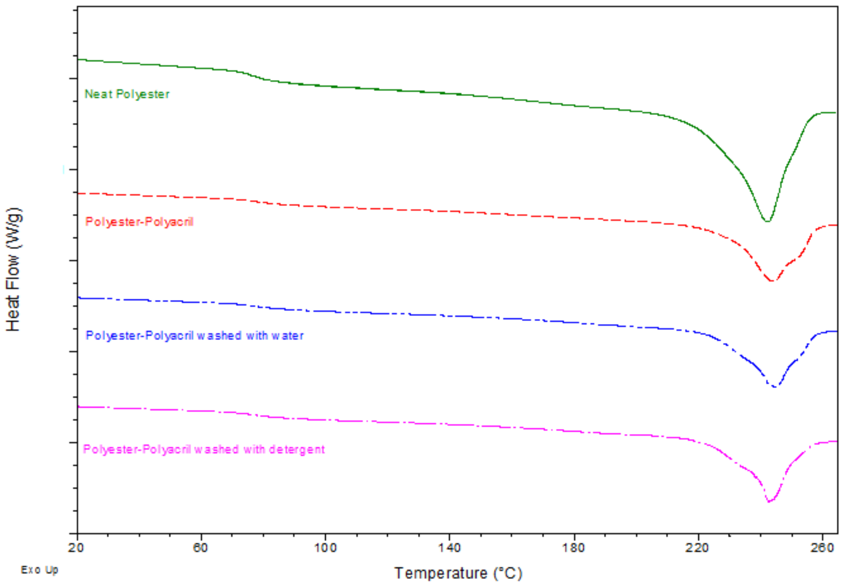


Figure 40. DSC heating curves of neat Polyester fibers and of Polyacril treated ones, before and after the washing with water and detergent.

Thermal stability of neat and treated Polyester fibers was analyzed using a thermogravimetric analyzer (TGA) Perkin Elmer Pyris. A small amount (about 2 mg) of each sample was placed in a platinum open pan and heated from 30 to 850 ◦C at 10 ◦C/min. High purity nitrogen was fluxed through the furnace at a flow rate of 40 mL/min. The curves of degradation obtained display for all the samples one characteristic degradation step, starting at about 380°C (loss of 2.5%) and with inflection point at about 435°C (loss of almost 83% of the total mass). No significant differences can be observed between the neat and the treated polyester samples since the low amounts of textile auxiliaries, confirmed by NMR analyses, do not affect the thermal stability of the fibers.

B 1.3.4 Washing resistance: Characterization and quantification of micro and nanoplastics

The solutions coming from the washing tests performed in Linitest were filtered in PVDF Millipore filters by using a peristaltic pump with tygon tubes. Such filters were analysed by a scanning electron microscope (SEM) in order to quantify the microplastic release, according to the counting method explained in the *Action A.2* (task A2.2.4).

The results are reported in Table 23. Two replicates of each sample have been measured for the treated Polyester. In the case of polyester treated with Polyacril 73 ECO and washed with detergent solution, one of the replicates was damaged and discarded. As expected, the washings with water release less microfibers than the ones with detergent solution. However, there is a reproducibility problem by using this procedure since the standard deviation values are significantly high for the two treated samples. Therefore, it is difficult to corroborate that by applying one of the selected auxiliaries the amount of microfibers can be reduced without performing more replicates. This will be taken into account in the Action B4.

Sample	Washing	N° of fibers/filter		
		Replicate 1	Replicate 2	Average
Polyester treated with Polysilk CTE	Water only	324	1015	**670**
	Detergent solution	917	3465	**2191**
Polyester treated with Polyacril 73 ECO	Water only	562	775	**668**
	Detergent solution	3750	-	**3750**
Woven PES 777 (control)	Water only	-	-	**240**
	Detergent solution	-	-	**1048**

Table 23. Counting results

[i] W. D. Schindler, P.J Hauser. Chemical Finishing of Textiles.
[ii] A. BETON, D. DIAS. Environmental Improvement Potential of Textiles (IMPRO-Textiles).

Name of the report:
Report on additives for detergents and washing liquor in lowering microplastics release

Number of the associated action: B2

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OCEAN CLEAN WASH

LIFE13 ENV/IT/001069

Mitigation of microplastics impact caused by textile washing processes

Introduction

The aim of this task is to prove experimentally if the use of new additives for detergents and for the washing liquor can improve the fibre conservation decreasing the fibres release.

In order to improve fabric care, different types of additives are commonly used in laundry formulations. These new additives, most of them polymers, presumably should prevent the damage of fabrics and fibres, so providing protection to garments. Other additives for the washing liquor, in this case, can be also useful in fabric care; for example, softeners decreasing the fibres friction or builders softening the water used during the washing cycle.

Objectives

The objectives of this action are the study of new additives for detergents and washing liquor, which may contribute to the reduction of fibre breakage, then to assess the effectiveness of laundry additives applied onto fabrics and, finally, to define if the optimum additives and formulations are capable of reducing "pilling" effect and achieving high smoother fabric surface without worsen other fabric characteristics.

Experimental Study

1. Description of the washing process

The washing process employed simulates the domestic washing, in which one cycle of the test simulates five domestic washing cycles. This test is based on the standard ISO 105 C06. The washing has been performed in Lini-test equipment. The machine is composed of a water bath containing a routable shaft which supports steel containers. The shaft/container assembly is rotating at an established frequency and at a defined temperature. The machine is presented in the next picture:

Figure 1. Lini-test machine

2. Fabrics and laundry products used

In order to study the influence of new additives for detergent and for the washing liquor in the micro-fibres release, some polymers, softeners and builders have been tested together with a commercial basic liquid detergent. Products have been tested on three different types of fabrics.

The fabrics used to carry out the tests have been:

- Woven Polyester 100% (ref. 777)
- Woven Poplypropylene 100% (ref. 983)
- Knitted Polyester, double knit jersey, disperse dyeable (ref. 720 WOB)

Pieces of each type of fabric were prepared taking into account the bath ratio employed for the test. Each piece of fabric was sewed on the sides with cotton thread in order to avoid interferences in the results caused by the release of microfibers from the thread.

The additives used are specified in the table below:

Table 1. New additives tested

	Type of Product	Code	Dose* (ml/15L water)
New additives for detergent	Polymer: Polyethylene glicol polyester	Polymer 1 (P1)	2% t.q. in detergent
	Polymer: Copolymer of acrylic acid and dialkyldimethylammonium chloride	Polymer 2 (P2)	1% t.q. in detergent
	Polymer: Wax crystal	Polymer 3 (P3)	2% t.q. in detergent
	Polymer: Mix of three-dimensional polymers	Polymer 4 (P4)	1% t.q. in detergent
	Polymer: Amino-functionalized polydimethylsiloxane	Polymer 5 (P5)	15% active matter in detergent
	Polymer: Polyalkylene glycol	Polymer 6 (P6)	1% t.q. in detergent
New additives for washing liquor	Commercial liquid builder: Active polycarboxylates	Builder 1 (B1)	83 ml/15 L water
	Builder: Aminopolycarboxylates	Builder 2 (B2)	225 g/15 L water
	Builder: Sodium citrate	Builder 3 (B3)	92 g/ 15 L water
	Commercial liquid softener: Cationic surfactants	Softener Liquid 1 (SL1)	40 ml/ 15 L water
	Commercial liquid softener: Cationic surfactants	Softener Liquid 3 (SL3)	40 ml/15 L water
	Commercial solid softener: Bentonite and sucrose	Softener Solid (SS1)	45 ml/15 L water

*Dose recommended by the manufacturer

All the additives used, excepting the liquid softener, have been tested together with a basic commercial liquid detergent, which dose is **65 ml/15 L water.** This detergent is the same used in the Task A.2.2.3 when the influence of the washing conditions in the release of micro and nano fibres was studied. The liquid softener has been tested without this liquid detergent because liquid softeners in general are incompatible with detergents due to its chemical nature. In a regular domestic washing cycle, the liquid softener is always dropped in the drum of the washing machine after the clothes have been washed with the detergent; otherwise, if they both were applied at the same time, their effectiveness would be reduced because of the interactions of the chemicals in their corresponding composition.

3. Washing conditions

Some pre-trials were made in order to check and set the definitive domestic conditions in lini-test based on ISO 105-C06:2010 and to check all equipment and filtration tests.

After all this work the actual conditions have been:

- Temperature: 40 ºC
- Time: 45 minutes
- Number of steel balls: 10
- Medium: distilled water, except in the case of builders, since their function is to catch the present ions in hard water; so the water used with builders has a hardness of around 21 ºdH.

4. Filtration

After the washing in the Lini-test, the samples were filtrated using Durapore® membrane filters from Merck Millipore with a pore diameter of 5 µm (ref. SVLP04700). Once the filtration was finished, the filters were exposed to 85 ºC during 30 minutes and, finally, analyzed with a Scanning Electron Microscope (SEM).

5. Counting method

The counting procedure developed is based on the acquisition of several electron micrographs of the filter surfaces using a scanning electron microscope, SEM, FEI Quanta 200 FEG.
SEM observations were performed in low vacuum mode (PH_2O = 0.7 torr), using a large field detector (LFD) and an acceleration voltage of 5-20 kV. The observations were performed on the whole filter without applying metal coating.
Taking into account the geometry and the dimensions of the used filters, 21 SEM micrographs were acquired for each filter. Every micrograph represents a squared area (A_S) of the filter surface, equal to 7.8 mm^2 (see *Figure 2*).
SEM micrographs were acquired along two orthogonal diameters of the circular filter (see *Figure 3*).

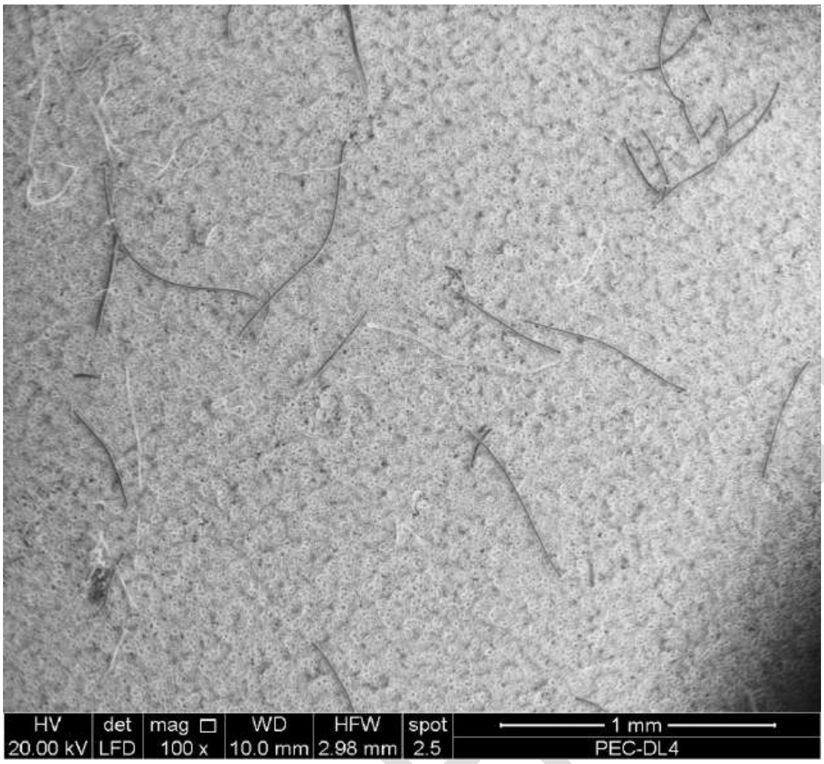

Figure 2. SEM micrograph of a squared area (A_S = 7.8 mm^2) of a filter containing PEC fibres

It was hypothesised that the fibre distribution is equal along all the traceable diagonals, thus 6 concentric circles were taken into account (see *Figure 4*):

- The first circle is right in the centre of the filter and it circumscribes the central square;
- The other 20 squares are included, four by four, in 5 annuli.

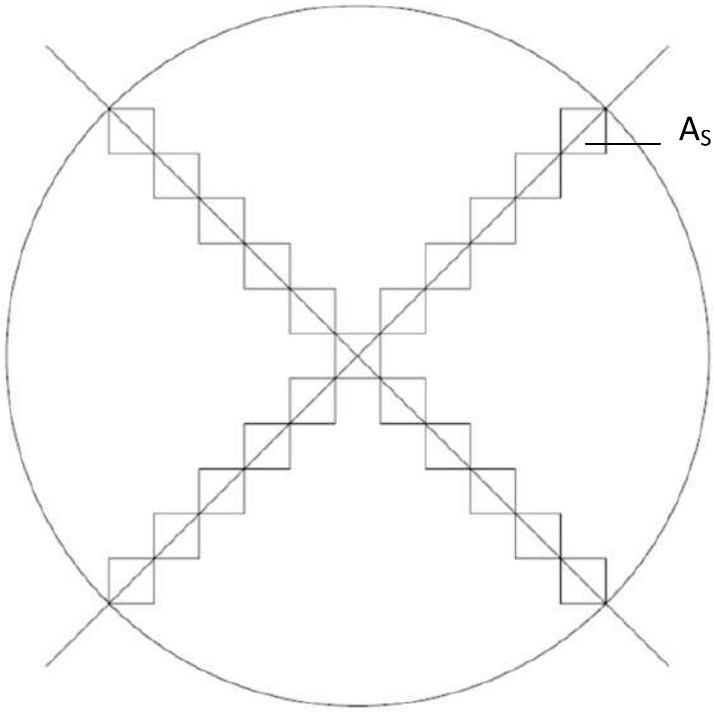

Figure 3. Position of the images along the filter

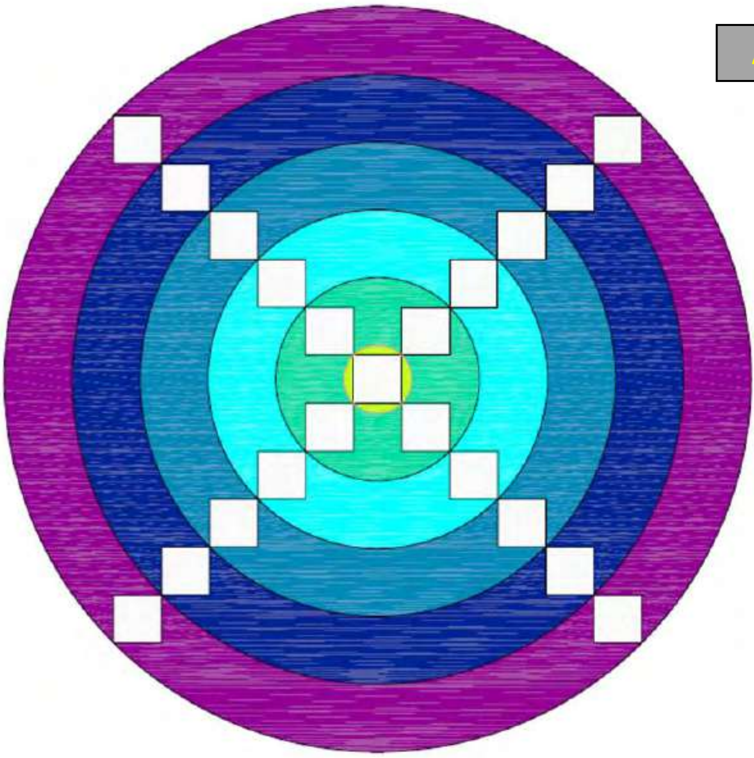

Figure 4. Concentric circles

The counting develops in 4 steps explained below:

1. Counting of the number of fibres in the first image in the centre of the filter (n_0) and division of this number for the area of the single square (A_S = 7.8 mm^2). In this way it is possible to obtain the number of fibres per area ($n_0/A_s= C_0$). Considering the area of the circle (A_0) that circumscribes the central square and multiplying this value for C_0, the number of fibres contained in the first circle is easily obtained ($N_0 = C_0$ x A_0).

2. In the first annulus, the number of fibres is counted in each of the four squares (n_1, n_2, n_3, n_4) and an average value is calculated $\left(\frac{n_1+n_2+n_3+n_4}{4} = n_A\right)$. As done in the previous step, this value is divided for A_S (n_A/A_S = C_1) and multiplied for the area of the considered annulus (A_1), obtaining the number of fibres contained in the first annulus (C_1 x A_1= N_1).

