



# Conducting fabrics of polyester coated with polypyrrole and doped with graphene oxide



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## ABSTRACT

Polyester (PES) has been coated with polypyrrole (PPy) to produce conducting fabrics. Graphene oxide (GO) has been used in different concentrations (10, 20 and 30% weight) as counter ion to neutralize the positive charges of the PPy structure. Fourier transform infrared spectroscopy with attenuated total reflection (FTIR-ATR), energy dispersive X-ray (EDX) and X-ray photoelectron spectroscopy (XPS) of the PPy/GO powders corroborated the incorporation of GO as counter ion due to the presence of O in the EDX spectrum, as well as an excess of C, arising from GO contribution. The doping level ( $N^+/N$ ) decreased with the GO content. Field emission scanning electron microscopy (FESEM) showed the formation of the PPy/GO coating and the incorporation of GO in the composite. Electrochemical impedance spectroscopy (EIS) in solid state and solution, cyclic voltammetry (CV) and scanning electrochemical microscopy (SECM) were used to test the electrical properties and electroactivity of the fabrics. There was a decrease in the electrical properties and electroactivity as the GO content increased. The conductivity of the fabrics could be tuned by varying the GO content.

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## 1. Introduction

The production of fabrics with new properties has received particular attention during recent years. Different properties such as electrical conduction [1], flame resistance [2], self-cleaning [3], thermal regulation [4], color change [5], solar energy production [6], photonic [7], antimicrobial [8], UV protecting [9] or even catalysis [10] have been reported. Electrical conduction has attracted special attention in the development of antistatic or smart textiles [1].

There are different methods to produce conductive fabrics, such as the incorporation of conductive particles [11], electroless deposition of metals [12], reduced graphene oxide coatings [13,14], graphene [15] or conducting polymers synthesized either chemically [16–18] or electrochemically [19,20]. During the formation of conducting polymers, positive charges are created in their structures. These charges need to be neutralized by molecules with negative charges (counter ion). Different counter ions, either organic or inorganic have been used for this purpose

[16–18]. Here, we report the use of graphene oxide as counter ion in conducting fabrics for the first time. Graphene oxide carries a negative charge in its structure due to the presence of carboxylic acids acquiring a negative zeta potential [21]. One of the main problems of fabrics coated with conducting polymers is the migration and loss of the counter ion when the fabrics are put in solution, mainly in basic pH where deprotonation of PPy occurs [17,18]. The immobilization of a macroscopic material (between several microns and nanometers) such as graphene oxide in a conducting polymer structure prevents its solubilization. The aim of this paper is to obtain conducting fabrics of polyester coated with PPy/GO and characterize them chemically and electrochemically.

Polyester was coated with PPy and different contents of GO (10, 20 and 30%). The chemical characterization of the PPy–GO coated fabrics was performed by FTIR-ATR, EDX and XPS. The electrical characterization of the fabrics was performed by EIS in solid state. FESEM was used to observe the morphology of the coatings and the incorporation of GO in the PPy structure. EIS in solution, CV and SECM were used to test the electroactivity of the different fabrics. PPy/GO pellets were also obtained by compacting the powder and these were analyzed by the majority of the mentioned techniques. Samples doped with an organic (AQSA) and an inorganic counter ion ( $PW_{12}O_{40}^{3-}$ ) were also obtained and characterized by SECM for studying the effect of the counter ion's nature.

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## 2. Experimental

### 2.1. Reagents and materials

All reagents used were of analytical grade.

For the synthesis: monolayer graphene oxide (GO) powder was acquired from Nanoinnova Technologies S.L. (Spain). Analytical grade pyrrole (Py), ferric chloride ( $\text{FeCl}_3$ ) and anthraquinone sulfonic acid sodium salt (AQSA) were purchased from Merck. Acetone was acquired from Prolabo. Analytical grade phosphotungstic acid ( $\text{H}_3\text{PW}_{12}\text{O}_{40}$ ) hydrate was supplied by Fluka.

Polyester fabrics characteristics were: fabric surface density,  $100 \text{ g m}^{-2}$ ; warp threads per cm, 55; weft threads per cm, 29. These are specific terms used in the field of textile industry and their meaning can be consulted in a textile glossary [22].

For the characterization: sulphuric acid ( $\text{H}_2\text{SO}_4$ ) and potassium chloride (KCl) were purchased from Merck. Hexaamineruthenium (III) chloride ( $\text{Ru}(\text{NH}_3)_6\text{Cl}_3$ ) and iron (III) sulfate pentahydrate ( $\text{Fe}_2\text{SO}_4)_3 \cdot 5\text{H}_2\text{O}$ ) were used as received from Acrös organics.

