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Synthesis of PPy/PW12O403- organic-inorganic hybrid material on polyester yarns and subsequent weaving to obtain conductive fabrics

Journal:	<i>Textile Research Journal</i>
Manuscript ID:	TRJ-10-0438.R3
Manuscript Type:	Original Manuscript
Keywords:	coatings < Chemistry, surface modification < Chemistry, fiber, yarn, fabric formation < Fabrication, polymer formation < Chemistry
Abstract:	<p>In this paper, we study the morphological, chemical and electrochemical characteristics of conductive fabrics obtained by weaving conductive yarns of polyester (PES) chemically coated with PPy (polypyrrole)/PW12O403-. The weaving process allows us to obtain conductive fabrics with higher dimensions than the traditionally obtained by in-situ chemical oxidation methods. Untwisted yarns and satin fabrics produced the most satisfactory results. The yarns of PES-PPy/PW12O403- as well as the fabric obtained after weaving, were chemically characterized by means of Fourier transform infrared spectroscopy with attenuated total reflection (FTIR-ATR) and energy dispersive X-ray (EDX). Scanning electron microscopy (SEM) was employed to observe the morphology of the coating as well as the formation of defects during the weaving process. Electrochemical impedance spectroscopy (EIS) was employed to measure the conductivity of the fabrics and the conductive nature by means of the phase angle. The process was satisfactory since the coating of polypyrrole was not excessively damaged by the weaving process.</p>

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3 **Synthesis of PPy/PW₁₂O₄₀³⁻ organic-inorganic hybrid material on polyester yarns**
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6 **and subsequent weaving to obtain conductive fabrics**
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12 **Abstract**
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15 In this paper, we study the morphological, chemical and electrochemical characteristics
16 of conductive fabrics obtained by weaving conductive yarns of polyester (PES)
17 chemically coated with PPy (polypyrrole)/PW₁₂O₄₀³⁻. The weaving process allows us to
18 obtain conductive fabrics with higher dimensions than the traditionally obtained by in-
19 situ chemical oxidation methods. Untwisted yarns and satin fabrics produced the most
20 satisfactory results. The yarns of PES-PPy/PW₁₂O₄₀³⁻ as well as the fabric obtained after
21 weaving, were chemically characterized by means of Fourier transform infrared
22 spectroscopy with attenuated total reflection (FTIR-ATR) and energy dispersive X-ray
23 (EDX). Scanning electron microscopy (SEM) was employed to observe the morphology
24 of the coating as well as the formation of defects during the weaving process.
25 Electrochemical impedance spectroscopy (EIS) was employed to measure the
26 conductivity of the fabrics and the conductive nature by means of the phase angle. The
27 process was satisfactory since the coating of polypyrrole was not excessively damaged
28 by the weaving process.
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50 **Keywords:** Polyester yarns, polypyrrole, polyoxometallate, conductive fabrics.
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1. Introduction

The development of textiles with new properties and applications has received a great deal during recent years; one of these properties is the electrical conductivity for its use in electrical textiles [1-3]. Different methods have been employed to produce electrical textiles; such as employing metallic fibers. The problem with metallic fibers is that the movements of bending and stretching that take place in the textiles can break them [1]. An alternative method to the employment of metallic fibers is the in-situ chemical synthesis of polypyrrole (PPy) on fabrics [4-13]. Applications of polypyrrole-based conductive fabrics are varied and numerous; such as antistatic materials [4], gas sensors [5], biomechanical sensors [6], electrotherapy [7, 8], heating devices [9-11] or microwave attenuation [12].

The drawback with in-situ polymerization is that only fabrics with small dimensions can be obtained. The continuous polymerization of polypyrrole on yarns by continuous vapour polymerization has been also reported in bibliography [14, 15]. Therefore, a continuous production process of conductive fabric could be obtained from yarns coated by continuous chemical vapor deposition of polypyrrole and their subsequent weaving process.

During the formation of polypyrrole, positive charges are created in its structure (polarons and bipolarons). These positive charges are responsible for the electrical conduction and must be compensated by anions (counter ions) to maintain the electroneutrality principle. Low size anions such as Cl^- have been employed as counter ions, but their stability is low [12]. If the counter ion is expelled from the structure (dedoping), there is a significant loss of electrical properties. To prevent this phenomenon, bulky anions with high molecular size have been employed [16]. Typical counter ions employed in polypyrrole-based conductive textiles and fibers are organic

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3 molecules of larger size; such as; anthraquinone sulfonic acid (AQSA) [5, 10, 12, 17-
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6 20], dodecylbenzene sulfonic acid (DBSA) [4, 13, 18], p-toluene sulfonic acid (PTSA)
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8 [5, 12, 18, 21], naphthalenedisulfonic acid (NDSA) [6, 12, 13, 18], benzenesulfonic acid
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10 (BSA) [7, 22], naphthalenesulfonic acid (NSA) [12, 13, 21], and anthraquinone
11
12 disulfonic acid [23, 24]. Inorganic molecules have been also used either as counter ions
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14 [25-28] or as particles to create nanocomposites with polypyrrole [29].
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16 Polyoxometallates (POMs) are small oxide clusters with several metallic ions
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18 coordinated by shared oxide ions, forming a highly symmetrical metal oxide cluster
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20 [25]. When they are combined with conducting polymers such as polypyrrole, a hybrid
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22 material is obtained [25, 26]. In addition to this, they also present catalytic properties
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24 [27, 28]. $PW_{12}O_{40}^{3-}$ is a molecule with high volume and charge [25], so its diffusion
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26 coefficient is low, and the exchange with anions present in the solution is prevented.
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28 However, very little has been reported on the use of inorganic anions as counter ions
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30 when obtaining conductive fabrics. In our previous work [20], we employed a
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32 polyoxometallate as counter ion ($PW_{12}O_{40}^{3-}$) to produce polypyrrole coated conductive
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34 fabrics. As commented previously, only fabrics with low dimensions can be obtained
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36 with in-situ polymerization of polypyrrole on the fabrics. In the present paper we study
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38 the synthesis of $PPy/PW_{12}O_{40}^{3-}$ on polyester threads and the posterior weaving of the
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40 conductive yarns obtained for producing conductive fabrics with higher dimensions.
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42 Also the influence of the twist of the yarns has been studied to evaluate the influence of
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44 this parameter on the conductivity of the yarns obtained after polypyrrole deposition, so
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46 that only the most conductive threads will be employed to weave conductive fabrics.
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48 PES yarns have been coated with $PPy/PW_{12}O_{40}^{3-}$ by in-situ chemical polymerization
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50 method to produce conductive yarns. The yarns and the fabrics have been chemically
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52 characterized by means of Fourier transform infrared spectroscopy with attenuated total
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3 reflection (FTIR-ATR), and energy dispersive X-ray (EDX). The detection of W in the
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5 EDX **analyses** indicates the formation of the hybrid material PPy/PW₁₂O₄₀³⁻. The
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7 morphology of the PPy coating and the formation of defects during the weaving process
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9 have been observed by scanning electron microscopy (SEM). Electrical resistance has
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11 been also measured, as well as its conductive properties by means of electrochemical
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13 impedance spectroscopy (EIS).
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22 **2. Experimental**

