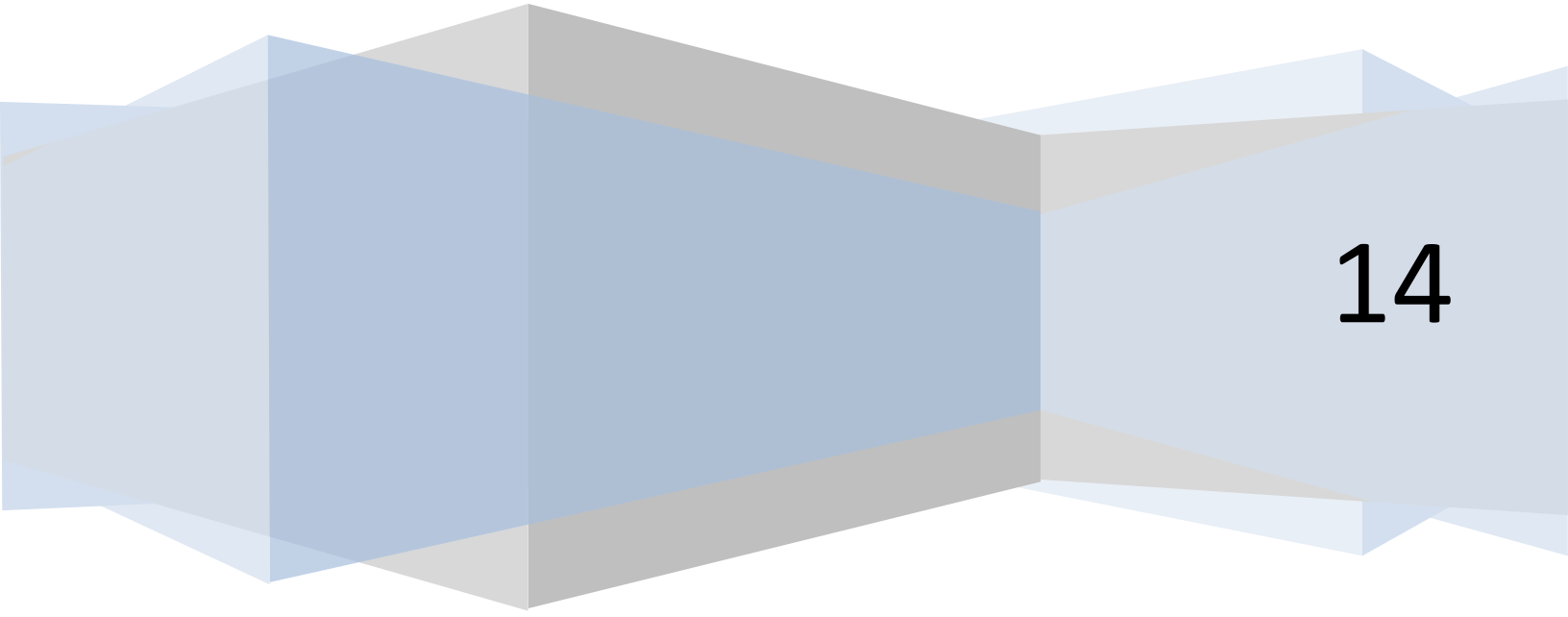
Graphene Synthesis Method vs. Voltage in a Dye Sensitized Solar Cell

Scientific Report

Dan Avila



14

Question:

How does the graphene synthesis method; sonochemical, H2O2 method, Tang Lau, electrochemical, and thermal; affect the electric potential in a dye sensitized solar cell?

Goals:

The goal of this project is to find the graphene synthesis method that has the highest electric potential in a dye sensitized solar cell when graphene is used as the electrode.

Hypothesis:

If the graphene synthesis method is manipulated as the electrode in a dye sensitized solar cell then the thermal reduction method will have the highest electric potential because it is the most popular method of producing graphene.

