# **ACT-CEMENTEGRITY Final Report**

The CEMENTEGRITY project was funded through the ACT programme (Accelerating CCS Technologies, Horizon2020 Project No 691712). Financial contributions from the Research Council of Norway (RCN), the Netherlands Enterprise Agency (RVO), the Department for Energy Security & Net Zero (DESNZ, UK), and Harbour Energy are gratefully acknowledged.

# 1. Identification of the project and report

Project title	<b>CEMENTEGRITY</b> : Development and testing of novel cement designs for enhanced CCS well integrity.	
Project ID	327311	
Coordinator	IFE Institute for Energy Technology	
Project website	www.cementegrity.eu	
Reporting period	2021-10-01 to 2024-12-31	

## Participants:

Organisation	Main contact(s) + E-mail	Role in the project
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ReStone AS	Astri Kvassnes – <u>astri@restone.no</u>	Collaborator (WP1, WP5), technical advice
Universitetet i Stavanger	Mahmoud Khalifeh – <u>Mahmoud.Khalifeh@uis.no</u>	WP-leader (WP6)
Delft University of Technology	Anne Pluymakers† – <u>anne.pluymakers@tudelft.nl</u> Guang Ye* – <u>g.ye@tudelft.nl</u>	National Coordinator (Netherlands) <sup>+</sup> WP-leader (WP3 <sup>+</sup> , WP4 <sup>*</sup> )
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Heriot Watt University	Benny Suryanto – <u>b.suryanto@hw.ac.uk</u>	National Coordinator (UK) WP-leader (WP5)
Harbour Energy	Oliver Czuprat – <u>Oliver.Czuprat@harbourenergy.</u> <u>com</u>	Funder, Technical advisor, industry perspective

## 2. Executive summary

CEMENTEGRITY performed research into the main mechanisms by which leakage might form through or along a wellbore seal in a CCS-reservoir, with the aim of identifying the critical properties of a cured cementitious sealant for ensuring long-term seal integrity. We studied five different sealant compositions, based on three different binder technologies and with different TRL's and identified three key abilities.

None of the five blends showed superior performance on all aspects tested. Based on this outcome, the best performance may be achieved by selecting individual blends for different parts of the wellbore system, tailoring the required performance based on the expected local conditions. Furthermore, the learnings from the CEMENTEGRITY project can lead to better all-round blends.

The ability to form and maintain a seal was investigated as part of WP5. This WP also investigated the use of electrical impedance spectroscopy methods for assessing and monitoring the integrity of the seal body, and of the seal-steel interface. Key findings are:

- Curing conditions, setting time of the sealant, and corrosion at the interface between sealant and steel casing were the dominant factors controlling apparent bond strength. Bond strength was also found to be related to compressive strength, but high compressive strength did not guarantee good bonding performance.
- 2) The elevated curing temperature and pressure was found influential in increasing both the shear-bond and compressive strengths of the sealants. The high curing pressure was particularly instrumental in reducing volumetric changes during the extended curing, hence elevating measured bond strength.
- 3) Electrical measurements showed interesting potential for assessing sealant permeability. When the steel casing was used as one of the electrodes, variations in response was observed and linked to the degree of corrosion at the sealant-steel interface. This demonstrates the potential of such methods for monitoring the sealant-steel interface.

The ability to resist exposure to  $CO_2$ -bearing fluids, i.e. liquids, gasses or supercritical fluids (taking into account the  $CO_2$ -phase diagram), under in-situ conditions was addressed in WP1 and WP2. WP1 performed forced-flow experiments using  $CO_2$ -saturated water and wet supercritical  $CO_2$ . Key findings are:

- Supercritical CO<sub>2</sub> penetrates faster than CO<sub>2</sub>-saturated fresh water and may impact a sealant's properties differently. When testing sealants for CCS, the exposure media (CO<sub>2</sub>-saturated water vs. supercritical CO<sub>2</sub>; brine vs. fresh water, etc.) should be selected to reflect expected well conditions.
- 2) Progression of the carbonation front through low permeability sealants based on Portland Cement appears to be dominated by diffusion rather than differential pressure, and this should be considered in any extrapolation calculations. On the other hand, fluid flow rates through a sample are controlled by the pressure gradient along the sample.
- 3) An indentation testing method based on the Rockwell testing method and cones was developed. Through calibration, this method provides a good indication of local unconfined compressive strength and Young's modulus values and can be used to map changes due to





exposure. However, about 5 mm distance between measurement points is needed to avoid interference, which limits spatial resolution.

WP2 performed batch experiments exposing sealant samples to  $CO_2$ -saturated water and wet supercritical  $CO_2$ , using clean  $CO_2$ ,  $CO_2$  with 1.6 mol% H<sub>2</sub>S, or  $CO_2$  equilibrated with concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Key findings regarding the impact of these impurities are:

- During exposure to CO<sub>2</sub>-saturated water, the presence of 1.6 mol% H<sub>2</sub>S in the CO<sub>2</sub>-phase only reduced carbonate precipitation and enhanced alteration depths by up to 1.5x. During exposure to wet supercritical CO<sub>2</sub>, the presence of 1.6 mol% H<sub>2</sub>S reduced carbonate precipitation and somewhat enhanced the alteration and degradation induced by CO<sub>2</sub>exposure.
- 2) During exposure to supercritical  $CO_2$  equilibrated with concentrated sulfuric acid, the presence of  $H_2SO_4$  resulted in reduced carbonate precipitation and enhanced alteration depths by up to 2x. The presence of  $H_2SO_4$  also resulted in (minor) degradation in the outer ~100-200  $\mu$ m of most samples.
- 3) In general, impurities had a more pronounced impact on sealants that were already more strongly affected by exposure to clean CO<sub>2</sub>, for both exposure to CO<sub>2</sub>-saturated water and to wet supercritical CO<sub>2</sub>.

The ability to withstand thermal shocks or cycling was investigated as part of WP3, considering both solid sealant samples, and the interface of compound sealant samples, where sealant was cast around a central stainless-steel tube. Here, the impact of confinement was also considered. Key findings are:

- Confinement is an important factor preventing thermal damage for solid sealant samples as well as for compound samples. Without confinement, solid sealant samples fracture when the thermal stress exceeds the tensile strength, and thermal stress creates leakage pathways along the micro-annulus around the central tube in a compound sample.
- 2) Thermo-mechanical damage can be related to basic thermomechanical properties. High tensile strength, high diffusivity, low Young's modulus, and a thermal expansion coefficient that is either low or similar to that or surrounding materials can help prevent thermally induced damage.
- 3) For compound samples under unconfined conditions, less damage at the sealant-steel interface may be expected when the thermal expansion coefficient of the sealant is closer to that of the steel.

WP6 developed a granite-based geopolymer sealant, tailored specifically for use in CCS wells. Key findings include:

- A one-part geopolymer formulation was developed, based on granite, Ground Granulated Blast-Furnace Slag (GGBFS), and potassium-silicate. The resulting sealant had a suitable slurry behaviour, good mechanical properties, low permeability, and a low Ca-content for enhanced ability to resist CO<sub>2</sub>.
- 2) The resulting sealant was tested under wide range of exposure conditions, including exposure to various brines and CO<sub>2</sub>-containing fluids. Macro- and micro-scale analyses of exposed samples demonstrated the strong chemical resilience of the geopolymer.





3) Hydraulic sealability testing indicated minor shrinkage of the geopolymer sealant after three months, impacting its seal quality when used as a plug. Future research will address the addition of an expanding agent to counteract shrinkage and improve sealability.

WP4 built a numerical model of the geopolymer sealant developed as part of WP6, and of the subsequent impact of exposure of this material to  $CO_2$ . Key findings are:

- 1) The GeoMicro3D simulation framework was extended to successfully model the reaction and microstructure development of a one-part granite-based geopolymer, such as sealant S5.
- 2) The resulting simulated geopolymer microstructural and compositional model was then used to model the impact of exposure of sealant S5 to CO<sub>2</sub> under in-situ down-well conditions.
- 3) The carbonation model was validated with experimental results and was able to successfully simulate carbonation depths by estimating the pH of the pore solution.

Finally, WP7 collated and compared all results from the above WP's, to synthesise project outcomes, and identify critical sealant properties that can help ensure long-term sealant integrity during CCS. In addition, WP7 discussed testing methods for exposing sealants to relevant deleterious conditions, and for measuring the identified critical properties. Key recommendations include:

- A list of critical properties was identified that should be measured before and after exposure to relevant conditions that could negatively impact a sealant material's integrity (such as exposure to CO<sub>2</sub> or thermal cycling), to identify changes in a sealant material, and ensure adequate properties are maintained.
- 2) When assessing the impact of exposure to deleterious effects (such as CO<sub>2</sub>-containing fluids or thermal cycling), in-situ conditions should be simulated as much as possible to ensure representative results.
- 3) When selecting measurement methods for sealant testing, optimal methods should be selected based on the availability and cost of measurements vs. the number of measurements to be performed, and the required accuracy under relevant in-situ conditions. Simpler measurements may suffice when (initially) comparing different materials, while more complex but more representative methodologies may be needed when acquiring input for predictive models.





#### 3. Role and contributions of each project partners

**IFE**: The project was coordinated by dr. Reinier van Noort at the IFE Reservoir Technology Department, who also acted as national coordinator for the Norwegian sub-project, and as leader for WP7. In addition, Reinier van Noort worked as one of two main investigators on WP2, which was led by dr. Gaute Svenningsen of the IFE Corrosion Technology Department, and Reinier was co-supervisor for the PhD-student at UiS (WP6). In WP2, Gaute designed and performed all exposure tests, while Reinier coordinated sample analyses and interpretation of the results. As part of WP7, Reinier organized four *Ceminars*, and he coordinated one webinar for SPE (with support from Prof. Mahmoud Khalifeh), and all non-academic dissemination (supported by Dr. Anne Pluymakers and Dr. Benny Suryanto). In WP6, Reinier co-supervised PhD-student Seyed Hasan Hajiabadi, in particular regarding sample analyses and interpretation of results. In addition, Reinier supported sample analysis and publication in WP1, and coordinated internal knowledge exchange where needed.

**Halliburton**: Gunnar Lende led WP1, which included exposure experiments on all five sealants, along with a wide range of analyses on both exposed and unexposed samples. Halliburton also prepared all samples required for testing in WP's 2, 3, 5, and 6, and coordinated the distribution of these samples, ensuring that all partners had the samples needed, and had samples that were prepared and cured identically. As part of W 1, Gunnar and his team developed the use of indentations to assess changes in sealant mechanical properties caused by exposure to CO2. Finally, Gunnar Lende supported the work done at the other partners with his deep experience in wellbore sealant development and use, in particular the work done in WP7.

**ReStone**: ReStone AS, through dr. Astri Kvassnes, collaborated with Halliburton to develop a new sealant design based on high-temperature blends using Portland Cement and the RePlug<sup>®</sup> material. This design was then tested by other WP's as part of the project. Furthermore, Astri collaborated directly with both Halliburton and Heriot-Watt in testing and assessing their materials and visited IFE for more detailed SEM analyses of the samples with RePlug<sup>®</sup> together with Reinier. Astri Kvassnes also supported WP7, by providing thoughtful input and reviewing the deliverables. They also provided insights based on their experience with innovation and developing a product from idea to commercially available new product.