23 2.1. Reagents

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25 Analytical grade pyrrole and ferric chloride were purchased from Merck. Normapur
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27 acetone came from Prolabo. Analytical grade phosphotungstic acid hydrate was
28
29 supplied by Fluka. Ultrapure water was obtained from an Elix 3 Millipore-Milli-Q
30
31 Advantage A10 system with a resistivity near to 18.2 MΩ cm.
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38 2.2. Chemical synthesis of PPy/PW₁₂O₄₀³⁻ on polyester yarns

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40 Chemical synthesis of PPy on polyester yarns was done similarly to the work reported
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42 in previous papers on polyester fabrics [20]. Two types of polyester yarns were
43
44 employed, one twisted and another untwisted. The length of the yarns employed was
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46 100 m approximately and different yarns were polymerized each time. Prior to reaction,
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48 polyester yarns were degreased with acetone in ultrasound bath and washed with water.
49
50 The pyrrole concentration employed was 2 g L⁻¹ and the molar ratios of reagents
51
52 employed in the chemical synthesis bath were pyrrole: FeCl₃: H₃PW₁₂O₄₀ (1: 2.5: 0.2).
53
54 The following stage was the adsorption of pyrrole and counter ion (PW₁₂O₄₀³⁻) (V= 1 L)
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56 on the yarns for 30 minutes without stirring. At the end of this time, the FeCl₃ solution
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3 (V= 250 mL) was added drop wise with a burette over approximately 60 minutes. The
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6 oxidation of the monomer took place during 150 minutes without stirring. Adsorption
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8 and reaction took place in a container of approximately 20 cm x 10 cm. The conductive
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10 yarns were washed several times with ultrapure water to remove PPy not attached to
11
12 fibers. Dall'Acqua et al. employed a washing machine to remove the excess of polymer
13
14 not attached to the fabric [24]. However, in our case the threads can become entangled
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16 if washed with a washing machine. This would make the subsequent weaving process
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18 difficult. The yarns were dried in a desiccator for at least 24 h before measurements
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20 were taken. The weight increase was measured obtaining a value near to 10 %.
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27 2.3. Weaving of the fabrics

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29 Two types of fabric structure (weave) were employed to observe the influence of the
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31 fabric structure on the conductivity of the fabrics, satin and serge (Figure 1). The satin
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33 employed was 7e3 (Figure 1-a) and the serge employed was 29e1, b,1,1,1,27 (Figure 1-
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35 b). Fabrics were woven in an electronic Jacquard loom (Dornier) with a wide of 140 cm.
36
37 The warp employed was twisted polyester (100 %) (350 turns per meter, Z direction)
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39 (Letex Gmbh) with a metric of 54 Nm and a density of 66 threads/cm. The weft
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41 employed was untwisted textured polyester (100 %) and coated with PPy/PW₁₂O₄₀³⁻
42
43 (Polyester SLG 2002 from Letex Gmbh) with a metric of 15 Nm and a maximum
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45 density of 33 picks/cm. Twisted threads (100 % PES, 110 turns per meter, Z direction)
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47 and coated with PPy/PW₁₂O₄₀³⁻ were also employed. After regulating the thread feeders
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49 to the minimum tightness, the process of weaving was done.
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58 2.4. FTIR-ATR spectroscopy

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Fourier transform infrared spectroscopy with horizontal multirebound attenuated total reflection (FTIR-ATR) was performed with a Nicolet 6700 spectrometer equipped with DTGS detector. An accessory with pressure control was employed to equalize the pressure in the different solid samples. A prism of ZnSe was employed. Spectra were collected with a resolution of 2 cm^{-1} and 400 scans were averaged for each sample.

2.5. SEM and EDX characterization

A Jeol JSM-6300 Scanning Electron Microscope was employed to observe the morphology of the samples and perform EDX analyses. SEM analyses were performed using an acceleration voltage of 20 kV. EDX measurements were done between 0 and 10 keV. Samples for SEM measurements were coated with Au employing a Sputter Coater Bal-Tec SCD 005. When EDX measurements were carried out, samples were coated with C.

2.6. Electrical resistance and electrochemical impedance spectroscopy measurements

Electrical resistance of the yarns and the fabrics was measured with a Goldstar DM-311 digital multimeter employing two metallic tweezers. The measurements were performed on the zones where the conductive threads are on the surface (weft floats). The resistance of the twisted and untwisted threads ($k\Omega$) coated with the hybrid material PPy/PW₁₂O₄₀³⁻ was measured with a multimeter (Fluke) at different distances (1, 3, 5, 10, 15 and 20 cm). The resistance of the satin and serge conductive fabrics ($k\Omega$) was measured at different distances (from 10 to 120 cm).

An Autolab PGSTAT302 potentiostat/galvanostat was employed to perform electrochemical impedance spectroscopy (EIS) analyses. EIS measurements were performed in the 10^4 - 10^{-2} Hz frequency range. The amplitude of the sinusoidal voltage

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3 applied was 10 mV. Measurements were carried out using two rectangular copper
4 electrodes (0.5 cm x 1.5 cm) separated by 1.5 cm and pressed on the dry textile sample.
5
6 The measured area of the textile was a square of 1.5 cm, so the measured impedance
7
8 modulus (Ω) when the material acts as a resistor, was equal to the surface resistivity
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10 (Ω/\square). The surface resistivity was only measured for the satin sample. To carry out the
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12 measurements accurately, a flat surface is needed. In the case of the serge, the structure
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14 of the background fabric is a plain. However, the plain suffers shrinkage and tends to
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16 roll up; consequently the surface resistivity cannot be measured accurately.
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27 3. Results and discussion