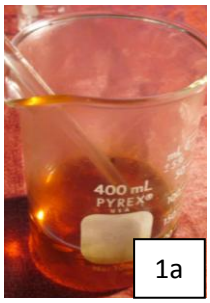
Materials:

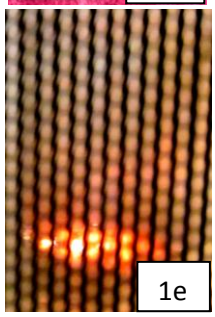
- 86ml sulfuric acid (75%)
- 17.2g graphite powder (99% from GS graphite)
- 3g iron(III) chloride
- 21g potassium permanganate
- 105ml sulfuric acid (96%)
- .34 ml ammonium hydroxide (30%)
- 550ml 10% hydrogen chloride
- 20ml Iodine tincture
- 1L hydrogen peroxide
- 250ml antifreeze
- 1tsp ascorbic acid
- 17ml hydrochloric acid (37%)
- 1ml polyethylene glycol
- 4ml nitric acid (70%)
- 5g titanium dioxide
- Granulated cylinder
- Beaker
- 3L glass container
- Glass pan
- Coffee grinder
- Food processor
- Stirring stick
- Pipette (1ml)
- 1.5in pipe nipple
- 1.5in. pipe caps (2x)
- PTFE tape
- 30mg ethanol (95%)
- 150ml ethanol(40%)
- 100ml hydrogen peroxide (30%)
- 10ml potassium iodide
- 40ml raspberry juice
- 11ml 30% sodium hydroxide
- AA battery (8x)
- Glass bowl
- Coffee filter
- Tape
- Alligator clips (4x)
- 12 medium binder clips
- 3inx3in glass (6x)
- Indium tin oxide coated glass
- Candle
- Ethylene glycol
- Work light
- Aluminum foil
- Vacuum filter
- Sonicator (35 Watt)
- Thermometer
- Multimeter

Procedure:

Expanded Graphite for Graphene Oxide and Liquid Phase Exfoliation

1. Add 3g Iron (III) Chloride to 86ml of 75% sulfuric acid and stir (1a).

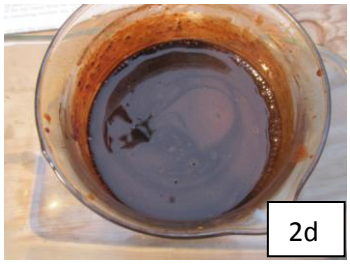




2. Add 17.2g of graphite powder and continue stirring (1b).
3. After thoroughly stirred add 6.2g potassium permanganate stirring until reaction dies down (1c).
4. Leave for one hour at 40C (104F).
5. Place in large container with two liters of water.
6. Once settled out pour off water on top keeping the settled remains.
7. Add another two liters of water and stir.
8. Repeat steps 6-7 two more times.
9. Place resulting solution onto a pan and heat at 60C (140F) for 10 hours or until dried into a powder (1d-this is graphite intercalated compound).
10. Add the ¼ tsp to a beaker and place in microwave for 20 seconds (1e)
11. Repeat step 10 until all of the potassium intercalated graphite compound is expanded(1f).

Graphene Oxide

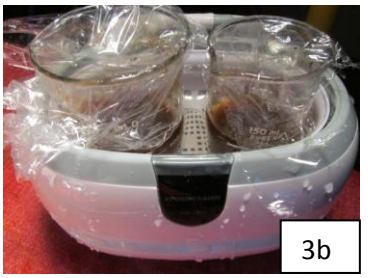
1) Add 15g potassium permanganate to 5g of the exfoliated graphite and stir.
2) Place the beaker containing the potassium permanganate and exfoliated graphite mixture into an ice bath (2a).
3) Add 100ml 96% chilled sulfuric acid to the beaker containing the potassium permanganate and exfoliated graphite and stir. (fumes)
4) Stir for 1 hour at a medium speed (2b).
5) Add 400ml of distilled water slowly over 30 minutes and stir constantly with the stirring stick (2C).
6) Place in oven at 90C (194F) for 1 hour (2d).
7) Let settle for 48 hours.
8) Pour off the top liquid from the beaker.
9) Pour the remaining mixture into a large glass container with 2L of tap water.



10) Let sit until the mixture is completely settled out (24 hours) and pour off the top liquid.
11) Add another 2 liters of tap water and repeat step 9.
12) Add remaining mixture to a beaker and allow to separate.

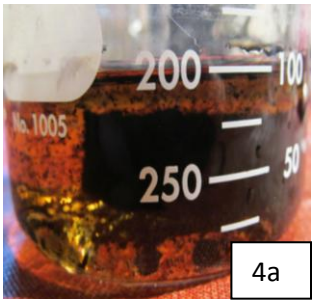

3a

13) Pour off top liquid and pour orange solution into a glass pan.
14) Allow to dry over two days then scrape from bottom of pan into a coffee grinder.
15) Grind in coffee grinder and then place powder on filter paper on a vacuum filter.
16) Filter with 500ml of a 10% hydrogen chloride solution using a vacuum filter (3a).
17) Wash with 1L distilled water with vacuum filter.
18) Spread out on to glass pan and let air dry for 1 day.
19) Weigh remaining powder and sonicate with distilled water in a ratio of 1g of powder per 250ml of distilled water for 23 hours in a closed lid container (3b).