**UiS**: Prof. Mahmoud Khalifeh, of UiS, led WP6, and was the main supervisor for the project's PhDstudent, Seyed Hasan Hajiabadi. Together, they developed a new geopolymer, based mostly on granite powders, and provided materials to Halliburton for sample preparation and testing by WP's 1, 2, 3 and 5. In addition, the UiS team also carried out a range of tests and analyses on their own material, and collaborated closely prof. Guang Ye's group at TU Delft, providing input needed for WP4. As part of this, PhD-students from UiS visited TU Delft, and carried out work there. Finally, Mahmoud supported the overall project using his network to help disseminate our work, in particular to industry.

**TU Delft**: Dr. Anne Pluymakers at the TU Delft Geoscience & Engineering Department was the national coordinator for the Dutch sub-project, and leader for WP3. In WP3, Anne and post-doc dr. Kai Li performed experiments exposing various sealant samples to thermal shocks or cycles, under different confinement conditions, and using different sample geometries. In addition, Anne and Kai performed CT analyses on samples from WP2. Anne also coordinated the collaboration on public outreach with ACT projects RETURN and SHARP, with articles in the Netherlands and Norway.

Prof. Guang Ye at the TU Delft Department of Materials, Mechanics, Management & Design led WP4, in which post-docs dr. Xiujiao Qiu and dr. Mayank Gupta built a numerical model for the hardening and subsequent CO2-exposure of the rock-based geopolymer sealant designed by UiS. In addition, Guang substituted for Anne as national coordinator when Anne was on parental leave (Aug-Dec 2022 and May – Sept 2024). Guang and Mayank supported work done in other WP's, including WP2 and WP7, through their deep knowledge of Portland Cement and other binders.





**EBN BV**: As a key partner in the Dutch large-scale CCS projects PORTHOS and ARAMIS, EBN BV was an important part of the project steering committee. Early in the project, EBN provided input regarding the conditions of the planned CO2-injection in PORTHOS, so that these conditions could be used to set experimental and numerical parameters, ensuring the applicability of project results. EBN also recommended the inclusion of a reference sealant, both to compare obtained results to and to represent cements used in legacy wells. Later in the project, EBN participated in WP7, ensuring the quality of the deliverables of this WP.

**Heriot-Watt University**: At Heriot-Watt University, Dr. Benny Suryanto was the national coordinator of the UK sub-project, and leader of WP5. In this WP, Benny performed bond strength tests using their patented method on all five sealants and analysed the results. He collaborated with post-doc dr. Gerry Starrs, who performed electrical resistivity and impedance spectroscopy measurements on curing and cured samples, to study how these properties change during curing, and whether electrical measurements can be used to assess interface quality. In addition, Benny collaborated with TU Delft, and supported them in their work on WP3, in particular with developing a push-out test measuring the shear-stress needed to push a stainless steel tube out of a sealant ring.

**Harbour Energy**: Harbour Energy (as Wintershall DEA) provided financial support to the Norwegian sub-project, topping up the Norwegian budget. In addition, Harbour Energy supported dissemination to industry, and ensured relevance of the work done.

The regular collaboration between the CEMENTEGRITY partners is reflected, for example, in coauthorships on many of the academic papers, conference presentations, and other outreach publications produced by the CEMENTEGRITY project.





## 4. <u>Short description of activities and final results</u>

## **Project overview**

CEMENTEGRITY performed experimental research, as well as numerical modelling, into which sealant properties can help ensure long-term seal integrity during CO<sub>2</sub>-injection and -storage, taking into account the chemical, mechanical, and thermal mechanisms that can negatively impact wellbore seals. To carry out this research, five different sealant compositions were selected, representing sealants based on different binder technologies, and sealants currently in use as well as compositions being developed as part of the project (see Table 1).

Sealant	Description
S1	Reference cement, consisting of Class G cement plus 35% BWOC silica flour.
S2	Low permeability composition based on Class G cement plus 35% BWOC silica flour, adding silica fume and MgO.
S3	Design based on S2, replacing 28.5% of the binder with RePlug <sup>®</sup> (olivine-based CO <sub>2</sub> -sequestering agent).
S4	Sealant composition based on Calcium Aluminate Cement (CAC).
S5	Geopolymer based on powdered granite with GGBFS and micro-silica.

The ability to form and maintain a seal without negatively affecting the surrounding materials is the most important property of a sealant in any application. This was directly addressed in WP5, where the integrity of the interface between a sealant and a steel tube inside of which this sealant was cured was measured as the force needed to push the cement plug out of this steel tube. WP5 also explored the use of electrical impedance spectroscopy methods as an alternative method for assessing and monitoring interface quality and integrity.

Common wellbore sealants based on Portland Cement (PC) may have limited chemical resistance against  $CO_2$ -containing (hydrous) fluids. The effects of chemical interactions when a sealant is exposed to  $CO_2$ -saturated water or (wet) supercritical  $CO_2$  were studied in WP1, while WP2 investigated how key impurities in the  $CO_2$ -stream (H<sub>2</sub>S and H<sub>2</sub>SO<sub>4</sub>) can alter this impact.

When cold  $CO_2$  is injected into a hot reservoir, or into a depleted hydrocarbon reservoir with low initial pressure, this can cause significant temperature and pressure changes in the injection area. If the injection is intermittent, these temperature changes may also be cyclic. The impact of such thermal changes on the integrity of the sealant materials themselves, as well as on the integrity of the interface between a steel tube and a surrounding cement ring were addressed in WP3.

In addition, considering the limitations and environmental footprint of PC, alternative materials, either as additions to a sealant design based on PC, or as full replacements of PC, were also considered as part of CEMENTEGRITY. As part of WP1, we developed a sealant design with a higher RePlug<sup>®</sup> content replacing PC with a CO<sub>2</sub>-binding agent. In WP6, we developed a geopolymer cement based mostly on powdered granite obtained as waste from mining operations. To support this development, WP4 built a numerical model simulating the hardening of this geopolymer material, as well as the reactions taking place when it is subsequently exposed to CO<sub>2</sub>.

Finally, the key results from all above WP's were analysed together to identify three key abilities for a successful sealant in CCS, as well as the material properties behind those abilities, that need to be considered when developing a new sealant, or selecting and tuning a sealant for a specific application.





## Deviations

The project deviated from the original plans in the following ways:

- WP1 added work on method development for using indentations to assess the progression of sealant carbonation, and the impact of carbonation on mechanical properties;
- WP1 carried out a larger number of exposure tests than originally planned, to provide a larger body of representative data;
- The exposure tests carried out in WP2 had a shorter duration than originally planned, to compensate for delays in constructing the required laboratory facilities;
- While WP3 originally planned to deliver two academic papers, due to the volume of results produced, this was split into three papers instead;
- WP3 did not include S5 in their experiments on solid samples (i.e., without central stainlesssteel tube), as samples of S5 were not available early in the project. The impact of thermal cycling on unconfined solid samples of S5, with and without brine, was addressed as part of WP6 (in collaboration with WP3).
- WP4 incurred delays due to difficulties in recruiting post-doc candidates, and because the first post-doc left when offered an industry job. Furthermore, additional experiments were needed that were not planned for to obtain accurate reaction kinetics. As a result, the planned model simulating the impact of thermal shocks/cycling was not completed.
- WP5 experienced initial delays due to the need to re-design test procedures to accommodate curing at elevated temperatures and pressure. This included altering the steel casing material to facilitate machining and potential corrosion. Interestingly, these changes led to extensive corrosion of the steel casing material in contact with the reference sealant, highlighting a possible long-term issue. Repeat tests were necessary in certain sealants due to their quick setting characteristics, indicating the need to engineer the setting to enhance bonding performance during temperature increases in cementing.
- While geopolymers are typically prepared by mixing solid precursors with a liquid activator consisting of caustic solutions of sodium/potassium silicate and/or hydroxide, these activators are a logistical challenge to their implementation. Therefore, WP6 reformulated their geopolymer to a "one-part" or "just add water" type, where the activator is instead added to the solid mixture, and then water is added on site to start the material. As this is more similar to how regular Portland Cement is mixed, a geopolymer binder formulated in this manner is more marketable.
- Completion of WP7 was delayed to the first quarter of 2025. However, while originally only two reports were planned as deliverables, an additional conference paper was published at the GHGT conference in October 2024. Furthermore, the final deliverable (D7.2), which was completed in 2025-Q1, had an expanded scope compared to the originally planned report, with a more thorough review and comparison of results from different WP's.





## WP1: Effects of dissolved and supercritical CO2

WP1 had two main scope categories – firstly mix and cure samples to be tested by IFE, TUD and HWU as part of the other WP's; secondly perform testing on the designs selected. As the other parties in their WP's did not require samples at the same time and further did not have the moulds required ready, these two activities were run in parallel through 2022 and 2023. Hence the curing schedule was re-organized frequently to obtain the necessary progress with the final project deadline in mind.

S1 through S4 were mixed as per API-10B-2 / ISO-10426-2 and Halliburton internal procedures. The S5 mixing procedure and materials were provided by UiS at a later stage. After mixing, entrained air was evacuated as much as possible by gentle stirring, and the moulds were filled as soon as possible afterwards.

Curing for WP1 was done in cylinder shaped split ring moulds made from AISI 316 stainless steel, located in large size autoclaves for the entire period plus controlled ramp down of temperature and pressure. The curing medium was fresh water. Curing conditions were chosen to 150°C and 300 bar (4350 psi) for 28 days, using fresh water (Norwegian tap water) as the pressure medium. This long curing time at high temperature provides documentation for high temperature stability and accelerates the curing process to near 100% completion. The latter is to ensure that ageing and storage between the curing and testing can be considered insignificant and therefore not an error source. Hindsight it may appear as S4 and S5 were still not 100% cured, something that might cause slight changes in the reference values versus the pre-exposure values of some material properties (such as mechanical strength and permeability). After curing, the samples were removed from the moulds and stored under water. In parallel with exposure testing, reference samples were cured in an autoclave in fresh water at 80°C and 69 bar for 90 or 180 days.

IFE required about Ø12 x H30 mm samples for their testing. So, it was decided to cure samples in 500 mm long pipes and cut to length afterwards. The pipes were sourced, and moulds prepared in the Halliburton EESSA laboratory on behalf of IFE. HWU manufactured the moulds themselves, and their personnel came to the Halliburton EESSA lab to participate in the moulding. Some of the moulds had integrated contact pins to measure the cement resistivity from fluid to solid phase. This required a slight modification of the autoclave to facilitate cable feed-through of 8 conductors which were routed to a data acquisition box that HWU provided. The EESSA lab measured and logged data for the 4-week curing period. The moulds and samples were then sent to HWU for further work.

## Test program

The general test program applied in WP1 consisted of pre-exposure tests, exposure and parallel reference sample curing, post exposure and reference tests.

Pre-exposure testing:

- 1. Mechanical properties at end of curing (reference).
  - For some designs uni-axial compressive load test, for some tri-axial compressive load tests.
    - a. Unconfined compressive strength.
    - a. Young's Modulus.
      - i. Strain interval used for the YM was 20 50 or 20 40 % of axial strain to failure (linear range).
    - b. (S3: Triaxial confined compressive strength and Young's Modulus, friction angle, cohesive strength).
    - c. Poisson's ratio.
    - d. Brazilian Tensile Strength.
- 2. Water permeability after curing (reference).
- 3. Rockwell A based indentation tests as required after curing (reference).