28 3.1. Resistance of the threads and the conductive fabrics

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30 Two types of polyester threads were employed, one twisted and another untwisted. The
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32 purpose was to study the influence of this parameter on the conductivity of the threads
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34 obtained after the formation of the polypyrrole coating. The polymerization of pyrrole is
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36 quite independent of the substrate employed. Only the polarity of the surface may have
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38 an effect on the adhesion of the conductive polymer. The adhesion is worse with fibers
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40 without polar groups (hydrophobic surfaces) (polyethylene or tetrafluoroethylene) [30,
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42 31]. In the case of polyester, the existence of C=O groups allows the formation of Van
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44 der Waals forces between the O^{δ-} (from C=O groups) and N^{δ+} (from polypyrrole).
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51 Figure 2 shows the values of resistance vs. distance for both types of threads (twisted
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53 and untwisted). As can be seen, the resistance of the untwisted thread is always lower
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55 than this of the twisted sample. For example at a measuring distance of 20 cm, the
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57 resistance of the untwisted thread is around 28 k Ω and for the twisted sample the value
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59 is around 100 k Ω . From these results, we concluded that the best thread to produce
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conductive textiles was the untwisted **one**. The twisted sample does not allow the proper diffusion of the reactants and oligomers formed during the polymerization to the inner fibers of the thread. The outer fibers that are in contact with the solution are well coated with PPy/PW₁₂O₄₀³⁻, however the inner fibers are not properly coated. On the other hand, the untwisted threads allow the formation of polypyrrole in all the fibers of the thread since there is no impediment for the diffusion of the reactants. The result is a lower resistance in the case of employing untwisted threads, so the untwisted threads were selected as the optimal threads for carrying out the polymerization of polypyrrole. Once the type of thread was selected according to the values of resistance obtained, the weaving process was carried out to produce the conductive fabrics. Two sorts of fabric structures were employed: the satin and the serge. Figure 3 shows the values of resistance vs. distance for both types of fabric structures. As can be seen the resistance values obtained for the satin fabric are always below the values of the serge fabric. For instance, at a measuring distance of 120 cm, the resistance of the satin fabric was around 5.3 kΩ and for the serge fabric the value was around 32.7 kΩ. The satin employed has better electrical conductivity since conductive threads form smaller floats than the serge employed. Higher floats produce less contact between the conductive threads since the structure is less compact. This produces a reduction in conductivity of the fabric in the case of the serge compared to the satin structure. Also, due to the higher dimensions of the floats in the case of the serge; there can be problems when measuring **electrical conductivity** since the measuring tweezers can introduce between the weft floats and touch non-conductive threads (warp).

3.2. FTIR-ATR spectroscopy

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3 Figure 4 shows the spectra of PPy/PW₁₂O₄₀³⁻ powders (a), untwisted PES yarns (b),
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5 untwisted PES yarns coated with PPy/PW₁₂O₄₀³⁻ (c), and the satin fabric after weaving
6
7 the untwisted conductive yarns (d). PPy/PW₁₂O₄₀³⁻ powders were analyzed to assign the
8
9 different bands of PPy and PW₁₂O₄₀³⁻. Contributions of PPy can be observed in Figure
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11 4-a at: 1540 cm⁻¹, associated to the pyrrole ring stretching vibration (C=C) [23, 24];
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13 1450 cm⁻¹, attributed to C–C stretching [23, 24]; 1300 cm⁻¹, C-N stretching [23, 24];
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15 1160, 1045 and 780 cm⁻¹ due to the stretching vibration of polypyrrole [23, 24].
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17 Additionally, other bands can be highlighted; such as: C–H in plane vibration (1090 cm⁻¹)
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19 ¹); C–H bending vibrations (966 cm⁻¹); and =C–H out of phase vibration (886 cm⁻¹)
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21 [32]. The only band attributed to PW₁₂O₄₀³⁻ can be observed at 1075 cm⁻¹ (P–O
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23 stretching) [33-35]. The other bands of PW₁₂O₄₀³⁻ are overlapped with those of PPy and
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25 cannot be discerned.
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32 In Figure 4-b, the spectrum of PES yarns is presented, different bands attributed to
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34 polyester can be observed; the most representative bands are: 720 cm⁻¹ (out of plane
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36 benzene group) [36]; 960 cm⁻¹ (C-O stretching of glycol) [36]; 1014 cm⁻¹ (in-plane
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38 vibration of benzene) [36]; 1090 cm⁻¹ (ester C=O stretching) [36]; 1236 cm⁻¹ (ester C=O
39
40 stretching) [36] and 1714 cm⁻¹ (C=O stretching of aromatic ester) [37].
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43 After coating PES yarns with PPy/PW₁₂O₄₀³⁻, different bands attributed to polypyrrole
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45 appeared in the spectrum (Figure 4-c); the same bands were observed after weaving the
46
47 conductive yarns to produce the satin conductive fabric (Figure 4-d). Observed bands
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49 which were not overlapped with bands of polyester are as follows:
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- 52 • 1540 cm⁻¹, associated to the pyrrole ring stretching vibration (C=C) [23, 24].
 - 53 • 1450 cm⁻¹, C-C stretching [23, 24].
 - 54 • 780, 1045 and 1160 cm⁻¹, associated with bending vibration of pyrrole [23, 24].
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3 The reduction of the different bands attributed to polyester substrate is also noticeable.
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5 The coating of polypyrrole partially avoids the penetration of the infrared radiation to
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7 the polyester substrate, so the intensity of the polyester bands diminishes.
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10 The P-O stretching band of the counter ion at 1075 cm^{-1} was not observed since it
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12 overlaps with the PES band at 1090 cm^{-1} . This is why EDX measurements were carried
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14 out to confirm the incorporation of the counter ion ($\text{PW}_{12}\text{O}_{40}^{3-}$).
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18 19 20 3.3. SEM and EDX characterization

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22 Figure 5 shows the micrographs of twisted PES yarns (Figure 5-a), and twisted PES
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24 yarns and coated with PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$ (Figure 5-b, c, d). In Figure 5-a, a twisted PES
25
26 yarn can be seen, and as can be appreciated the yarn is composed of a bundle of fibers.
27
28 We can also see the twist applied to the yarn during the spinning process. In Figure 5-b,
29
30 the twisted PES yarn after the polymerization of PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$ on its surface is shown.
31
32 Apparently all the yarn is coated with polypyrrole, as can be appreciated in this
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34 micrograph. The presence of globular polypyrrole aggregates is also noticeable on the
35
36 surface of the fibers. These aggregates could not be removed after washing with water
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38 several times. In our previous paper, it was also demonstrated that after domestic
39
40 washing tests its presence was reduced although it was still noticeable [20]. Figure 5-c,
41
42 d show micrographs of the inner fibers of the yarn. On the inner fibers, the coating is
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44 not as good as on the outer fibers. As it can be seen in these micrographs, insufficient
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46 coverage can be observed in some fibers. The layer has not been completely formed in
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48 some zones of the inner fibers due to the twist applied. This makes the fiber bundles
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50 compact and hinders the penetration of the reactants and the oligomers. The presence of
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52 these defects causes a loss of conductivity in relation to the untwisted yarns, as it has
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54 been pointed out previously.
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3 Figure 6 shows the micrographs of untwisted PES yarns (Figure 6-a), untwisted PES
4 yarns coated with PPy/PW₁₂O₄₀³⁻ (Figure 6-b, c), and the satin fabric obtained after
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Figure 6 shows the micrographs of untwisted PES yarns (Figure 6-a), untwisted PES yarns coated with PPy/PW₁₂O₄₀³⁻ (Figure 6-b, c), and the satin fabric obtained after weaving the untwisted conductive yarns (Figure 6-d, e, f). Prior to the polymerization of PPy/PW₁₂O₄₀³⁻, the fibers of polyester are smooth (Figure 6-a). After the formation of the PPy/PW₁₂O₄₀³⁻ coating, the appearance of a continuous and uniform layer of polypyrrole on the fibers of untwisted polyester can be seen (Figure 6-b). The treatment of textile fabrics with polypyrrole does not affect the tactile properties of the textile since inter-fiber bonding does not occur [38]. The layer of polypyrrole formed can be better appreciated in Figure 6-c, where we can see the continuous layer of polypyrrole and the presence of polypyrrole with globular morphology superficially. As in the case of the twisted yarns, the presence of polypyrrole aggregates which could not be removed from the polyester fibers during the washing stage is also noticeable. In the case of the untwisted yarns, all the fibers are properly coated with PPy/PW₁₂O₄₀³⁻ since there is no hindrance for the penetration of the reactants.

