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# Polymerising pyrrole on polyester textiles and controlling the conductivity through coating thickness

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#### Abstract

The surface resistance of polypyrrole coated polyester fabrics was investigated and related to coating thickness, which was controlled by adjusting the reactant concentrations. The thickness of the coating initially increased rapidly followed by a steady increase when the concentration of pyrrole was larger than a concentration of approximately 0.4 mg/ml. The surface resistance decreased from  $10^{6} \Omega$  to  $10^{3} \Omega$  with increase in pyrrole concentration within 0.2 mg/ml until the concentration reached a value of about 0.4 mg/ml, above which the rate of decrease diminished. The effect of initial treatment with monomer or oxidant prior to polymerisation reaction with regards to thickness and surface resistance was minimal. The immersion time of the textile into the monomer solution prior to polymerisation reaction did not have a significant effect on the abrasion resistance.

Keywords: conducting polymers, textiles, resistivity, coatings.

#### 1. Introduction

Coating conducting polymers on textiles has received considerable attention due to their potential applications in the areas of electromagnetic shielding [1,2], chemical

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sensors [3,4] and heating fabrics [5]. Polyester is one of the most commonly used substrate [1, 6-10]. Other textiles such as nylon [6, 11-13], polyethyleneterephthalate [14], cotton [15], glass wool [16] and wool [15, 17] have also been used as substrates for conducting polymers. The coating technique is often based on in-situ chemical polymerisation of monomers such as pyrrole and aniline in the presence of textiles. It was reported that the in-situ polymerisation process consists of two competitive processes at the initial stage of the polymerisation reaction; physical adsorption of oligomers and polymers onto the textile fibre and polymerisation in solution [11]. The formation of the conductive polymer coating on the textile substrate starts from deposited oligomers, which form islands in the initial stage and finally grow and cover the surface of the substrate completely. Since polymerisation takes place both in solution and on the surface of the substrate, the thickness of the coating layer does not necessarily have a linear relationship with the amount of the reactant. However, the thickness of the conducting layer is a very important factor to decide the conductivity and final application of the textile composite. In this study, the conductivity was investigated and related to thickness of the conducting polymer. The thickness of the polypyrrole (PPy) on the polyester textile was adjusted by changing the reactant concentrations. In addition, the thermostability properties were investigated and related to the reaction conditions.

#### 2. Experimental Details

Reagent grade chemicals were used without any purification. A woven polyester fabric with an average fibre diameter of 18.5 µm was used. The fabric samples were washed in acetone before polypyrrole coating to remove any grease and plastic reagent from the fabric. Infrared spectra were collected using a Perkin Elmer System

2000 Fourier transform infrared spectrometer equipped with a SPECAC model 11900 variable angle attenuated total reflection accessory and a narrow band Mercury Cadmium Telluride detector. All data were recorded using a Germanium Internal Reflective Element set at a 45° angle of incidence. Spectra were collected at a resolution of 4 cm<sup>-1</sup> and 256 scans were co-added. Scanning Electron Microscopy (SEM) images were obtained by using a LEO 1530 field emission gun scanning electron microscope using an accelerating voltage of 5kV at a working distance of around 15mm.

The mean thickness of the polypyrrole coating on Polyethyleneterephthalate (PET) fibres was determined by the Optical Fibre Diameter Analyser (OFDA), which measures the width of fibres by use of a projection microscope and a digital camera. At least 20,000 individual fibres were measured on each fabric. The diameters of both polypyrrole coated fibres and uncoated fibres were determined and the mean thickness of polypyrrole layer was calculated by the difference in radius. In order to clearly identify the conducting polymer layer the fibre cross-section was then prepared for Transmission Electron Microscopy (TEM) study. The samples were embedded by using Spurr's firm resin, polymerised at 60°C, for 18 hours in a hot air oven. Sections approximately 100 nm thick were cut using a DiATOME 45 degree angle ultra diamond knife, operating at a cutting speed of 1mm/second. Sections were collected on deionized water and mounted on 200 mesh copper grids and examined by using a JEOL JEM-2010 Transmission Electron Microscope at an accelerating voltage of 100 keV, using a magnification of 2000X, which was further increased by approximately 19 times when the digital image is collected by the GATAN system. Hence the image had a final magnification approaching 38K (38,000X).

