

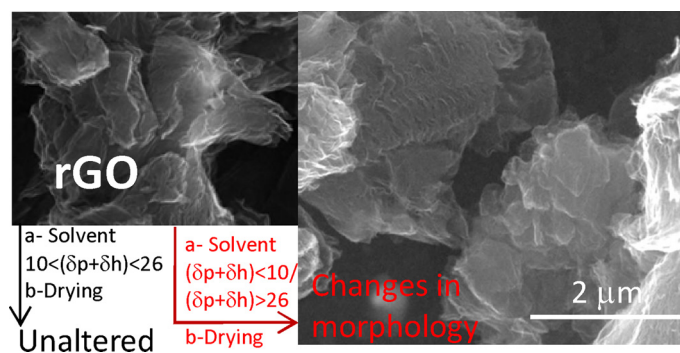
Morphological changes in graphene materials caused by solvents

Laura Fernández-García, Ana M. Pérez-Mas, Patricia Álvarez*, Clara Blanco, Ricardo Santamaría, Rosa Menéndez, Marcos Granda

Instituto Nacional del Carbón INCAR-CSIC, P.O. Box 73, 33080, Oviedo, Spain



GRAPHICAL ABSTRACT



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ABSTRACT

The aim of this work is to determine the changes in morphology caused in thermally reduced graphene oxide (rGO) after it has been mixed with organic molecules with different surface properties. The morphology of the dried rGO after being mixed with 16 organic solvents was evaluated using statistically SEM analysis. The results obtained indicate that this process led, in some cases, to an increase both in the number of stacked layers and in the corrugation of each rGO sheet. The largest modifications were obtained after contact with organic solvents undergoing intramolecular interactions mainly governed by the hydrogen-bonding interactions (measured as the Hansen δh parameter) and the dipole-dipole molecular interactions (expressed by the Hansen δp parameter). On the other hand, the morphology of the rGOs remained unaltered when solvents with values of $\delta p + \delta h$ in the range 10–26 MPa^{1/2} were used. These results represent a useful guide for the preparation of graphene-based composites in which the morphology of the graphene in the mixture is a crucial parameter which affects their characteristics.

1. Introduction

Graphene is a two-dimensional carbon material with a one-atom-thick planar sheet of sp^2 bonded carbon atoms that are densely packed together in a honeycomb crystal lattice. Graphene is thought to possess remarkable properties, such as a high thermal conductivity, superior

mechanical properties and excellent electronic transport properties [1,2]. These intrinsic graphene properties have generated enormous interest in the scientific community due to their possible implementation in a large number of devices [3]. These include future generations of high-speed, radio-frequency logic devices, thermally and electrically conducting reinforced nanocomposites, ultra-thin carbon films,

* Corresponding author at: Instituto Nacional del Carbón, CSIC, Department of Chemistry of Materials, P.O. Box 73, 33080, Oviedo, Spain.
E-mail address: par@incarc.csic.es (P. Álvarez).

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Table 1
Sedimentation behavior and morphological SEM parameters calculated for rGO in different solvents.

Solvent	Sedimentation			SEM morphology				
	LnA(t) = A ₀ + LnB(t)			[con]3h	[con]5h	Al ^a (nm)	Ac ^b (%)	S ^c (%nm)
	A ₀	B	R ²					
N-Methyl-pyrrolidinone	-0.9293	-0.0642	0.98	0.33	0.18	2000	0.1	200
Tetrahydrofurane	-0.2218	-0.9084	0.97	0.2	0.14	1700	0.4	680
Chlorobenzene	-0.2685	-1.0412	0.97	0.15	0.1	3900	0.8	3120
Hexane	-1.4202	-1.6876	0.95	0.002	< 0.001	3000	0.9	2700
Dimethyl sulfoxide	-0.2822	-1.242	0.97	0.11	0.08	3000	0.6	1800
chloroform	-0.3558	-1.1153	0.98	0.09	0.05	3900	0.7	2730
Toluene	-0.3652	-1.2535	0.95	0.09	0.05	3200	0.9	2880
Benzaldehyde	-0.1052	-1.075	0.98	0.25	0.2	3000	0.3	900
Dimethylformamide	-0.2711	-0.9983	0.99	0.16	0.1	2700	0.5	1350
Methanol	-0.5309	-1.1374	0.96	0.06	0.025	4000	0.8	3200
Dichloromethane	-0.4208	-0.8121	0.97	0.14	0.06	1500	0.3	450
Ethanol	-0.5669	-1.1697	0.96	0.06	0.015	4000	0.8	3200
Acetone	-0.3074	-1.1298	0.97	0.13	0.07	2000	0.5	1000
Isopropylalcohol	-0.158	-1.0563	0.98	0.21	0.16	1900	0.5	950
Ethyl acetate	-0.2611	-1.1909	0.95	0.13	0.09	2900	0.6	1740
Dimethylacetamide	-0.1775	-1.2224	0.98	0.17	0.13	2400	0.5	1200
acetonitrile	-0.4945	-1.0022	0.99	0.09	0.03	2300	0.4	920

a) Average length of the stacking. b) Average corrugation. c) Sedimentation factor (Al/Ac).