## Exposure

This was carried out in two different setups: a) Twin setup that uses two independent laboratory pumps to control pressure and rate for each channel. One channel flowed  $CO_2$ -saturated fresh water; the second flowed water-wetted supercritical  $CO_2$ . Duration was 6 months. b) a 6-channel setup where 2 groups of 3 samples each were flowed with  $CO_2$ -saturated fresh water. Durations were 3 and 6 months. All samples were confined in Hassler type core holders ensuring flow through the sample. In addition, 90 day batch exposure tests were carried out on cylinders of all sealants at 80 °C and 48 bar (700 psi).

#### Post exposure testing

As only a limited number of cylinders was available after exposure, the following tests were selected. From the twin cell:

- a) 1 cylinder exposed to scCO<sub>2</sub> flow for 180 days: water permeability and indentation mapping.
- b) 1 cylinder exposed to CO<sub>2</sub>satH<sub>2</sub>O flow for 180 days: water permeability and indentation mapping.

From the 3x2 cell:

- c) 3 cylinders exposed to  $CO_2satH_2O$  flow for 90 days.
  - o 1: indentation mapping
  - 1: Brazilian Tensile Strength (cut into four discs, giving four measurements)
  - 1: uniaxial compressional load
  - (Young's Modulus (YM), Poisson's Ratio (PR), Unconfined Compressive Strength (UCS)
- d) 3 cylinders exposed to  $CO_2 satH_2O$  flow for 180 days.
  - o 1
  - : indentation mapping
  - 1: Brazilian Tensile Strength (cut into four discs, giving four measurements)
  - o 1: uniaxial compressional load

(Young's Modulus (YM), Poisson's Ratio (PR), Unconfined Compressive Strength (UCS)

- e) Optional Phenolphthalein pH image of cross-section after CO<sub>2</sub>-exposure (indicator of Ca(OH)2 presence).
- f) 3 reference samples for each composition (kept under water at 80 °C and 69 bar without CO<sub>2</sub> for 0, 90 and 180 days)

For each sealant pre-, post- and reference-samples were tested. The flow tests in the twin-cell setup provided comparative data between  $scCO_2$  and  $CO_2satH_2O$ , both for flow potential and for progression of  $CO_2$  impacted (both carbonated and bi-carbonated/detrimentally damaged) front. This setup was also used for pre- and post- exposure water permeability. The flow tests in the 2 x 3 cell setup provided samples for mapping of progression of  $CO_2$  impacted front with  $CO_2satH_2O$  after 3 and 6 months, plus samples for testing of mechanical properties.

Mapping of the  $CO_2$  impacted front progression was obtained by visual observation with or without phenolphthalein as indicator for S1 – S3, plus indentation testing for all sealants. An indentation test protocol was developed during this project, where S1 was used to find the most rational test matrix, and this was implemented for S2 – S5. An alternative test protocol was also developed for use when the samples are radially exposed. This was used on one additional sample set that had been exposed to  $CO_2$ satH<sub>2</sub>O for 3 months in a traditional autoclave setup.

Severable comparison systems were applied in an attempt to normalize the data to compare the sealants regardless of their different properties. Change factors were applied for mechanical properties, permeability and carbonation progression.





## Development of a higher RePlug<sup>®</sup> content blend (S3)

The S3 blend was originally designed to maximise the protective effects of olivine in the blend by increasing the amount of RePlug<sup>®</sup> incorporated over previous compositions (from 19% to 28.5 wt. % of the dry binder mixture), while approaching the theoretical minimum amount of Portland Cement binder. After curing, the blend showed an acceptable permeability (0.18-0.19 x 10-18 m<sup>2</sup>). However, as expected, the blend had lower, though still acceptable, UCS (34-35 MPa) compared to blends with significantly higher PC content. The Young's modulus was also relatively low (9.2-9.9 GPa). S3 showed good sealability. S3 also showed very good resistance against thermal shock, even without confinement, most likely due to its low elastic modulus and high thermal diffusivity (0.80 mm<sup>2</sup>/s). However, compared to other PC-based sealants the blend was affected to greater depth by flow-through exposure with CO<sub>2</sub>. In batch exposure tests, S3 was impacted more strongly than S2, but typically less than S1. Considering microstructural observations and measured UCS, the clinker content of the blend was likely somewhat low, and the silica content rather high. Therefore, the blend would likely benefit from increasing the clinker content, mostly by lowering the silica flour (e.g. quartz) content rather than the olivine content, as olivine also provides a source of silica, and it is evident that the finer grains of RePlug<sup>®</sup> dissolved in the matrix.

#### Summary of observations

The most significant observations from the indentation testing are that:

- S1 and S2 are the only sealants that remained partly unaffected after exposure, by the definition of no change in indentation compared to the reference samples. S4 is the only sealant that has no detrimental damage at all. S3 was impacted the deepest, and also showed deepest detrimental damage depth.
- There seems to be a correlation between indentation depth, Young's Modulus, and unconfined compressive strength. However, the current dataset is too small to provide a substantial confidence level.
- The 90 days batch exposure tests showed that none of the sealants had any change in hardness radially (from depth 7 mm inwards) in the axial midpoint of the cylinder. All sealants had hardening at the top level, this was least significant for S1 and S5, while S2, S3 and S4 showed about the same change.
- The permeability data can be summarized: S1 and S2 clearly have the lowest permeability values and also show the largest reduction ratios due to exposure. S2 shows a substantial reduction from initial to 180 days reference. S3 shows no particular improvement. S4 has comparably high initial values, still well within D010 specifications, but also shows a significant reduction. S5 shows a significant reduction from initial to 180 days reference, with no improvement post exposure.
- In future studies developing blends containing RePlug<sup>®</sup>, we should pursue an intermediate blend to achieve better all-round results.

#### Publications

Lende, G., E. Sørensen, S. Jandhyala, R. van Noort (2024) State of the art Test Method to Quantify Progression Rate of Carbonation of Wellbore Sealing Materials. SPE Europe Energy Conference and Exhibition 2024, 28/06-29/06, Turin (I).





# WP2: Impact of CO<sub>2</sub> with impurities on integrity of wellbore sealants during CCS

While the impact of exposure to combinations of  $CO_2$  and water (or brine) on wellbore sealant integrity has been the subject of considerable research reported in the literature, the potential additional impact of impurities in  $CO_2$  on wellbore seal integrity has only seen minor investigation. Therefore, the impact of impurities in  $CO_2$  on the integrity of the five sealants in CEMENTEGRITY was investigated in WP2. We performed batch exposure tests, exposing sealant cylinders to  $CO_2$ -saturated water and wet supercritical  $CO_2$  at 80 °C and 10 MPa for up to 16 weeks. Exposures were performed using clean  $CO_2$ ,  $CO_2$  with 1.6 mol% H<sub>2</sub>S, and  $CO_2$  equilibrated with concentrated H<sub>2</sub>SO<sub>4</sub>. After exposure, changes in the sealant composition and microstructure were investigated using Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS), as well as Computed Tomography scanning (CT-scanning).

In order to perform these exposure experiments, a new apparatus was constructed (Figure 1) consisting of five parallel pressure vessels built of titanium, to enable experimentation with corrosive (and hazardous) chemicals such as  $H_2S$ . Sample holders were designed such that samples could be placed on two levels within these vessels, to enable simultaneous exposure to  $CO_2$ -saturated water (near the bottom of the vessel) and wet supercritical  $CO_2$  (near the top of the vessel). For exposure to clean  $CO_2$  or  $CO_2$  with  $H_2S$ , samples were placed on both levels, the vessel was then partially filled with water, and pressurised with  $CO_2$ . For exposure to  $CO_2$  with  $H_2SO_4$ , samples were only placed on the top shelf, while small vials containing saturated  $H_2SO_4$  were placed on the bottom shelf, and the vessel was pressurized with  $CO_2$  without adding additional water.



Figure 1. Apparatus constructed for WP2 exposure tests, with five parallel exposure vessels.





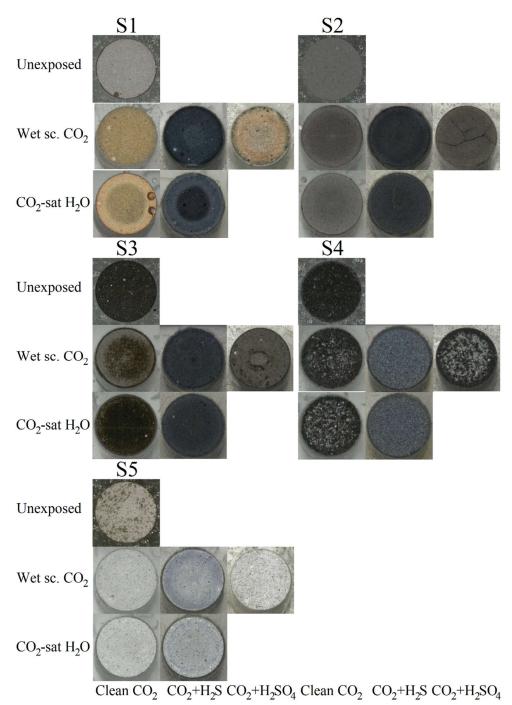


Figure 2. Optical scans of samples exposed for 16 weeks to wet supercritical CO2 or CO2-saturated water, using clean CO<sub>2</sub>, CO<sub>2</sub> with H<sub>2</sub>S or CO<sub>2</sub> with H<sub>2</sub>SO<sub>4</sub>.

Figure 2 shows optical scans of samples of all five sealants exposed for 16 weeks. A thorough analysis of the changes experienced by the exposed samples has been published or submitted for publication in two academic publications: Van Noort et al (2025) presents the results of exposure to clean  $CO_2$ ; Van Noort et al (submitted) presents the results of exposure to  $CO_2$  with  $H_2SO_4$ .





## Key findings

- All samples were fully penetrated by CO<sub>2</sub> within four weeks, resulting in carbonation of all free Ca(OH)<sub>2</sub> where relevant (i.e., for sealants S1-S3 which were based on PC). Further impacts on microstructure and composition were observed at sample surfaces, and varied with exposure duration and conditions, and sealant composition.
- Alteration fronts, representing changes in composition and microstructure beyond direct carbonation of free Ca(OH)<sub>2</sub> did not depend strongly on sealant permeability. In general, for PC-based sealants, alteration depths were more limited after exposure to wet supercritical CO<sub>2</sub> than after exposure to CO<sub>2</sub>-saturated water.
- Degradation of sealant integrity was observed mostly after exposure to CO<sub>2</sub>-saturated water, but was absent, or much more limited after exposure to (wet) supercritical CO<sub>2</sub>. Comparison of S1 and S2 suggests that reducing sealant permeability resulted in reduced degradation depth and intensity.
- Tailoring sealant composition to change how exposure to CO<sub>2</sub> impacts sealants can help improve long-term sealant integrity, by either adding additives that can result in more stable carbonates (such as olivine S3); developing a sealant in which Ca is not an integral part of the main gel structure (S5); or using a sealant that is inert when exposed to CO<sub>2</sub> and water (S4).
- When sealants were exposed to CO<sub>2</sub>-saturated water, the presence of H<sub>2</sub>S enhanced alteration depths (by up to a factor 1.5), and resulted in reduced carbonate precipitation (especially in S1).
- When sealants were exposed to wet supercritical CO<sub>2</sub>, the presence of H<sub>2</sub>S resulted in reduced carbonate precipitation, and, for sealants S1 and S5, increased alteration depths.
- For sealants exposed to supercritical CO<sub>2</sub>, the presence of  $H_2SO_4$  resulted in up to 2x deeper, but more diffuse alteration, as well as minor degradation at the sample surface, to depths of about 100-200  $\mu$ m.
- The above impacts of impurities, in general, were less significant for sealants that were also less reactive when exposed to clean CO<sub>2</sub>.
- For the sealants and impurities considered here, the impact of impurities was very limited, and these should not be expected to significantly affect integrity of an intact seal. However, when CO<sub>2</sub> flows along a leakage pathway, these impurities may affect how the leakage pathway develops, potentially exacerbating the leakage, and/or inhibiting carbonate precipitation that could otherwise lead to self-sealing of the leakage pathway.