Figure 6-d shows the satin fabric after weaving the untwisted polyester yarns coated with PPy/PW₁₂O₄₀³⁻. The vertical fibers are the conductive fibers and the horizontal fibers are the non-conductive polyester fibers. The horizontal fibers appear as brighter zones due to the accumulation of electrons as consequence of the limited polyester conductivity. The weaving process did not produce significant damage to the polypyrrole coating. Only small areas of the coating had been affected by the entire process as can be seen in Figure 6-e. The damaged areas may correspond with the zone of the yarn that is caught by the tweezers of the loom. Figure 6-f shows a fiber damaged by the weaving process, in which the layer of polypyrrole had been partially removed. This allowed the estimation of the thickness of the PPy/PW₁₂O₄₀³⁻ coating (Figure 6-f, inset). The thickness of the coating was approximately 400 nm.

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Figure 7 shows the EDX characterization of the layer of polypyrrole in two different zones; a zone with polypyrrole coating and a zone where the coating of polypyrrole had been damaged due to the weaving process. The EDX spectrum in a zone with polypyrrole coating (Figure 7-a), showed the presence of W, P and O arising from the counter ion ($\text{PW}_{12}\text{O}_{40}^{3-}$). The presence of Fe was also noticeable; this contribution is due to the use of FeCl_3 as an oxidant during the polymerization process. The EDX analysis of the zone where the layer of polypyrrole had been scraped off, showed the decrease of the intensity of the bands of W, P and O (Figure 7-b). The presence of these zones was minimal as mentioned previously, so the weaving process can be considered as a good method to obtain conductive fabrics of large dimensions.

3.4. Electrochemical impedance spectroscopy

Figure 8-a, shows the impedance modulus ($|Z|$) at the different frequencies of the satin fabrics of PES and PES-PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$. As it was explained previously, the impedance modulus (Ω) is equivalent to the surface resistivity (Ω/square), as a square of 1.5 cm was measured. The sample of polyester presents a value of the impedance modulus at low frequencies of around 10^{11} - 10^{12} Ω , typical value of insulating materials. The fabric containing conductive yarns of PPy-PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$, showed values of the impedance modulus lower than 1700 Ω , more than seven orders of magnitude lower than the values of polyester fabric. In our previous work [20], washing fastness and rubbing fastness tests of PES-PPy/ $\text{PW}_{12}\text{O}_{40}^{3-}$ conductive fabrics showed an increase of the surface resistivity of x 2 and x 15 respectively.

An interesting application for conductive textiles is as antistatic materials. Charging of the surface of fibers is excluded with surface resistivity below $5 \cdot 10^9$ Ω/\square [39], so the surface resistivity of our conductive fabrics makes this application feasible.

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3 Figure 8-b shows the phase angle for the different samples measured. The phase angle
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5 obtained for the polyester sample was around 90°, a typical value of insulating
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7 materials; the data at low frequencies is not shown since noise due to the large values of
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9 impedance modulus were observed. The conductive fabric showed values of 0° of phase
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11 angle, this value is typical of conductive materials. So the conductive fabrics act as a
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13 conductive material with a determinate resistance.
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20 3.5. Textile prototypes

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22 Another application for conductive fabrics involves the incorporation of light emitting
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24 diodes (LEDs), for example a dress wired with 50 LEDs has been reported in the
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26 bibliography [1]. Figure 9-a shows two satin fabrics connected to a power supply, one
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28 fabric was connected to the positive pole and the other to the negative pole. The fabrics
29
30 had 1.4 m of length and different LEDs were placed at different distances. After this, the
31
32 two metallic contacts of the LEDs were put in contact with each fabric to close the
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34 circuit; the LEDs were illuminated at a voltage of 3-5 V. Figure 9-b and 9-c, show two
35
36 serge structures in the same fabric, one serge structure was connected to the positive
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38 pole and the other to the negative pole. In this case, the metallic contacts of two LEDs
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40 were introduced inside the yarns of the conductive fabrics. As can be seen, the two
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42 LEDs were also illuminated in this type of structure at a voltage of 5 V. The LEDs can
43
44 be removed and connected in other parts of the conductive textile without any problem.
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46 Figure 9-d shows a circuit with LEDs incorporated; the circuit was embroidered (with
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48 an industrial embroiderer Tajima TMM-HC606) with twisted conductive yarns of PES-
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50 PPy/PW₁₂O₄₀³⁻ on a leather support. It was not possible to embroider with untwisted
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52 yarns, therefore twisted yarns were employed. In the case of the embroidered textile the
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3 voltage applied to illuminate the LEDs was 12 V since the yarns were less conductive
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5 than untwisted yarns as mentioned previously.
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10 11 12 13 **4. Conclusions**

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15 The weaving process of conductive yarns of polyester coated with PPy/PW₁₂O₄₀³⁻ has
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17 been shown to be a good method to obtain conductive fabrics with higher dimensions
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19 than the conductive fabrics obtained by in-situ chemical oxidation methods. The type of
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21 yarn and the sort of fabric structure also influence the conductivity of the fabric. The
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23 best results were obtained with untwisted yarns of polyester and satin structure. The
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25 coating of the yarns has been characterized by means of FTIR-ATR, showing the
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27 presence of different bands attributed to the polypyrrole coating. EDX analyses showed
28
29 the presence of W, P and O bands arising from the counter ion. SEM micrographs
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31 showed that all the fibers were coated by a continuous and smooth layer of polypyrrole.
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33 The weaving process only produced minor defects due to the deterioration of the
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35 polymer coating in the zones where the tweezers of the loom grasped the yarns. EIS
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37 measurements showed the conductive nature of the fabrics obtained. Compared with
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39 polyester, a reduction of the impedance modulus of more than seven orders of
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41 magnitude was obtained. The phase angle obtained for the conducting fabrics was 0°,
42
43 typical value of conductive materials. These values of surface resistivity would allow
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45 the use of these conductive fabrics as antistatic materials, where a surface resistivity
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47 below $5 \cdot 10^9 \Omega/\square$ is required.
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49 LEDs have been also incorporated in the conductive fabrics and they were illuminated
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51 at 5 V. Therefore, the conductive fabrics can serve as an electrical conductor to mount
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53 and connect LEDs and, at the same time they can be easily removed and connected
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3 again. The same principle is applicable for the incorporation of other devices **such as:**
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5 chips, antennae, wireless devices, etc. More work is in progress to evaluate the
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7 durability of the coating as well as its possible improvement by plasma treatment [40].
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10 11 12 13 14 15 **Acknowledgements**