3b

20) Pour off top golden solution to a separate beaker (this is graphene oxide).

Sonochemical



4a

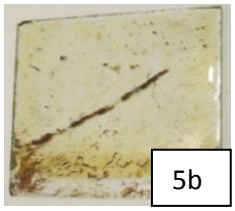
1) Add ½ tsp of ascorbic acid powder to 100ml of graphene oxide in a beaker and stir.
2) Add .34ml of 30% ammonium hydroxide (using the pipette) to mixture while stirring.
3) Sonicate for 23 hours (Results in 4a).
4) Put through food processor for 1 minute.
5) Set aside in bottle labeled "Sonochemical". This is graphene in solution.

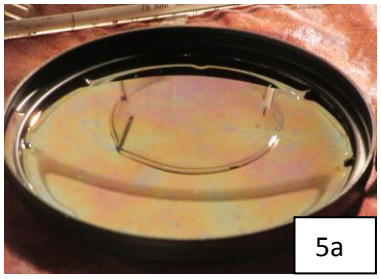
H2O2 Method

1) Add ¼ tsp of graphite intercalated compound with 50ml hydrogen peroxide (30%).
2) Place on a hot plate on low for 1 hour to dry and add 50ml of hydrogen peroxide.
3) Set aside in bottle labeled "H2O2". This is graphene in solution.

Tang Lau Method

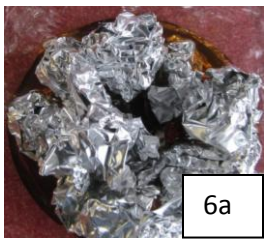
1) Attach one pipe cap to nipple with PTFE tape between.
2) Mix together 70ml water and 12g sugar in a beaker.
3) Pour into the pipe fitting and screw on top fitting with PTFE tape.
4) Place in oven at 210C (410F) for 3 hours.
5) Pour the liquid into a small glass bowl.
6) Dip the 3in x 3in glass through the water and remove trapping the top floating layer (5a). Anneal the piece at 350C (662F) in a kiln (5b).

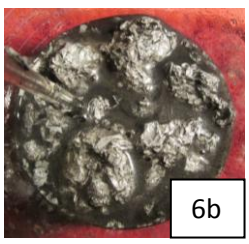

5b


5a

Nascent Hydrogen Reduction

1) Add 25ml of Graphene Oxide to 75ml of distilled water in a large beaker and stir.
2) Crinkle up about enough aluminum foil to fill the beaker and add to the beaker (6a).


6a


6b

3) Add 50ml of hydrochloric acid to the mixture and leave for 1 hour at 40C (6b)
4) Stir about every 15 minutes.

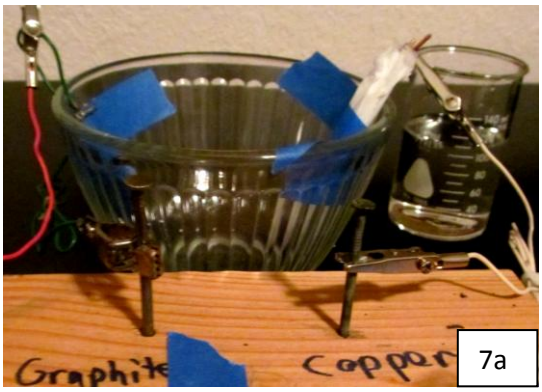
5) Pour the remaining mixture into a large glass container with 2L of tap water.
6) Let sit until the mixture is completely settled out (24 hours) and pour off the top liquid.
6b
7) Add another 2 liters of tap water and repeat step 6 two more times.
8) Pour remaining liquid into a separate bottle and label.

Liquid Phase Exfoliation

1) Add the 150ml of 40% ethanol and 5g exfoliated graphite together in a beaker and stir.
2) Sonicate for 4.5 hours.
3) Set aside in bottle labeled "Liquid Phase Exfoliation". This is graphene in solution.