## Publications

Van Noort, R., G. Svenningsen (2024) Impact of CO<sub>2</sub> with impurities on integrity of wellbore cements during CCS. EGU 2024, 14/04-19/04, Vienna (A).

Van Noort, R., G. Svenningsen, K. Li, A. Pluymakers (2025) Exposure of five cementitious sealant materials to wet supercritical CO<sub>2</sub> and CO<sub>2</sub>-saturated water under simulated downhole conditions. International Journal of Greenhouse Gas Control 144, doi: 10.1016/j.ijggc.2025.104380

Van Noort, R., G. Svenningsen, K. Li, (submitted) Experimental study on the impact of H<sub>2</sub>S and H<sub>2</sub>SO<sub>4</sub> in CO<sub>2</sub> on five different sealant compositions under conditions relevant for geological CO<sub>2</sub>-storage. *Submitted to Geoenergy Science and Engineering*.





## WP3: Thermomechanical behaviour of wellbore sealants

During CCS, periodic injection of cold CO<sub>2</sub> (seafloor temperatures) into warm subsurface reservoirs results in thermal stress that may lead to the formation of leakage pathways. WP3 had the objective to expose sealant specimens to thermal shocks and cycling, to observe thermally-induced cracking of the intact material, as well as the potential for leakage along the annular contacts between sealant and wellbore steel. We answered the question *"How do thermal shocks/cycles impact the integrity of the sealant material itself, and of the interface between a sealant sheath around a stainless-steel pipe; and what is the effect of confinement in suppressing this"* through the development of a novel experimental program to study the five CEMENTEGRITY sealants. In addition, we measured thermomechanical properties, and determined which are key.

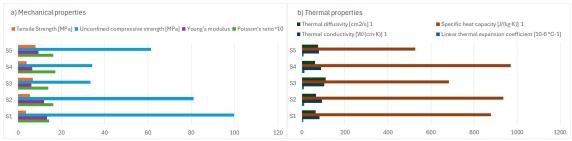


Figure 3. a) mechanical properties of the dried sealants; b) thermal properties of the dried sealants.

## Methodology

Halliburton (WP1) prepared two sample types: solid cylinders and composite cylinders, both 30 mm diameter and 70 mm height. In the composite cylinders a stainless-steel pipe (AISI 316L) was cured in the centre of the samples, with outer diameter 6 mm and wall thickness of 1 mm. We stored all samples as received at room temperature (~20°C) in the water they were sent in until usage. In half of the solid samples, we drilled a central borehole of 4 mm diameter. Before the thermo-mechanical experiments, samples were dried for 48 hours in an oven. Where applicable, the samples to be tested were measured with Computed X-ray Microtomography (~30 $\mu$ m/voxel resolution) pre-test. We measured the mechanical and thermal properties plus the porosity (He-Pycnometry) of representative dried and intact sealant samples for sealant S1-S5 to establish a baseline for comparison. Pre-thermal shock characterization indicated that the five sealant compositions represented a diverse array of initial mechanical properties (Figure 3a) and thermal properties (Figure 3b).

Four types of experimental protocols were executed on all five CEMENTEGRITY compositions:

- Type A: unconfined tests on intact cylinders, without and with a central borehole;
  - $\circ$  Type A1: intact, oven-heated samples quenched in a cold-water bath,
  - Type A2: cold flow-through through a central borehole for samples inside an oven.
- Type B: confined tests on intact cylinders, without and with a central borehole;
- Type C: unconfined tests on composite samples;
- Type D: confined tests on composite samples.

The four protocols were aligned as much as possible, and followed the generic format shown in Figure 4. All samples were tested mechanically pre- and post-shock, to measure either UCS (Type A, B, intact samples) or push-out stress (Type C, D). Sealant S5 was only tested in Type C and D experiments as samples were not available when Type A and B were carried out. The generic procedure was to heat the sample to 120°C (Type A, B) or 60°C (Type C, D), and shock with 5 or 20°C cold water in either 8 or 16 cycles (see Figure 4). Post-shock, where possible a microstructural measure was used to determine the effectiveness of cracking and/or porosity creation. For all sealant types one or several samples were imaged with micro-tomography, and for the composite samples pre- and post-shock flow-through tests of the interface were performed. Type C experiments were performed in a custom designed jig placed in an oven, while a triaxial apparatus was especially adapted and fitted with a new, custom-designed piston set for Type D experiments.





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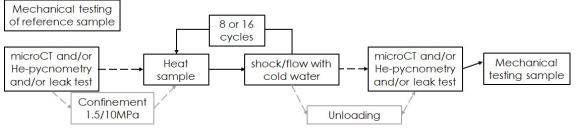


Figure 4. Format of the test protocol for Type A-D.

#### Results

**Type A)** Only S3 was undamaged after the thermal shock procedure. Both quenching (A1) and flowthrough (A2) induced additional cracks and voids in sealants S1, S2, and S4. In S4 samples the cracks connected to form a potential leakage pathway in this cm-scale sample. The visible mechanical damage in S1, S2 and S4 coincided with a decrease in UCS post-thermal shock procedure, as well as increased porosity (based on He-pycnometry). Quenching led to a 2x larger UCS reduction than flow-through.

**Type B)** Even under relatively low confinement (1.5 MPa), thermal shocking did not lead to any microstructural changes. The post-shock UCS increased slightly for all compositions, attributed to a slight decrease in porosity (which was confirmed by He-pycnometry). Control samples which were confined without thermal shock procedure, showed similar property changes.

The bulk thermal stress in type A and B experiments is calculated as Thermal stress = -E \*  $\gamma$  \*  $\Delta$ T, where E is the Youngs Modulus,  $\gamma$  the thermal diffusivity and  $\Delta$ T the imposed temperature difference. Comparing the generated thermal stress with the tensile strength shows that only for S3 the generated thermal stress remained significantly below the tensile strength in both confined and unconfined experiments (Figure 5a). (See also Li & Pluymakers, 2024; Li et al, submitted1.)

**Type C)** We applied 3 bar of  $N_2$  to the sealant-steel interface, to check for the existence of a microannulus , and to have a quantitative indication of leaking vs sealing. CT-scanning was used to assess sealant integrity. Any mechanical damage within the first 5-10 voxels next to the pipe is invisible due to the high density of steel. None of the samples exhibited visible cracks after thermal shock. Penetration rates indicate S3 remained unaffected by the thermal treatment, whereas all other sealants are affected negatively (Figure 5b). S1 and S5 experienced major damage; S2 and S4 minor damage. A comparison between thermal and mechanical properties indicates that if the thermal expansion coefficient of the sealant is closer to that of steel, less debonding is induced due to thermal cycling. As in all cases the generated thermal stress is lower than the tensile strength, there are no cracks in the sealant. (See also Li et al, submitted2.)

**Type D)** For all sealants, penetration rates after thermal cycling under confinement are equal or less than before. As the samples were fully cured, this must be related to closure of the gap due to viscous/plastic processes. Indeed, reference samples that were confined for the same duration as the shocked samples showed similar decreases in penetration rates. This indicates that improved sealing due to confining pressure alone has a bigger impact than the thermal shock procedure.

#### Limitations

All procedures are executed first unconfined to represent a worst-case scenario. This also allows to determine if using confining pressure is worth the extra time needed to perform the tests. In-situ confinement pressures up to 10's of MPa can be expected, but since 10 MPa led to a full negation of unconfined thermomechanical effects, also 1.5 MPa was used. In each test type, we applied temperature cycles with instantaneous temperature drops up to 100°°C, and with intervals between cycles just enough to recover the original temperature (12 min). This temperature difference may occur in a scenario with seafloor transport and subsurface injection, but the short time interval between cycles is atypical. Last, the number of cycles used in all procedures was limited, with a maximum of 16 cycles. Mechanics dictates that with more cycles more damage will accumulate (e.g.





Miner, Cumulative Damage in Fatigue, 1945; Fatemi and Yang, 1998; International Journal of Fatigue 20), and any potential for subcritical damage accumulating might not be observed in these tests.

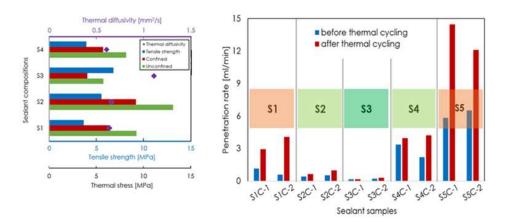


Figure 5. a) Linking thermomechanical properties for the solid sealant samples; b) N2-penetration rates for unconfined samples before and after thermal cycling. Colour coding indicates the impact of thermal treatment.

#### Conclusions

- Without confinement, if the thermal stress exceeds the tensile strength, samples fracture. Confinement reduces potential for thermally-induced cracks.
- Without confinement, S3 alone is unaffected by thermal cycling. S1 and S5 experience most damage and S2 and S4 experience minor damage. When the thermal expansion coefficient of the sealant is closer to that of the steel, less damage should be expected.
- With confinement, the cement sheath is contracted closer to the steel range as steel during cooling phases. This decreases potential of debonding at interfaces.
- Thermo-mechanical damage can be related to basic thermomechanical properties. High tensile strength, high diffusivity, low Young's modulus, low expansion coefficient led to better sealants. For the cement/steel interface, an expansion coefficient close to that of steel leads to better sealing at the interface.

#### Publications

Li, K. & A. Pluymakers (2024) Effects of Thermal Shocks on Integrity of Existing and Newly-Designed Sealants for CCS Applications. International Journal of Greenhouse Gas Control, doi: 10.1016/j.ijggc.2024.104103.

Li, K., A. Pluymakers, R. van Noort (submitted1) Effects of Confined Thermal Cycling on Sealants with Different Thermomechanical Properties for CCS. *Submitted to Scientific Reports*.

Li, K., M. Friebel, A. Pluymakers (submitted2) Thermo-mechanical Behaviour of the Sealant-steel Interface under Thermal Cycling for CCS. *Submitted to: Geomechanics for Energy and the Environment*.





# WP4: Numerical simulation of reaction and microstructure development of novel sealant for carbon capture and storage

WP4 focused on the development of sealant S5, a one-part granite-based geopolymer created in WP6. The key objective of WP4 was to establish three numerical simulation frameworks. The first framework models the reaction and microstructure development of sealant S5, while the second simulates the changes in microstructure due to  $CO_2$ -exposure under Carbon Capture and Storage (CCS) well conditions, including carbonation depth. A third framework was to address the impact of thermal cycling on microstructural integrity, but this was not completed due to time constraints.

## GeoMicro3D Framework for Microstructure Development (Gupta et al, 2025)

WP4 extended the GeoMicro3D simulation framework to model the reaction and microstructure development of one-part granite-based geopolymers like sealant S5. The GeoMicro3D model incorporates four main components: 1) initial particle packing, 2) particle dissolution, 3) ion transport, and 4) nucleation and growth of reaction products. Sealant S5 is composed of a variety of precursors and activators – slag, granite, microsilica,  $K_2SiO_3$ , and KOH – each with distinct particle size distributions. The model begins by accounting for the particle size distributions and shapes of the solids, preparing the initial simulation domain with the Anm material model.<sup>1</sup>

To better understand the dissolution kinetics of these solids, dissolution experiments were conducted. The dissolution of slag, granite, microsilica, and  $K_2SiO_3$  was examined by dissolving 0.1 g of each solid in 100 ml of alkaline solution with varying concentrations of potassium hydroxide (0.1, 0.5, 1.0, and 2.0 mol/l). The pH values for these solutions ranged from 12.89 to 14.22. Samples were taken at multiple time intervals (5, 15, 30, 60, 120, 240, and 1220 minutes) and analyzed using ICP-OES to determine the concentrations of Si, Al, and Ca.

