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ARTICLE Alkali Treatment to Maximize Adhesion of Polypyrrole Coatings for Electro-Conductive Textile Materials

Zehra Yildiz^{1*} Ismail Usta¹ Bandu Madhukar Kale² Graham B. Mellen³ Youjiang Wang³

1. Faculty of Technology, Department of Textile Engineering, Marmara University, 34722, Istanbul, Turkey

2. Department of Material Engineering, Technical University of Liberec, Studentska 1402/02 46117 Liberec, Czech Republic

3. School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, 30332 Georgia, USA

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1. Introduction

Polyesters are one of the most important classes of synthetic polymers that are presented in fibers, elastomers, high-performance composites, engineering thermoplastics, and thermosetting resins ^[11]. It has found widespread applications in textile industry including medical and automotive textiles, protective clothing, sports and active wear because of its excellent resistance to chemicals, stretching, abrasion and wrinkling properties ^[2]. Although it exhibits good properties, it has also undesirable hydrophobic and inactive surface characteristics that cause problems in coating and finishing processes. In order to remedy these problems, polyester fiber surfaces have been treated by at-

ABSTRACT

In this paper polyester fabrics were pretreated with alkaline solution to improve the ability for the fabric surface to bond with polypyrrole (PPy) coating layer. In situ chemical oxidative polymerization of pyrrole monomer was performed on alkali treated polyester fabrics. Then the fabrics were characterized by FTIR and XRD analysis. The tensile properties of the yarns in both warp and weft directions were measured after alkali treatment and PPy coating processes. The abrasion resistance test was performed on PPy coated fabrics with and without alkali treatment. The surface electrical resistivity of PPy coated fabrics were searched. The electromagnetic shielding effectiveness (EMSE) properties of fabrics in terms of reflection, absorption and transmission behaviors were also investigated. A significant EMSE value increase (about 27%) was obtained with alkali treatment.

mospheric plasma ^[3], acidic ^[4], enzymatic ^[5, 6], and alkaline ^[7-9] solutions. The alkali treatment of polyester fibers has been widely investigated for the enhancement of dyeability, handle, hydrophilicity, and moisture regain properties of the fibers. During the alkali treatment, hydroxyl anions cause the hydrolytic scission of ester bonds of the polyester polymeric chains, resulting in changes in the polymerization degree and overall crystallinity ^[6].

Recently, usage of electronic devices and wireless systems has increased in daily life, and more attention is paid to electromagnetic interference (EMI) notion. Electromagnetic waves emitting from the electronic devices may lead to malfunction of other electronic equipment and affect human health negatively. Thus the undesirable radiation

Zehra Yildiz,

^{*}Corresponding Author:

Faculty of Technology, Department of Textile Engineering, Marmara University, 34722, Istanbul, Turkey; Email: zehra.yildiz@marmara.edu.tr

should be minimized by shields. Textile substrates are inherently insulator but they can be converted into conductive material by introducing metal fillers/coating ^[10,11] to the structure or by coating the surface with conductive polymers ^[12,13]. Metal coated materials exhibit very high electromagnetic shielding effectiveness (EMSE) about 40-100 dB but their shielding property is mainly based on reflection instead of absorption property because of their shallow skin depth. In contrast, conductive polymer coated textile surfaces show an absorption dominant shielding property towards electromagnetic waves ^[14].

Among the conductive polymers, polypyrrole (PPy) is the most promising one because of its high conductivity, good environmental stability, ease of synthesis, high temperature resistance, and less toxicological properties [14-16]. A number of studies have been conducted on PPy synthesis directly onto textile materials. In situ chemical oxidative polymerization of pyrrole monomer onto polyester fabrics has been performed using para-tolene-2-sulphonic acid monohydrate as dopant and ferric chloride hexahydrate as oxidant. The PPy coated polyester fabrics showed on average 8 dB EMSE value in 1-18 GHz frequency range [17]. Cotton fabrics have been coated by globular PPy and PPy nanotubes with in situ polymerization technique. The conductivity loss after washing and chemical cleaning processes has been investigated. Samples coated with PPy nanotubes exhibited higher fastness property against washing ^[18]. In order to enhance the adhesion of PPy layer on textile surfaces, various studies have been performed. For instance, polyamide 6.6 fabrics have been treated with ethanol, tetrachloroethylene, and formic acid at different times and temperatures. The most effective method on PPy deposition has been found as ethanol treatment ^[19]. In another researches, cold plasma discharge has been used to activate the polyester fabric surface prior to the PPy deposition [20,21]. Pyrrole monomer has been also functionalized by triethoxysilane to enhance the adhesion of PPy layer on polypropylene and viscose fabric surfaces ^[22]. For the catalytic activity enhancement of cotton and linen fabrics, nickel coating via electroless plating method has been employed before the in situ PPy deposition ^[23,24]. Cotton and polyester fabrics that are used in cell cultivation have been treated with NaOH in order to increase the adhesion of PPy layer [25].

