



Chemical and electrochemical study of fabrics coated with reduced graphene oxide



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ABSTRACT

Polyester fabrics coated with reduced graphene oxide (RGO) have been obtained and later characterized by means of chemical and electrochemical techniques. X-ray photoelectron spectroscopy showed a decrease of the oxygen content as well as an increase of the sp^2 fraction after chemical reduction of graphene oxide (GO). The electrical conductivity was measured by electrochemical impedance spectroscopy (EIS) and showed a decrease of 5 orders of magnitude in the resistance (Ω) when GO was reduced to RGO. The phase angle also changed from 90° for PES-GO (capacitive behavior) to 0° for RGO coated fabrics (resistive behavior). In general an increase in the number of RGO layers produced an increase of the conductivity of the fabrics. EIS measurements in metal/sample/electrolyte configuration showed better electrocatalytic properties and faster diffusion rate for RGO specimens. Scanning electrochemical microscopy was employed to test the electroactivity of the different fabrics obtained. The sample coated with GO was not conductive since negative feedback was obtained. When GO was reduced to RGO the sample behaved like a conducting material since positive feedback was obtained. Approach curves indicated that the redox mediator had influence on the electrochemical response. The $Fe(CN)_6^{3-/4-}$ redox mediator produced a higher electrochemical response than $Ru(NH_3)_6^{3+/2+}$ one.

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1. Introduction

The production of fabrics with new properties such as thermal regulation [1], flame resistant [2], self-cleaning [3], electrical properties [4], etc., has attracted great attention during the last years. Among these properties the electrical conduction has attracted particular attention. Different methods have been employed to provide the fabrics with electrical conductivity. For example the use of metallic fibers inserted in the fabric, chemical metallization of fibers [5], the extrusion of fibers with conductive particles such as carbon derivatives [6] or the synthesis of conducting polymer films on the fabrics [7–9].

On the other hand, the discovery of graphene and its derivatives has opened a new era in the field of physics and materials science. The amazing electronic, mechanical, optical or thermal properties shown by this material open the door to a range of different applications [10–17]. Different methods have been employed for the production of graphene and its derivatives [16–18]. The first method reported by Novoselov et al. [10] was the mechanical exfoliation from graphite that produced high quality graphene sheets.

However, the quantity of graphene obtained was minimal, reason why other methods such as chemical vapor deposition or chemical methods were developed. Among them, the chemical methods have been proposed as an alternative for the production of larger quantities of graphene. One of these methods is the production of graphene oxide (GO) by the oxidation of graphite. However, GO is electrically insulating, reason why it has to be reduced afterwards by chemicals to produce reduced graphene oxide (RGO) [18]. RGO is electrically conducting due to the restored sp^2 bonds networks. Graphene derivatives have also been found of interest to produce composite conducting materials [19,20] with such low filler content as 0.1% [19].

Fabric materials have the advantage of possessing a high surface area as well as a good flexibility and mechanical properties, what makes them interesting supports to deposit other materials. Regarding conducting fabrics coated with graphene and its derivatives very little has been reported [21–23]. Yu et al. deposited graphene directly on the fabrics and later electrodeposited MnO_2 to produce supercapacitors [21]. Other strategy employed is to adsorb graphene oxide (GO) sheets on the surface of the fabric and later reduce them employing chemicals such as hydrosulfite [22] or hydrazine [23]. Graphene oxide sheets are adsorbed on the surface of the fabric due to the attraction forces existing between the functional groups of the fabrics and the oxidized groups of graphene oxide sheets. In the present paper the last strategy has been employed to produce reduced graphene oxide

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coated on polyester fabrics (which is the synthetic fiber most widely employed in textile industry).

Together with the synthesis of graphene coated fabrics, the electrochemical characterization of these materials is another field to be studied. In this paper we have employed two electrochemical techniques; electrochemical impedance spectroscopy (EIS) and scanning electrochemical microscopy (SECM), to characterize the RGO coated fabrics obtained. SECM is a powerful technique with several applications in different fields such as the study of metals corrosion [24], biological systems [25], etc. EIS has been shown also as a useful technique to characterize the electrical properties of conducting fabrics [26].

2. Experimental

2.1. Reagents and materials

All reagents employed were of analytical grade.

2.1.1. For the synthesis

Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) was acquired from Merck. Monolayer graphene oxide (GO) powders were acquired from Nanoinnova Technologies S.L. (Spain). Polyester fabrics were acquired from Viatex S.A. and their characteristics were: fabric surface density, 140 g m^{-2} ; warp threads per cm, 20 (warp linear density, 167 dtex); weft threads per cm, 60 (weft linear density, 500 dtex). These are specific terms used in the field of textile industry and their meaning can be consulted in a textile glossary [27].

2.1.2. For the characterization

Sulphuric acid (H_2SO_4) and potassium chloride (KCl) were purchased from Merck. Hexaammineruthenium (III) chloride ($\text{Ru}(\text{NH}_3)_6\text{Cl}_3$) 98%, $\text{K}_3\text{Fe}(\text{CN})_6$ 99% and $\text{K}_4\text{Fe}(\text{CN})_6$ 99% were used as received from Acrös Organics.