The dissolution of Si from slag was found to be correlated with the pH of the solution and the NBO/T ratio (non-bridging oxygen to tetragonal oxygen ratio). An equation was developed to estimate the dissolution rate of Si ions from slag as a function of pH and NBO/T ratio:

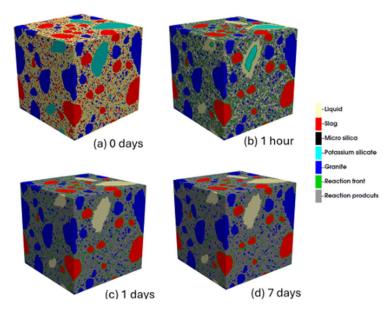
$$Log_{(r+,si)} = -0.1934pH * \frac{NBO}{T} + 0.5981pH + 6.4288 * \frac{NBO}{T} - 23.381$$

The dissolution of Al from the slag is taken to be stoichiometric to Si in the slag. The dissolution of Ca from the slag was also estimated and linked to the pH of the solution. Ca-dissolution was found to take place in two different stages. Initial non steady state Ca-dissolution lasted for about 30 minutes and was followed by steady state dissolution. The dissolution of other ions such as Na, K, Mg were linked to the dissolution of Ca. Similarly, the dissolution of K and Si from K<sub>2</sub>SiO<sub>3</sub>, Si from microsilica were also estimated from the dissolution experiments, giving an overall dissolution rates of different solids present in the mix. Further GeoMicro3D models the transport of different ions in the microstructure of the paste using the Lattice Boltzmann method – Multi relaxation time (LBM-MRT). The diffusion coefficient at different nodes is estimated based on the amount of solid volume fraction at that node.

<sup>&</sup>lt;sup>1</sup> Zuo, Ye (2021) Lattice Boltzmann simulation of the dissolution of slag in alkaline solution using real-shape particles, Cement and Concrete Research 140.







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following the ion transport, the nucleation of reaction products are modelled using classical nucleation theory. Nucleation of a product at any location starts to occur when the nuclei of a reaction product reaches a size due critical to supersaturation of the ions in the solution. The precipitation of reaction products was modelled using thermodynamic modelling. After combining the four component of the modelling the reaction degree, amount and type of reaction products, ion concentration in the pore solution and the microstructure of the sealant was predicted.

Figure 6: Simulated microstructure of the one-part granite-based geopolymer after 0 days, 1 h, 1 day, and 7 days of curing

## Carbonation Model for CO<sub>2</sub> Exposure (Gupta et al, in preparation)

The information obtained from the microstructure and reaction products was subsequently used to model the reaction and deterioration of sealant S5 under  $CO_2$ -exposure. This carbonation model consists of four modules: 1)  $CO_2$  dissolution, 2) gel dissolution, 3) ion transport, and 4) product precipitation.

Dissolution of  $CO_2$  in the pore solution was modelled using thermodynamic principles. Ion transport, including  $CO_2$  and other species, was modelled using LBM-MRT, leading to a reduction in the solution's pH. At lower pH, reaction products such as C-(N,K)-A-S-H, zeolites, and hydrotalcite begin to dissolve. Gel dissolution was modelled based on the transition state theory, where the dissolution rate of the gel is linked to the degree of saturation of the products. The presence of carbonates in the pore solution leads to the precipitation of calcium and magnesium carbonates, and the formation of silica gels or low-calcium gels in the microstructure. The precipitation of these products was also modelled thermodynamically.

To validate the carbonation model, cylindrical specimens (12 mm diameter) were exposed to wet supercritical  $CO_2$  at elevated temperature and pressure for 3 hours and 16 hours. The carbonation depths were determined using the phenolphthalein test (Figure 7). The model successfully simulated the carbonation depth by estimating the pH of the pore solution, and the results closely matched the experimental data.

## **Key findings**

Two numerical simulation frameworks were built to simulate microstructure development and carbonation of the sealants. The key findings are given below:

1. The initial input microstructure is built using the Anm model, considering real particle shapes and particle size distributions of the solid components, i.e., slag,  $K_2SiO_3$ , micro silica, and granite.

2. The forward dissolution rate of different elements from slag (Si, Ca, and Al), micro-silica (Si),  $K_2SiO_3$  (Si) materials are calculated from the dissolution experiments, which are further used as input for GeoMicro3D.

3. The precipitation of the products in the microstructure of the paste is modelled using classical nucleation probability and thermodynamic modelling which can quantitatively predict the formation





of different reaction products such as C-(N,K)-A-S-H, Nat(K), Natrolite and MA-OH-LDH in the microstructure.

4. The DOR of slag and volume of reaction products are estimated using SEM-BSE. The simulated DOR for slag and volume of reaction products shows good agreement with the experiments, proving the rationality of the model.

5. The carbonation model built use this information as an input, to simulate the dissolution of  $CO_2$ , transport, precipitation of products and ultimately the carbonation depth.

6. The built carbonation model can simulate the microstructural changes due to chemical reaction of  $CO_2$  and the precipitated gels. Model could also simulate the changes in the porosity and precipitation of different carbonated products and silica gel.

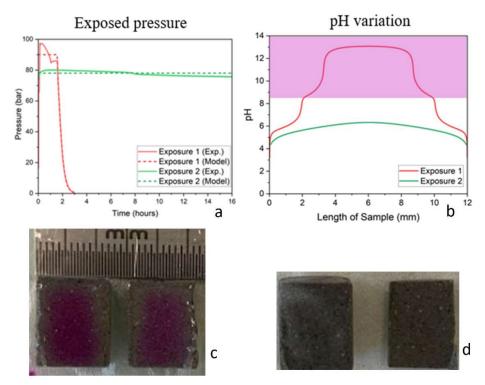


Figure 7: (a) Monitored and modelled exposure conditions for the samples; (b) modelled pH curves on a cross-section through each sample, (c&d) carbonation depths determined using phenolphthalein test for exposures 1 (c) and 2 (d).

## Publications

<u>Gupta, M., X. Qiu, M. Omran, M. Khalifeh, G. Ye (2025) Reaction and microstructure development of</u> <u>one-part geopolymer for wellbore applications – An experimental and numerical study. Cement and</u> <u>Concrete Research 188, 107738.</u>

Gupta, M., S.H. Hajiabadi, F. Aghabeyk, Y. Chen, R. van Noort, M. Khalifeh, G. Ye (in preparation) A 3-D reaction transport model to simulate microstructural changes of rock-based geopolymer exposed to wet supercritical CO<sub>2</sub>.





# WP5: Interfacial and bulk properties of cement sealants

In light of the anticipated timelines for the operation of CCS storage facilities, it is crucial to minimise  $CO_2$  leakage for optimal performance. Ensuring the wellbore plug adheres effectively to the casing is thus of significant importance. WP5 aims to evaluate the bond performance of CEMENTEGRITY wellbore sealants to metal casings, with emphasis placed on technically simple test procedures that can be further implemented in standard cement or concrete testing laboratories.

## Method

Test samples were first designed to replicate miniature versions of plugged well-bores, each comprising an outer cylindrical steel casing and an internal cement plug (Figure 8a). The samples were cured at elevated temperature and pressure (Figure 8b), followed by push-out shear-bond tests at 80°C (Figure 8c). Non-destructive testing, utilising electrical measurements, was also performed on the sealant samples during and after curing to determine the duration required for the sealants to achieve near or full hydration. Additional measurements were taken post-curing to provide information on the sealant properties and the quality at the sealant/casing interface. This complemented the primary shear-bond test and other investigative techniques in other work packages. All sealant samples were prepared at Halliburton in Norway, with the mixes prepared by Halliburton staff according to the API 10B-2. Each was cast into pre-prepared moulds and subjected to the enhanced curing regime before being airlifted and subsequently tested at HWU in the UK.

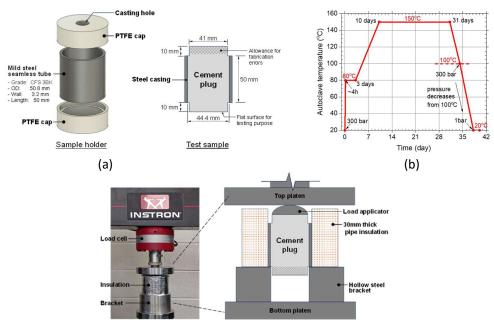


Figure 8. Schematic diagrams of (a) test sample; (b) curing regime; and (c) shear bond test.

## During curing

Figure 9 depicts a typical response during the enhanced curing process. During the first 3 days at 80°C, resistance gradually increased due to early pore development from cement hydration (Stage I). As the temperature increased (Stage II), resistance decreased. Accelerated hydration then caused resistance to rise again (Stage III). The rate of increase became more pronounced around 130°C (Stage IV) and at 150°C (Stage V), likely due to the contribution of silica flour. At the constant temperature 150°C, resistance continued to increase albeit at a lower rate (Stage VI), indicating ongoing pore structure development. Towards the end of this stage, the resistance gradually levelled off. The other sealants exhibited different characteristics but followed similar stages of response, confirming that maintaining 150°C over 3 weeks was generally sufficient.





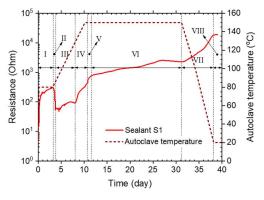


Figure 9. Resistance profile of sealant S1 during high-temperature curing.

#### Post curing

Figure 10 shows typical bond testing responses, with all samples showing an increase in mean bond stress with increasing displacement until failure. The bond strengths and overall stiffnesses varied, with both parameters decreasing in the order: S1 < S3 < S2 < S4. Mean bond strengths ranged from 0.54 to 4.66 MPa. This order differs from the compressive strengths measured after bond testing, which were S4 < S3 < S1 < S2, with means values ranging from 22.2 to 70.4 MPa. Subsequent physical examination revealed contrasting conditions on the inner surfaces of the steel casings: S1 samples showed extensive corrosion, while S2 and S3 samples showed minimal to no corrosion. Further testing of S1 with stainless-steel casing indicated 17% to 28% reduction in mean bond strengths. Repeated testing of S4 with retarding agent to regulate its very rapid setting significantly enhanced the bond with steel casing, resulting in a fivefold increase in value. The bond strength of S1 cured under laboratory and ambient pressure was much lower (only 0.91 MPa), highlighting the benefits of the enhanced curing. Final batch of testing using sealants S1(R) and S5 showed that S5 samples attained a mean bond strength of 10.2 MPa, while the repeat S1(R) samples ranged from 9.4 to 10 MPa. Although the reasons for these elevated strengths were unclear (e.g., whether it was caused by variations in the starting material properties, or induced during preparatory procedures), it was confirmed that S5 had the smoothest surface at the casing interface with no signs of corrosion.