16
17 Tejidos Notex S.L. and its team, especially Pedro Romero Snr, Pedro Romero Jnr, José
18
19 Peris and Eliseo Vilaplana **are acknowledged** for providing the resources for the
20
21 elaboration of the fabrics. Paco Tormo and Juanfran Tormo of Bordados Tormo S.L. are
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23 also acknowledged for providing machinery for embroidering.
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32 **Funding**

33
34 This work was supported by the Spanish Ministerio de Ciencia y Tecnología and
35
36 European Union Funds (FEDER) [grant number CTM2010-18842-C02-02]; and
37
38 Universidad Politécnica de Valencia [grant number PAID-06-10]. J. Molina is grateful
39
40 to the Conselleria d'Educació (Generalitat Valenciana) for the FPI fellowship. A.I. del
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42 Río is grateful to the Spanish Ministerio de Ciencia y Tecnología for the FPI fellowship.
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Figure captions

Figure 1. Types of weave employed: (a) Satin 7e3, (b) serge 29e1, b,1,1,1,27.

Figure 2. Resistance ($k\Omega$) vs. measuring distance (cm) for untwisted and twisted threads coated with PPy/PW₁₂O₄₀³⁻.

Figure 3. Resistance ($k\Omega$) vs. measuring distance (cm) for conductive fabrics obtained by weaving untwisted yarns of polyester coated with PPy/PW₁₂O₄₀³⁻, satin and serge fabrics.

Figure 4. (a) FTIR-ATR spectra of PPy/PW₁₂O₄₀³⁻ powders, (b) untwisted PES yarns, (c) untwisted PES yarns coated with PPy/PW₁₂O₄₀³⁻, and (d) satin fabric of PES-PPy/PW₁₂O₄₀³⁻; resolution 2 cm⁻¹, 400 scans.

Figure 5. SEM micrographs of: (a) twisted PES yarn (x150); (b) twisted PES yarn coated with PPy/PW₁₂O₄₀³⁻ (x100), (c) (x1500); (d) (x2000).

Figure 6. SEM micrographs of: (a) untwisted PES yarn (x200); (b) untwisted PES yarn coated with PPy/PW₁₂O₄₀³⁻ (x200), (c) (x5000); (d) satin fabric obtained by weaving untwisted PES-PPy/PW₁₂O₄₀³⁻ yarns (x50); (e) (x500); (f) (x500), inset x60000.

Figure 7. SEM micrographs and EDX analysis of a satin fabric PES-PPy/PW₁₂O₄₀³⁻: (a) Zone with PPy/PW₁₂O₄₀³⁻ (x500); (b) Zone without PPy/PW₁₂O₄₀³⁻ (x500).

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6 Figure 8. Bode plots for the PES satin fabric and the satin fabric after weaving PES-
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8 PPy/PW₁₂O₄₀³⁻ yarns. Textile measured area 1.5 cm x 1.5 cm. Frequency range from 10⁴
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10 Hz to 10⁻² Hz.
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15 Figure 9. Images showing LEDs mounted on different fabrics: (a) satin, (b) and (c)
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17 serge, (d) circuit embroidered in leather.
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For Peer Review

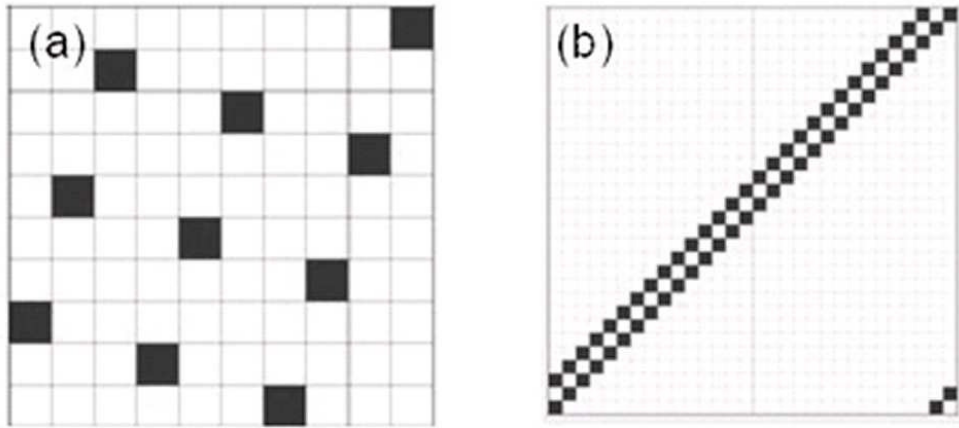


Figure 1. Types of weave employed: (a) Satin 7e3, (b) serge 29e1, b,1,1,1,27.
80x35mm (300 x 300 DPI)

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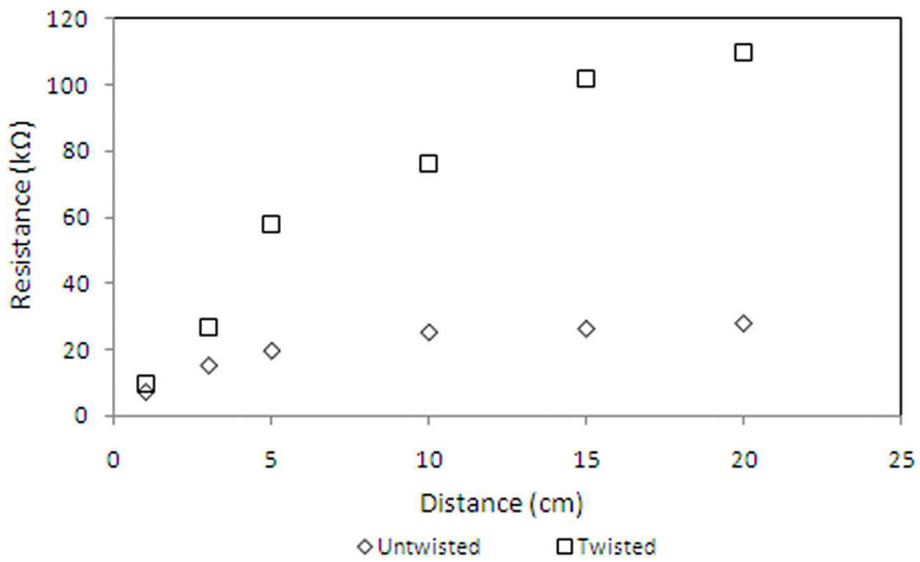