Electrochemical Reduction

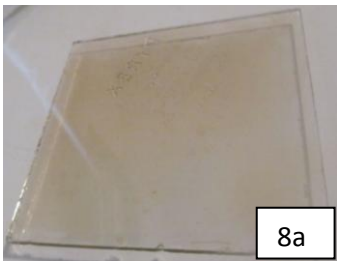
1) Connect the volt battery to two alligator clips with tape.
2) Connect two AA batteries together in series using tape and then to two alligator clips to make a 3volt battery.
3) Drill a hole through the graphite stick.
4) Bolt the stick to a holder and place on side of glass bowl.
5) Surround the copper wire cathode with a coffee filter and tape off.
6) Tape the copper cathode on the opposite side of the bowl as the graphite stick (7a).
7) Mix together 100ml distilled water, 2.4g sulfuric acid, and 11ml sodium hydroxide solution in a beaker.
8) Pour the mixture into the bowl with graphite and copper.
9) Apply the 3v battery with the positive on graphite and negative on copper for 1 minute.



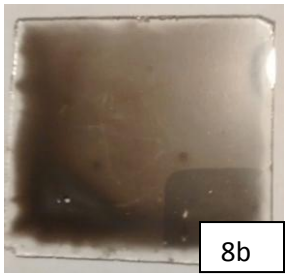
7a

10) Apply the 10v battery with the positive on graphite and negative copper for 2 seconds. Apply the 10v battery with the negative on graphite and positive on copper for 5 seconds.
11) Repeat step 10 continuously for 10 minutes.
12) Pour the mixture into a beaker.
13) Weigh solution in beaker and add 1% polyethylene glycol and 1% isopropyl by weight to the mixture in a separate beaker and stir.
14) Sonicate for 23 hours.

Thermal Reduction

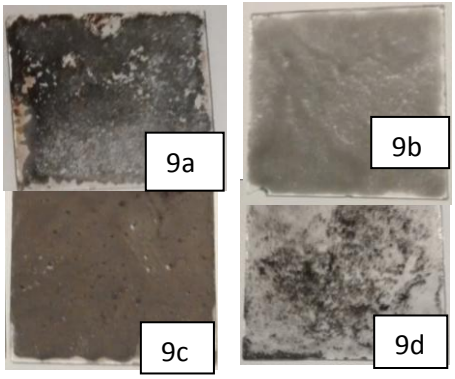

8a

1) Use pipette to evenly spread 1ml of graphene oxide onto one 3inx3in piece of glass and place on hot plate for 15 minutes on low (8a).
2) Place the pieces of glass into oven and bake at 350C (662F) for 1 hour.
3) Remove from oven and let cool for 1 hour (8b).


8b

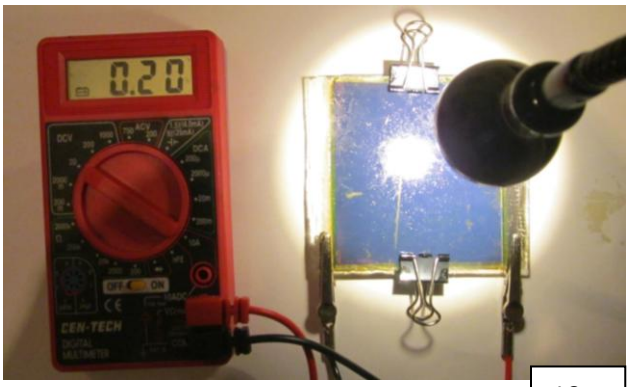
Spin Casting and Annealing

1) Use pipette to spread 1ml of solution onto a 3inx3in piece of glass and place on hot plate on low for 15 minutes.
   - Sonochemical(9a)
   - Nascent hydrogen(9b)
   - Hydrogen peroxide(9c)
   - Liquid phase exfoliation(9d)
2) Heat the pieces of glass at 200C for 1 hour.



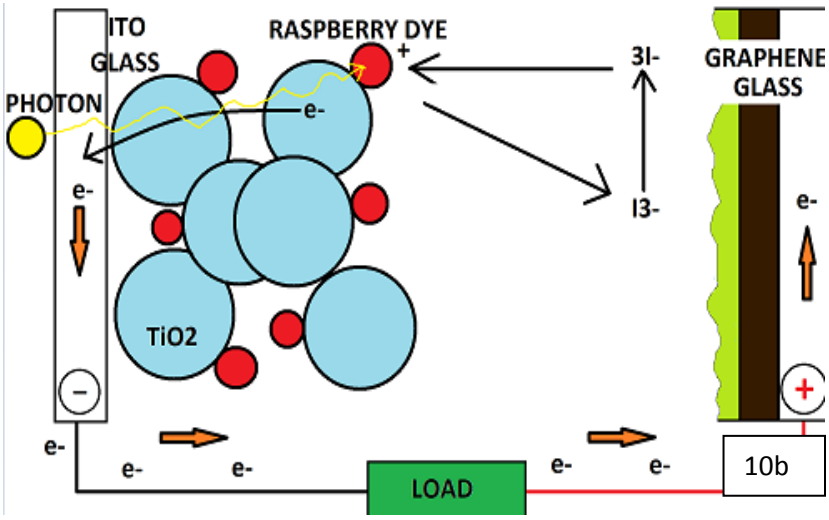
Dye Sensitized Solar Cell Assembly and Testing