3. The previous step is repeated for the other 5 annuli, obtaining N_2, N_3, N_4, N_5.

4. Finally N_0, N_1, N_2, N_3, N_4 and N_5 are added together giving the total number of fibres on the filter surface.

Task B.2.1. Study of new additives for detergent products and their contribution to the reduction of fibre breakage

1. Selection of new additives for detergent products

In order to select the proper additives to improve the fibre care, the composition of representative commercial detergents from the European market was studied in the Task A.2.2.1. From the big amount of compounds that can be found in a regular liquid detergent, those which can carry out the function of fabric care are polymers.

After an intensive research (see in detail in Task A.2.2.2) several polymers were selected from different suppliers.

Polymers selected for the study

The polymers tested to decrease the amount of fibres released to the ocean during laundry washing are described below:

- **Polymer 1 (P1): Polyethylene glycol polyester**. Boosts cleaning and keeps fabric colours bright. Increasing surface hydrophilicity reduces the interaction between synthetic fabric and greasily oil (Figure 5), so it allows a cold wash performance leading less fibre breakage. It creates a fiber protection that prevents soil deposition wash after wash, making it easier to remove tough stains and maintain fabric color and whiteness.

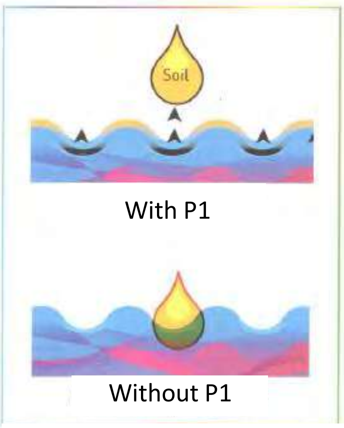

Figure 5. Repellent effect between fabrics and soil using P1. Source: Solvay.

- **Polymer 2 (P2): Copolymer of acrylic acid and dialkyldimethylammonium chloride.** Antistatic and film forming properties. Provides excellent affinity to fiber and is suitable for both standard and high efficiency (HE) laundry products.

- **Polymer 3 (P3): Wax Crystal**. Builds a thin wax layer in the fiber, protecting it from friction.

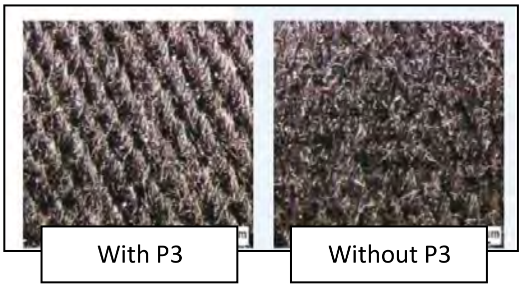

Figure 6. Test results after rubbing (200 cycles). Source: BASF.

- **Polymer 4 (P4): Mix of three-dimensional polymers**. Offers protection to the polyester and increases the liberation of soil during the washing cycle.

- **Polymer 5 (P5): Amino-functionalized polydimethylsiloxane.** Reduces friction between fibers and between fibers and metal thanks to the flexibility of their Si-O-Si backbone, which anchores on the fibers forming loops. The resulting silicone loops have a high mobility which means that the treated fibres can easily sliced past each other reducing fibre/metal friction.

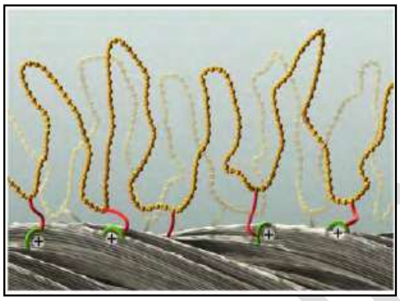

Figure 7. Silicone polymer (P5) anchored to fabric fibres forming loops. Source: Wacker.

- **Polymer 6 (P6): Polyalkylene glycol.** This functional fluid can be used as a thickener and lubricant in textile processes.

2. Methodology

Polymers are additives commonly used in laundry detergents. Thus, our polymers were mixed with a commercial basic liquid detergent (the same in all cases) in the dose recommended by the suppliers (see *Table 1*). This way, a washing cycle using a liquid detergent containing one of the polymers selected for our study in each case could be simulated. Once the basic detergent and the polymer were mixed, the resulting mix was dosed according to the recommendations of the detergent's fabricant (65 ml/15L water). In each washing only one polymer was tested, never more than one was mixed at the same time with the detergent. All the washes were carried out at the same washing conditions (see page 5, *3. Washing conditions*). The fabrics washed are described above (see page , *2. Fabrics and laundry products used*); however, the three types were not tested with all the polymers because of lack of time. Therefore, P1 and P2 were tested with the three fabrics, but the rest of them were tested only with woven polyester, since is the fabric which showed more fibres release.

After washing, the fibres released during the washing cycle were filtered and dried at 80 ºC during 30 minutes. Then, the fibres were counted.

3. Results

The results of this method are reported in *Table 2*. In the collected micrographs, besides the synthetic fibres, additional cotton fibres were detected (see *Figure 8*). Such fibres belong to the cotton thread used to sew the sides of the piece of fabric washed in the Lini-test machine. Of course these fibres were excluded during the counting procedure.

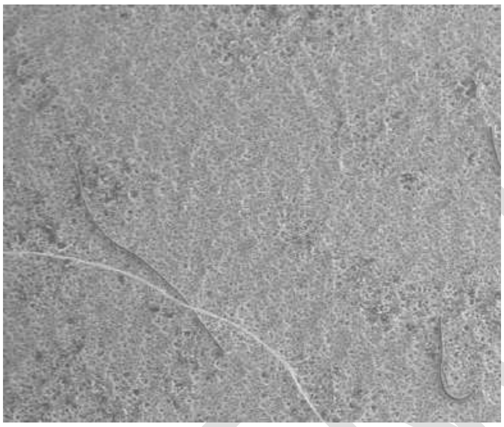

Figure 8. Magnification of a SEM micrograph showing a cotton fibre (white)

Table 2. Counting results

Sample	N° fibres/filter	Mean	Sample	N° fibres/filter	Mean	Sample	N° fibres/filter	Mean
PEC C0.1	1033	1090						
PEC C0.2	1147							
PEC P1.1	~~793~~	1866	**PEP P1.1**	462	630	**PP P1.1**	1264	1418
PEC P1.2	1715		**PEP P1.2**	797		**PP P1.2**	1572	
PEC P1.3	2016							
PEC P2.1	~~1998~~	972	**PEP P2.1**	516	498	**PP P2.1**	804	905
PEC P2.2	1065		**PEP P2.2**	480		**PP P2.2**	1005	
PEC P2.3	879							
PEC P3.1	1168	1231						
PEC P3.2	485							
PEC P3.3	2038							
PEC P4.1	714	753						
PEC P4.2	792							
PEC P5.1	607	537						
PEC P5.2	~~1168~~							
PEC P5.3	466							
PEC P6.1	585	671						
PEC P6.2	~~1902~~							
PEC P6.3	757							

*The strikethrough numbers have been omitted in the calculation of the mean because of the difference with the rest of results.

According to results some trends can be observed; some of the polymers tested seem to improve the fabric care reducing the number of fibres released. These polymers are Polymer 2, Polymer 4, Polymer 5 and Polymer 6.

The detergent mixed with Polymer 2 has released around 100 fibres less than this detergent alone (compared with PEC C0). In the case of Polymer 4 the reduction of fibres broken is around 300, while it is about 400 with Polymer 6 and around 500 with Polymer 5.

The numbers of fibres highlighted in red may be affected by error since, as visible in *Figure 9-12*, there are a lot of little fibre fragments whose nature is not of straightforward attribution: actually, it is not possible to distinguish between plastic and cotton fibres. The reported numbers refer to the only distinguishable ones.

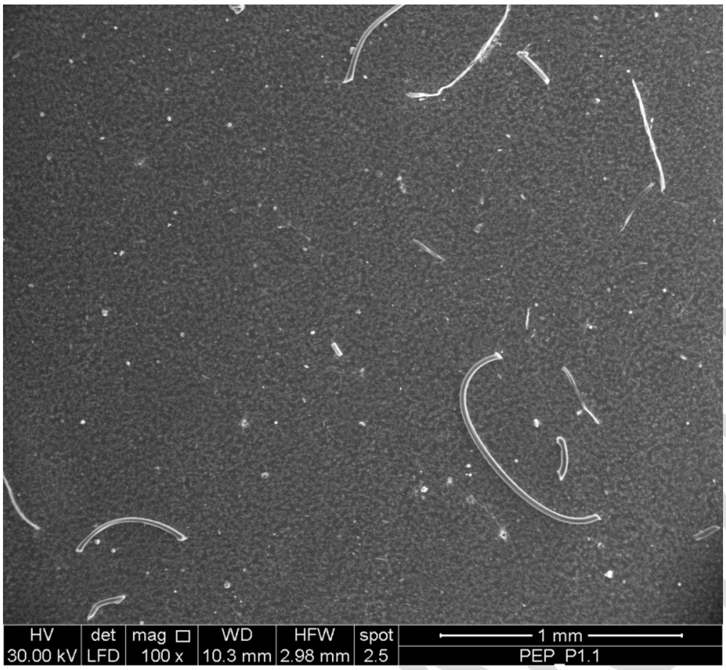

Figure 9. SEM micrograph of the filter PEP_P1.1

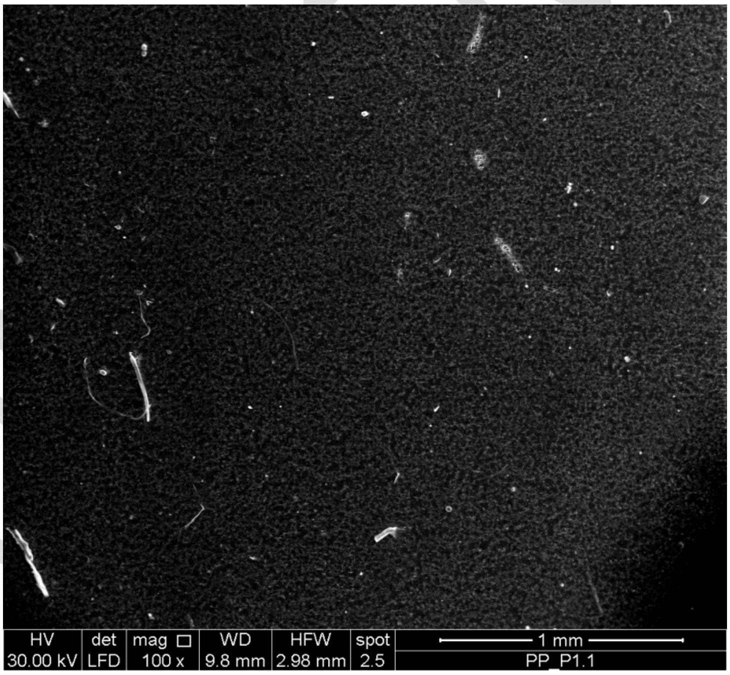

Figure 10. SEM micrograph of the filter PP_P1.1

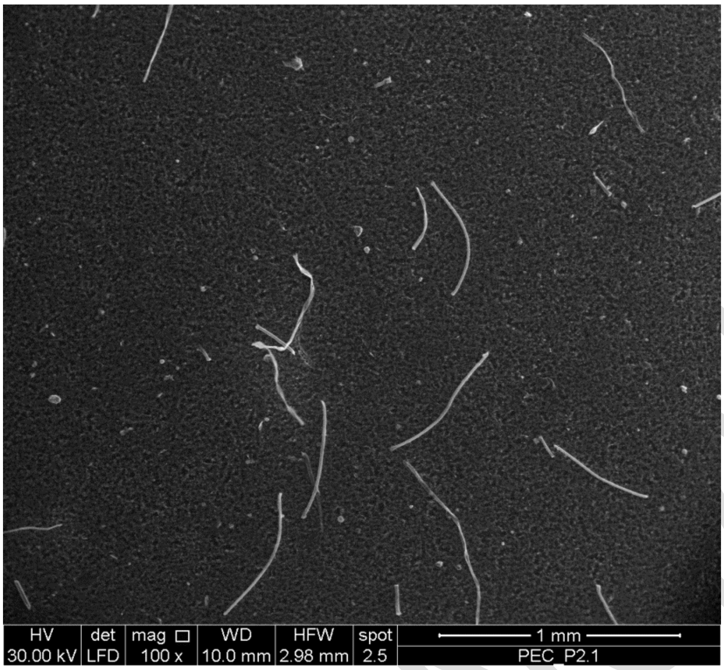

Figure 11. SEM micrograph of the filter PEC_P2.1

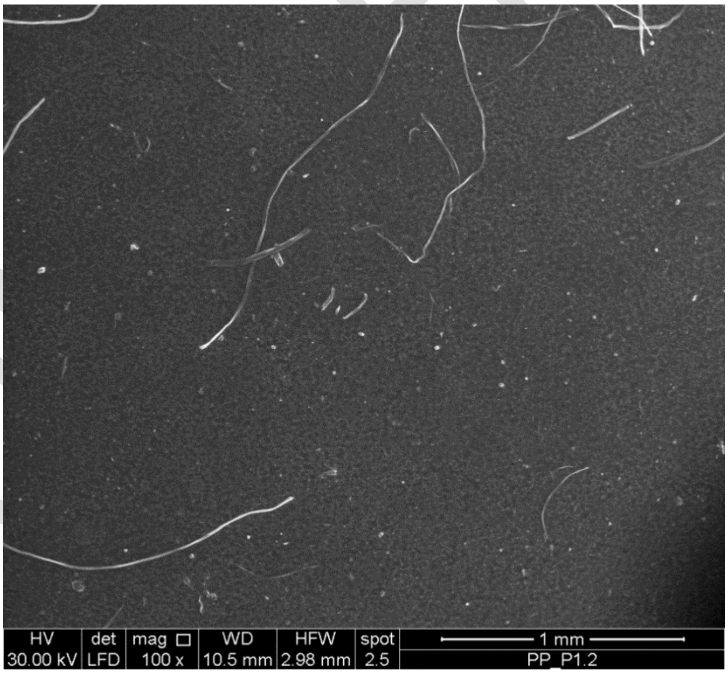

Figure 12. SEM micrograph of the filter PP_P1.2

Legend:

TEXTILES
PEC: Woven polyester
PEP: Knitted polyester
PP: Woven polypropylene

LAUNDRY PRODUCTS
C0: Basic liquid detergent product
P1: Basic liquid detergent product + Polymer 1
P2: Basic liquid detergent product + Polymer 2
P3: Basic liquid detergent product + Polymer 3
P4: Basic liquid detergent product + Polymer 4
P5: Basic liquid detergent product + Polymer 5
P6: Basic liquid detergent product + Polymer 6

4. Conclusions

Several trends in the micro-fibers release have been observed after analysing the filters through the SEM. Once the results have been obtained and studied, some tendencies can be drawn for PEC fabric, since there is no reference for the rest of fabrics and not all the polymers have been tested with the others fabrics:

- Not all polymers have decreased the fibres release; however good results have been obtained with Polymer 2, Polymer 4, Polymer 6 and Polymer 5 (in this order from worst to best).
- Therefore, the use of some polymers may reduce the fibre breakage, but not all the polymers with fabric care claim are useful from the point of view of minimizing the release of micro and nanofibres.

Task B.2.2. Study of new additives for washing liquor and their contribution to the reduction of fibre breakage

1. Selection of new additives for washing liquor

In order to decrease the emission of micro and nanofibres to the oceans, two different kinds of new additives for the washing liquor were selected for the trials: builders and softeners. They both are capable of reducing the fibres breakage in two different ways.

On the one hand, the function of builders is to chelate the positive ions from hard water (most of them calcium and magnesium ions) which is important because most of the tap water used in domestic washings is hard water. These ions can bind the anionic surfactants from the detergent and cause their precipitation forming deposits onto fabrics; besides, the calcium ion has the property of acting as "glue" between the stain and the fabrics surface making it harder to remove, so more detergent or more friction is needed to clean clothes favouring the release of fibres. As is shown in *Figure 13* the chelation of calcium through builders facilitated the dilution of the stain into the washing liquor.