Figure 2. Resistance (kΩ) vs. measuring distance (cm) for untwisted and twisted threads coated with PPy/PW12O403-.
119x72mm (300 x 300 DPI)

Review

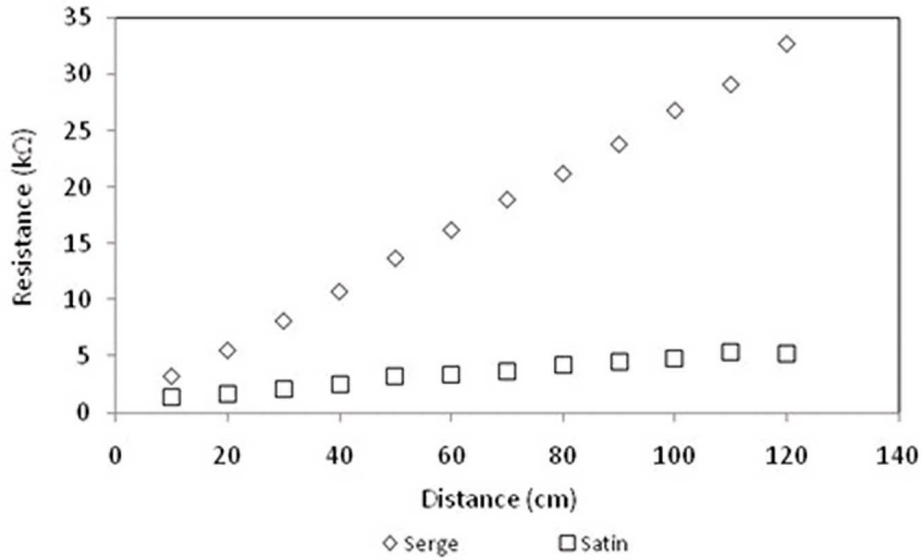


Figure 3. Resistance (kΩ) vs. measuring distance (cm) for conductive fabrics obtained by weaving untwisted yarns of polyester coated with PPy/PW12O403-, satin and serge fabrics. 119x72mm (300 x 300 DPI)

Review

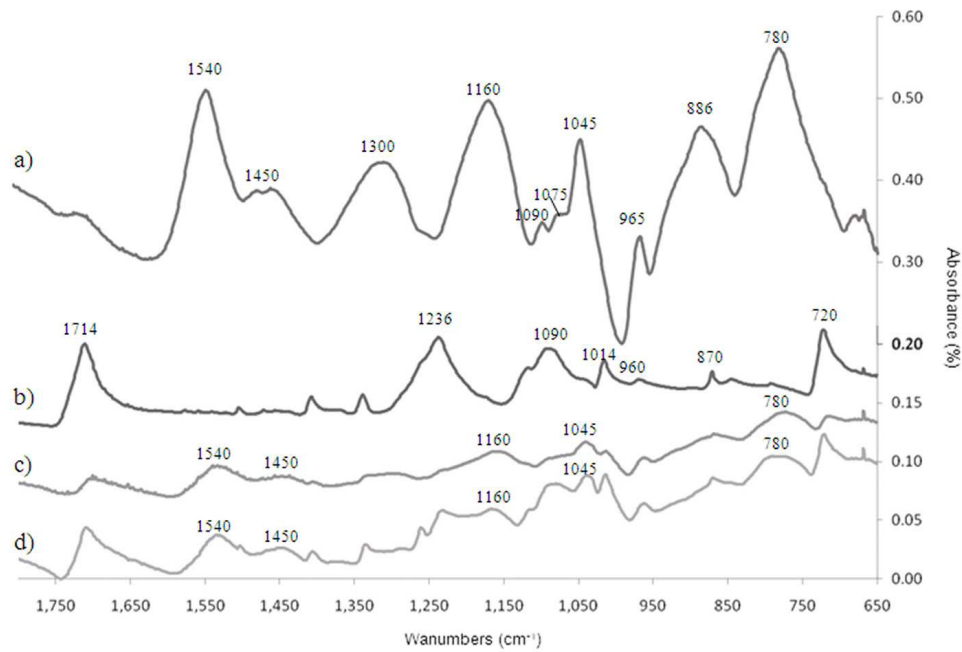


Figure 4. (a) FTIR-ATR spectra of PPy/PW12O403- powders, (b) untwisted PES yarns, (c) untwisted PES yarns coated with PPy/PW12O403-, and (d) satin fabric of PES-PPy/PW12O403-; resolution 2 cm⁻¹, 400 scans. 119x82mm (300 x 300 DPI)

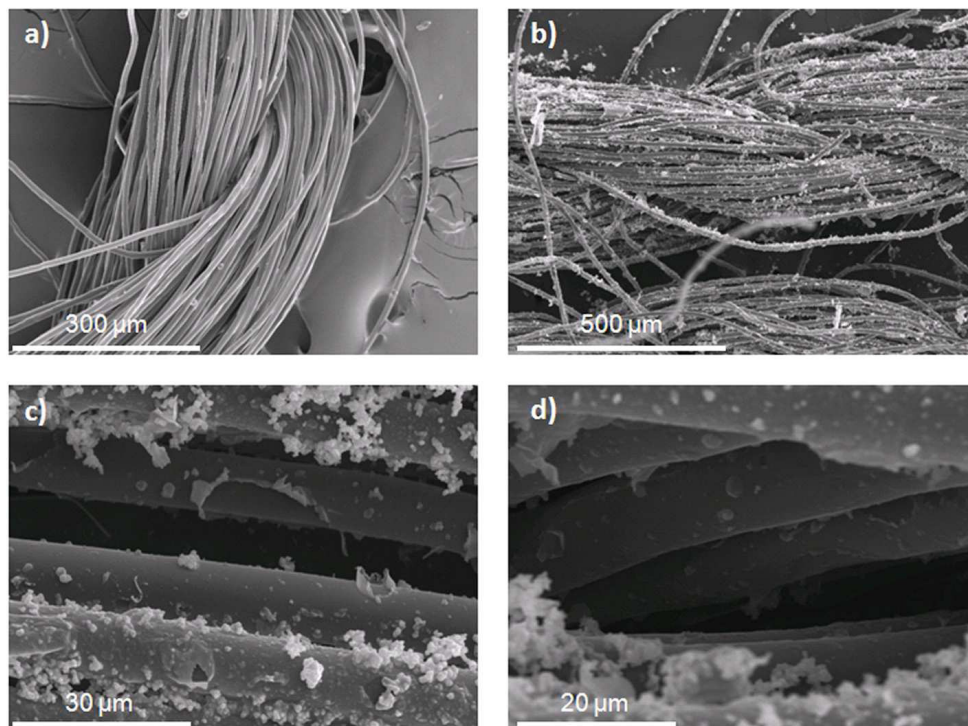


Figure 5. SEM micrographs of: (a) twisted PES yarn (x150); (b) twisted PES yarn coated with PPy/PW12O403- (x100), (c) (x1500); (d) (x2000).
119x90mm (300 x 300 DPI)