1) Stir together 25.4ml ethanol and 5g titanium dioxide in a beaker.
2) Stir slowly with magnetic stirrer for 5 hours then add 1ml nitric acid.
3) Use 3 pieces of tape to form a box inside the indium tin oxide glass.
4) Use a pipette to place 1ml titanium dioxide ethanol paste on the open end of the glass so that it could spread to the entire cell.
5) Use a stirring stick to roll out titanium dioxide paste in an even coat.
6) Remove the tape.
7) Place piece on a hot plate on high for 15 minutes.
8) Remove allow to cool for 30 minutes.
9) Place in dish with 40ml raspberry juice and let sit 12 hrs.
10) Remove and wash with distilled water followed by ethanol.
11) Mix 120mg of iodine and 830mg of potassium iodide together until it browns and add 10ml ethylene glycol.



10a

12) Pour 1ml of ethylene glycol potassium iodide solution onto the titanium dioxide and spread evenly.
13) Clamp together the thermally reduced graphene coated glass and the indium tin oxide glass, offset by 1 cm, using two binder clips.
14) Attach multimeter to the anode and cathode with alligator clips.
15) Place 2.5in below light(ITO side up) and record the volts from cell (10a).
16) Pull apart cell and add another .5ml of electrolyte to the TiO2 side.

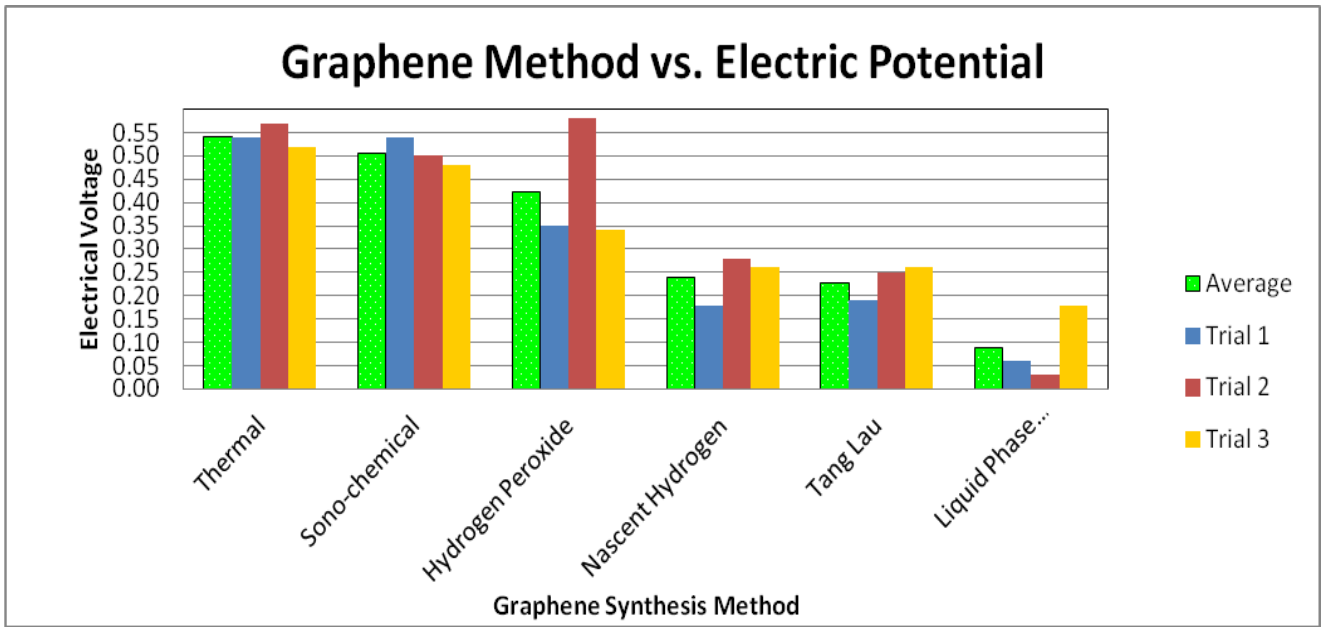


10b

17) Repeat steps 13-16 with the:
    - Electrochemical method
    - Sonochemical method
    - Tang Lau method
    - Nascent hydrogen method
    - Liquid phase exfoliation method
18) Repeat steps 13-17 two more times.