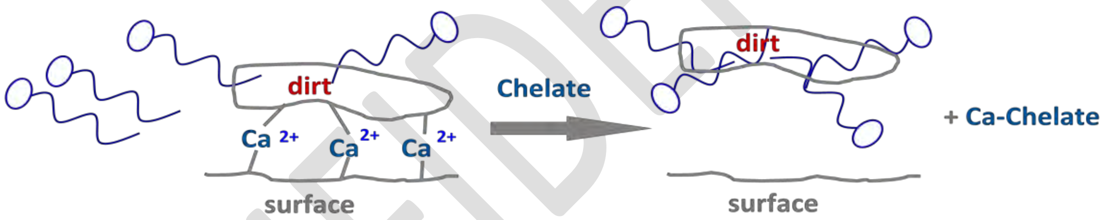

Figure 13. Chelating action of builders on Ca ions (Source: AkzoNobel)

Also, hard water forms lime deposits on fabrics, which may cause their abrasion; hence continuous washings in hard water can cause significant damage to fibres. Builders are capable of reducing all these effects thanks to their capacity of sequester the ions from hard water avoiding them cause all these problems. Builders can also cause deposits, but the amount is less than without using builders during the washing cycle. The amount of depositions depends on the type of builder:

- Carbonates: soften the water
- Citrates: soften and descale
- Aminopolycarboxylates (GLDA or MGDA): soften, descale and bind heavy metals.

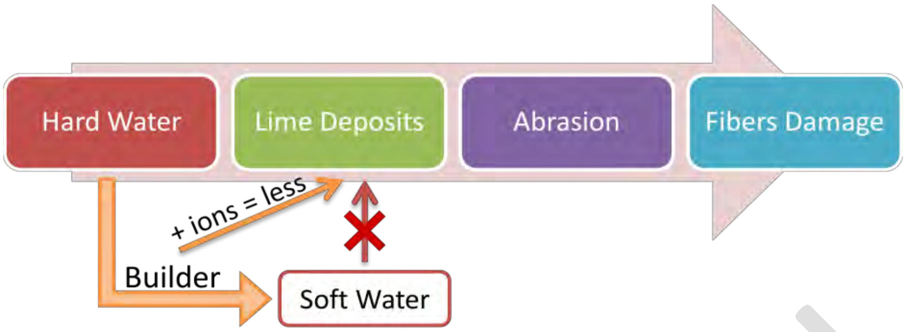

Figure 14. Action of builders related to lime deposits

On the other hand, the function of softeners is to reduce the friction between fibres, therefore their breakage is decreased.

Products selected for the study

The builders tested to decrease the amount of fibres released to the ocean during laundry washing are described below:

- Commercial builder 1 (B1): This is a liquid product used to soften the hard water from the washing machine avoiding the formation of lime deposits. It is based on active polycarboxylates.
- Builder 2 (B2): This is a raw material based on aminopolycarboxylates (trisodium salt of methylglycinediacetic acid) which sequesters heavy metal ions and it is also an effective builder for phosphate-free detergents. It softens water to prevent lime soaps from being formed and removes hardness ions from textile fabrics in the neutral and soda-alkaline pH ranges.
- Builder 3 (B3): This is sodium citrate, which prevents the formation of lime scale in the washing machine.

In the case of softeners, two different types were selected:

- Commercial liquid softener (SL3): another liquid softener (SL1) was tested in action A.2.2.1 and, since good results were obtained, we decided to confirm them with another one. Liquid softeners are based on cationic surfactants.
- Commercial solid softener (SS1): based on bentonite and sucrose.

The reason why a solid softener was selected is the moment of their application: liquid softeners are applied after detergents and, therefore, the protection of the fibre starts late in the washing cycle, thus the fibre breakage has already started once the softener is dropped. Solid softeners, however, are applied at the same time than detergents. Thereby, they can protect fibres from the beginning of the washing cycle

1. Methodology

The fabrics were washed using the new additive together with the same basic commercial liquid detergent used in Task B2.1, except in the case of the liquid softeners (SL1 and SL3) because of the incompatibility between detergents and liquid softeners. The dose used for the additives and the detergent was the recommended by the fabricants (see *Table 1*). The water used for softeners was distilled water, while in the case of builders the water had a hardness of 21 ºdH. In each washing only one additive was tested and all the washes were carried out at the same washing conditions (see page 5, *3. Washing conditions*). The fabrics washed are described above (see page , *2. Fabrics and laundry products used*); however, the three types were not tested with all the additives because of the lack of time. Therefore, B1, SL1 and SL3 were tested with the three fabrics, but the rest of them were tested only with woven polyester, since is the fabric which showed more fibres release.

After washing, the fibres released during the washing cycle were filtered and dried at 80 ºC during 30 minutes. Then, the fibres were counted.

2. Results

The results of this method are reported in *Table 3*. In the collected micrographs, besides the synthetic fibres, additional cotton fibres were detected (see *Figure 15*). Such fibres belong to the cotton thread used to sew the sides of the piece of fabric washed in the Lini-test machine. Of course these fibres were excluded during the counting procedure.

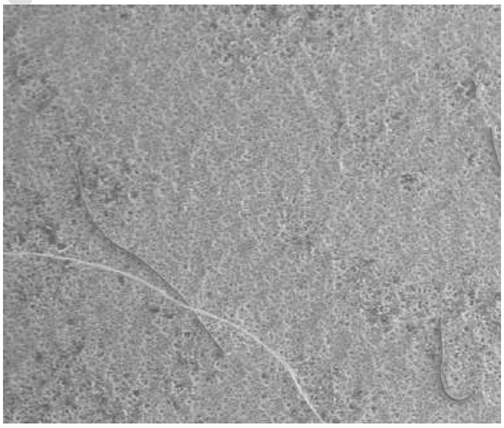

Figure 15. Magnification of a SEM micrograph showing a cotton fibre (white)

Table 3. Counting results

Sample	N° fibres/filter	Mean	Sample	N° fibres/filter	Mean	Sample	N° fibres/filter	Mean
PEC C0.1	1033	1090						
PEC C0.2	1147							
PEC B1.1	1853	1853	**PEP B1.1**	2299	2202	**PP B1.1**	993	935
PEC B1.2	-		**PEP B1.2**	2105		**PP B1.2**	877	
PEC B2.1	435	1025						
PEC B2.2	1589							
PEC B2.3	1049							
PEC B3.1	750	1006						
PEC B3.2	1261							
PEC B3.3	~~2310~~							
PEC SS1.1	893	775						
PEC SS1.2	658							
PEC R1	123	123	**PEP R1**	100	100	**PP R1**	187	187
PEC SL1.1	674	674	**PEP SL1.1**	177	177	**PP SL1.1**	830	830
PEC SL3.1	163	217	**PEP SL3.1**	313	311	**PP SL3.1**	830	694
PEC SL3.2	272		**PEP SL3.2**	308		**PP SL3.2**	558	

*The strikethrough numbers have been omitted in the calculation of the mean because of the difference with the rest of results

According to results some trends can be observed; builders seem do not have any effect in fibres release (just a very slight improvement), except in the case of Builder 1 which may increase it (compared with PEC C0). However, the result of the Builder 1 is not conclusive, since as visible in *Figure 16-17*, there are a lot of little fibre fragments whose nature is not of straightforward attribution: actually, it is not possible to distinguish between plastic and cotton fibres. The reported numbers refer to the only distinguishable ones. Moreover, only one replicate has been analyzed.

In the case of softeners, Solid softener 1 seems to decrease the number of fibres release (compared to PEC C0); however the results are not conclusive, since they may be affected by error probably due to the kind of detergent mixture. As shown in *Figure 18-19,* some fibres are completely or partially covered and so not safely distinguishable. On the other hand, liquid softeners seem to produce a not significant increase of the fibres released with all the fabrics tested (compared to PEC, PEP and PP R1). Therefore, liquid softeners seem do not have any effect on fibres breakages, although they may are not proper for the mitigation of this problem, since they are not compatible with detergents (they are added after the detergent during the washing cycle).

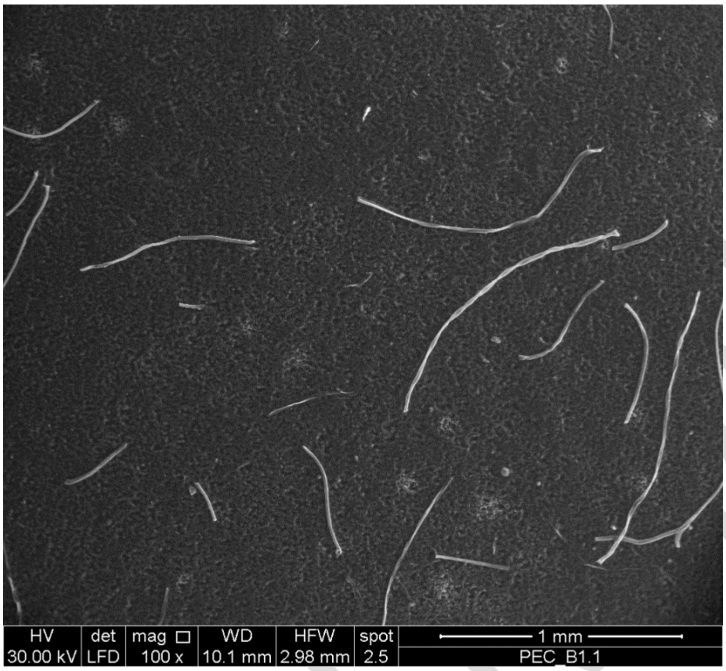

Figure 16. SEM micrograph of the filter PEC_B1.1

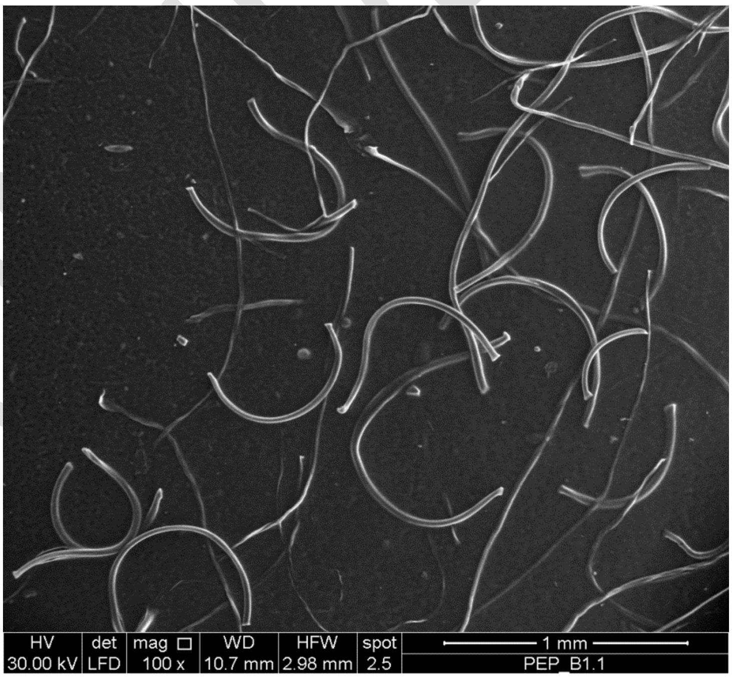

Figure 17. SEM micrograph of the filter PEP_B1.1

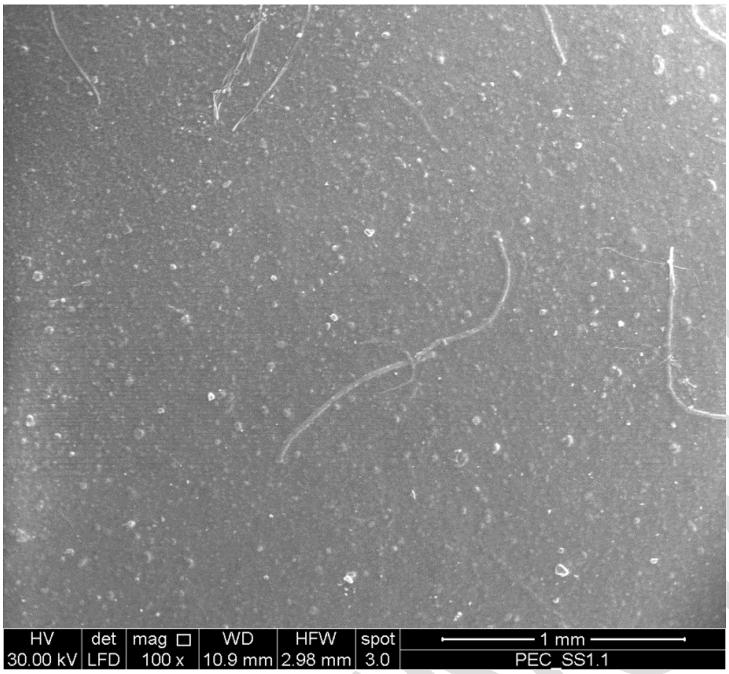

Figure 18. SEM micrograph of the filter PEC_SS1.1

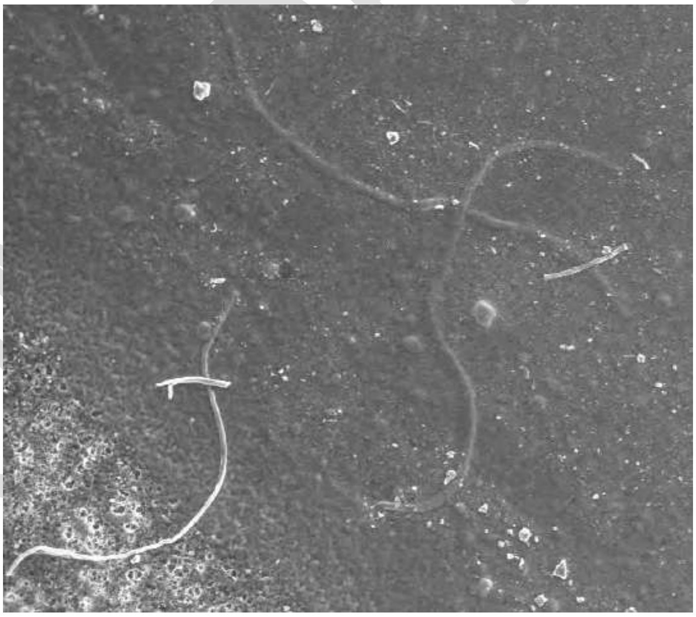

Figure 19. Magnification of a part of a SEM micrograph of the filter PEC_SS1.1, displaying some fibres completely or partially covered by the detergent mixture

Legend:

TEXTILES
PEC: Woven polyester
PEP: Knitted polyester
PP: Woven polypropylene

LAUNDRY PRODUCTS
C0: Basic liquid detergent product
B1: Basic liquid detergent product + Builder 1
B2: Basic liquid detergent product + Builder 2
B3: Basic liquid detergent product + Builder 3
SS1: Basic liquid detergent product + Solid softener 1
R1: Distilled water
SL1: Liquid softener 1
SL3: Liquid softener 3

3. Conclusions

Several trends in the micro-fibers release have been observed after analysing the filters through the SEM. Once the results have been obtained and studied, some tendencies can be drawn for PEC fabric in the case of builders B2 and B3 and solid softener, since there is no reference for the rest of fabrics (in the case of B1, SL1 and SL3 all the fabrics have been tested):

- Builders seem do not have any effect in the fibres release, only Builder 1 seems to increase the number of fibres broken, while in the case of Builder 2 and Builder 3 just a very slight improvement has been observed.
- Solid softener 1 seems to decrease the fibres breakage; however the results are not conclusive because of the difficulty found in fibres counting.
- Softener Liquid 1 and Softener Liquid 2 seem do not have any effect on fibres release; however they are not the most proper for the mitigation of this problem, since they are not compatible with detergents (they are applied after the detergent during the washing cycle).

Name of the deliverable:

"Environmental Baseline Scenario"
(Confidential)

Number of the associated action: C2
Involved partners: LEITAT

(31/12/2015)

mermaids.
OCEAN CLEAN WASH

LIFE13 ENV/IT/001069

Mitigation of microplastics impact caused by textile washing processes

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Abbreviations list

ELCD - European reference Life Cycle Database

ILCD - International Reference Life Cycle Data System

ISO - International Organization for Standardization

LCA - Life cycle assessment

LCI – Life Cycle Inventory

PEF - Product Environmental Footprint

SETAC - Society of Environmental Toxicology and Chemistry

1. Introduction and context of the study

"Some scientific studies have shown that litter found in oceans and inland waters is dominated by plastics and microplastics. Litter in the marine and coastal environment is known to have negative effects in 633 species. Plastics' components may be toxic or develop endocrine effects. Hence there is also a risk of pollutants accumulating in the marine food web. Furthermore, microplastics can act as a transport medium for pollutants, invasive species and pathogens. Some of these plastics come from the fragmentation of plastic bottles, abrasion of tyres and textiles (by laundry). Fibres from clothing and other textiles are also possible source of microplastics in water bodies[1]*. An estimated 1,900 microfibers can get rinsed out of a single piece of synthetic clothing each time it's washed, and these microplastic fibres might be the biggest contributors to ocean pollution"*[2].