Considering the all represented studies from literature, alkali treatment has never been used to improve the electromagnetic shielding activity of PPy coated fabrics. In this study, chemical oxidative polymerization of pyrrole monomer was carried out on polyester woven fabric surfaces. In order to increase the PPy amount and enhance the overall coating quality, samples were pre-treated by alkaline solution prior to the PPy coating. The experimental process can be seen in Scheme 1. By changing the alkali treatment duration, the surface electrical resistivity, EMSE, tensile properties, and abrasion resistance were all investigated.



Scheme 1. Schematic representation of the production of carboxyl groups on PET surface by hydrolysis, and formation of PPy layer on the fabric surface

2. Experimental

In the experimental process, a plain-weave polyethylene terephthalate (PET) fabric (95 g/m², 40 warp/cm, 32 weft/cm) was used. Sodium hydroxide (NaOH), hydrochloric acid (HCl), ethanol, and reagent grade pyrrole (Py) monomer were all purchased from Sigma-Aldrich and used as received. Ferric chloride hexahydrate (FeCl₃.6H₂O) was supplied from Merck.

Alkali treatment was applied to the fabrics prior to the PPy coating process. Fabric samples were cut into 10x10 cm dimensions and immersed in an aqueous solution of NaOH (4 mol/L) at 70 °C for various durations (0.5-2.5 h). They were then washed with ethanol and 1 M HCl solution respectively in order to neutralize the fabric surface, and dried in open air ^[7]. The weight loss after alkali treatment process was measured as about 6.5 %. The in-situ chemical oxidative polymerization process was performed as follows; fabric sample was laid out in a glass vessel containing 1 M Py solution in distilled water at room temperature. After 0.5 h mixing with magnetic stirrer, 0.5 M FeCl, solution in distilled water was added to the vessel dropwise in 30 minutes to initiate the reaction. The polymerization reaction was ended after 2 h when the characteristic black color of PPy was observed in the whole fabric surface. Several washing cycles were applied to the samples with ethanol, 1 M HCl solution, and distilled water, and then the samples were dried in open air [26].

The FTIR spectra (Perkin Elmer Spectrum 100 FTIR) were obtained from the fabric surfaces after alkali treatment and PPy coating processes with the help of an ATR sampling holder. X'Pert³ X-ray diffractometer (PANalytical, USA) was used to analyze crystal structure of untreated, alkali treated and PPy coated fabrics in steps of 0.017. The formation of a PPy layer in both samples with and without

alkali treatment, was investigated by a light microscopy (OLYMPUS CH-2) equipped with image capturing software (Kameram KSCMOS/1). Surface electrical resistivity of the PPy coated samples were measured using a Keithley 6517A Electrometer/High Resistance Meter instrument according to the ASTM D257-07 standard [27]. A Network Analyzer (ROHDE&SCHWARZ) was used for EMSE measurement with the coaxial transmission line method in 0-3 GHz according to ASTM D4935-10 standard ^[28,29]. Tensile testing from the yarns was performed using an Instron 4411 in both warp and weft directions on the alkali treated samples both before and after PPy coatings ^[30]. Each sample was measured 5 times and the average of these results were taken in order to increase the accuracy of the tensile testing. Abrasion resistance test was performed on PPy coated fabrics without alkali treatment and with 2 h alkali treatment according to ISO 5470-1 standard by using a Martindale pilling and abrasion instrument [31].

3. Results and Discussion

3.1 FTIR Spectroscopy

Figure 1 shows the FTIR spectra of the polyester fabrics both before and after alkali treatment as well as PPv coating processes. Considering the untreated and alkali treated fabric samples, there was no significant changes in FTIR spectra. Only slightly changes were observed in peak intensities after alkali treatment process. These intensity changes can be explained by the conformational changes of crystalline and amorphous regions of polyester's structure after the alkali treatment [32]. In the spectra of untreated polvester fabric, the strong carbonyl stretching band at 1719 cm⁻¹ was slightly moved to a lower wavenumber (1717 cm⁻¹) due to the 2 h alkali treatment ^[7]. In the spectra of PPy coated polyester fabric, some newly formed peaks were observed, verifying the PPy formation. The peaks at 735 and 1094 cm⁻¹ are the characteristic peaks of PPv, correspond to the N-H plane inner deformation peaks that occur in the deprotonation process. The peaks at 1037 and 1303 cm⁻¹ come from the =C-H in plane bending vibration from the PPy ring. The peak at 911 cm⁻¹ refers to =C-H out of the plane bending vibration while the peak at 1547 cm⁻¹ belongs to C=C stretching, respectively ^[33, 34].



