

Nanographene oxide high yield production and uses in phototherapy

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Graphene is increasingly attracting interest from the scientific and business community, due to its great potential for the development of new high-value technologies in the scientific and industrial environment. Current methods for graphene oxide (GO) production, like mechanical exfoliation, chemical exfoliation, chemical vapor deposition, and others, are not capable of producing nanosized GO with high yield and concentrations, having water stability, and being biocompatible. Therefore, improvement of the methods is necessary to achieve higher yield and higher concentrations of materials that meet the quality specifications demanded for different industrial applications, especially in areas related to biomedicine. Among the limitations in the production of graphene from current production methods are high cost, low efficiency and low reproducibility on a high scale. [1,2] Herein, single layer nano-sized graphene oxide (GOn) was produced through the modified Hummers method, followed by ultrasonication using a custom-built industrial grade system with technical specifications that allowed to achieve materials with the desired characteristics, for biomedical applications, in very high concentrations with a simple process.

Particle size was determined by transmission electron microscopy (TEM) and dynamic light scattering (DLS). Surface charge was measured using a zeta potential analyser. Oxidation degree was characterized by X-ray photoelectron spectroscopy (XPS) and Fourier-transform infrared spectroscopy (FTIR). Thermal stability of the samples was determined by thermogravimetric analysis (TGA; 30-1000 °C, 10 °C min⁻¹, under N₂ flow). Biocompatibility was evaluated using human foreskin fibroblasts (HFF-1) and by assessing cell viability through resazurin assay. Single layer GOn was obtained with mean lateral dimensions of 99 ± 43 nm (52 % <100 nm, 99 % <200 nm). Original GO size was of 1178 nm ± 479 nm. GOn dispersion showed colloidal stability with zeta potential values around -39.4 ± 1.8 mV, at neutral pH and a concentration of 8 mg mL⁻¹. After 6 months no decrease in particle stability was observed. XPS analysis revealed that GOn oxygen atomic percentage (at.%) was of 30% and that its carbon at.% was of 70%, also a typical FTIR spectra was obtained, confirming that a material with the desired chemical functionalities was produced. TGA analysis revealed that a first step of 25% weight loss occurred between 141 °C and 200 °C, due to the degradation of thermolabile oxygen-containing functional groups. Also, a second step of 5% weight loss occurred between 200 °C and 548 °C, corresponding to the combustion of the carbon skeleton. The material revealed to be biocompatible at concentrations (100 – 250 µg mL⁻¹) above the usual amount used for biomedical applications or that can be release *in vivo* by implants containing those. At our team it has been characterized for biomedical applications in skin disease and cancer phototherapy, as produced, modified or incorporated in pharmaceutical formulations. This work will also be presented.

In sum, a biocompatible single layer nanosized material was obtained with high yield and at high concentrations, which presented stability for at least 6 months kept at room conditions. Currently, materials with such characteristics are not available commercially. Therefore, we are seeking translation to industry and exploring their applications in the biomedical field and other areas.

References

- [1] Costa-Almeida, R. *et al.*, Polymers, 12 (2020), 1840.
- [2] Pinto, A.M. *et al.*, Carbon, 99 (2015), 318.

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