Post-curing electrical measurements, presented in Nyquist format, revealed typical impedance responses with a low-frequency spur and a high-frequency arc for all sealants except S4, which exhibited a more complex response due to the emergence of an intermediate arc. Conductivity, derived from impedance data, represents direct ionic conduction through the connected porosity and can thus be related to material permeability. Conductivity was observed in the order S2 < S1 < S3 < S4 < S5, with values ranging from 0.036 S/m to 0.097 S/m. Conductivity is, however, influenced by the pore-fluid conductivity, which can affect the bulk conductivity value, although this is not expected to differ significantly among the three PC-based sealants. Of these, S2 is expected to be less permeable than S1, while S3 is likely more permeable. Due to the use of chemical activators, S5 is expected to have significantly higher ionic content (e.g., 2-3x), hence its high conductivity value. The higher conductivity of S4 suggests that it is more permeable than the PC-based sealants.





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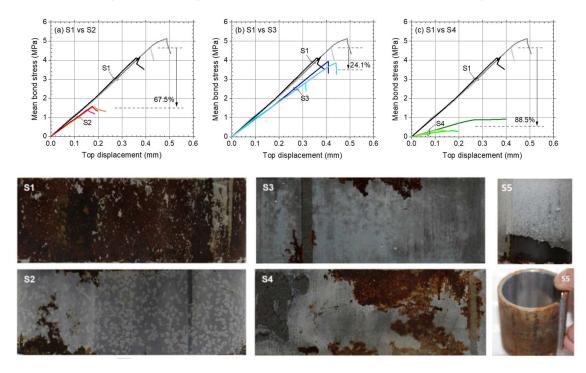


Figure 10. Results of bond tests and conditions at the sealant/casing interface.

#### Summary

The enhanced curing significantly increased both the shear-bond and compressive strengths of the sealants. Bond strengths ranged from 0.54 MPa for S4 to 4.66 MPa for S1. S1 (Repeat) and S5 samples exhibited more than twice the bond strengths, but the reasons for this significant increase were unclear. Of the other four sealants, S3 displayed superior bond strength despite its moderate compressive strength. Compressive strength influenced bond strength to some extent, but did not guarantee good bonding, as observed with the S2 samples. The high bond strength of S1 was due to corrosion at the casing interface, likely caused by internal confining pressure, which is expected to diminish over time. Regulating the setting time in S4 (e.g., preventing rapid setting and allowing sufficiently longer setting time) proved beneficial and could potentially be applied to other sealants to enhance their bonding performance with metal casing.

## Publications

Suryanto, B., G. Starrs, A.J.S. Kvassnes. (submitted) Bonding performance of cement sealants designed for CCS applications. *Submitted to Measurement*.

Starrs, G., B. Suryanto (in preparation) The electrical properties of cementitious sealants for use in CCS applications. *To be submitted to Measurement*.





## WP6: Development of rock-based geopolymers for CCS applications

The main objective of WP6 was to develop a rock-based geopolymer for CCS applications. Geopolymers are inorganic, SiO<sub>2</sub>-based binders, that can benefit from enhanced capabilities as wellbore sealants in CCS due to the geopolymeric structure and low calcium content of the system. WP6 contained four deliverables, and resulted in 4 journal publications, and 2 conference papers. Furthermore, one PhD-student successfully defended his thesis on 14<sup>th</sup> February 2025:

https://uis.brage.unit.no/uis-xmlui/handle/11250/3174104

#### D6.1 Initial CCS geopolymer design; laboratory preparation

The initial design of the geopolymer (GP) system was developed through a comprehensive literature review that systematically investigated the key factors influencing the chemomechanical durability of various GP formulations. This review identified optimal parameter values affecting GP performance and critically assessed existing research gaps, providing recommendations for targeted future scientific investigations. Based on the findings along with the results obtained from previous laboratory research conducted by the research team at University of Stavanger (UiS), the initial mix design for the targeted geopolymer system was formulated. For further details, please refer to DOI: 10.1021/acsomega.3c01777.

#### D6.2 Rheology and early strength development

Following modifications to the initial mix design—particularly through the optimization of retarder content—a series of experiments was conducted to evaluate the rheological behavior, pumpability, and stability of the slurries. Additionally, the strength of the solidified geopolymer samples was assessed using uniaxial compressive strength (UCS) testing. Notably, the impact of critical yet often overlooked elements, such as magnesium (Mg) content, was examined as part of this experimental series. Furthermore, in-situ mechanical strength assessments were conducted under simulated pressure-temperature conditions representative of CCS wellbores through a series of triaxial tests. These tests were complemented by a set of micro-scale analyses to gain deeper insights into the microstructural alterations occurring under optimized conditions. The results of these analyses can be found at the following links:

- <u>https://doi.org/10.1016/j.geoen.2023.212375</u>
- https://doi.org/10.3997/2214-4609.202310742

## D6.3 Geopolymers aged up to 6 months (strength; microstructure)

In this phase, we investigated the response of the granite-based GP system to NaCl and MgCl<sub>2</sub>-based brines, simulating conditions typical of CO<sub>2</sub> storage wellbores in aquifers and depleted oil and gas reservoirs. This research employed imposed-flow techniques and micro-scale analytical methods (XRD, SEM-EDS, FTIR, BET, ICP-MS, etc.) to assess the effects of brine exposure on the GP material. The results are documented in the following publications and presentations:

- <u>https://doi.org/10.1016/j.cemconcomp.2024.105511</u>
- https://doi.org/10.2118/223651-MS
- <u>Hajiabadi et al, 2023 TCCS12</u>.

In the second part of this phase, a comprehensive investigation was conducted to assess the performance of the granite-based one-part GP system under water- and CO<sub>2</sub>-saturated conditions over a 3- to 6-month period. This assessment included evaluating the impact of CO<sub>2</sub> on mechanical properties at various scales, supported by detailed micro-scale analyses. The results have been published here:

• https://doi.org/10.1016/j.geoen.2025.213919





## D6.4 Long-term properties of geopolymer as a seal (sealability; annular and plug)

Deviation Noted in D6.4: Hydraulic sealability tests (plug test) were conducted on the mix design for durations of 1 and 3 months. Results indicate that the material undergoes slight shrinkage after a 3-month curing period, suggesting the need for an expansive agent. The deviation pertains to the type of test conducted; a plug test was performed instead of an annular plug test due to challenges in ensuring reliability within the setup design (e.g., aspect ratio and large-scale relevance). As a result, we were unable to conduct the annular plug test.

#### Integrating Experimental Data and Numerical Simulation:

As the developed rock-based geopolymer were novel, as result of tight collaboration between WP4 (University of Delft) and WP6 (University of Stavanger), the reaction and microstructure development of the geopolymer were numerically simulated by use of GeoMicro3D. The results were published in the journal Cement and Concrete Research : <u>https://doi.org/10.1016/j.cemconres.2024.107738</u>

#### **Publications**

Hajiabadi, S.H. (2025) Granite-Based Geopolymers for Zonal Isolation of Carbon Capture and Storage (CCS) Wells – Improvement and Characterization. PhD-thesis, University of Stavanger.

Hajiabadi, S. H., M. Khalifeh, R. van Noort (2025) Durability Assessment of a Granite-Based One-part Geopolymer System Exposed to CO2-Water Conditions: Implications for CO2 Geosequestration. Geoenergy Science and Engineering 252, doi:10.1016/j.geoen.2025.213919.

Hajiabadi, S.H., M. Khalifeh, R. van Noort (2025) Hydro-Mechanical Behavior of a Granite-Based Geopolymer Sealant Exposed to MgCl<sub>2</sub>-Brine: Implications for CO<sub>2</sub> Geosequestration. SPE/IADC International Drilling Conference and Exhibition 2025, 04/03-06/03, Stavanger (N).

Hajiabadi, S. H., K. Li, A. Pluymakers, R. van Noort, M. Khalifeh (2024) Exploring the durability of a granite-based geopolymer sealant for carbon capture and storage: evaluating sealing performance under thermal shocks in brine environments. 17th Greenhouse Gas Control Technologies Conference 2024 (GHGT-17) proceedings, https://ssrn.com/abstract=5010201.

Hajiabadi, S. H., M. Khalifeh, R. van Noort (2024) Stability analysis of a granite-based geopolymer sealant for CO<sub>2</sub> geosequestration: In-situ permeability and mechanical behavior while exposed to brine. Cement and Concrete Composites, doi: 10.1016/j.cemconcomp.2024.105511.

Hajiabadi, S. H., M. Khalifeh, R. van Noort (2023) Multiscale insights into mechanical performance of a granite-based geopolymer: Unveiling the micro to macro behavior. Geoenergy Science and Engineering, doi: 10.1016/j.geoen.2023.212375.

Hajiabadi, S. H., M. Khalifeh, R. van Noort, P. H. Silva Santos Moreira (2023) Review on Geopolymers as Wellbore Sealants: State of the Art Optimization for CO<sub>2</sub> Exposure and Perspectives. ACS Omega, doi: 10.1021/acsomega.3c01777.

Hajiabadi, S.H., R. van Noort, M. Khalifeh (in preparation) Ability of a granite-based geopolymer sealant to withstand exposure to CO2 with H2S or H2SO4 in the presence of H2O. Paper providing a deeper exploration of the impact of  $CO_2$  on S5 mineral composition and microstructure.





# WP7: Identification of critical properties and suitable testing methods for sealants in CCS applications

The main tasks of WP7 were (1) to coordinate dissemination of all project results, in particular beyond the regular, academic channels (such as journals and academic conferences); and (2) to synthesise the results obtained by the other WP's to identify critical properties for sealants, that can improve long-term wellbore seal integrity, and also propose suitable methods and procedures for assessing these properties, and the impact thereon of exposure to  $CO_2$  and other potentially deleterious mechanisms that may occur during CCS.

## **Dissemination beyond academic**

One key component of project dissemination was the establishment and maintenance of a project website, at <u>www.cementegrity.eu</u>, through which project results and other items are shared publicly. It is planned to maintain this website for three more years (until 2028), and to update the Results page when further work is published.

A second important avenue for dissemination beyond regular academic dissemination was the organisation of seven webinars. During the project period, four open webinars were organised, including the final, Concluding *Ceminar*. *Ceminars* were open to all, and project participants presented the results they had obtained in the preceding period. Two *Ceminars* were held in 2023, around the mid-point of our project, while two more were held in 2024. Recordings of all *Ceminars* are shared publicly through the CEMENTEGRITY website. In addition, CEMENTEGRITY organised two private webinars for regulatory authorities, to share our work and findings and to initiate a discussion to help identify key findings from a regulator point of view, and how best to disseminate these findings to them. Furthermore, CEMENTEGRITY held one additional webinar as part of the Society of Petroleum Engineers (SPE) webinar series on CCS, in November 2024.

Public outreach was also pursued through three popular science articles. The first article outlining the CEMENTEGRITY project and its key findings was published in September 2024 in First Break – an industry magazine published by the European Association of Geoscientists and Engineers (EAGE) (Van Noort et al, 2024). In addition, CEMENTEGRITY collaborated with two other ACT-projects, RETURN and SHARP, to jointly write and publish a short popular science article about our projects and findings, to be published through national channels. The resulting articles were published on the Norwegian website geoforskning.no in December 2024 (Key advances in CCS through ACT - Geoforskning.no ), and in the Dutch Geo.brief in February 2025 (Pluymakers et al, 2025).