Surface resistance was measured according to the American Association of Textile Chemists and Colourists Test Method 76-1995 [18]. Two rectangular copper electrodes ( $20x30 \text{ mm}^2$ ) separated by 20 mm were placed on the fabric sample ( $30x60 \text{ mm}^2$ ) by a 1kg mass. The resistance was recorded with a Fluke 83 III Multimeter. Surface resistance ( $R_s$ ) is given by:

$$R_s(\Omega / square) = \frac{W}{D}R'$$
(1)

Where, R' is the resistance measured by the multimeter, W and D are the width of the sample and the distance between the two electrodes respectively.

The flat abrasion resistance of coated fabrics was conducted using a Martindale abrasion tester. Each specimen was tested for 1000 abrasion cycles with a pressure of 9 kPa. The colour difference of the samples before and after abrasion tests was determined using a Datacolor Spectraflash 600. The resistance change due to abrasion was calculated using Equation (2).

Decrease of conductivity (%) = 
$$\frac{R - R_0}{R_0} \times 100$$
 (2)

Where, *R* is the resistance after heat treatment and  $R_0$  is the resistance at room temperature.

The thermostability tests were carried out by exposing the samples to 150°C and 200°C for 5 and 15 minutes respectively and observing the corresponding change in surface resistance.

Polymerisation of pyrrole (Py) on textiles has been carried out by oxidation of the monomer in the presence of polyester substrate as reported in literature [11, 12, 14, 19]. The fabric sample was immersed in pyrrole and anthraquinone-2-sulfonic acid sodium salt (AQSA) solution and kept at 4°C for a period of 30 minutes, followed by addition and thorough mixing of the oxidant solution to initiate polymerisation. The polymerisation reaction was performed at 4°C for a period of 2.5 hours. The coated fabric was then washed with water and dried at ambient temperature. The molar ratio of the monomer, dopant and oxidant was kept constant at 1:0.6:2.5 and the textileliquor ratio were kept at 1:50 (w/v g:ml). Different concentrations of pyrrole were used by changing the ratio of pyrrole to textile from 2% to 12% (w/w) in increments of 2%. The procedure was repeated by exchanging the order of monomer and oxidant treatments to the substrate, whereupon, the textile was first soaked into the ferric chloride solution for 30 minutes, and the monomer and dopant solution were then added to start the reaction. In addition, the immersion time of the textile into monomer/dopant solution was varied while keeping the other reaction conditions the same.

#### 3. Result and Discussion

#### **Polymerisation and characterization**

In this study, the polyester textile was initially immersed into either pyrrole/AQSA (monomer/dopant) or the ferric chloride (oxidant) aqueous solutions for a period of time before the polymerisation reaction took place. Mixing the reactant solutions initialised the polymerisation reaction. Two competitive processes occurred simultaneously in the reaction; polymerisation on the surface of the substrate and in the solution. It was observed that the colour changed from white to deep green and

finally black, indicating increase of the polymer deposition during the reaction. At very low monomer concentrations (<0.2 mg/ml) bulk polymerisation was not observed. However, higher concentration of reactants ( $\geq 0.4 \text{ mg/ml}$ ) led to polymers formed in the reaction solution and on the walls of the reaction vessel. The coating process was attributed to a physical adsorption mechanism [11]. It is reasonable to assume that a larger quantity of polymer is adsorbed on the surface of the textile compared to the quantity polymerised in solution due the larger surface area of the textile substrate. Reaction rates and transport of reactants in the solution were determined by the reactant concentrations and temperature. Higher reaction rate led to excess of oligomers that could not be effectively transported onto fibre surface, resulting in formation and accumulation of polymer particles in the solution. When the reaction was carried out in a static vessel, lower reaction temperature decreased both the reaction speed and the transport rate of reactants in the solution. If the rate of polymerisation reaction is suppressed more than the rate of reactant transportation at low temperature, the deposition of the polymer on the surface of the textile would be increased.