The main objective of the MERMAIDS project is to contribute to the mitigation of the impact caused by microplastics particles resulting from laundry wastewater on European seas ecosystem's, by demonstrating and implementing innovative technologies and additives for laundry detergent processes and finishing textile treatments. For this reason, the aim of the action C2 called "*Monitoring of the environmental problem targeted*" is to collect data in order to determine the environmental performance of a domestic laundry process considering the microplastics release.

This document is previous to the complete LCA Report and it is aimed to define the system boundaries and the assumptions on which the LCA will be based, as well as to identify what are the main environmental impact categories related to a current and domestic laundry process on which the MERMAIDS solutions will be compared. This allows the selection of the main environmental indicators to be addressed in the future LCA. Therefore, a baseline scenario for a current laundry process have been defined and assessed in this study. The baseline scenario considered for LCA has been outlined it comprising only the laundry process.

Existent literature and technical databases have been used for the environmental assessment of a current laundry process which allowed identifying the environmental impact categories on which the complete LCA will be based. The outcomes obtained are very helpful for supporting the data collection along the project. The results of this study will be updated once the complete LCA is performed. They will be fully delivered on September 2016.

The figure 1 shows the life cycle of a general laundry process.

[1] Essel, R. et al. Sources of microplastics relevant to marine protection in Germany. Germany. 2015.

2 Browne, Mark. Accumulation of Microplastic on Shorelines Worldwide: Sources and Sinks. American Chemical Society. 2011

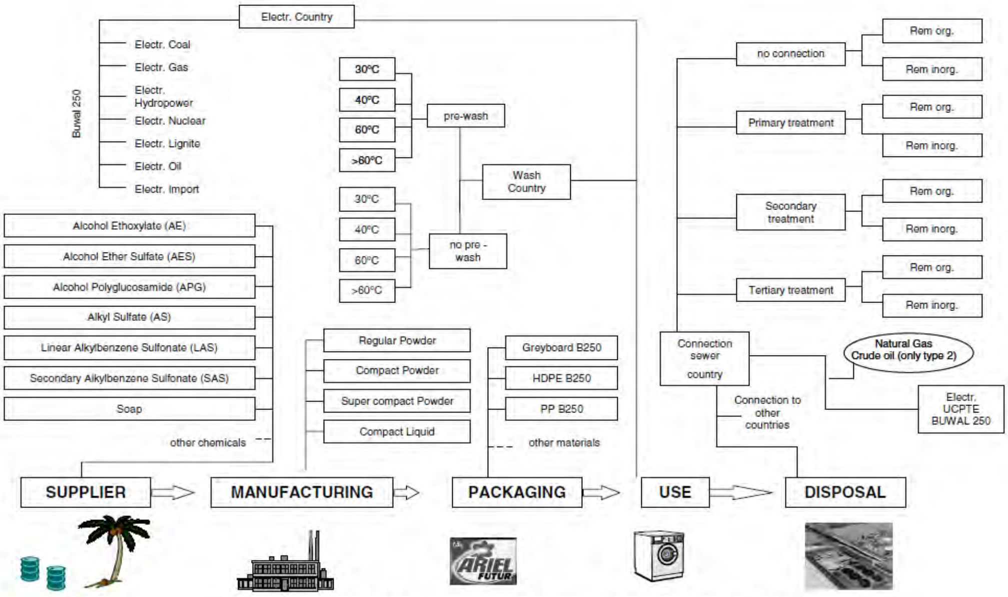

Fig. 1: Structure of the life cycle of 'laundering' as organised in the SimaPro software

Figure 1. Life cycle of general laundry process Source: A database for the Life Cycle Assessment of Protect and Gamble Laundry Detergents

2. Objectives

The environmental solutions addressed in MERMAIDS are being developed and implemented in order to reduce the microplastics release into the sea. The new additives for laundry detergent products and innovative additives to improve finishing fibres treatments will avoid garments' microplastics removal during laundry process.

Within this context, a full Life Cycle Assessment (LCA) will be carried out in order to compare the environmental impact of the innovative solutions developed in the MERMAIDS project versus a current laundry process (baseline scenario).

The methodology for LCA is explained in this document, which starts with the **definition of the goal and scope** on which the assessment is focused and that directly depends on the nature and magnitude of the processes to be analyzed. Hence, this LCA is comparative and focused, on the one hand, on conventional laundry process on which the MERMAIDS solutions will be contrasted; and, on the other hand, on the innovative additives which are assessed in Mermaids. The **baseline scenario** for the comparative LCA has been simulated and described, which mean the current situation to be compared with the future MERMAIDS solutions. This is essential to measure any environmental improvement or variation produced throughout the implementation of the MERMAIDS solutions.

Below is described the **LCA methodology** proposed for this evaluation, whose first step is outlined in regard to the goal and scope definition, and focused on the boundaries of the system to be analyzed. Besides, an assessment of the **environmental impact of a current laundry process** is presented so that it could establish the mentioned baseline scenario for future LCA.

Noteworthy that the results of this deliverable could be modified as necessary for developing the future complete LCA, once the MERMAIDS process is fully defined.

3. Conceptualization of Life Cycle Assessment

Life cycle assessment is a methodology for assessing quantitative environmental analysis of products and services, which allows quantifying the potential environmental impacts of a product or service generated in all its life cycle. The most important applications are:

- Analysis of the contribution of the life cycle stages to the overall environmental load, usually with the aim of prioritizing improvements on products or processes.
- Comparison between products and processes for internal or external communications.

LCA is a relatively young method that became popular in the early nineties. In recent years life cycle thinking has become a key focus in environmental policy making. LCA provides the more quantitative and scientific basis for all these new concepts. In many cases LCA feeds the internal and external discussions and communications, being a tool to communicate the environmental impacts of products and business processes.

The first definition for LCA was done by the Society of Environmental Toxicology and Chemistry (SETAC). According to SETAC, LCA can be defined as:

"A process to evaluate the environmental burdens associated with a product, process, or activity by identifying and quantifying energy and materials used and wastes released to the environment; to assess the impact of those energy and materials used and releases to the environment; and to identify and evaluate opportunities to affect environmental improvements. The assessment includes the entire life cycle of the product, process or activity, encompassing, extracting and processing raw materials; manufacturing, transportation and distribution; use, re-use, maintenance; recycling, and final disposal".

Figure 2.Life cycle of general laundry process Source: A database for the Life Cycle Assessment of Protect and Gamble Laundry Detergents

The proposed methodology has been developed in accordance with the standard ISO framework for LCA (ISO 14040:2006 and ISO 14044:2006)[3], as well as the Guidelines of International Reference Life Cycle Data System (ILCD) Handbook[4] and Product Environmental Footprint (PEF)[5].

Calculations have been done using the software SimaPro 8, and taking as a basis the Ecoinvent Database v3 and the ILCD impact assessment method, which was released by the European Commission, Joint Research Centre in 2012. It supports the correct use of the characterization factors for impact assessment as recommended in the ILCD guidance document "Recommendations for Life Cycle Impact Assessment in the European context - based on existing environmental impact assessment models and factors.

The standardized methodology for LCA establishes four interconnected steps, represented in the Figure below.

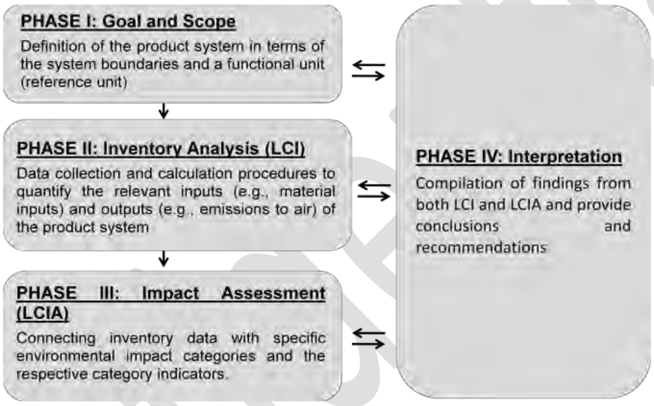

Figure 3. Methodology LCA studies according to ISO 14044

The four interrelated basic stages shown in figure 3 have been established:

Goal definition

Goal definition is the first step of the environmental assessment, and sets the overall context for the study. The purpose of clearly articulating goals is to ensure that the analytical aims, methods, results and intended applications are optimally aligned, and that a shared vision is in place to guide participants in the study.

3 ISO 14040:2006 Environmental management -- Life cycle assessment -- Principles and framework ISO 14044:2006 Environmental management -- Life cycle assessment -- Requirements and guidelines

4 European Commission Joint Research Centre. ILCD Handbook: General guide for Life Cycle Assessment (http://lct.jrc.ec.europa.eu/assessment/publications)

5 Product Environmental Footprint (PEF) Guide. European Commission (EC), Joint Research Centre (JRC). Institute for Environment and Sustainability (IES). 2012

Scope definition

Defining the scope of the study refers to describing in detail the system to be evaluated along with the associated analytical specifications. Scope definition must be in alignment with the defined study goals and the requirements. The unit of analysis (functional unit), reference flow, system boundaries, cut-off rules and environmental impact categories for product environmental footprint shall be identified and clearly described.

The unit of analysis, also called the "functional unit", describes qualitatively and quantitatively the function(s) or the service(s) provided by the product, as well as its duration. The reference flow is the amount of product necessary to provide the defined function. It constitutes the flow(s) to which all other input and output flows in the analysis quantitatively relate. The reference flow can be expressed in direct relation to the functional unit or in a more product-oriented way.

The system boundaries define which parts of the product life cycle and which associated processes belong to the analyzed system (i.e. are required for providing its function as defined by the functional unit). The system boundary should be defined following general supply-chain logic, including all phases from raw material extraction through processing, distribution, the use phase and end-of-life treatment of the product, as appropriate to the intended application of the study.

In principle, all processes and flows that are attributable to the analyzed system are to be included in the system boundaries. However, not all these processes and elementary flows may be quantitatively relevant. The cut-off criterion to be applied is what modeled flows must be account according to the relative contribution of each of the environmental impact categories considered.

Environmental impact categories refer to specific categories of environmental impacts considered in a product environmental study. These are generally related to resource use or emissions of environmentally problematic substances, such as greenhouse gases or toxic chemicals. Environmental impact assessment methods use models for quantifying the causal relationships between the material/energy inputs and emissions associated with the product life cycle and each environmental footprint impact category considered. Each impact category hence has an associated, stand-alone environmental footprint impact assessment method.

Life Cycle Inventory

The inventory analysis of the LCA comprises the data collection and the calculation procedures to quantify the inputs and outputs (energy, raw materials, air, water, soil, etc.) through the system boundaries. To make the analysis easier, the system is divided in several interconnected subsystems.

Life Cycle Impact Assessment

The impact assessment is the phase in which the set of results from the inventory analysis are processed in terms of potential environmental impacts. It consists of four distinct steps: classification (classification of inventory flows into different impact categories: resource depletion, human health and ecological consequences, etc.), characterization (common numeric values for each impact category), normalization (in relation to the actual magnitude of the impacts) and weighting (based on value judgments, weighting among different impact category in order to obtain an unique punctuation of the global environmental impact). The steps classification and characterization are obligatory whereas normalization and weighting are optional.

Interpretation of LCA results

A critical interpretation of the results will be done in order to verify its reliability. In this step the completeness, sensitivity and consistency of data gathered and results obtained will be done. The interpretation of the results will help to define the most relevant environmental impacts and the stages where attention has to be paid in order to minimize the impact.

4. Goal and Scope definition for LCA

The general goal of this LCA is to analyze the environmental performance of the innovative additives for laundry detergent products and finishing textile treatments outlined from MERMAIDS and to compare these results with the environmental results of a current laundry process.

As explained above, the goal and scope definition establishes the LCA boundaries on which the environmental assessment will be focused; i.e., the system, the functions, the functional unit, the life cycle stages, the environmental impacts to be investigated, the methods, the interpretation approaches and the assumptions to be undertaken.

4.1 Goal definition

A clear goal definition is essential for a correct later interpretation of the results. In this project, the LCA is performed covering the following two key objectives:

- **To estimate the project's environmental impact** based on the dimension microplastic release arising from laundry process.
- **To compare the potential environmental impacts between a current laundry process and a MERMAIDS process**, in order to establish the environmental benefits of these innovative MERMAIDS solutions.

To achieve these objectives, a baseline scenario is necessary to be defined in order to be able to make such comparison between both scenarios, current and MERMAIDS-based.

This baseline scenario is defined in the section 5 of this deliverable.

4.2 Scope definition

A regular LCA includes all four stages that summarize a product or process life cycle: raw material acquisition, manufacturing, use/reuse/maintenance, and recycle/waste management. To determine whether one or all of the stages should be included in the scope of the LCA, the following has been assessed: the goal of the study, the required accuracy of the results, and the available time and resources.

In this case, LCA is only focused on a domestic laundry phase. The assessment will compare the conventional laundry process versus the innovative MERMAIDS solutions.

Figure 4. LCA compared current laundry process versus MERMAIDS solutions

4.2.1 Functional Unit

Since this LCA is used to compare two types of laundry processes, the basis of comparison should be equivalent unit, thus each system should be defined so that an equal amount of product or equivalent service is delivered to the consumer. The functional unit serves as a reference unit to be considered along the life cycle, to measure and display the LCA results and to improve the accuracy of the study and the usefulness of the results. In MERMAIDS, the functional unit needs to be easily measurable and comparable into both laundry processes.

The one selected is:

4,5 kg of laundry load of fabric (polyester 100%)

This selection of laundry load is based on different LCA studies where an average between 4 and 5 kg of laundry load is observed regardless of the type of used fabric. The survey on domestic washing habits carried out within the task A1.1 of this MERMAIDS project has been also considered for the functional unit selection.

4.2.2 System boundaries

Each step or process can be viewed as a subsystem of the total product system. The boundaries of subsystems are usually defined by life-cycle stage categories. The following flow diagram illustrates the outlined system boundaries, where this analysis is limited.

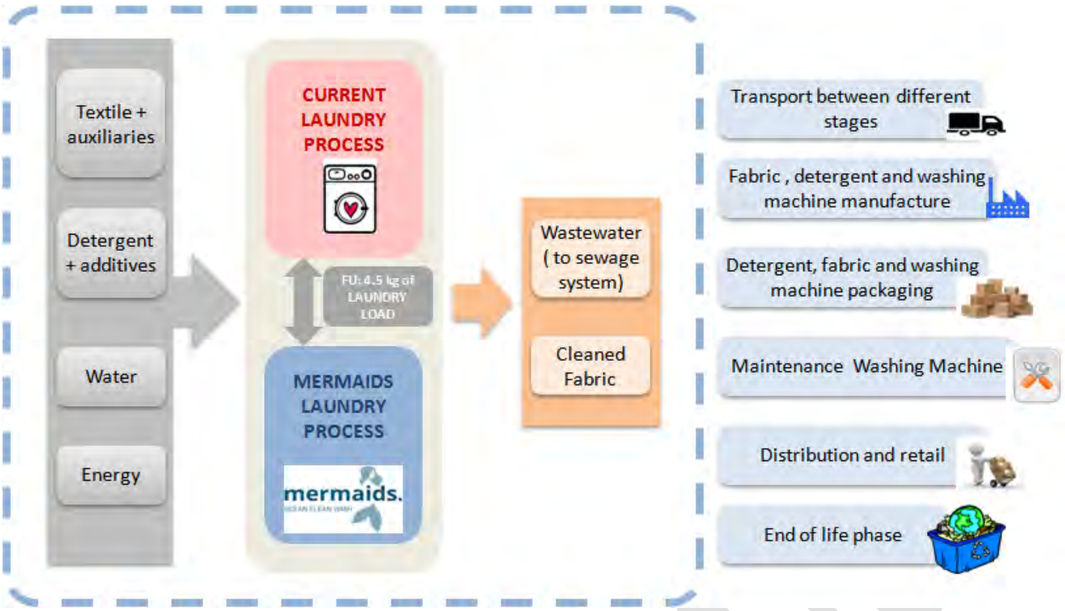

Figure 5. General system boundaries; current laundry process versus MERMAIDS solutions

This Figure 5 reflects the whole framework on which the LCA is sited. The intermittent thick blue line marks the boundary between included and excluded processes. Unit processes inside of the system boundary link together to form a complete life cycle picture of the required inputs (material and energy) and outputs (wastewater release) of the system.