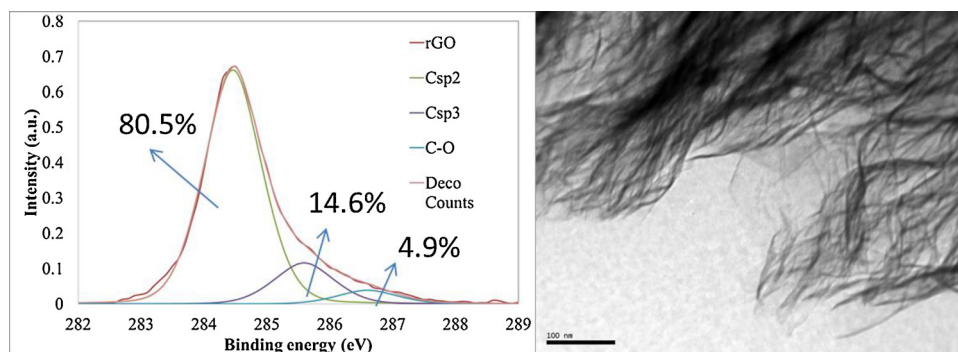


Fig. 1. Characterization of the as obtained rGO material; a) C1s XPS spectra and deconvolution curves, b) TEM image.

electronic circuits, sensors, and transparent and flexible electrodes for displays and solar cells [4–7].

Nowadays one of the most widely used methods to prepare graphene materials on a large scale is by the oxidation and thermal exfoliation/reduction of graphite. The resultant graphene material is a corrugated solid that exhibits lower mechanical, thermal and electrical properties than those of other graphene materials obtained, for example, by CVD. This is because the sp² carbon lattice after its initial oxidation is not fully restored by the thermal treatment. However, these graphene materials have been demonstrated to substantially improve the properties of certain graphene-based devices, as in the case of polymer/graphene nanocomposites which show superior mechanical, thermal, gas barrier, electrical and flame-retardant properties compared to the neat polymer [8–13]. The improvement of the physico-chemical properties of the nanocomposites depends on the distribution of the graphene sheets in the polymer matrix as well as on the interfacial bonding between the graphene layers and polymer matrix [14]. It has also been reported that the polarity, molecular weight, hydrophobicity, reactive functional groups, etc., present in the polymer, graphene/graphite and solvent are important factors that must be given due consideration in the preparation process [15]. However, as far as the authors are concerned, there is still a lack of information about these aspects of graphene behavior.

We have addressed these issues by focusing our attention on the solubility parameters of the materials involved. This approach, although rarely reported for graphene materials, has been considered an

effective method to investigate the dispersion of nanomaterials [16]. In recent years, it was successfully applied to determine how the solubility of functionalized reduced graphene materials increases in water and in organic solvents by means of selecting the adequate surfactant [17]. This methodology was also used with pyrrole-functionalized graphene materials which allowed to further investigate the theoretical insight on the nature of the involved molecular interactions [18]. Regarding the preparation of stable graphene solutions without the use of surfactants, several authors also see a relationship between the solubility parameters of the solvents and their capacity to maintain monolayers or few layers of graphene materials in suspension [19,20]. These authors mainly focus on the selection of the most convenient solvent which maximizes the yield of single or few-aggregated graphene layers in suspension. By selecting solvents with adequate solubility parameters an improvement in the liquid-exfoliation of graphite or in the reduction of graphene oxide by hydrazine was achieved. However, they do not present any information concerning the possible effects on graphene morphology caused particularly by those solvents with less suitable solubility parameters. Yet this is of particular importance for example, in the preparation of graphene-based composites where two or more materials with pre-determined solubility parameters have to be mixed. In these cases, it is desirable that the properties of the graphene, measured usually in a solid state or in a homogeneous solution be transferred to the final composite. Modification of the morphology of the graphene by its simple mixture with an organic substance is a matter of great concern, which needs to be analyzed in detail.

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