When needed, solutions were deoxygenated by bubbling nitrogen ( $\text{N}_2$  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 PPy/GO on polyester fabrics

Chemical synthesis of PPy/GO on polyester textiles was carried out similarly to previous reports [18]. Size of the samples was approximately  $4 \text{ cm} \times 6 \text{ cm}$ . Polyester was degreased with acetone before use. Pyrrole concentration employed was  $2 \text{ g l}^{-1}$  ( $\sim 0.02 \text{ M}$ ). The molar relation of the oxidant ( $\text{FeCl}_3$ ) used was (1:2.5) (pyrrole:  $\text{FeCl}_3$ ). Different concentrations of GO were used as counter ion (10, 20 and 30% respect to the pyrrole mass). The solution containing pyrrole and GO (80 ml) was placed in an ultrasound bath for 30 min to allow the exfoliation of the GO flakes. The second stage was the adsorption of pyrrole and GO on the PES fabric during 30 min. After this time, the  $\text{FeCl}_3$  solution (20 ml) was added in drops and the reaction elapsed over 150 min at room temperature with magnetic stirring. Adsorption and reaction elapsed in a precipitation beaker. The obtained conducting fabric was washed with water to remove unbound PPy/GO. The conducting fabrics were dried in a desiccator for at least 24 h before measurements were performed. Organic (AQSA) and inorganic ( $\text{PW}_{12}\text{O}_{40}^{3-}$ ) molecules were also used as counter ions to compare the results obtained with those of GO. Hence, PPy/AQSA and PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$  powders were obtained and collected for measuring their electrochemical behavior by SECM.

### 2.3. Preparation of PPy disks

PPy/GO, PPy/AQSA and PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$  powders (0.1 g) were pressed in a press (98.1 kN) to obtain discs (13 mm diameter) that were employed to carry out the different measurements.

### 2.4. FTIR-ATR

FTIR-ATR with horizontal mono-rebound attenuated total reflection accessory was performed with a Nicolet 6700 Spectrometer equipped with deuterated triglycine sulfate detector. An accessory with pressure control was used to equalize the pressure in the different solid samples. A prism of ZnSe was used and spectra were collected with a resolution of  $4 \text{ cm}^{-1}$ , 400 scans were averaged for each sample. GO powders were characterized to assign their different contributions. PES fabrics uncoated and coated with PPy/GO (10, 20 and 30% GO content) were characterized. In addition, PPy/GO powders (10, 20 and 30% GO content) were also characterized.

### 2.5. X-ray photoelectron spectroscopy (XPS)

XPS measurements were performed on a VG Scientific ESCALAB200 A equipment with PISCES software for data acquisition and analysis. For analysis, an achromatic Al (K $\alpha$ ) X-ray source operating at 15 kV (300 W) was used, and the spectrometer, calibrated with reference to Ag 3d5/2 (368.27 eV), was operated in CAE mode with 20 eV pass energy. Data acquisition was performed with a pressure lower than  $10^{-6}$  Pa. Spectra have been charge corrected to give the adventitious C1s spectral component (C–C, C–H) a binding energy of 285 eV. High-resolution spectra were collected using an analysis area of  $\approx 1 \text{ mm}^2$ . The peaks were constrained to have equal FWHM to the main peak. This process has an associated error of  $\pm 0.1 \text{ eV}$ . Spectra were analyzed for elemental composition using CasaXPS software (version 2.3.15). Deconvolution into sub-peaks was performed by least-squares peak analysis software, XPSPEAK version 4.1, using the Gaussian/Lorentzian sum function and Shirley-type background subtraction. The best mixture of Gaussian–Lorentzian components was defined based on the instrument and resolution (pass energy) settings used as well as the natural line width of the specific core hole.

### 2.6. Field emission scanning electron microscopy (FESEM) and energy dispersive X-ray (EDX)

A Zeiss Ultra 55 FESEM was used to observe the morphology of the samples using an acceleration voltage of 3 kV. Energy dispersive X-ray (EDX) measurements were performed between 0 and 10 kV.

### 2.7. Electrical characterization by electrochemical impedance spectroscopy (EIS)

An Autolab PGSTAT302 potentiostat/galvanostat was used to perform EIS analyses. EIS measurements were performed in the  $10^5$ – $10^{-2}$  Hz frequency range. The amplitude of the sinusoidal voltage used was  $\pm 10 \text{ mV}$ . Measurements were carried out in a two-electrode arrangement, in which the sample was located between two round copper electrodes ( $A = 1.33 \text{ cm}^2$ ). In the second configuration, two rectangular copper electrodes ( $0.5 \text{ cm} \times 1.5 \text{ cm}$ ) separated by 1.5 cm and pressed on the fabric sample were used. The measured area of the fabric with this configuration was a square of 1.5 cm so the measured impedance modulus ( $\Omega$ ) was equal to the surface resistivity ( $\Omega/\square$ ) [19,20].

### 2.8. Electrochemical impedance spectroscopy (EIS) measurements in solution

A standard three-electrode design was used to measure the EIS response of PES-PPy/GO samples in 0.1 M  $\text{H}_2\text{SO}_4$  solutions. An asymmetrical configuration metal/sample/electrolyte was used. The sample was mounted on a stainless steel plate (used to produce the electrical connection). An Ag/AgCl (3.5 M KCl) electrode and stainless steel rod were used as reference electrode and counter electrode, respectively. The effective area used was  $0.28 \text{ cm}^2$ .

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

### 2.9. Cyclic voltammetry (CV) measurements

An Autolab PGSTAT302 potentiostat/galvanostat was used to perform CV measurements in 0.1 M  $\text{H}_2\text{SO}_4$  medium. To produce the electrical contact with the fabrics, they were located between two Ti plates. The measurements were performed in a three-electrode

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