The system boundaries have been defined following the general supply chain (shown in figure 1) including only the laundry phase; while packaging, transport, manufacture, distribution and retail, maintenance and end of life phases have been excluded of the analysis:

- **Laundry phase**: in this system, detergent use (including ingredients), polyester (100%) fabric, water and energy consumption have been taken into account, as well as the amount of wastewater release after laundering.

Table 1 shows a detailed list of inclusions and exclusions for system boundaries in Mermaids.

Included into the system boundaries	Excluded from the system boundaries
+Water consumption +Energy consumption (hot water and electricity) +Finishing textile treatments (modified textile) +Detergent ingredients +Additives for detergent products +Use of the washing machine (laundry use phase) +Wastewater release (after laundry use)	-Transport phase -Washing machine manufacture, including materials -Maintenance of washing machine -Packaging phase -Distribution and retail phase -End of life phase

Table 1. List of inclusions and exclusions for LCA System boundaries

The figures 6 and 7 show the limits for finishing textile treatments and detergent formulation. Both inputs will be considered not for the LCA baseline scenario but for the future complete LCA, once the Mermaids laundry process are well defined to determine what is the necessary data for comparison.

Figure 6. System boundaries of finishing textile treatments

Figure 7. System boundaries of detergent formulation

For the baseline definition, the selected model comprises only the laundry process for 4.5 kg of polyester (100%) fabric including detergent use, energy and water consumption:

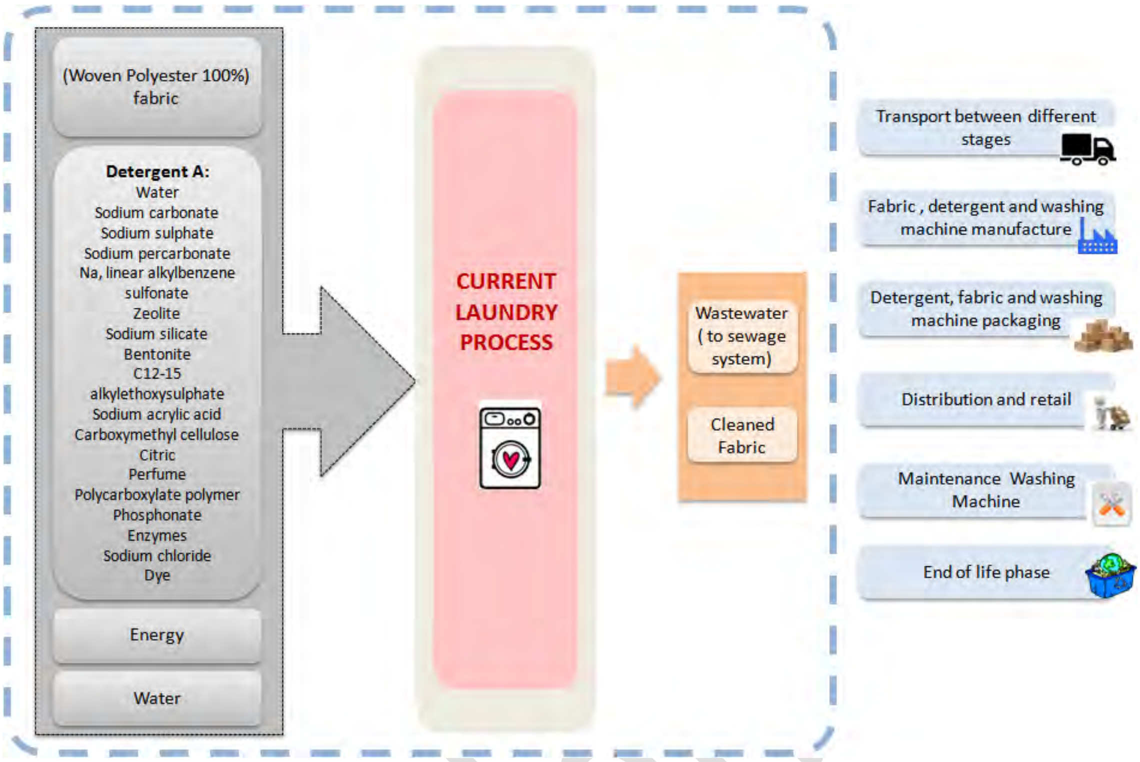

Figure 8. Detailed system boundaries in Baseline Scenario (A) [6]

More information regarding the current laundry (baseline scenario) is gathered below in chapters 5 and 6 of this document.

4.2.3 Target audience

The target audience of the present study is the scientific community (RTD professionals), maritime, textile and laundry detergent products industry, but also thematic related governmental agencies.

4.2.4 Limitations of the study

The limitations of the project are the following:

- Representativeness and quality of inventory data for the laundry phase.
- Some substances are not available in the LCA Databases used. In those cases, similar or equivalent substances are selected for LCA analysis. However, some substances can not be modelled and in this case they cause uncertainty in the impact assessment.

[6] The system boudnaries for the baseline scenario A is the same as the baseline scenarios B and C, but with different detergent ingredients.

5. Current laundry process

As mentioned previously, the baseline scenario considered for LCA comprises all the current laundry process.

A brief definition of a domestic laundry process is provided in this section as: "Laundry is the washing of clothing and linens. The material that is being washed, or has been laundered, is also generally referred to as laundry. Laundry processes include washing (usually with water containing detergents or other chemicals), agitation, rinsing, drying and pressing (ironing). The washing will often be done at a temperature above room temperature to increase the activities of any chemicals used and the solubility of stains, and high temperatures kill micro-organisms that may be present on the fabric. Washing machines are the device designed and used to provide clean clothes while retaining the quality of the material. The use of the washing machine however, is a highly energy and water intense process that results in a number of negative impacts on the environment."[7]

5.1 Selection of the baseline scenario

In this section, three baseline scenarios have been defined and assessed. These scenarios have been based on existing literature and they have followed the availability of data needed for the corresponding analysis. The purpose of these scenarios is to perform a thorough analysis of the environmental impacts of different current laundry processes. This current laundry process has taken into account the washing of fabric, detergent use, energy and water consumption.

The type of fabric selected is woven polyester 100%. The woven polyester (Ref.777)[8] has been selected to be the worst quality textile in terms of pilling formation (see Preliminary action A.2.1 of the MERMAIDS project). Moreover, in clothing textile production weight the market is dominated by cotton, which accounts for more than 43% of all fibers, and followed by polyester (16%). So it would be an important improvement to tackle the problematic of this fabric.

Finally, a current laundry process has been analyzed in terms of environmental impacts in the following section 6. Thus major inputs and outputs of the system (Figure 8) have been estimated and mostly based on existing literature.

[7] https://en.wikipedia.org/wiki/Laundry

[8] Report on the innovative textile additives to perform lab scale finishes, characterization and pilot plant implementation of thereof B1. MERMAIDS PROJECT. LEITAT and POLYSSTEC .30/10/2015

6 Environmental impact of current laundry process

This chapter involves the environmental assessment of a current and domestic laundry process to be compared as baseline scenario during the full LCA performed later at the end of the project.

6.1 Life Cycle Inventory

Life cycle inventory (LCI) is a "gate to gate" accounting (compilation and quantification) of the environmentally significant inputs and outputs of the system throughout its life cycle (Figure 9). The environmental burdens in this study include woven polyester (100%) fabric, detergent use, energy and water consumption associated with the laundry's life cycle. LCA data is converted to the study's functional unit.

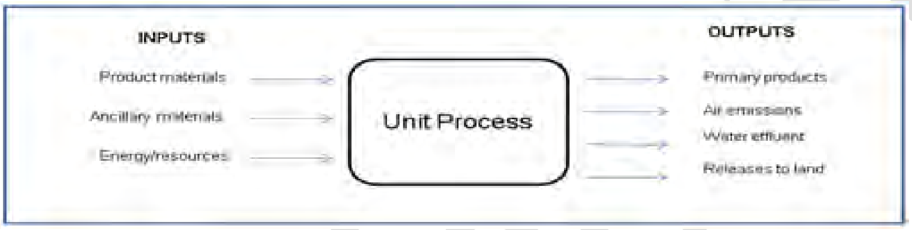

Figure 9. Inventory inputs and outputs scheme

The inputs and outputs of the process have been gathered and quantified. For the baseline scenario generic data is limited and based on the existent LCA studies and also Ecoinvent database has been used. Detergent, textile and washing machine manufacture, packaging, distribution and retail, transport and end of life stages have not been considered of high relevance for this study.

<u>**Assumptions**</u>

For the inventory the following **assumptions** have been considered:

- The units of measurement used are: Energy: kWh / Mass: g / Water: L (litters)
- The characteristics of a conventional laundry performance are:
  - Domestic and standard washing machine
  - Average wash temperature around 40ºC in Europe

  The washing machine considered refers to regular washing machine used in domestic houses. However, professional washing machines used in industries are out of the scope of this study due to a lack of data.
- Inputs and outputs are in line with the Table 1 of list of inclusions and exclusions.
- Due a lack of data in wastewater release, it is necessary to estimate the approximately amount of wastewater produced from one laundry wash cycle. It has to be considered

that a 60% of the water is absorbed by the washed fabric (this percentage is calculated through real and technical trials carried out by LEITAT in the B1 task of the MERMAIDS project).

- The potential effect of microplastics fibres release has not been assessed in a quantitative way in this LCA analysis. Other methodologies are being studied in order to incorporate these microplastics release in the final life cycle impact assessment.
- Data about detergent formulations is scarce owing to they contain confidential quantitative information . Detergent data, therefore has been obtained through existing LCA of laundry detergent studies.

6.1.1 Life Cycle Inventory: laundry phase

As it is mentioned before, laundry phase has considered the washing of woven polyester (100%) fabric, detergent use, energy and water consumption. In this section, these parameters are being described.

A life cycle inventory has been done in order to obtain environmental data of a laundry process. The three baseline scenarios selected for a laundry process are summarized in table 2.

Current laundry process[9]	Baseline A	Baseline B	Baseline C
Washing machine load:		4,5 kg of fabric	
Washing machine energy consumption	0,62 kWh	0,67kWh	0,63 kWh
Washing machine water consumption:	55,1L	78L	59,8L
Detergent consumption:	75g (See table 4)	75 g (See table 5)	75 ml (confidential data
Wastewater release:	22,4L	31,2L	23,92L

Table 2. Summary of the main baseline scenarios [10].

[9] All these data is based and calculated taking into account different LCA studies.

[10] Arendorf, J. et al. Revision of European Ecolabel Criteria for Laundry Detergents. Preliminary Report. October 2014. / Beton, A. et al. Environmental Improvement Potential of Textiles (IMPRO Textiles). / Sustainable Innovation. Household and Personal care Today. Cost-neutral replacement of surfactants with enzymes- a short-cut to environmental improvement for laundry washing. Denmark. . 4/2007

Detergent formulation and use

There is no standard laundry detergent formulation. A large number of different ingredients can be used in a variety of combinations giving rise to different formulations. Sometimes, this quantitative information is confidential therefore it is difficult to obtain it from their manufacturers.

Generally, all laundry detergents contain the following categories of ingredients but in different concentrations: surfactants, builders, bleaching agents and auxiliary agents. The table 3 shows the general characteristics of a laundry detergent[11].

Product formulation	Function	Concentration (WT%)
Surfactant	Wetting agent, soil removal, soil/film prevention, sheeting action, soil dispersion, drying aid	10-15%
Builder	Sequestration, soil suspension, alkalinity, emulsification, soil pepitization	30-85.5%
Bleach	Soil removal, stain removal, sanitation, disinfection	7-21%
Defoamer (optional)	Foam prevention, wash efficiency	0-1%
Colour, perfume (optional)	Aesthetic enhancement	0-1%
Water (optional)	Solvent, carrier, flow property	Balance

Table 3. Generic laundry detergent formulation. Source: Oakende Hollins, based on data from www.isditproductveilig.nl

All the information related to the composition of a current liquid and powder detergents, is presented in the A.2 Report of the MERMAIDS project. This report is based on the influence of commercial laundry agents and washing conditions on microplastics release reduction by use of commercial detergents, additives and changing laundering conditions.

Detergent ingredients of the Baseline Scenario A:

The table 4 shows an example of the main ingredients of laundry detergent for the Baseline scenario A.

Ingredient	Detergent (g)	Ecoinvent data
Water	5.85	Water, completely softened, at plant /RER S
Sodium carbonate	16.63	GLO: Sodium carbonate from NH3Ch production, at plant
Sodium sulphate	14.92	Sodium sulphate, powder, production mix, at plant/RER S
Sodium percarbonate	9.95	Sodium percarbonate, powder, at plant/RER S
Na, Linear alkylbenzene sulfonate	6.52	Alkylbenzene, linear, at plant/RER S

[11] Arendorf, J. et al. Revision of European Ecolabel Criteria for Laundry Detergents. Preliminary Report. October 2014

Zeolite	5.28	Zeolite, powder at plant / RER S
Sodium silicate	3.53	Layered sodium silicate, SKS-6, powder, at plant/RER S
Bentonite	3.36	Bentonite, at processing/ DE S
C12-15 alkylethoxysulphate (3EO)	2.31	RER: fatty alcohol sulphate mix, at plant
Sodium acrylic acid	1.11	Acrylic acid {RER}| production | Alloc Def, S
Carboxymethyl cellulose	0.92	Carboxymethyl cellulose, powder, at plant /RER S
Citric	0.74	Citric acid {RER}| production | Alloc Def, S
Polycarboxylate polymer	0.43	Polycarboxylates, 40% active substance, at plant/RER S
Sodium chloride	0.05	Sodium chloride, powder, at plant /RER S
Unknown[12]	3.40	Empty process

Table 4. Ecoinvent data inventory for a laundry detergent frame formula. Baseline scenario A. Source[13].

Note that the detergent formulation used for this baseline scenario A is just an example among the several kind of scenarios that could be used by considering data previous 2012. Also note that some components of considered detergents tend to decrease in current detergents due to environmental restrictions, etc (e.g. perborate...).

Detergent formulation of the Baseline Scenario B:

The table 5 offers another example of the laundry detergent composition for the Baseline scenario B.

Ingredient	Detergent (g)	Ecoinvent data
LAS	6.60	Alkyl benzene sulphonate, linear, petrochemical at plant/RER U
Ethoxylated fatty alcohol	3.60	Ethoxylated fatty alcohol
Sodium Soap	2.40	Sodium Soap
Protease, lipase, amylase and cellulose	0	Protease, lipase, amylase and cellulose
Sodium silicate	2.30	Layered sodium silicate, SKS-6, powder, at plant/RER S
Zeolite	21.30	Zeolite, powder at plant / RER S
Sodium carbonate	8.70	Sodium percarbonate, powder, at plant/RER S
Sodium salt of a copolymer from acrylic and maleic acid	1.80	Sodium salt of a copolymer from acrylic and maleic acid
Phosphonate	2.10	Phosphonate
Sodium perborate tetrahydrate	15.00	Sodium sulphate, powder, production mix, at plant/RER S
TAED (EDTA)	2.30	TAED (EDTA)
Sodium sulfate	4.90	Sodium sulphate, powder, production mix, at plant/RER S

12 Due to the lack of data in Ecoinvent, there is an empety process of three substances, which causes uncertainty in the impact assessment because they are not considered.

13 Arendorf, J. et al. Revision of European Ecolabel Criteria for Laundry Detergents. Preliminary Report. October 2014

CMC	0.90	Carboxymethyl cellulose, powder, at plant /RER S
Optical whitener	0,20	Optical whitener
Foam inhibitor	2,90	Foam inhibitor

Table 5. Ecoinvent data inventory for a laundry detergent frame formula. Baseline scenario B. Source[14]

Note that the detergent formulation used for this baseline scenario B is an example which could be changed over the last few years to be more environmentally friendly.

Detergent formulation of the Baseline Scenario C:

The detergent formulation of the baseline scenario C is only an example of the liquid detergent composition and it is not representative for all existing liquid detergents used in the market. This detergent formulation has not been shown in this section due to it contains quantitative and confidential information. It is only used to model and assess the baseline scenario C.

Washing phase

The inputs considered during the washing phase are: water and energy consumption since they are necessary resources for the washing action. The amount of water and energy consumption are based on available information from literature. Other inputs, such as energy for drying and ironing have not been taken into account as they are optional and not directly related to this analysis.

For the future LCA, the wastewater composition will be considered in order to measure the presence of microplastics released at the end of a washing cycle. It is necessary to know the quantity of microplastics released into wastewater during a current laundry process in order to compare with the presence of microplastics at the end of the project when the MERMAIDS solutions will be applied. The wastewater release has not been assessed for the baseline scenario.