Planche et al [20] has studied the reaction kinetics of the polymerisation reaction in water, with ferric chloride as the oxidant of pyrrole. They have shown that a first order kinetics with respect to pyrrole and second order with respect to the FeCl<sub>3</sub> take place in the polymerization reaction. The consumption of the pyrrole follows equation (3).

$$\frac{d[Py]}{dt} = \beta[Py][Fe]^2$$
(3)

Here  $\beta$  is a constant that relates to the ratio of pyrrole and ferric chloride and reaction condition. The recovery of polypyrrole stabilized and kept at a constant value when

the reaction reached to 4 hours. This result is similar to our experimental observations, as there is no polypyrrole formed within the filtered reaction solution after 4 hours in the presence of the textiles.

Figure 1a. SEM image of the polypyrrole coated textile ( $C_{pyrrole} = 0.8 \text{ mg/ml}$ ).

Figure 1b. SEM image of the polypyrrole coated textile ( $C_{pyrrole} = 0.8 \text{ mg/ml}$ ).

Figure 1c. TEM image of the coated fibre ( $C_{pyrrole} = 0.3 \text{ mg/ml}$ ).

SEM images in Figures 1a and 1b show the morphology of the conducting polypyrrole film on the surface of the textile fibre. A similar morphology of the coated surface was obtained irrespective of the reaction sequence; whether the textile was first immersed in pyrrole/dopant or oxidant solution prior to the polymerisation reaction. Some polypyrrole particles, which are polymerised within the solution and deposited on the surface of the fibre were also observed (Figure 1b). These deposits are not adherent to the surface and can be washed off the surface.

TEM image of the cross-section of the coated fibre (Figure 1c) confirms that a layer of conducting polymer was formed on the surface of the polyester fibre. Although TEM is a direct and a more precise way of measuring thickness, the scope of the TEM image is too small to get a statistically accurate result of the thickness of the coating layer. In addition, this method is expensive and time consuming. Due to the fact that the thickness measured on TEM is very close to the OFDA statistical result, OFDA method can provide an easier method of estimating the coating thickness.

#### Average coating thickness

As shown in Figure 2, the polypyrrole coating thickness increases with pyrrole concentration. The higher rate of increase of thickness at lower pyrrole concentrations (Figure 2) suggests a faster deposition of the polymer on the textile surface and reduced bulk polymerisation. When the monomer concentration is larger than 0.4 mg/ml, the rate of increase of the thickness diminishes and polymerisation in the reaction solution is observed. The decrease in the deposition rate is attributed to the rapid reaction rate in higher monomer concentrations resulting in excessive polymer/oligomer generation in the solution. The concentration of approximately 0.4 mg/ml indicates a balance point for transportation and consumption of the monomer on the surface of the textile. In addition, the coating procedure does not seem to have a significant effect on thickness, as a similar trend in the variation of coating thickness with pyrrole concentration is observed when the textile is first immersed into the oxidant solution prior to polymerisation (Figure 2).

Figure 2. Change in thickness of polypyrrole coating with pyrrole concentration.

#### **Surface resistance**

As shown in Figure 3, the surface resistance of the coated PET fabric varies between mega ohms to hundreds of ohms with increase in concentration of reactants. The decrease in surface resistance is due to the increase in the thickness of the conducting polymer layer. The rate of decrease of surface resistance in lower reaction concentrations ( $\leq 0.4 \text{ mg/ml}$ ) is faster than in higher concentrations. The resistance decreases from 10<sup>6</sup>  $\Omega$  to 10<sup>3</sup>  $\Omega$ , about 3 orders of magnitude, with a small increase in the pyrrole concentration. It continues to decrease with further increase in the pyrrole concentration with about one order of magnitude decrease in the range of 0.5-1.5 mg/ml, and finally less than an order of magnitude when the concentration is larger than 1.5 mg/ml. A similar tendency was observed in both of the coating procedures, where the substrate was initially exposed to monomer and oxidant solutions respectively. The reaction conditions, sequence, thickness of the coating, substrate material and the structure of the textile influence the electrical properties of the resulting conductive fabric. A thicker conducting polymer layer also improves the inter-fibre electrical contact, which increases the overall conductivity of the textile. At very dilute reactant concentrations, there were no detectable electrical conductivity until a threshold concentration of about 0.2 mg/ml is reached, after which a sudden rise in conductivity is observed, similar to that observed for the conductive particlefilled thermoplastic composites [21-24]. The rate of increase in conductivity diminished with further increase in reactant concentration. The higher rate of decrease of resistance in the low reaction concentration range suggests that electrical contact is insufficient among the fibres, particularly in warp-weft cross-overs, forming a 'bottleneck' in conductivity. The concentration of 0.2 mg/ml is a critical concentration for the conductivity of this coating system.