When needed, solutions were deoxygenated by bubbling nitrogen (N_2 premier X50S). Ultrapure water was obtained from an Elix 3 Millipore-Milli-Q Advantage A10 system with a resistivity near to $18.2\text{ M}\Omega\text{ cm}$.

2.2. Synthesis of reduced graphene oxide on polyester fabrics

Polyester fabrics were coated with reduced graphene oxide (RGO) similarly to Fugetsu et al. [22]. GO solution was employed mixing GO monolayer powders in an ultrasound bath for 30 min, solutions of 3 g L^{-1} of GO were obtained. The first stage of the synthesis was putting in contact the GO solution with the fabric to allow the adsorption of GO sheets on the surface of the fabrics, this stage lasted 30 min. After this time, fabrics with GO were dried during 24 h in ambient conditions. The second stage of the synthesis was the reduction of GO to RGO. Fabrics coated with GO were placed during 30 min in a solution containing the reducer (0.5% weight of sodium dithionite at approximately 90°). This time was considered sufficient to achieve the conversion of GO to RGO as other studies have shown [22,28]. After the adsorption and after reduction process, the samples were washed several times to remove graphene oxide and reduced graphene oxide sheets loosely attached to the fabrics. Samples with different number of RGO coatings (1, 2, 3, 4) were obtained (PES-1G, PES-2G, PES-3G, PES-4G) repeating the procedure mentioned above. Samples coated with graphene oxide (PES-GO) were also obtained for comparison of the different results.

2.3. Scanning electron microscopy (SEM)

A Jeol JSM-6300 scanning electron microscope was employed to observe the morphology of the samples using an acceleration

voltage of 10 kV. Samples were coated with Au employing a Sputter Coater Bal-Tec SCD 005.

2.4. X-ray photoelectron spectroscopy measurements (XPS)

XPS analyses were conducted at a base pressure of 5×10^{-10} mbars and a temperature around -100°C . XPS spectra were obtained with a VG-Microtech Multilab electron spectrometer by using unmonochromatized $\text{Mg K}\alpha$ (1253.6 eV) radiation from a twin anode source operating at 300 W (20 mA, 15 kV). The binding energy (BE) scale was calibrated with reference to the C 1s line at 284.6 eV. C 1s and O 1s core levels XPS spectra were analyzed for the different fabrics (PES-GO, PES-1G, PES-2G, PES-3G, PES-4G).

2.5. Electrochemical impedance spectroscopy measurements

An Autolab PGSTAT302 potentiostat/galvanostat was employed to perform electrochemical impedance spectroscopy (EIS) analyses. EIS measurements were performed in the 10^5 – 10^{-2} Hz frequency range. The amplitude of the sinusoidal voltage was ± 10 mV. Measurements were carried out in two types of electrode configuration.

- (1) EIS analyses in a two-electrode arrangement were performed to evaluate the electrical properties of dry samples. A metal/sample/metal symmetrical configuration was employed placing a mercury drop on the different fabrics that were located on a stainless steel plate. The electric contact was done with a stainless steel rod contacting with the mercury drop, the other electric contact was made through the stainless steel plate.
- (2) A standard three-electrode design was employed to measure the EIS response of PES-GO and PES-RGO samples in 0.1 M H_2SO_4 solutions. An asymmetrical configuration metal/sample/electrolyte was employed. Sample was mounted on a stainless steel plate (employed to produce the electrical connection). An Ag/AgCl (3 M KCl) electrode and stainless steel rod were employed as reference electrode and counter electrode, respectively. The electrolyte resistance values were obtained replacing the textile working electrode by a stainless steel one for each different pH solution.

The experimental results were also fitted using a non-linear least squares fitting minimization method by ZView software (version 2.7).

2.6. Scanning electrochemical microscopy (SECM)

SECM measurements were carried out with a scanning electrochemical microscope of Sensolytics. A three-electrode configuration cell consisting of a 100- μm -diameter Pt ultramicroelectrode (UME), a Pt wire auxiliary electrode and an Ag/AgCl (3 M KCl) reference electrode. Solutions of $\text{Ru}(\text{NH}_3)_6\text{Cl}_3$, $\text{K}_3\text{Fe}(\text{CN})_6$ and $\text{K}_4\text{Fe}(\text{CN})_6$ 0.01 M in 0.1 M KCl (supporting electrolyte) were prepared. Thus, two types of redox mediators, $\text{Ru}(\text{NH}_3)_6^{3+/2+}$ and $\text{Fe}(\text{CN})_6^{3-/4-}$ were selected to see the influence of the redox mediator on the electrochemical responses obtained. All the experiments were carried out in inert nitrogen atmosphere. The substrates were samples (0.5 cm \times 0.5 cm) cut from the different fabrics (PES, PES-GO, PES-1G, PES-2G, PES-3G, PES-4G). These samples were glued to microscope slides with epoxy resin. Approach curves were obtained by recording the tip current as the Pt UME tip was moved in z direction. Approach curves give us an indication of the electroactivity of the surface. These curves were compared to the theoretical ones (positive and negative feedback models).

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