LEITAT with CNR partner have made a series of tests in order to determine the presence of microplastics at the end of a current washing cycle. For these assays, the following issues and assumptions have been considered:

- LEITAT has carried out some pre-trials in order to check and set the definitive domestic conditions in lini-test based on ISO 105-C06:2010 and to check all equipment and filtration tests.
- The washing conditions have been:

[14] Sustainable Innovation. Household and Personal care Today. Cost-neutral replacement of surfactants with enzymes- a short-cut to environmental improvement for laundry washing. Denmark. . 4/2007.

- Temperature: 40ºC
- Time: 45 minutes
- number of steel balls:10
- Medium: distilled water

- Some assays have been carried out changing the washing conditions in order to observe changes in the microfibers release. The detergent used has been a commercial liquid detergent with a dose of 65ml/15L of water, which washing pH is 8.1.
- At laboratory level, the woven polyester selected is polyester ref 777. (The woven polyester (Ref.777) has been selected to be the worst quality textile in terms of pilling formation).
- After the washing in the Lini-test, the samples were filtrated using Durapore® membrane filters from Merck Millipore with a pore diameter of 5 µm (ref. SVLP04700). Once the filtration was finished, the filters were exposed to 85 ºC during 30 minutes and, finally, analyzed the presence of microplastics with a Scanning Electron Microscope (SEM).
- 1.000 microplastics/ g of fabric (for 1 g of fabric of woven polyester 100%) are released during the washing process.
- Linear mass density of a microplastics (theoretical data) is 1 *denier (*1 g per 900 metres of fibre).
- Polyester microplastic length: 36 mm (LEITAT experimental data).
- It is assumed that 250000 microplastics weigh 1 g.

According with all these considerations the approximately number of microplastics released to wash a 4.5kg of woven polyester 100% fabric is estimated with a range between 15 to 30 g microplastics/ laundry load. (Note that the quantity of the microplastics released could significantly vary if it is washed with other type of textiles as polypropylene, polyamide and knitted polyester).

6.1.2 Data quality

Data concerning the laundry process are provided from different existing studies as it has been mentioned before. The LCI data on electricity and water consumption from the scenarios have been gathered from the Ecoinvent database. The European electricity grid mix (electricity, medium voltage, production RER, at grid/RER S) and the domestic consumption of tap water (Tap water, at user {Europe without Switzerland}| market for | Alloc Def, S) have been considered, respectively.

6.2 Life Cycle Impact Assessment

The base methodology chosen for Impact assessment is the ILCD method. The ILCD 2011 Midpoint method was released by the European Commission, Joint Research Centre in 2012. It supports the correct use of the characterisation factors for impact assessment as recommended in the ILCD guidance document "Recommendations for Life Cycle Impact Assessment in the European context - based on existing environmental impact assessment models and factors.[15] This LCIA method includes 16 midpoint impact categories. For this study, the following have been selected to be considered relevant for the system studied. Detailed information on this impact categories are detailed in the table 6.

Impact category	Description	Model/ Method	Analyzed
1 - Climate change	Global Warming Potential calculating the radiative forcing over a time horizon of 100 years.	IPCC 2007.	✓
2 - Ozone depletion	Ozone Depletion Potential (ODP) calculating the destructive effects on the stratospheric ozone layer over a time horizon of 100 years.	World Meteorological Organization (WMO) 1999.	✓
3 - Human toxicity, cancer effects	Comparative Toxic Unit for humans (CTUh) expressing the estimated increase in morbidity in the total human population per unit mass of a chemical emitted (cases per kilogramme). Specific groups of chemicals require further works.	USEtox.	✓
4 - Human toxicity, non-cancer effects	Comparative Toxic Unit for humans (CTUh) expressing the estimated increase in morbidity in the total human population per unit mass of a chemical emitted (cases per kilogramme). Specific groups of chemicals require further works.	USEtox.	✓
5 - Particulate matter	Quantification of the impact of premature death or disability that particulates/respiratory inorganic have on the population, in comparison to PM2.5. It includes the assessment of primary (PM10 and PM2.5) and secondary	Rabl and Spadaro 2004.	✓

[15] The ILCD 2011 Midpoint method was released by the European Commission, Joint Research Centre in 2012. It supports the correct use of the characterisation factors for impact assessment as recommended in the ILCD guidance document "Recommendations for Life Cycle Impact Assessment in the European context - based on existing environmental impact assessment models and factors

	PM (incl. creation of secondary PM due to SOx, NOx and NH3 emissions) and CO.		
6 - Ionizing radiation HH (human health)	Quantification of the impact of ionizing radiation on the population, in comparison to Uranium 235.	Frischknecht et al. 2000.	✓
7 - Ionizing radiation E (ecosystems)	[note: this method is classified as interim; see reference for explanation]: Comparative Toxic Unit for ecosystems (CTUe) expressing an estimate of the potentially affected fraction of species (PAF) integrated over time and volume per unit mass of a radionucleide emitted (PAF m3 year/kg). Fate of radionucleide based on USEtox consensus model (multimedia model). Relevant for freshwater ecosystems.	Garnier-Laplace et al. 2008.	
8 - Photochemical ozone formation	Expression of the potential contribution to photochemical ozone formation. Only for Europe. It includes spatial differentiation	van Zelm et al. 2008.	✓
9 - Acidification	Accumulated Exceedance (AE) characterizing the change in critical load exceedance of the sensitive area in terrestrial and main freshwater ecosystems, to which acidifying substances deposit. European-country dependent.	Seppälä et al. 2006 and Posch et al. 2008.	✓
10 - Terrestrial eutrophication	Accumulated Exceedance (AE) characterizing the change in critical load exceedance of the sensitive area, to which eutrophying substances deposit. European-country dependent.	Seppälä et al 2006 and Posch et al. 2008.	✓
11 - Freshwater eutrophication	Expression of the degree to which the emitted nutrients reaches the freshwater end compartment (phosphorus considered as limiting factor in freshwater). European validity. Averaged characterization factors from country dependent characterization factors.	ReCiPe version 1.05.	✓
12 - Marine eutrophication	Expression of the degree to which the emitted nutrients reaches the marine end compartment (nitrogen considered as limiting factor in marine water). European validity. Averaged characterization factors from country dependent characterization factors.	ReCiPe version 1.05.	✓
13 - Freshwater ecotoxicity	Comparative Toxic Unit for ecosystems (CTUe) expressing an estimate of the potentially affected fraction of species (PAF) integrated over time and volume per unit mass of a chemical emitted (PAF m3 year/kg). Specific groups of chemicals require further works.	USEtox.	✓
14 - Land use	Soil Organic Matter (SOM) based on changes in SOM, measured in (kg C/m2/a). Biodiversity impacts not covered by the data set.	Mila i Canals et al. 2007.	
15- Water resource depletion/ Total freshwater consumption, including rainwater	Freshwater scarcity: Scarcity-adjusted amount of water used.	Swiss Ecoscarcity 2006.	✓
16 - Mineral, fossil & renewable resource depletion	Scarcity of mineral resource with the scarcity calculated as 'Reserve base'. It refers to identified resources that meet specified minimum physical and chemical criteria related to current mining practice. The reserve base may encompass those parts of the resources that have a reasonable potential for becoming economically available within planning horizons beyond those that assume proven technology and current economics.	van Oers et al. 2002.	✓

Table 6. Impact categories of ILCD LCIA method

In general terms, LCA studies consulted previously indicate that for a current laundry process, the energy consumption together with detergent use are the inputs with the major environmental impacts in the life cycle[16].

6.2.1 Results of the BASELINE SCENARIOs (LCIA)

This section presents the Life Cycle Impact Assessments of the considered baseline scenarios and it is based on the data obtained from the inventory stage. The environmental impacts have been assessed by the impact categories listed from table 6. The results obtained for each baseline scenario are:

Environmental impact of the Baseline scenario A:

Impact categories	Unity	Water	Detergent	Energy
Climate change	kg CO2 eq	0,01963574	0,09864725	0,31119666
Ozone depletion	kg CFC-11 eq	1,51E-09	9,26E-09	1,47E-08
Human toxicity, cancer effects	CTUh	2,41E-09	1,45E-08	2,23E-08
Human toxicity, non-cancer effects	CTUh	6,63E-09	3,49E-08	8,13E-08
Particulate matter	kg PM2.5 eq	1,07E-05	5,46E-05	0,00014766
Ionizing radiation HH	kBq U235 eq	0,0076869	0,02634551	0,23837887
Ionizing radiation E (interim)	CTUe	1,35E-08	8,08E-08	7,35E-07
Photochemical ozone formation	kg NMVOC eq	5,67E-05	0,00026544	0,00064188
Acidification	molc H+ eq	0,00011147	0,00054841	0,00161582
Terrestrial eutrophication	molc N eq	0,00019468	0,00099039	0,00226379
Freshwater eutrophication	kg P eq	1,08E-05	4,77E-05	0,00026414
Marine eutrophication	kg N eq	1,98E-05	0,00014566	0,00025613
Freshwater ecotoxicity	CTUe	0,30767541	0,97033945	2,0402749
Land use	kg C deficit	0,08537529	0,23056178	0,14794861
Water resource depletion	m3 water eq	0,0579828	0,05026748	0,3903662
Mineral, fossil & ren resource depletion	kg Sb eq	1,43E-06	2,05E-06	1,29E-06

Table 7. Environmental impacts of the laundry phase (baseline A)

The figure 10 of the distribution of environmental impacts of the laundry phase (baseline scenario A) shows that energy consumption is the responsible of the major impact contributions in fourteen out of sixteen categories, except on mineral, fossil & renewable resource depletion (27%) and land use (32%). In all of them electricity has a more than a 50% of impact contribution. Ionizing radiation E is the impact category which records a higher impact contribution due to electricity consumption (88%).

[16] Arendorf, J. et al. Revision of European Ecolabel Criteria for Laundry Detergents. Preliminary Report. October 2014./ Beton, A. et al. Environmental Improvement Potential of Textiles (IMPRO Textiles)./ Koerner, M.; Shulz, M.; Powell, S. and Ercolani, M. The Life Cycle assessment of clothes washing options for city west water's residential customers. Australia. 2010./ Saouter, E. and Van Hoof, G. A database for the Life Cycle Assessment of Protect and Gamble Laundry Detergents. Belgium. 2001.

Otherwise, for two out of sixteen categories, the detergent use is the responsible of the major impact contributions. These percentages of environmental impact contribution range it from 43% (mineral, fossil & renewable resource depletion) to 50% (land use category).

Finally, the water consumption has low environmental impact contribution (maximum of 30% on mineral, fossil & renewable resource depletion category).

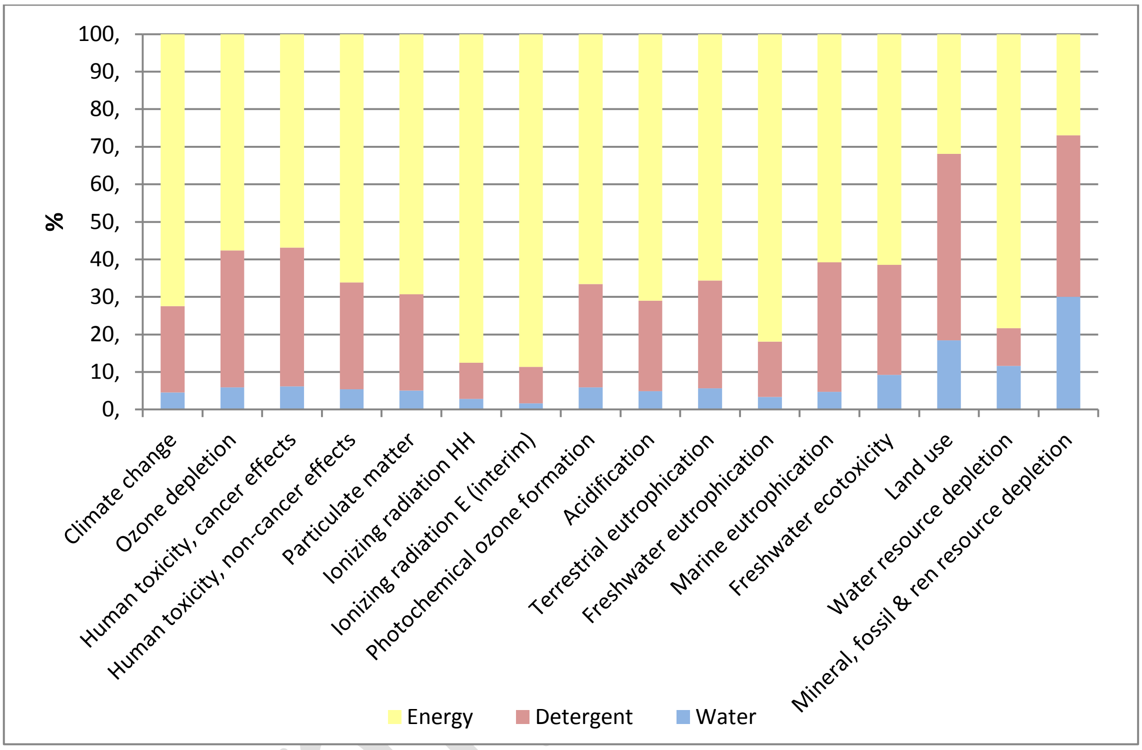

Figure 10.Environmental impacts of the laundry phase (%, baseline A)

The potential environmental impacts of the detergent formulation are analysed in detail in the figure 11. It can be seen that the zeolite is the major contributor in fifteen out of sixteen categories, except on marine eutrophication (16%).

The bentonite has the higher environmental impact contribution in Land Use category (55%). The sodium carbonate has the higher environmental impact contribution in marine eutrophication (44%). And the sodium percarbonate contributes to the category of freshwater eutrophication with 24%. It is important to recall that some ingredients could be replaced by other components to be more environmentally friendly.

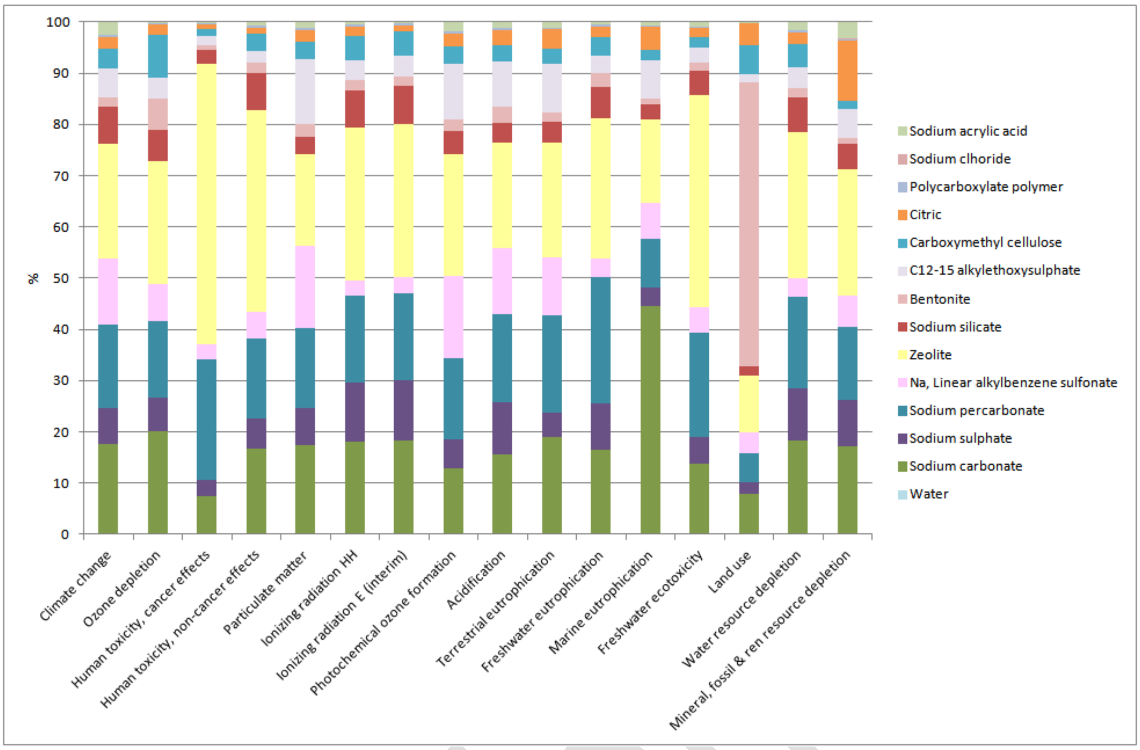

Figure 11. Environmental impacts of 75g of the detergent formulation A(%, baseline A)

Environmental Impact of the Baseline scenario B:

Impact categories	Unity	Water	Detergent	Energy
Climate change	kg CO2 eq	0,02779651	0,21441758	0,32119579
Ozone depletion	kg CFC-11 eq	2,13E-09	2,10E-08	4,13E-08
Human toxicity, cancer effects	CTUh	3,41E-09	4,24E-08	8,05E-09
Human toxicity, non-cancer effects	CTUh	9,38E-09	8,72E-08	3,75E-08
Particulate matter	kg PM2.5 eq	1,51E-05	0,00011764	0,00013017
Ionizing radiation HH	kBq U235 eq	0,01088164	0,06012482	0,16207505
Ionizing radiation E (interim)	CTUe	1,92E-08	1,86E-07	3,42E-07
Photochemical ozone formation	kg NMVOC eq	8,03E-05	0,00061181	0,00094802
Acidification	molc H+ eq	0,0001578	0,00109332	0,00212686
Terrestrial eutrophication	molc N eq	0,0002756	0,00206025	0,00331554
Freshwater eutrophication	kg P eq	1,53E-05	0,0001015	5,84E-05
Marine eutrophication	kg N eq	2,80E-05	0,00023211	0,00031294
Freshwater ecotoxicity	CTUe	0,43554777	2,4205943	1,0482525
Land use	kg C deficit	0,12085795	0,20676346	0,20470048
Water resource depletion	m3 water eq	0,08208091	0,11306486	3,0004538
Mineral, fossil & ren resource depletion	kg Sb eq	2,03E-06	4,11E-06	1,82E-06

Table 8. Environmental impacts of the laundry phase (baseline B)

The figure 12 of the distribution of environmental impacts of the laundry phase (baseline scenario B) shows that energy consumption is the responsible of the major impact contributions in ten out of sixteen categories: water resource depletion, ionizing radiation HH, ionizing radiation E, ozone depletion, acidification, terrestrial eutrophication, photochemical ozone formation, climate change, marine eutrophication and particulate matter. In all of them electricity has a more than a 49% of impact contribution. Water resource depletion is the impact category which records a higher impact contribution (94%) due to electricity consumption.