Data:

Graphene Synthesis Method	Trial 1	Trial 2	Trial 3	Average
Thermal	0.54	0.57	0.52	0.54
Sonochemical	0.54	0.5	0.48	0.51
Hydrogen Peroxide	0.35	0.58	0.34	0.42
Nascent Hydrogen	0.18	0.28	0.26	0.24
Tang Lau	0.19	0.25	0.26	0.23
Liquid Phase Exfoliation	0.06	0.03	0.18	0.09



Conclusion

The purpose of this experiment is to determine which method of graphene synthesis produces the most volts in a dye sensitized solar cell. The results indicate that thermally reduced graphene produces the highest voltage. Thermally reduced graphene produces the highest voltage with .54 volts. In comparison, the liquid phase exfoliation graphene synthesis method produces the lowest voltage with .09 volts. In conclusion, thermally reduced graphene produces the highest voltage, producing 0.45 volts greater than the liquid phase exfoliation graphene synthesis method.

Abstract

The purpose of this experiment is to determine which method of making graphene has the highest voltage in a dye sensitized solar cell. This affects the scientific world by making the dye sensitized solar cells even more efficient and economical so they may compete with photovoltaic cells. The problem with the current dye sensitized solar cell is that they are made of indium tin oxide (electrode) and platinum (catalyst) which isn't economical. The answer to this problem is using graphene as a combined catalyst and electrode because graphene is more flexible, transparent, conductive, economical and environmentally friendly.

Six methods were used to produce graphene: sonochemical, hydrogen peroxide, Tang Lau, liquid phase exfoliation, thermal and nascent hydrogen reduction. All materials are commonly available materials. The graphene was bonded to glass with high temperature. The solar cell anode is made by applying atitanium dioxide-ethanol paste to indium tin oxide coated glass, heating, soaking in raspberry juice, and applying an electrolyte.

The results indicate that thermally reduced graphene produces the highest voltage with 0.54 volts. The liquid phase exfoliation method produces the lowest voltage with 0.09 volts. My hypothesis was proven correct because the thermally reduced graphene produces the most voltage. This contributes to the area of dye sensitized solar cells because this shows that to achieve the highest efficiency thermally reduced graphene should be used as the electrode. My objective was achieved; this project has determined that thermally reduced graphene produces the most voltage in a dye sensitized solar cell.

Acknowledgements:

I would like to thank all the people who have helped me with my project such as my parents for allowing me to use some garage space, my chemistry teacher Greg Fetrow for supplying me with various chemicals, my friend Cameron Hohimer for allowing me to borrow his Sonicator, and RS graphite for suppying me with high quality graphite at such a low price.

Bibliography

1) UC Center for Laboratory Safety Standard Operating Procedure "Nitric Acid" and "Sulfuric Acid"
2) Iowa State University Materials Safety Data "Potassium Permanganate", "Sodium hydroxide solid, pellets, or beads", "Ethyl Alcohol, Denatured", and "Ammonium Hydroxide"
3) msdssearch.dow.com "Liquid Caustic Soda [Sodium Hydroxide] Safe Handling and First Aid"
4) mdk12.org "Chemicals: Managing, Handling and Disposing"; and 3m.solutions.com "3M™ Organic Vapor Cartridge/Filter 60921, P100 Respiratory Protection 60/Case"
5) Nano Letters 2008 Vol. 8, No. 1 323-327 "Transparent, Conductive Graphene Electrodes for Dye-Sensitized Solar Cells" by Xuan Wang, Linjie Zhi, and Klaus Mullen.
6) Materials Letters 109 (2013) 207-210 "Mass Production of Graphene Oxide from Expanded Graphite" by Ling Sun and Bunshi Fugetsu
7) International Journal of Engineering and Advanced Technology (IJEAT) ISSN: 2249 – 8958, Volume-2, Issue-3, February 2013, "Graphene Oxide Thin Films: A Simple Profilometer for Film Thickness Measurement", by Satish bykkam, K.Venkateswara Rao, Ch.Shilpa Chakra, V.Rajendar, Rotte Naresh kumar, and J.Ananthaiah