Scalable process for automated production of graphene oxide

Rune Wendelbo

Abalonyx AS, Forskningsveien 1, 0314 Oslo, Norway
rw@abalonyx.no

Abstract
Abalonyx has developed a scalable process for the efficient production of high purity nano-particulate graphene derivatives Graphene Oxide (GO) and Reduced Graphene Oxide (RGO), and we are now ready to build and operate a production plant. The Abalonyx Graphene Oxide (GO) process is a modification of the "Hummers method" [1], with subsequent thermal reduction, "flashing" to RGO. The products have been extensively characterized, comprising SEM, XRD, IR, Raman, XPS, AFM, TEM, TGA, BET and DLS. AFM analysis indicates that the GO is single layer, whereas Raman results indicate an average of 2-3 layer particles.

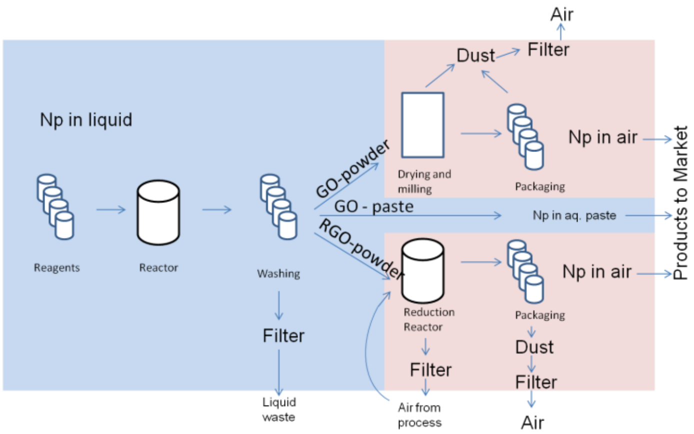

Figure 1.
Schematic process overview, including waste streams.

An extensive range of graphites were screened until we found a graphite raw-material that gives superior quality over the other graphites. The production process has been optimized so that it is safe, reproducible and fully scalable. The problems related to heat balance have been solved and we have designed a reactor that can produce up to 4 Kg of GO in one batch. The process has been partly automated, with plans for complete automation. The GO process has been run daily for 1 month with 100 g batches of graphite, producing 500 g batches of GO-paste with about 30 wt% GO [2].

It is well known that the Hummers method is difficult to control reproducibly. We have identified and addressed the following parameters as being critical.

1. Raw material. Out of a large number of graphites tested, we have identified one product - that gives high quality graphene oxide.
2. Reactor design. A cost efficient reactor that is perfectly scalable has been designed
3. Addition of reagents. The procedure for addition of the reagents has been optimized
4. Reaction temperature profile has been optimized.
5. Reaction time. The time for each reaction step has been optimized
6. Safety. Safety features have been designed to avoid runaway reaction
7. Automation. The most labor-intensive steps have been automated
8. Storing. Safe storage without product deterioration
9. Know-how. We possess extensive know-how related to the process

The production cost estimate (Figure 2) for a fully automated process has been worked out based on experience from our pilot reactor, reagent market prices, labor need and the following assumptions:

1. Reagent costs: 30 Euro per Kg at small volumes, decreasing to 20 Euros per Kg at max volumes.
2. Personnel: One person full time up to 4 Kg/day, then one extra person for each 10 X increment.
3. Personnel cost: 200 Euro per day per person
4. Infrastructure: From 200 Euro per day increasing by a factor 2 for each 10 X volume increase.
5. Waste management: 100 liters of acid waste per Kg product. Estimated cost 0.6 Euro/liter
6. Production plant cost: 200kE for 0.4 Kg/day capacity. Then 3 X more for each 10 X increment
7. Depreciation: Full write off/pay down over 4 years = 1000 working days.

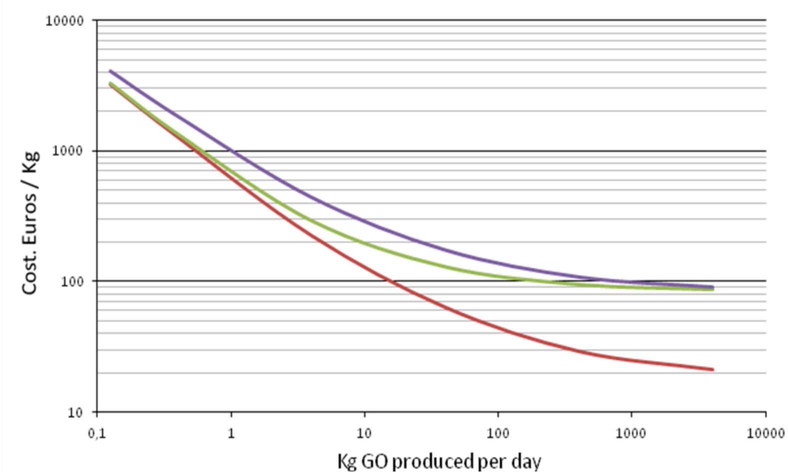

Figure 2.Estimated production cost for GO as a function of volume per day. Red line: Net cost. Green line: Net cost including cost for waste management. Blue line: Net cost including cost for waste management and cost of 4 year depreciation

From the above Figure, it can be seen that – under the present assumptions – waste management represents 75 % of total costs at high production levels, where as the contribution from depreciation is only about 5 %. Thus, if the acid waste can be neutralized and deposited at the site, a production cost of about 22 Euros/Kg for graphene oxide is achievable.

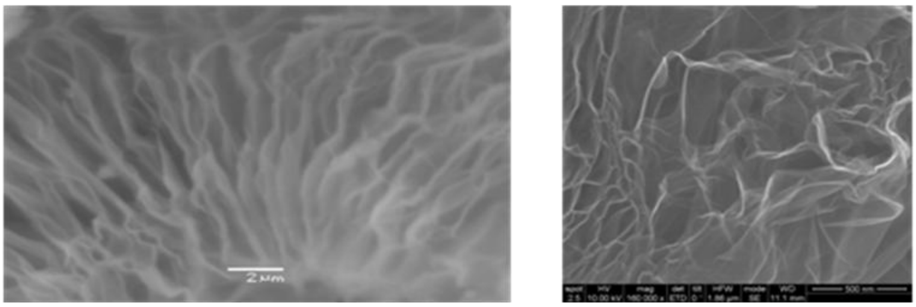

Figure 3.
Left: Freeze dried GO.
Right: Thermally reduced RGO

Potential end uses of our material include batteries [3] and ultracapacitors, paints, composites and printed electronics. We are presently building network to parties along the different value chains.

References

[1] Hummers, W.S., and Offeman, R.E., J. Am. Chem. Soc., 80 (1958),1339–1339.
[2] Wendelbo, R. and Fotedar, S. Norw. Patent application No. 20120917.
[3] Wendelbo, R. and Fotedar, S. Norw. Patent application No. 20121111.