Otherwise, for six out of sixteen categories, the detergent use is the responsible of the major impact contributions. These percentages of environmental impact contribution range it from 38% (land use category) to 75% (human toxicity, cancer effects category).

Finally, the water consumption has a small environmental impact contribution (maximum of 25% on mineral, fossil & renewable resource depletion category).

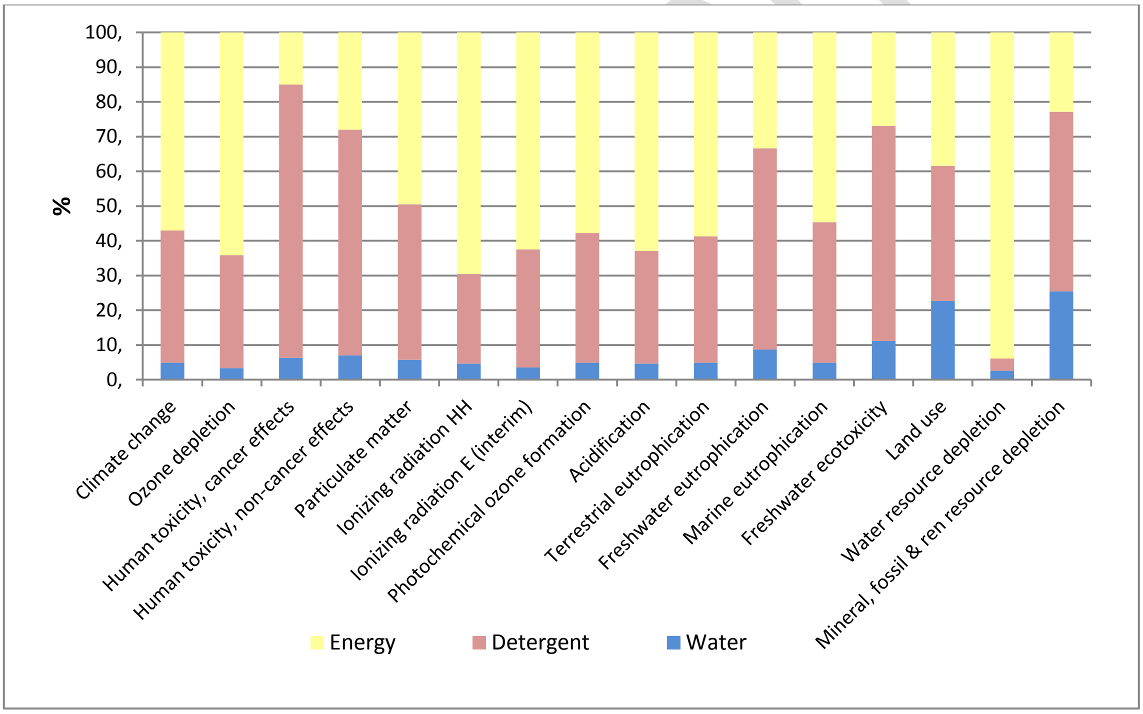

Figure 12. Environmental impacts of the laundry phase (%, baseline B)

The potential environmental impacts of the detergent formulation are analysed in detail in the figure 13. It can be seen that the Zeolite is the major contributor in all the impact categories. Zeolite contributes to the categories of freshwater ecotoxicity and human toxicity, cancer effects with the higher impacts: 67% and 76 % respectively.

The sodium perborate is the second component with the higher environmental impact contribution in climate change (25%) and ozone depletion (27%). It is important to recall that some ingredients could be replaced by other components to be more environmentally friendly.

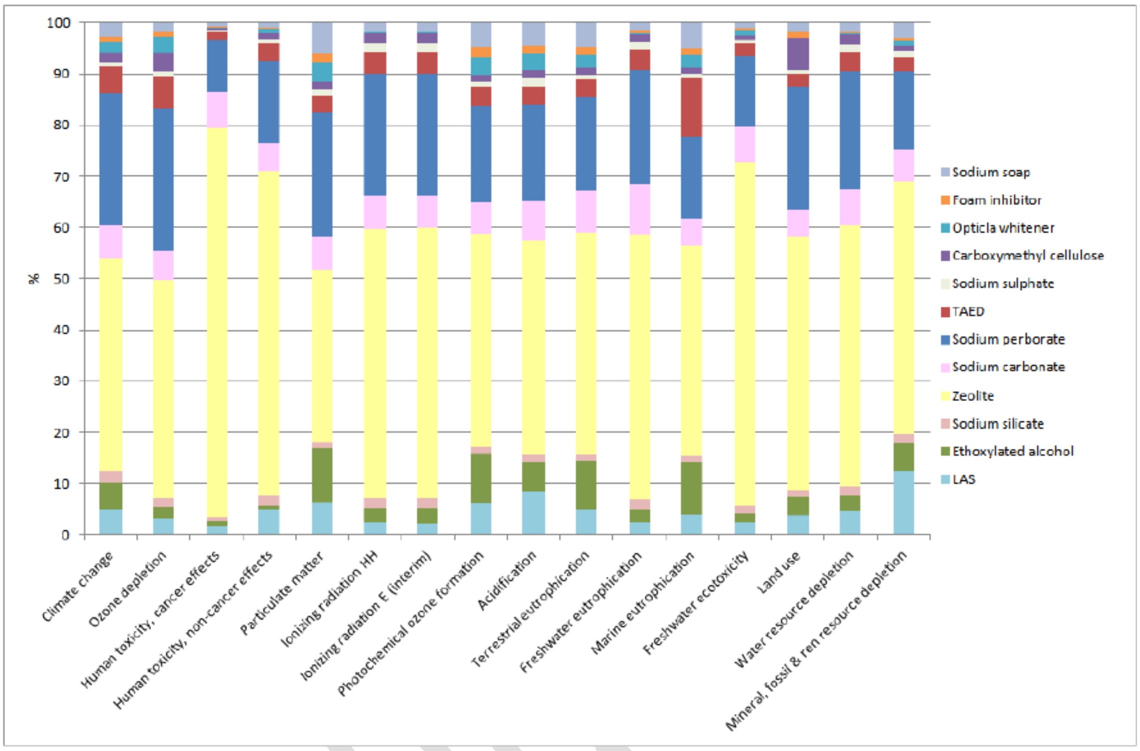

Figure 13. Environmental impacts of 75g of the detergent formulation B (%, baseline B)

Environmental Impact of the Baseline scenario C:

The figure 14 of the distribution of environmental impacts of the laundry phase (baseline scenario C) shows that energy consumption is the responsible of the major impact contributions in twelve out of sixteen categories: ionizing radiation E, ionizing radiation HH, freshwater eutrophication, human toxicity, cancer effects, human toxicity, non-cancer effects, water resource depletion, climate change, acidification, freshwater ecotoxicity, particulate matter and photochemical ozone formation. In all of them electricity has a more than a 55% of impact contribution. Ionizing radiation E is the impact category which records a higher impact contribution (92%) due to electricity consumption.

Otherwise, for four out of sixteen categories, the detergent use is the responsible of the major impact contributions. These percentages of environmental impact contribution range it from 50% (marine eutrophication) to 86% (land use category).

Finally, the water consumption has a small environmental impact contribution (maximum of 11% on water resource depletion category).

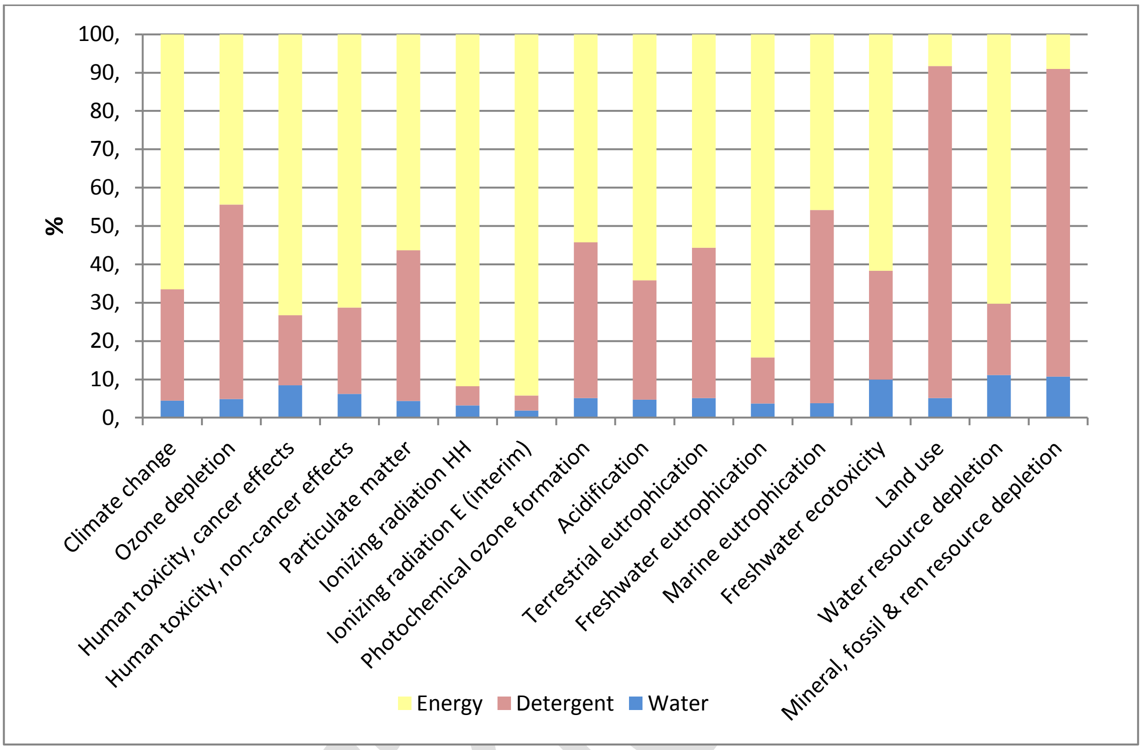

Figure 14. Environmental impacts of the laundry phase (%, baseline C)

7. Conclusions

The conclusions from the Life Impact Assessment of the baseline scenarios are:

- The LCA boundaries for the targeted system have been defined in figure 8 and the functional unit has been established as 4.5 Kg of laundry load fabric *(of woven polyester 100%).*
- The inputs of the laundry process with the largest contribution to environmental impacts are detergent use and energy consumption. The high contribution of the energy consumption is due to the energy consumed to heat water for the wash cycle. The major contribution of detergent use is owing to it requires sodium compounds, as well as surfactants, which are potentially harmful for human health, water and terrestrial ecosystems. The impacts of water consumption are very low.
- The approximately number of microplastics released to wash a 4.5kg of woven polyester 100% fabric is estimated with a range between *500 to 2000 grams of microplastics.* (the assumptions and washing conditions are explained in the section 6.1.1).
- These assessments have demonstrated that future LCA should pay special attention to the following environmental impact categories identified as the major potential impact during the laundry use:

1. **Ionizing radiation HH /and E(interim)**
2. **Water resource depletion**
3. **Freshwater eutrophication**
4. **Human toxicity (cancer and non-cancer effects)**
5. **Mineral, fossil and resource water depletion**
6. **Marine eutrophication**
7. **Freshwater ecotoxicity**
8. **Climate change**
9. **Ozone depletion**
10. **Land use**

- These impact categories will be assessed in the complete LCA that will be performed once the new textile additives and innovative detergent additives are decided (expected - Sep.2016). The analysis and comparison of the selected impact categories in such LCA will allow measuring the achievement of the three main environmental objectives of the MERMAIDS solutions:
  - To demonstrate innovative additives to improve finishing textiles and finishing fibres treatments to avoid garments' microplastic removal in laundry process.
  - To demonstrate innovative additives for detergent products and laundry products to avoid garments' microplastic removal in laundry process.
  - To reduce at least 70% of the total amount of microplastic fibres that is currently discharged in laundry wastewater.

- Regarding the examples of powder detergent ingredients which should receive special consideration in the future LCA are, among others:
  - **Zeolite**
  - **Sodium percarbonate**
  - **Sodium perborate**
  - **Sodium carbonate**

- Regarding with the baseline scenario C which presents an example of laundry phase with liquid detergent, the category of ingredients which should receive special consideration in the future LCA are surfactants. Noteworthy that the detergent composition is confidential and for this reason is not possible to offer in detail the exactly detergent ingredients and their environmental contribution.

It is necessary to take into account that some ingredients presented in detergents A and B could be replaced. For this reason, detergent industry has been studying different ingredients which improve detergent efficiency and replace those components more harmful to the environment by others with less environmental damage.

Significant benefits with respect to water emissions, eutrophication and climate protection will be also pursued.

Noteworthy that the selection of environmental impact categories will be updated in the future LCA, as necessary, in accordance to the data from the MERMAIDS solutions.

In conclusion, the goal and scope for the future complete LCA have been established as well as the environmental impact categories on which it will be focused. This assessment will demonstrate the environmental performance and viability of finishing textile treatments and new detergent additives assessed by the MERMAIDS project.

