A Robust, Scalable Process for Automated Production of Highly Dispersed Graphene Oxide and its use in transparent conductive coatings

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A robust, scalable process for the production of graphene oxide has been established based on a modification of the process of Hummers and Offeman [1]. The raw material graphite was chosen based on an extensive screening of different source materials, the process was optimized and automated and scaled to 100 g. Automation involved feeding of reagents, controlling temperature in each step, monitoring and logging of relevant data. In the pilot production phase, the synthesis was repeated 20 times, with excellent reproducibility, to produce a total of about 3 Kg of graphene oxide. A key test of quality is the suspension stability test. Our product forms a stable suspension in water over months, (Figure 1).

The product has been extensively characterized with TEM, XPS, SEM, Raman, XRD, TGA, BET and light scattering. The results indicate an average of about 2 layers per particle, with a substantial fraction of single layers. The raw graphite oxide has about one oxygen per carbon, but this ratio can be tuned to lower ratios by partial thermal or chemical reduction.

Chemically reduced graphene oxide (RGO) has been used to prepare transparent conduction coatings using a high throughput set-up for layer by layer deposition. Suspensions were prepared of RGO and magnesia nano-particles respectively, in 24-well microtiter plates, and a robotic arm was used to dip 24 glasslides forth and back between the two suspensions, washing water, a drop removal station, an oven and stations for on line measurement of conductivity and white light transmission (Fig. 2). Samples on glass slides are shown in Figure 3.
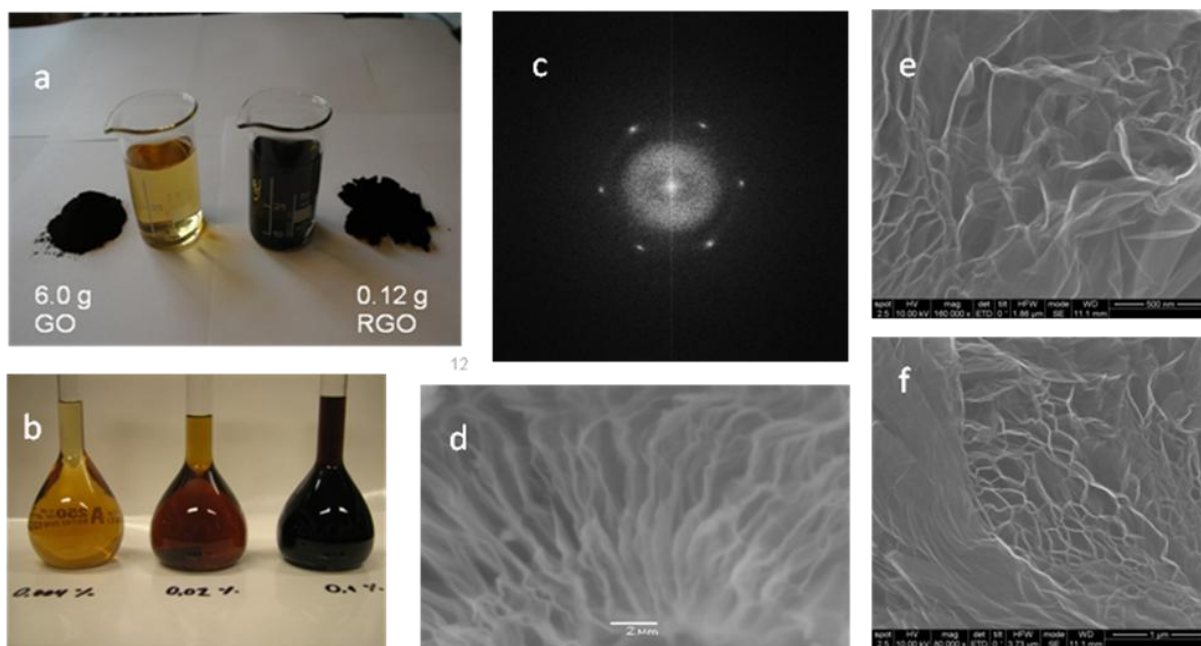

Figure 1. Graphene oxide and reduced graphene oxide. a) dispersions and dry powders. b) stable suspensions of GO. c) Electron diffractogram of single layer. d) freeze dried GO. e) thermally reduced GO 160,000 X and f) thermally reduced GO 80,000 X.
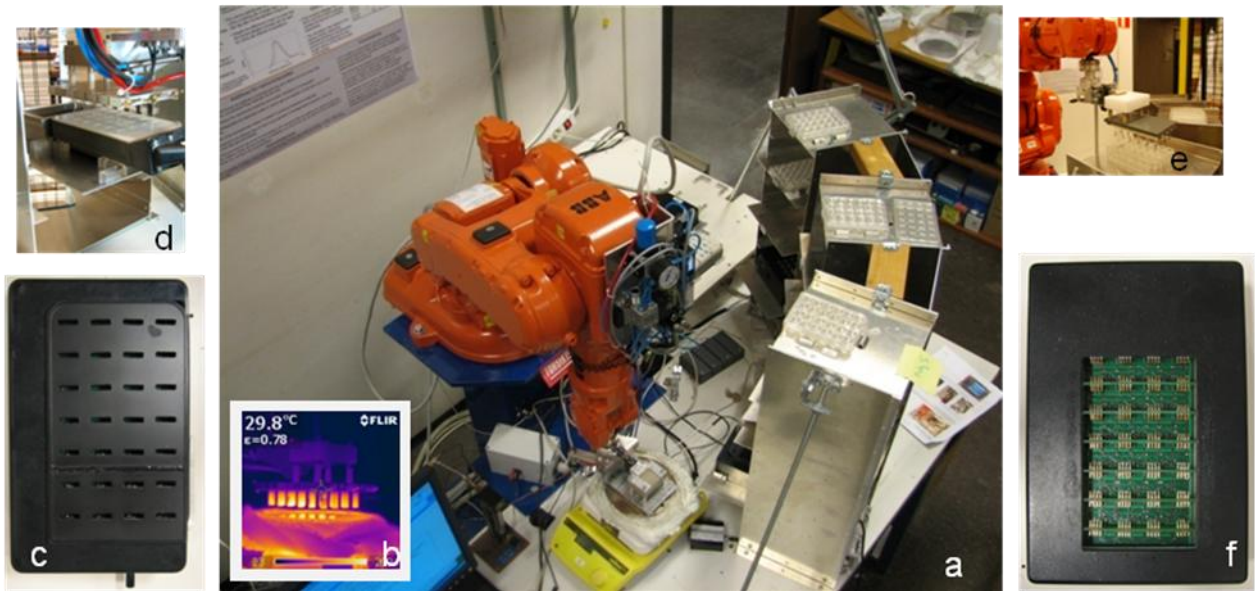
Figure 2. Robotic set-up for layer-by-layer deposition of particles and ions. a) robotic arm with well-plates and oven, b) hot samples withdrawn from oven, c) station for measurement of light transmission, d) drop removal station, e) dipping station detail, and f) station for online four-point measurement of conductivity.

Figure 3. Examples of transparent conducting cotings prepared. Grey samples contain reduced graphene oxide and magnesiaand greenish samples contain Cu-Zn-disulfide.

A further activity has now been started involving precipitation of metal oxides and phosphates on GO and RGO for the use in Li-ion battery electrode coatings.

References

[1] Hummers, W.S. and Offeman, R.E., *J. Am. Chem. Soc.*, **1958**, *80* (6), pp 1339–1339.
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