Figure 3. Surface resistance versus pyrrole concentration of a PPy coated PET fabric.

#### **Immersion time**

Prolonging immersion time of textiles in the monomer solution was initially thought to improve the penetration of monomer into the fibre, resulting in polymerisation initiating within the fibre followed by polymer growth out of the surface of the fibre. This could improve the interfacial interaction between polypyrrole and textile; hence improve the abrasion fastness of the conductive polymer layer. An experiment was carried out by varying the immersion time of the textile in pyrrole solution and observing the changes in resistance and fastness. As shown in the Figure 4, prolonging the immersion time of the textile in the monomer did not improve the conductivity of the textile as the surface conductivity of the textile is determined by the thickness of the coating and inter-fibre interaction.

Figure 4. Resistance versus immersion time of the textile in the monomer solution

The abrasion fastness test was carried out by a procedure similar to a standard test method applied to dyed textiles. The overall colour and resistance changes before and after the abrasion fastness treatment are listed in Table 1. After abrasion treatment, both the absorption intensity in the entire visible range and the conductivity decreased. These results suggest that the prolonged immersion time of the PET fabric in the reactants did not improve abrasion fastness as the PET fibre cannot be swelled

in aqueous solution to allow pyrrole to migrate into the fibre. A better way would be to establish chemical bonding between conductive polymer and PET fabric substrate.

Table 1. Effect of the immersion time on the fabric abrasion fastness

#### Thermostability

The thermostability was investigated by observing the changes in the surface resistance upon heat treating PPy coated samples at 150°C and 200°C for 15 and 5 minutes respectively. After heat treatment, the resistance of the sample made from low concentrations of pyrrole increased, especially when the concentration of pyrrole was less than 0.4 mg/ml (Figure 5a). As low concentration of pyrrole resulted in a thin conducting layer, the higher rate of increase in the resistance after heat treatment is attributed to damage in the thinner polymer layer, causing poor electrical contact between adjacent fibres. Although dedoping is a possibility for the increased surface resistance as a result of heating, AQSA is much less likely escape from the polymer matrix compared to other dopants such as chloride. The sample treated at 150°C exhibits better stability than the sample treated at 200°C. The increase in resistance was more pronounced for samples heat treated at 200°C.

Figure 5a. Effect of pyrrole concentration on the surface resistance at two different aging temperatures (Initial treatment by pyrrole).

Figure 5b. Effect of pyrrole concentration on the surface resistance at two different aging temperatures (Initial treatment by ferric chloride).

As the concentration of pyrrole increases from about 0.5 to 1.2 mg/ml, the relative surface resistance does not change with pyrrole concentration for the sample initially treated by pyrrole/AQSA solution prior to the polymerisation reaction (Figure 5a). However, the relative surface resistance of the fabric initially treated by the ferric chloride solution continues to decrease as the concentration of pyrrole increases from about 0.5 to 1.5 mg/ml. This may be attributed to the trace Cl<sup>-</sup> ion from ferric chloride, which adds to PPy chain, thus disrupting the conjugation. Faster degradation can be seen at concentrations larger then 1.5 mg/ml. The increase in the degradation rate at higher pyrrole concentrations may be attributed to increase in the thickness of the polypyrrole layer with pyrrole concentration. As the coating thickness increases it is easily peeled off the fibre surface due to thermal expansion of polyester fibres (Figure 6), reducing the effectiveness of electrical contact among the fibres and hence causing increase in resistance. The SEM images for the heated samples show electrical charging in some parts of the fibre, which appear as bright areas (Figure 6). The electrical charging occurs due to high electron accumulation and indicate low electrical conductivity in that area, which confirm the thermal degradation of the conducting polymer. The longer heating time and higher temperature result in increase charging on the sample surface. The loss in conductivity was attributed to oxidation and deprotonation at N-atom of the pyrrole ring [25-28]. Some parts of the coating was found to be considerably damaged by heat treatment, which manifested as electrical charging on the fibre surface as seen in the SEM images in Figure 6. This