## Identifying key abilities and critical properties, and suggesting best methods

The second task of WP7 was to synthesise results from the other six WP's, together with a literature review, to identify the critical properties for a (cementitious) sealant, that can help maintain long-term seal integrity during and after CO<sub>2</sub>-injection and -storage operations. The findings from this work were published two Deliverables, and one conference presentation with article (GHGT Calgary – Van Noort et al, 2024). CEMENTEGRITY Deliverable D7.1 – Van Noort, 2024 is published, and the second Deliverable (CEMENTEGRITY Deliverable D7.2 – Van Noort, 2025) is currently being reviewed by our Technical Advisory Board. Based on the work done, three key abilities were identified that a cementitious material must assume upon hardening, in order to be used successfully as a sealant in a CCS well: 1. Ability to form and maintain a seal against other materials; 2. Ability to resist exposure to chemical stressors (i.e., CO<sub>2</sub>-containing fluids); 3. Ability to withstand physical impacts (esp. thermal shock or cycling). Furthermore, the material must maintain each of these abilities when even exposed to any deleterious effects related to CCS.

Based on these Key Abilities, as well as the overview of existing regulatory documents presented in D7.1, the following Critical Properties were identified:





Permeability					
		Low permeability required;			
		Further reducing permeability may improve ability to withstand			
		CO <sub>2</sub> -exposure.			
Mecha	nical properties				
-	Compressive strength	Sufficiently high			
-	Tensile strength	High			
-	E-modulus	Low			
-	Poisson's ratio	Suitable (0.1-0.3)			
-	E/C-ratio	Low			
Volum	Volumetric behaviour				
-	During curing	No shrinkage;			
		Expansion preferred.			
-	Over time	No shrinkage;			
		Expansion preferred.			
Therm	al properties				
-	Thermal diffusivity	High			
-	Thermal expansion	Suitable (i.e., similar to surrounding materials)			
	coefficient				
Mass	Mass				
		Mass changes from exposure indicate ongoing reactions that			
		may cause (or lead to) degradation.			
Compo	osition				
-	Chemical,	Chemical, mineralogical, and microstructural changes should be			
-	Mineralogical,	assessed to determine the depth to which the material is			
-	Microstructure.	affected by various changes, and to what degree these changes			
		are deleterious to the material's integrity as a sealant.			

Table 2. Critical material properties for sealants considered for application in CCS wells.

Finally, different methods for assessing the three Key Abilities are discussed and (where possible) compared in Deliverable 7.2. Methods for measuring the Critical Properties are also discussed in both Deliverable 7.1 and Deliverable 7.2. It is suggested that the selection of testing methods for any project should be based on balancing the required accuracy and precision against available resources.

#### Publications

Pluymakers, A., R. van Noort, E. Skurtveit, A. Barnhoorn, P. Cerasi (2025) <u>Onderzoek naar CO<sub>2</sub>-opslag:</u> wat gebeurt er in Nederland? Geo.brief 2025-1, pp. 20-22.

Pluymakers, A., R. van Noort, E. Skurtveit, A. Barnhoorn, P. Cerasi (2024) <u>Key advances in CCS through</u> <u>ACT - Geoforskning.no</u>

van Noort, R., M. Gupta, S. H. Hajiabadi, M. Khalifeh, A. Kvassnes, K. Li, A. Pluymakers, G. Starrs, B. Suryanto, G. Svenningsen, G. Ye (2024) Development and testing of novel cement designs for enhanced CCS well integrity. 17th Greenhouse Gas Control Technologies Conference 2024 (GHGT-17) proceedings, https://ssrn.com/abstract=5010396.





van Noort, R. (2024) Overview of current standards and common other testing methods used in wellbore sealant assessment. CEMENTEGRTIY Deliverable 7-1.

van Noort, R. (2025) Critical properties and testing methods for sealants in CCS applications. CEMENTEGRITY Deliverable 7-2.





## 5. Project impact

#### Contribution to the facilitation of the emergence of CCUS:

CEMENTEGRITY has deepened our understanding of sealant integrity during CCS, and of what may be important sealant properties to help ensure long-term seal integrity. Furthermore, CEMENTEGRITY has created a roadmap for sealant testing for CCS (or other applications). These outcomes will help increase confidence in well integrity and safety, and therefore in CO<sub>2</sub>-storage in general. Furthermore, a better understanding of what properties are critical for a well-functioning sealant for CCS may help increased safety and reduce costs, both through more targeted material development and placement, and preventing remediation costs. Finally, we have shown the capabilities of alternative sealant materials with a lower CO<sub>2</sub>-footprint than regular cements as CCS well sealants. The rock-based geopolymer binder used in sealant S5 also has potential as a binder in concrete and may thus form the basis for a low-CO<sub>2</sub> (or even CO<sub>2</sub>-negative) alternative building material to regular PC-based concrete.

#### Strengthening the competitiveness and growth of European companies:

As part of CEMENTEGRITY, two new sealant compositions were developed and tested: one by the University of Stavanger (S5), and one as part of a collaboration between ReStone AS and Halliburton (S3). Both these materials will have a lower environmental footprint than a regular PC-based sealant and will have beneficial qualities for use during CCS. S5 will form (part of) the basis for a new start-up company to be founded soon and may also form the basis for the development of alternative concretes with much smaller environmental footprint.

In addition, an indentation-based testing methodology was developed in the Halliburton laboratories and is now being implemented by them in other projects as well.

#### Other environmental or socially important impacts, such as public acceptance:

As a very focused project with a large experimental part, CEMENTEGRITY did not directly address social challenges such as public acceptance. However, in collaboration with other ACT-funded projects (ACT-RETURN and ACT-SHARP), we published popular science articles about our work in Norway (geoforskning.no) and in the Netherlands (geo.brief). CEMENTEGRITY also published an article about our work in the EAGE journal First Break.

## Chances for commercializing the technology further:

ReStone is working on commercializing the RePlug<sup>®</sup> technology used as the main innovative additive in S3 in a number of similar blends. The project period for CEMENTEGRITY has been essential for ReStone AS. We have worked extensively with supply-companies and operators both in the USA, in Europe as well as in the Middle East, making wells safer with regards to CCS. We have had our material pumped in all three regions in commercial wells as pilots, working towards having prosperous business connections there. We have seen great impact of the papers published from the partners in CEMENTEGRITY, as our customers appreciate the independent research and testing. Even poorer outcomes in some tests are beneficial as they make it clear where our upper limits for blend compositions lie. We are incorporating the results into our existing Technology Qualification Report that we utilise when we work with our customers on commercial projects.

UiS is continuing work on developing rock-based (one-part) geopolymers such as S5 in this project. They are currently working with their Technology Transfer Office (Valide) to license this technology to a new startup company. Once this is completed, the startup plans to apply for Qualification and Verification funding calls through NFR (or similar agencies). The first objective will be to de-risk the technology development, in order to accelerate commercialization. In these efforts, rock-based one-part geopolymers will be developed not only as wellbore sealants, but also as main binders in building materials, such as concrete, to replace Portland Cement.

Halliburton has already used the indentation methodology established in CEMENTEGRITY in other (commercial) projects. Here, the ability to map the impact of exposure to  $CO_2$  on sealant (mechanical)





properties on a much smaller scale has been important in enhancing the assessment of candidate sealants' abilities to withstand  $CO_2$ .

#### Gender issues:

Providing equal opportunities to all genders is of importance to us and to all participating entities in the project. However, we were limited by the availability and interest of people with relevant expertise. Out of the sixteen people working on CEMENTEGRITY, four were women, and none disclosed other gender minorities. Anne Pluymakers was one of our three national coordinators, and Xiujiao Qiu was one of the four junior researchers hired as part of the project. Astri Kvassnes and Jill Klaussen represented ReStone AS, of which Astri is the current CTO.





## 6. Implementation

The main objective of the CEMENTEGRITY project was to address the potential leakage of CO<sub>2</sub> through or along wellbore seals. This was identified as one of the main challenges to subsurface CO<sub>2</sub>-storage in the Priority Research Directions composed at the Mission Innovation CCUS Challenge Workshop held in Houston in 2017 (PRD S-9; Houston, 2017).<sup>2</sup> CEMENTEGRITY addressed the main mechanisms by which leakage could develop through or along a wellbore seal and worked to identify the key properties of a wellbore sealant that can help ensure long-term seal integrity during CCS.

The results of our project will help ensure long-term sealant integrity, and therefore contribute to increased security of geological CO<sub>2</sub>-storage as part of CCS. This can support the required rapid development of CO<sub>2</sub>-storage noted as a priority by SET-plan IWG-9.<sup>3</sup> Furthermore, CEMENTEGRITY also studied a sealant representative of sealants found in many legacy wells (S1). Therefore, the outcomes of our study will help in examining and characterising legacy wells, and in better identifying the needs for remediation, thus reducing remediation costs where possible.<sup>4</sup>

Engaging industry (and regulators) was a key aspect of enhancing the impact of CEMENTEGRITY. This was done through our existing professional networks, in particular by organising open *Ceminars* where CEMENTEGRITY partners presented progress updates on their work. Each of these *Ceminars* was attended by ca. 30-60 students, (academic) researchers, and industry professionals from around the world. In addition, we organised two webinars specifically for regulatory authorities from Norway, the Netherlands, the UK and Danmark, to discuss our work in context of their needs, and to better understand how to present our work such that it would be of use to them. In particular, this has helped steer how the work done in WP7 was reported. Finally, in addition to regular academic conferences, CEMENTEGRITY work was also presented at more industry-oriented workshops, such as those organised by the SPE or EAGE, to widen our audience. CEMENTEGRITY also presented a webinar through SPE to address their world-wide, mostly industry-based, audience. This was attended by about 90 different people.

The ISO/IOGP TC-67, SC3, WG2 has been briefed underway of the methods applied in WP1, and parts of this work are now included in a technical report which is being drafted by IOGP with the aim of resulting in an ISO standard for  $CO_2$  exposure.

<sup>&</sup>lt;sup>4</sup> <u>https://ccus-setplan.eu/wp-content/uploads/2024/05/2.1-Recommendations-on-CCUS-RI-priorities-for-</u> Horizon-Europe-work-programme-without-appendices.pdf





<sup>&</sup>lt;sup>2</sup> <u>https://mission-innovation.net/wp-content/uploads/2018/09/Accelerating-Breakthrough-Innovation-in-Carbon-Capture-Utilization-and-Storage-\_0.pdf</u>

<sup>&</sup>lt;sup>3</sup> <u>https://ccus-setplan.eu/wp-content/uploads/2024/05/2.1-Recommendations-on-CCUS-RI-priorities-for-</u> Horizon-Europe-work-programme-without-appendices.pdf

## 7. Collaboration and coordination within the Consortium

#### Internal collaboration

Trans-national collaboration between the CEMENTEGRITY partners was a foundational component of our project. Central preparation of samples for WP's 1, 2, 3 and 5 by Halliburton (Norway) required direct coordination between Halliburton and the receiving partners, while the impedance measurements during curing performed as part of WP5 even required Heriot-Watt researchers to be present at Halliburton during sample preparation. Furthermore, for S5, UiS developed recipes early in the project, and then provided these and materials to Halliburton for samples to be prepared and cured for the other WP's.

However, direct collaboration between WP's and partners continued throughout the project. For example, ReStone collaborated directly with Halliburton in developing a new formulation based on a higher content of their RePlug<sup>®</sup> material, and subsequently participated in analysing the results obtained. ReStone also collaborated directly with IFE in WP2, to better investigate the impact of CO<sub>2</sub>-exposure on the sealant composition with their materials (i.e., S3), including a visit of Astri Kvassnes to IFE to collaborate on studying the samples in SEM.