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Handbook for zero microplastics from textiles and laundry

mermaids.
OCEAN CLEAN WASH

Good practice guidelines for the textile industry

1. Explanation of the topic and purpose of the guidelines

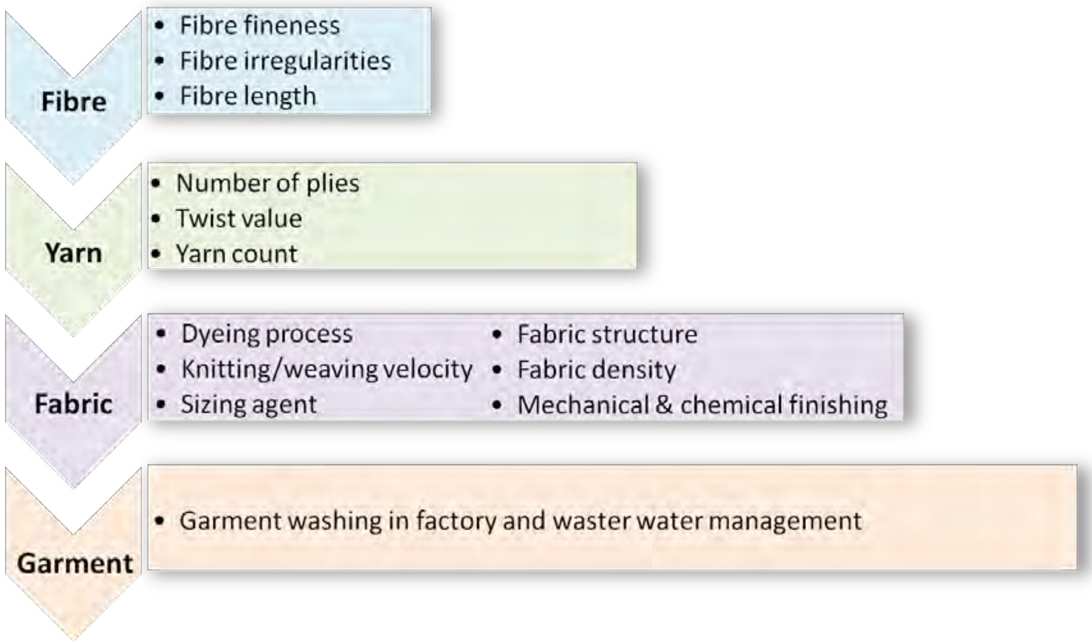
Polyester and acrylic are the main synthetic fibres identified as microfibers pollutants coming from the textile industry and found into the oceans[1]. Some characteristics of the production process of synthetic fibres and resulting characteristics of the fibres may be responsible of creating more microfibers release during laundry. The aim of these guidelines is to provide the synthetic fibres manufacturers, the textile auxiliaries manufacturers and others textile industries with good practices to reduce the microfibers production and their release to the environment. This should be done as far as possible and it needs to be suitable with their manufacturing process and final product requirements.

This guideline is indicated for any industry in the textile chain (synthetic fibres manufacturer, textile auxiliary manufacturer, spinning /weaving/knitting companies, finishing (chemical or mechanical) companies and textile retailers. This document can be useful for continuous synthetic fibres manufacturers in case the fabric they produce is finished by any mechanical treatment responsible of cutting and raising the fibres from the fabric surface to give a warm and softer hand.

[1] Microplastics from washing machine wastewater are polluting beaches, "Science for Environment Policy": European Commission DG Environment News Alert Service, edited by SCU, The University of the West of England, Bristol, 2012.

2. Key issues identified

The key issues identified for the textile industry include material and process modification at four different stages which are the fibre, the yarn, the fabric and the garment. This issues are detailed in the figure below.



3. Good practices

Good practices are given for each step in the production of synthetic fibres. For each practice presented in the next part, the main barriers are identified and the policies which could be implicated are mentioned. The practices are advices that should be taken as far as possible. Moreover, they should besuitable with the production process and the final product requirements.

POLYMER SYNTHESIS AND CHIPS PRODUCTION

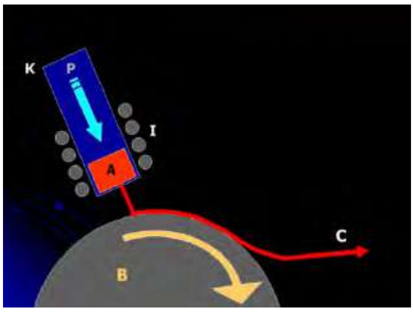
Process description:

Preparation of viscous PES liquid from solid polymers or from monomers. The viscous fluid is then extruded, dried and broken into chips.

Good practices guidelines: N/A

Main barriers: N/A

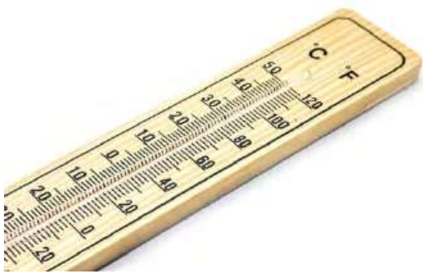
MELT SPINNING PROCESS



Process description:

The chips of PES are heated and extruded through a spinneret (metal disk with fine holes). The hardening PES liquid coming outside the holes is forming the resulting PES filaments. The filament (called tow at this step) are then wounded on a spool.

Good practices guidelines:



The **melting process conditions** (lower and graduated melting temperatures) could be adjusted in order to preserve the fibres mechanical properties (tensile strength...).

The **fibre fineness** should be increased to reduce yarn propensity to form protruding microfibers.

The **fibre irregularities** would increase the friction between fibres and avoid the release of microfibers from the yarn. The holes of the spinneret could present some irregularities instead of having a circular diameter.

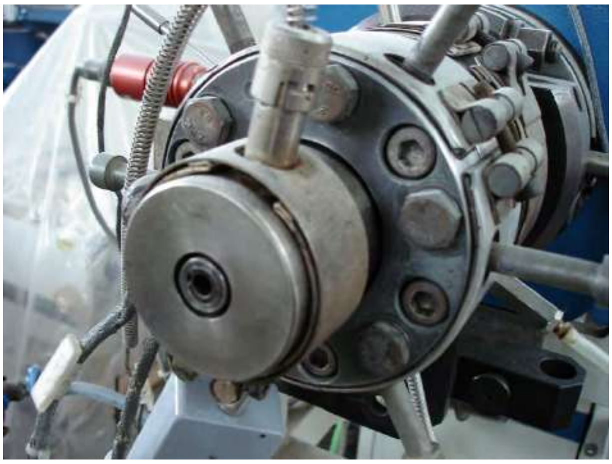
Main barriers:

Working at lower temperatures will increase the production times.

The modification of the yarn characteristics would probably not answer the customer requirements.

DRAWING, STRETCHING, TEXTURING, INTERMINGLING AND DRYING

Process description:



Drawing is made on the spool to increase the strength of the fibre. Stretching increase the fibre length from three to four times its original length. Texturing gives the fibres a crinkled structure. Intermingling is done to provide a specified number of knots per meter on the fibre length. Drying is the last step before cutting the filaments into specific lengths of a few centimetres.

Good practices guidelines:

The different **processes conditions** should be adjusted in order to preserve a good fibre tensile strength. A high tensile strength of the yarn will reduce its probability of breakage and release during washing.

The **cutting length** of the resulting fibres should not be too low to avoid the release of microfibers from the yarn.

With the contribution of the LIFE financial instrument of the European Comunity!

Main barriers:

Process modification feasibility depends on the client requirements and products specifications.

SPINNING (YARN)

Process description:

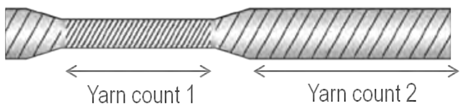
The cut filaments are carded to parallelize the fibres and a rope-like material is obtained. The rope is introduced in a spinning machine to reduce its diameter until obtaining a yarn and giving it a twist for more cohesion.

Good practices guidelines:

Yarns with **continuous fibres** have lower incidence on microfibers formation than yarns made of discontinuous or staple fibres.

Plied yarns have lower incidence on microfibers formation than single yarns.

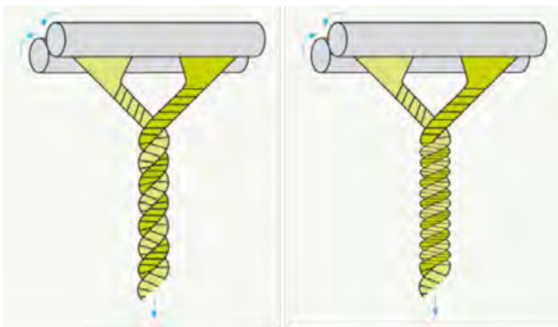
Yarns with **high twist** have lower incidence on microfibers formation than yarns with low twist.



A **low linear density of the yarn** (yarn count) is better than high yarn count as the number of microfibres released will increase with the yarn count due to a larger amount of fibres per cross section.

Main barriers:

The yarns specification has to respond to client requirements and the final product properties depends on these parameters.



Low twist

High twist recommended to avoid short fibres release from the yarn structure during laundry

DYEING

Process description:

The spools of twisted yarns are introduced in a dyebath containing specific dyes to the polymer type. Then the spools are dried before knitting or weaving.

Good practices guidelines:

Avoid garment dyeing, as it has more impact on the microfibers release than **yarn dyeing**.

Main barriers: N/A

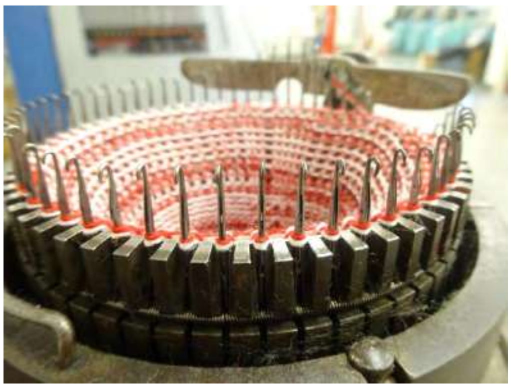
KNITTING AND WEAVING

Process description:

The dyed spools can be knitted (on circular or rectilinear machine) or woven. During this step, the yarn is submitted to mechanical actions and erosion onto the machine parts (needles, yarn carrier, weft thread transporter, warp threads movements, etc.).

Good practices guidelines:

The fibres are damaged during the knitting process by the action of the yarn carrier and the needles. The yarn **carrier velocity** may be reduced to reduce the fibres damage.



For the weaving process, the **quantity or the nature of the sizing agent** could be optimized and the **velocity** of the weft transporter could be reduced.

High density fabrics (number of yarns per unit length) are better (tighter structure) than low density fabrics to avoid the microfibersrelease.

Plain weave fabrics have lower incidence on microfibers formation than twill weave fabrics.

Main barriers:

The reduction of the velocity of the weaving and knitting process will increase the production time of the factory.

MECHANICAL FINISHING

Process description:

Napping is done to obtain a fuzzy effect on the fabric surface. The process consists in raising the fibres from the textile surface with revolving cylinders with bristles. Then, during **shearing**, the protruding fibres are cut at the same height.

Good practices guidelines:

The napping **process conditions** may be modified to reduce the mechanical action of the cylinders on the fabric and reduce the fibres weakening.

If not already done, the cut fibres resulting from the shearing process may be recollected and this **waste management** should be done in the factory.

Singeing mechanical finishing is a good option to avoid the microfibers formation on fabric surface.

Main barriers:

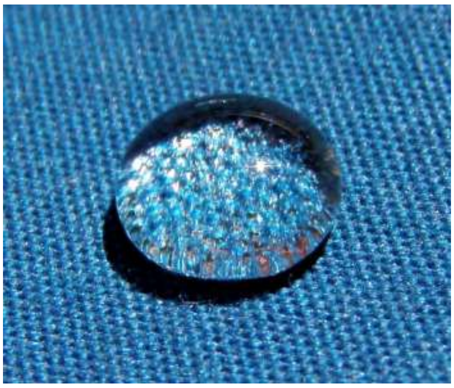
The difficulties to adjust the napping machine parameters or the modification on the machine won't respond to the client requirements.

Implications for policy and practice:

Set a policy for napping process conditions and waste management after shearing process.

FINISHING

Process description:



The finishing step is performed in a textile companies which apply textile auxiliaries products to provide a functionality to the fabric (water repellence, softness, antimicrobial, etc.).

Good practices guidelines:

Finishing agents with smoothening or softening properties capable of protecting the fabric surface can be used during the finishing process. The finishing agents should also have good washing fastness to 1) avoid dragging more fibres during laundry, 2) be effective during the whole product lifetime and 3) avoid contaminating domestic waste water.

Main barriers:

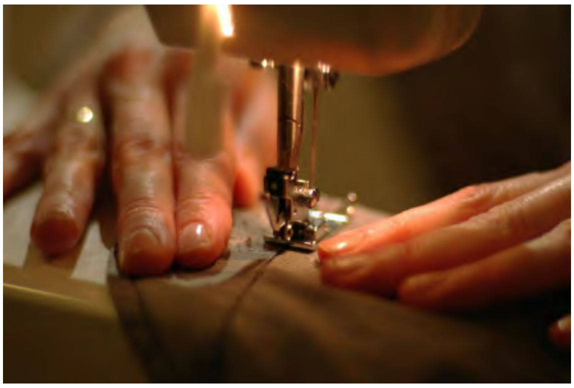
The protective finishing agent must be compatible with the others finishing treatments of the textile.

Implications for policy and practice:

The environmental impact of the finishing agent must be evaluated as well and some policies could regulate its maximum concentration per square metre of fabric.

MANUFACTURING

Process description:



The manufacturing process is the last step where the fabric is cut following a pattern and then sewed into a garment.

Good practices guidelines:

In general, most of the microfibres are released during the first wash of the garment. A **preliminary washing** of the manufactured garment can be done before selling it in order to eliminate the microfibers broken during the textile processes and present on the fabric surface. These microfibers would be then recollected by an **efficient water treatment system**.

Main barriers:

Textile manufacturing companies may not have the required facilities for industrial washing and water treatment. It might require high investment for small manufacturers.

The actual water treatment plants may not be equipped with a system capable of collecting the shortest microfibers.

Implications for policy and practice:

To set a new policy for textile water treatment plants allowing the controlled release of shortest microfibers.

Good practice guidelines for detergents manufacturers

1. Explanation of the topic and purpose of the guidelines

Part of the microplastics pollution found in the oceans comes from the washing in laundry machine of synthetic fabrics. The microfibers release comes from the breakage of the fibres forming fabrics, which can be produced during the washing cycle. Therefore, laundry products and the washing conditions can be involved in this issue. The aim of these guidelines is to provide detergents manufacturers some good practices and advices in order to produce laundry products able to participate in the reduction of the microfibers released during the automatic washing of fabrics.

2. Key issues

This guideline is indicated for laundry products manufacturers, from detergents to laundry aids. Also, it can be of interest for raw materials manufacturers and suppliers. The main key issues developed in this guideline are presented in the following table:

Table 1. Main key issues of this guideline

Washing Conditions	Dissemination
Laundry products	Awareness campaigns
Temperature	
Additives	
New detergents	

3. Good practice

After carrying out several experimental tests with the aim of identifying which kind of laundry products have influence in the microfibers release and how the pollution of microplastics in oceans issue can be mitigated from a detergency approach, some recommendations for detergents manufacturers have been elaborated.

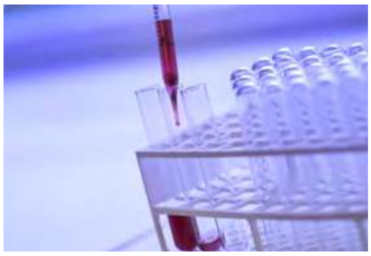
- ✓ **Focus the production of detergents for synthetics on liquid light duty detergents.** In our experiences, the use of heavy-duty detergents produces a bigger microfibers release, due to they are more aggressive with fabrics; especially in the case of powder detergents, which increase the friction in the fibres facilitating their breakage.

- ✓ **Focus the production of other laundry products for synthetics on liquid formats.** The previous recommendation is extensible to all laundry products; for instance, when a powder oxy-product was compared with a liquid one, the powdered aid produced a bigger amount of microfibers released than the liquid one.

- ✓ **Recommend powder products (detergents and aids) only for cotton.** Since most of products for delicates and synthetics are liquid and the final decision is in the consumer hands, a way to reinforce this practice could be to advice the use of powder products only in cotton fabrics.

- **Focus the production of detergents effective at low temperatures.** The temperature is a factor to be taken into account regarding the release of microfibers; to wash clothes at low temperature can decrease the number of microfibers released during the laundry. In addition of helping in the mitigation of this issue, it also supposes a reduction in the energy consumption.

- **Use of additives capable of protecting fibres from their breakage.** After testing several additives with fabric care, it has been observed that some of them are capable of reduce the number of microfibers released during the washing cycle.



➔ Main barriers: it is very difficult to obtain a detergent capable of removing stains efficiently and, at the same time, capable of protecting the fibres from their breakage. However, the idea is to use additives for minimizing the influence of the detergent in the emission of this pollutant, not for avoiding it. More research is needed for develop new additives proper for this purpose.

➔ Implications for policy and practice: the environmental impact of these additives should be evaluated and the concentration allowed regulated.

Long-term Recommendation

- ✓ **Develop detergents not or less aggressive with textile finishing products.** Some textile additives can be used to protect fabrics from their fibres breakage and these finishing products may be detached from the fabric surface during the washing cycle. The idea here, would be to develop detergents capable of minimize or even avoid this to happen.

→ Main barriers: first is needed to find proper textile finishings and, then, some research will be necessary for obtaining this kind of detergents.

- ✓ **Aware the consumer about this issue**. For example, including information about this topic in the label or even using it as a claim in products that follow these recommendations; this way not only the consumer is become aware about that, but also it may suppose an added value for the product.