may also result from the difference in thermal expansion coefficients of the fibre and PPy, leading to thermal cracking and peeling effects.

Figure 6. Typical SEM micrograph of polypyrrole coated PET fabric heat treated at 150°C for 15 minutes.

As shown in Figure 7, Fourier Transform Infrared (FTIR) spectra of the conducting fabric confirmed that the vibration band at 1559 cm<sup>-1</sup> decreased after heating at 150°C and the band is completely concealed by the background vibrations of polyester after heating at 200°C. The vibration at 1559 cm<sup>-1</sup> is attributed to the conjugated -C=N-/-C=C- stretch of pyrrole ring of the polypyrrole and the decrease in intensity of the band upon heating suggests that the conjugated system of the polypyrrole is damaged. Similar results were reported in literature [29].

Figure 7. FTIR spectra of polypyrrole coated (AQSA doped and oxidized by FeCl<sub>3</sub>) polyester fabric before and after heat treatment.

#### 2. Conclusion

The thickness of the polypyrrole on the polyester textile surface was controlled by adjusting the monomer concentration. Surface resistance changed as the thickness of the coating layer varied from 0.1 to 0.6  $\mu$ m. The pre-polymerisation treatment of textile in the monomer/dopant and oxidant solutions has a minor effect on the

thickness and conductivity of the resultant textile. The immersion time of the textile in the pyrrole solution did not have any significant effect on the abrasion fastness of polypyrrole coated PET fabric. The concentration of about 0.4 mg/ml indicated a critical point for transportation and consumption of the monomer on the surface of the textile. Above this concentration the rate of increase of coating thickness slowed down and bulk polymerisation was observed. There was no detectable electrical conductivity until a threshold concentration of about 0.2 mg/ml is reached, beyond which a sudden rise in conductivity was observed. SEM and FTIR observations revealed that thermal degradation started in isolated patches on the coating by damaging the conjugated system of the PPy.

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### Figures



Figure 1a. SEM image of the polypyrrole coated textile ( $C_{pyrrole} = 0.8 \text{ mg/ml}$ ).



Figure 1b. SEM image of the polypyrrole coated textile ( $C_{pyrrole} = 0.8 \text{ mg/ml}$ ).



Figure 1 c. TEM image of the coated fibre ( $C_{pyrrole} = 0.3 \text{ mg/ml}$ ).



Figure 2. Change in thickness of polypyrrole coating with pyrrole concentration.



Figure 3. Surface resistance versus pyrrole concentration of a PPy coated PET fabric.



Figure 4. Resistance versus immersion time of the textile in the monomer solution



Figure 5a. Effect of pyrrole concentration on the surface resistance at two different aging temperatures (Initial treatment by pyrrole).



Figure 5b. Effect of pyrrole concentration on the surface resistance at two different aging temperatures (Initial treatment by ferric chloride).



Figure 6. Typical SEM micrograph of polypyrrole coated PET fabric heat treated at  $150^{\circ}$ C for 15minutes (C<sub>pyrrole</sub> = 2.4 mg/ml).



Figure 7. FTIR spectra of polypyrrole coated (AQSA doped and oxidized by FeCl<sub>3</sub>) polyester fabric before and after heat treatment.

Immersion time ( h )	Total colour difference	Decrease of conductivity due to abrasion
	before and after abrasion	(%)
0	3.36	30
2	1.67	14
5	0.55	22
10	1.44	36
17	1.54	28
26	1.69	28

Table 1. Effect of the immersion time on the fabric abrasion fastness

#### **Figure and Table Captions**

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