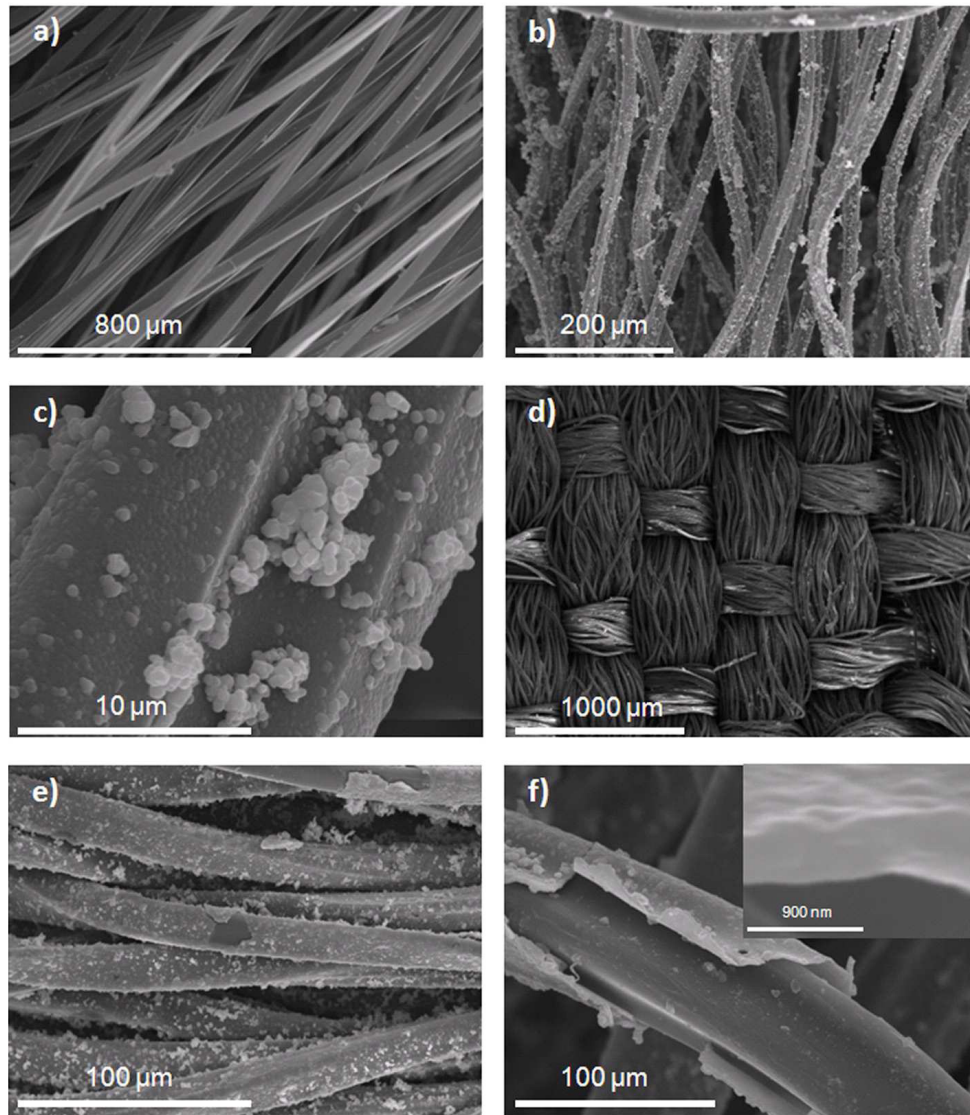


Figure 6. SEM micrographs of: (a) untwisted PES yarn (x200); (b) untwisted PES yarn coated with PPy/PW12O403- (x200), (c) (x5000); (d) satin fabric obtained by weaving untwisted PES-PPy/PW12O403- yarns (x50); (e) (x500); (f) (x500), inset x60000.
119x138mm (300 x 300 DPI)

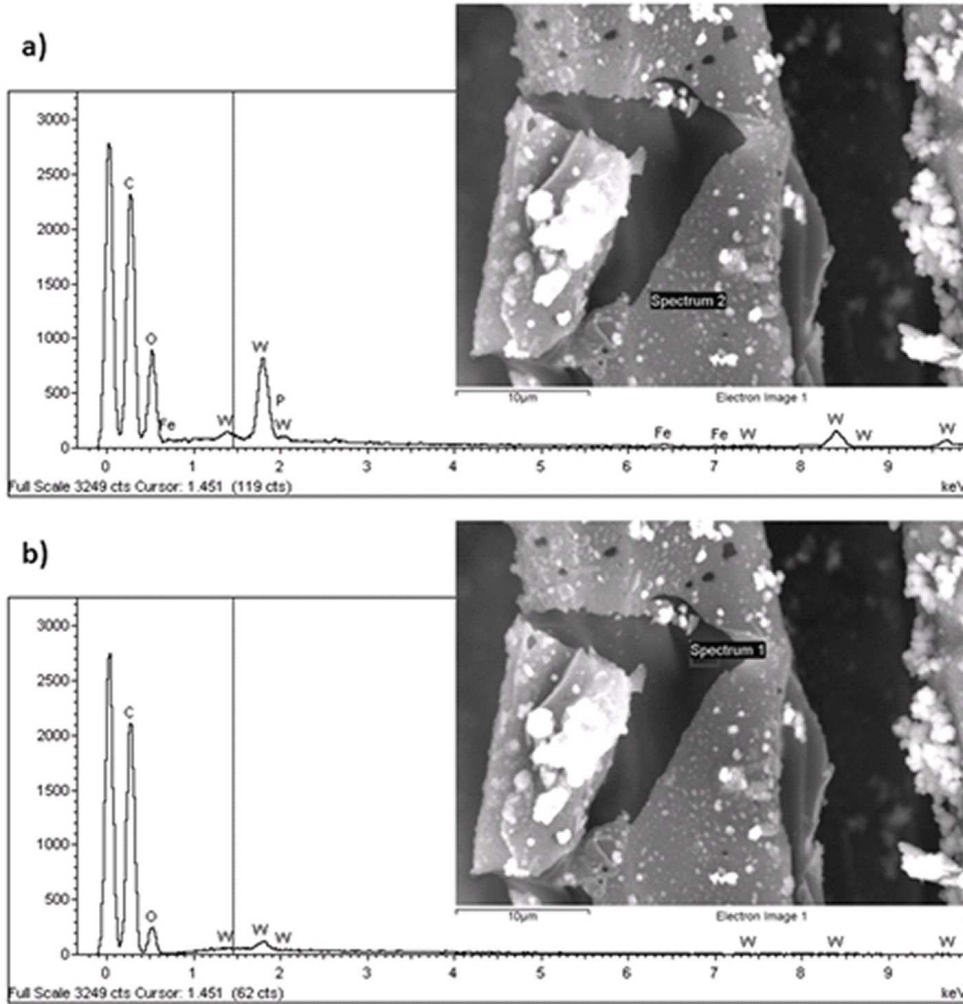


Figure 7. SEM micrographs and EDX analysis of a satin fabric PES-PPy/PW12O403-: (a) Zone with PPy/PW12O403- (x500); (b) Zone without PPy/PW12O403- (x500). 119x124mm (300 x 300 DPI)