Based on direct collaboration with Heriot-Watt University, TU Delft (WP3 – Kai Li and Anne Pluymakers) included a push-out test to assess the impact of exposure to thermal shock on their compound samples. This test uses similar principles as the patented bond strength test used in WP5. TU Delft (Kai Li and Anne Pluymakers) also collaborated with IFE on WP2, performing CT-scanning on all WP2 samples exposed for 16 weeks, as well as on unexposed reference samples, and collaborating on interpretation of these scans. Finally, WP3 collaborated with WP6, supporting UiS in carrying out thermal shock tests on S5.

Direct collaboration was also required between WP4 and WP6, as WP4 built a numerical model of the rock-based geopolymer that WP6 was developing. This model required input on compositions and reaction rates. These collaborations lead to an exchange of students between the two universities, also beyond CEMENTEGRITY. As WP4 needed input on carbonation reactions and rates, they also received direct input and samples from WP2 (IFE). Furthermore, some additional short-term exposure tests were performed by IFE to provide data needed by TU Delft as input for their models. EBN also provided a detailed overview of a CO<sub>2</sub>-well, and the in-situ conditions within it, providing researchers in WP4 with a better understanding of the system to be modelled.

The direct collaboration between IFE and UiS included co-supervision of the PhD-student trained in this project. IFE also sent all exposed samples of S5 to UiS for deeper analysis of the impact of  $CO_{2^-}$  exposure.

Finally, towards the end of the project, WP7 coordinated a closer collaboration between all partners to enable comparisons of results and methods applied within the other WP's to expose and assess sealant materials, and identify the critical parameters.

Many of these collaborations are reflected in joint authorships in publications and other disseminations performed as part of CEMENTEGRITY. Furthermore, the collaborations initiated for this project have also led to contact and collaboration in other projects or proposals, as well as new activities.

Collaborating on a highly specialized topic with a multidisciplinary team meant that close communication was indispensable for ensuring thorough understanding of the work performed. Direct sharing of progress and findings between all partners was ensured through regular online meetings (*Ceminars*) where all WP's briefly presented progress, while selected partners gave more extensive





presentations of their activities and findings, and direct collaborations were often generated from these exchanges.

#### Management structures and governance procedures

The requirement by ACT, that a Consortium Agreement was in place before the Funding Agreement was signed and the project could start, has helped ensure that all partners agreed on key issues, and that potential problems, such as the need to publish results while also protecting Intellectual Property, were identified and addressed before the project started.

As per the Consortium Agreement, the project was steered by a General Assembly, with representation from all partners. In addition, a (voluntary) Technical Advisory Board was in place to help ensure that project results are relevant and applicable for actual CCS projects currently underway. Regular contact between the Project Manager and WP leaders (in order to prepare quarterly Traffic Light Reports, as well as additional contact where needed) ensured that the Project Manager was kept up to date on progress, and potential problems were identified early, and solved (where possible).

No large problems or challenges arose during the project, and the management structure and governance procedures functioned well for our project.

#### Added value of trans-national collaboration

The trans-national collaboration enabled by ACT allowed the CEMENTEGRITY consortium to create added value by bringing together leading research institutes and industry from the Netherlands, Norway and the United Kingdom; and to combine research groups with a wide range of highly complementary expertise, in terms of both skills and equipment. This unique combination of capabilities, and good, close collaboration between all partners, was key to the success of the project.

Furthermore, while Norway, the Netherlands, and the UK have all progressed far towards developing large (commercial) geological CO<sub>2</sub>-storage projects in the North Sea, there are significant differences between these projects. For example, while Norway's Longship project will soon start storing CO<sub>2</sub> in a saline aquifer, the Dutch Porthos project aims to store CO<sub>2</sub> in a depleted gas reservoir. As such, these projects deal with different conditions and different challenges to seal integrity. Having a transnational collaboration with industry participation from both the Netherlands and Norway ensured that the CEMENTEGRITY project included these different national perspectives in one strongly integrated project, enhancing the breadth, and thus value, of our results.

Finally, having an international partnership widened the reach of dissemination of project results, thus helping enhance our impact, and the potential for uptake of our findings.

New collaborations were developed between partners that previously did not work together, and already this has led to the development of new collaborations (for example through project proposals) beyond CEMENTEGRITY.





## 8. Dissemination activities (including list of publications)

## **Published articles:**

Hajiabadi, S. H., M. Khalifeh, R. van Noort (2025) Durability Assessment of a Granite-Based One-part Geopolymer System Exposed to CO<sub>2</sub>-Water Conditions: Implications for CO<sub>2</sub> Geosequestration. Geoenergy Science and Engineering 252, doi:10.1016/j.geoen.2025.213919.

Van Noort, R., G. Svenningsen, K. Li, A. Pluymakers (2025) Exposure of five cementitious sealant materials to wet supercritical CO<sub>2</sub> and CO<sub>2</sub>-saturated water under simulated downhole conditions. International Journal of Greenhouse Gas Control 144, doi: 10.1016/j.ijggc.2025.104380.

Hajiabadi, S.H., M. Khalifeh, R. van Noort (2025) Hydro-Mechanical Behavior of a Granite-Based Geopolymer Sealant Exposed to MgCl<sub>2</sub>-Brine: Implications for CO<sub>2</sub> Geosequestration. SPE/IADC International Drilling Conference and Exhibition 2025, 04/03-06/03, Stavanger (N).

Gupta, M., X. Qiu, M. Omran, M. Khalifeh, G. Ye (2025) Reaction and microstructure development of one-part geopolymer for wellbore applications – An experimental and numerical study. Cement and Concrete Research 188, 107738.

van Noort, R., M. Gupta, S. H. Hajiabadi, M. Khalifeh, A. Kvassnes, K. Li, A. Pluymakers, G. Starrs, B. Suryanto, G. Svenningsen, G. Ye (2024) Development and testing of novel cement designs for enhanced CCS well integrity. 17th Greenhouse Gas Control Technologies Conference 2024 (GHGT-17) proceedings, https://ssrn.com/abstract=5010396.

Hajiabadi, S. H., K. Li, A. Pluymakers, R. van Noort, M. Khalifeh (2024) Exploring the durability of a granite-based geopolymer sealant for carbon capture and storage: evaluating sealing performance under thermal shocks in brine environments. 17th Greenhouse Gas Control Technologies Conference 2024 (GHGT-17) proceedings, https://ssrn.com/abstract=5010201.

Van Noort, R., A. Pluymakers, K. Li, B. Suryanto, G. Starrs (2024) Development of tailored wellbore sealants for CCS and other geological storage applications. First Break 42, p. 89-94.

Suryanto, B., G. Starrs, A.J.S. Kvassnes (2024) Assessment of Bond Strength of Various Cementitious Sealants for CCS Well Applications. Proceedings of Joseph Aspdin 200 International Symposium, Innovations in Binder Technology, p. 91-94.

Starrs, G., B. Suryanto (2024) The electrical properties of cementitious sealants for subsea CCS applications. Proceedings of Joseph Aspdin 200 International Symposium, Innovations in Binder Technology, p. 54-57.

Lende, G., E. Sørensen, S. Jandhyala, R. van Noort (2024) State of the art Test Method to Quantify Progression Rate of Carbonation of Wellbore Sealing Materials. SPE Europe Energy Conference and Exhibition 2024, 28/06-29/06, Turin (I).

Hajiabadi, S. H., M. Khalifeh, R. van Noort (2024) Stability analysis of a granite-based geopolymer sealant for CO<sub>2</sub> geosequestration: In-situ permeability and mechanical behavior while exposed to brine. Cement and Concrete Composites, doi: 10.1016/j.cemconcomp.2024.105511.





Li, K. & A. Pluymakers (2024) Effects of Thermal Shocks on Integrity of Existing and Newly-Designed Sealants for CCS Applications. International Journal of Greenhouse Gas Control, doi: 10.1016/j.ijggc.2024.104103.

Hajiabadi, S. H., M. Khalifeh, R. van Noort (2023) Multiscale insights into mechanical performance of a granite-based geopolymer: Unveiling the micro to macro behavior. Geoenergy Science and Engineering, doi: 10.1016/j.geoen.2023.212375.

Hajiabadi, S. H., M. Khalifeh, R. van Noort, P. H. Silva Santos Moreira (2023) Review on Geopolymers as Wellbore Sealants: State of the Art Optimization for CO<sub>2</sub> Exposure and Perspectives. ACS Omega, doi: 10.1021/acsomega.3c01777.

# **Conference presentations:**

Hajiabadi, S.H., M. Khalifeh, R. van Noort (2025) Hydro-Mechanical Behavior of a Granite-Based Geopolymer Sealant Exposed to  $MgCl_2$ -Brine: Implications for  $CO_2$  Geosequestration. SPE/IADC International Drilling Conference and Exhibition 2025, 04/03-06/03, Stavanger (N).

Van Noort, R., M. Gupta, S. H. Hajiabadi, M. Khalifeh, A. Kvassnes, K. Li, A. Pluymakers, G. Starrs, B. Suryanto, G. Svenningsen, G. Ye (2024) Development and testing of novel cement designs for enhanced CCS well integrity. GHGT-17 2024, 20/10-24/10, Calgary (Ca).

Hajiabadi, S. H., K. Li, A. Pluymakers, R. van Noort, M. Khalifeh (2024) Exploring the durability of a granite-based geopolymer sealant for carbon capture and storage: evaluating sealing performance under thermal shocks in brine environments. GHGT-17 2024, 20/10-24/10, Calgary (Ca).

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# **Dissemination in preparation:**

Van Noort, R., G. Svenningsen, K. Li, (submitted) Experimental study on the impact of H<sub>2</sub>S and H<sub>2</sub>SO<sub>4</sub> in CO<sub>2</sub> on five different sealant compositions under conditions relevant for geological CO<sub>2</sub>-storage. *Submitted to Geoenergy Science and Engineering*.

Li, K., A. Pluymakers, R. van Noort (submitted1) Effects of Confined Thermal Cycling on Sealants with Different Thermomechanical Properties for CCS. *Submitted to Scientific ReportsC*.





Li, K., M. Friebel, A. Pluymakers (submitted2) Thermo-mechanical Behaviour of the Sealant-steel Interface under Thermal Cycling for CCS. *Submitted to: Geomechanics for Energy and the Environment*.

Van Noort, R. and V. Yarushina (accepted) Comparison of forced-flow and batch exposures of a representative wellbore cement to  $CO_2$ , and implications for extrapolation from laboratory to field. *To be presented at the Trondheim CCS Conference 2025, 16/06-19/06, Trondheim (N).* 

Kvassnes, A.J.S. and R. van Noort (accepted) An integrated and experimental study of a high olivinecontent Portland Cement blend, designed for CCS wells. *To be presented at the Trondheim CCS Conference 2025, 16/06-19/06, Trondheim (N).* 

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# Public outreach and other dissemination

Van Noort, R., X. Qiu, K. Li and G. Starrs (2023) *Ceminar* #1. Open CEMENTEGRITY Webinar, 2023-03-16. <u>Recordings</u>.

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Van Noort, R., K. Li, B. Suryanto (2024) Enhancing Sealant Durability and Integrity During CCS – The CEMENTEGRITY Project. Webinar for SPE, 2024-11-14.

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## **Deliverables & Reports:**

van Noort, R. (2025) Critical properties and testing methods for sealants in CCS applications. CEMENTEGRITY Deliverable 7-2.

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Suryanto, B. & G. Starrs (2025) Characterisation and monitoring of oil-well cements under simulated CCS well conditions. CEMENTEGRITY Deliverable 5-1 - 5-6.

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