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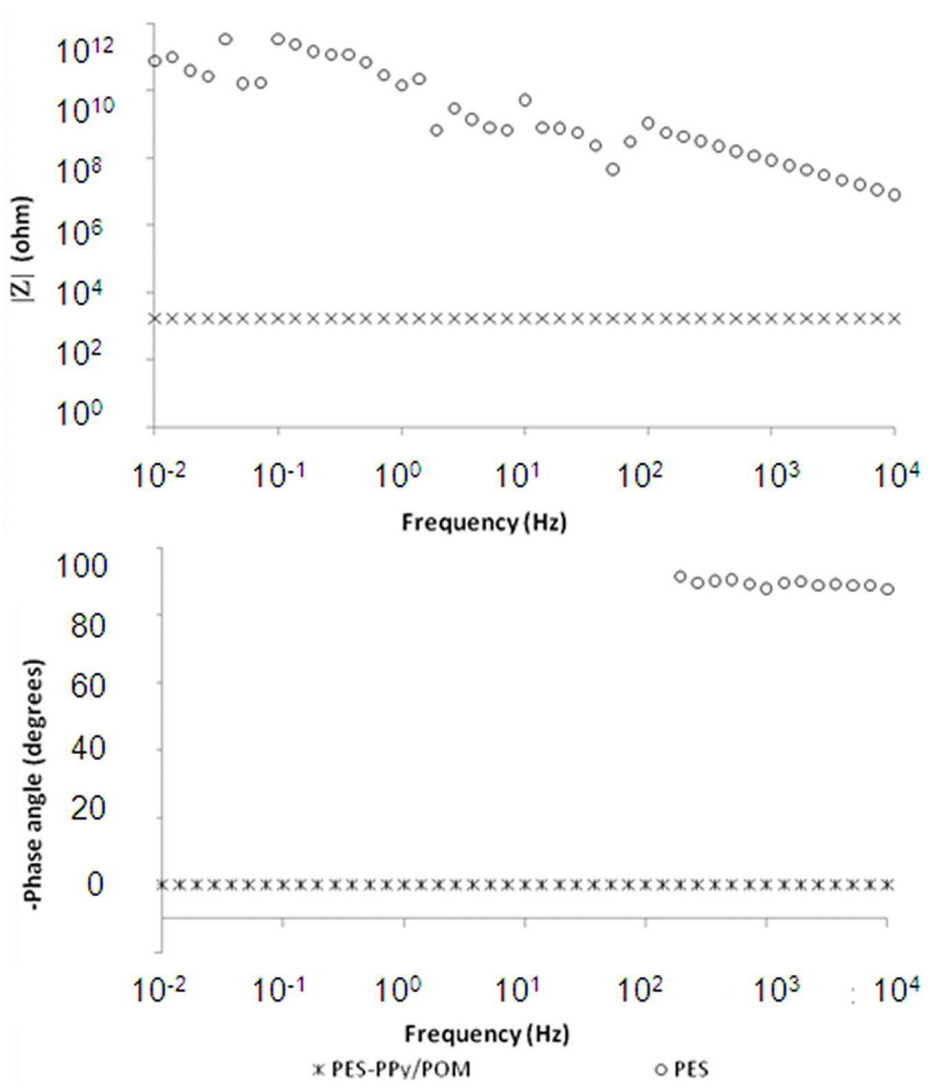


Figure 8. Bode plots for the PES satin fabric and the satin fabric after weaving PES-PPy/PW12O403-yarns. Textile measured area 1.5 cm x 1.5 cm. Frequency range from 104 Hz to 10-2 Hz. 119x136mm (300 x 300 DPI)

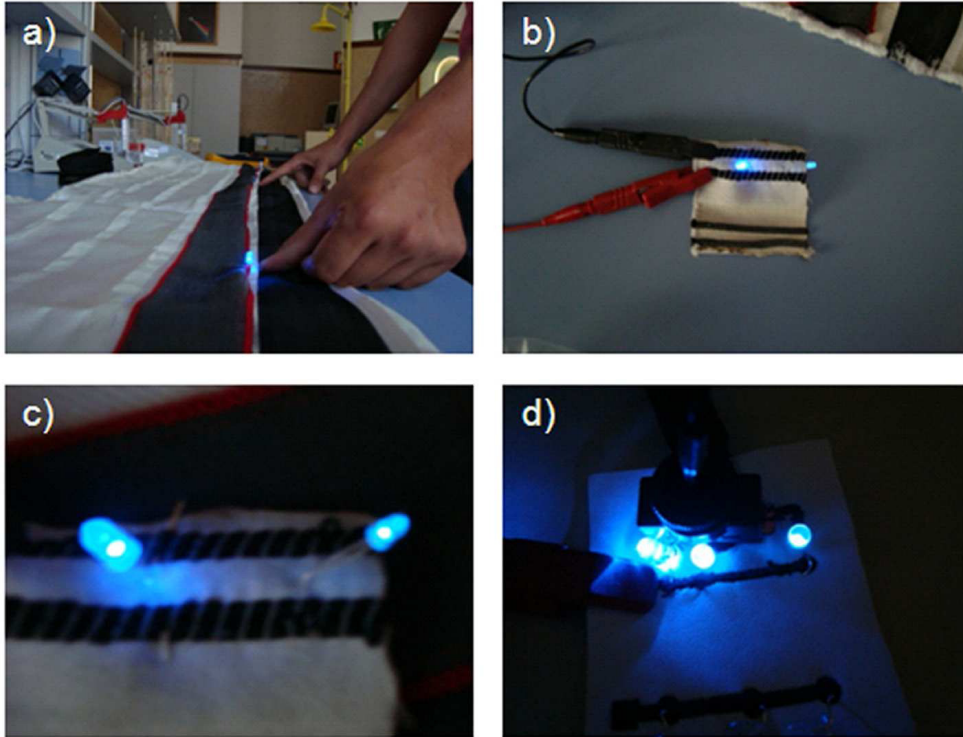


Figure 9. Images showing LEDs mounted on different fabrics: a) satin, b) and c) serge, d) circuit embroidered in leather.
119x91mm (300 x 300 DPI)

Review

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