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Figure 1. FTIR spectra of untreated, 2h alkali treated, and PPy coated polyester fabric samples

3.2 X-Ray Diffraction Analysis

The effect of alkali treatment and PPv coating on polvester fabric was analyzed using X-ray diffraction analysis with a scanning range in 2Θ from 10 to 40° at a scan speed of 10° per minute. Figure 2 (a) and (b) shows X-ray diffraction patterns of untreated, alkali treated and PPv coated polyester fabrics. Figure 2 (a) shows that there is no significant change in polyester after alkali treatment. Apparently, the characteristic peaks of polyester at 18°, 23° and 26° are presented in all X-ray diffraction patterns ^[35]. According to the characteristic XRD patterns of PPv, a broad peak, indicating the amorphous structure of PPy, should be observed in around 23° [16]. Figure 2 (b) does not show any distinct peak of PPv suggesting that the characteristic peak of PPy in diffraction pattern is merged with peaks of polyester in the range between 23-26°. Therefore, it can be said that alkali treatment does not affect the crystal structure of polyester and the peaks of PPy almost overlap with polyester peaks.



Figure 2. (a) X-ray diffraction patterns of untreated, alkali treated and PPy coated polyester fabrics. (b) Peak resolution diffraction patterns of untreated and PPy coated polyester fabrics

3.3 Light Microscopy Imaging

The light microscopy images of the PPy coated samples without alkali treatment (a) and with 2h alkali treatment (b) can be seen in Figure 3. When the sample is directly coated with PPy, the PPy formation occurs only on the surface layer of the fabric. PPy particles cannot penetrate among the warp/weft yarns, and agglomeration of PPy particles can be observed (Figure 3a). Whereas when the alkali treatment was applied prior to the PPy coating process, a more uniform PPy coating can be observed without any agglomeration (Figure 3b). Due to the hydrolysis of polyester surface

by sodium hydroxide solution, carboxyl groups are formed on polyester structure. The formation of strong hydrogen bonding (Scheme 1) between the newly formed COOgroups of the alkali treated polyester and NH groups of the PPy, supplies a good penetration of PPy layer on polyester without any agglomeration (Figure 3b)^[7].



Figure 3. Light microscopy images of the PPy coated samples (a) without and (b) with alkali treatment

3.4 Surface Electrical Resistivity

Table 1 shows the weight uptake and surface electrical resistivity values of the samples after PPy coatings for various alkali treatment times. Accordingly, when a fabric without any alkali treatment was coated with PPy, a weight uptake of 15.8 % was observed. In alkali treated samples, the weight uptake value reached up to 59.7 %. Weight uptake values increased monotonically with increasing alkali treatment time. Considering the surface electrical resistivity values, the lowest resistivity in other words the highest conductivity was observed when the fabric was treated for 2h in alkaline solution prior to the PPy coating process. The sample with 2 h alkali treatment showed more than ten times higher conductivity in comparison to the sample without alkali treatment. Although the sample with 2.5 h alkali treatment showed a great level of PPy uptake, it also showed a lower electrical conductivity. This is due to the excessive PPy agglomeration and non-uniform coating on the fabric surface.

Table 1. Weight increment and surface electrical resistivi-

Alkali Treatment Time (h)	Weight Uptake After PPy Coating (%)	Surface Electrical Resis- tivity (ohm/sq)				
No treatment	15.8	2.9 x 10 ³				
0.5	22.3	1.9 x 10 ³				
1	26.4	1.4 x 10 ³				
1.5	32.1	5.2 x 10 ²				
2	37.3	4.8 x 10 ²				
2.5	59.7	5.6 x 10 ²				

ty values of the samples after PPy coating

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3.5 EMSE Measurement

The EMSE values of the PPy coated fabric samples with various alkali treatment times can be seen in Figure 4. Accordingly, all the samples showed a better EMSE results in the 2000-2500 MHz frequency range. The highest EMSE value of 12.06 dB was observed at 2115 MHz in the 2 h alkali treated sample, whilst the sample without alkali treatment showed the lowest EMSE value of 9.5 dB at this frequency. Alkali treatment leads an up to 27 % increase in EMSE results, due to the deeply penetration and good bonding of PPy layer on polyester structure.



Figure 4. EMSE values of PPy coated fabrics with various alkali treatment durations

Table 2 shows the shielding effectiveness (SE), absorption (A), reflection (R) and transmission (T) values of PPy coated fabrics with various alkali treatment times. Results were given specifically at the frequencies of 1005, 1500, 1800, and 2505 MHz because they are the mostly exposed

 Table 2. Shielding effectiveness (SE), absorption (A), reflection (R) and transmission (T) values of the PPy coated fabrics for various alkali treatment durations

	Frequency (MHz)															
Alkali Treat- ment Time (h)	1005				1500			1800			2505					
	SE (dB)	Α	R	Т	SE (dB)	Α	R	Т	SE (dB)	Α	R	Т	SE (dB)	Α	R	Т
0	8.18	0.73	0.12	0.15	8.72	0.70	0.16	0.14	8.73	0.78	0.08	0.14	9.02	0.85	0.03	0.12
0.5	8.73	0.72	0.14	0.14	9.28	0.69	0.19	0.12	9.25	0.78	0.10	0.12	9.26	0.85	0.03	0.12
1	9.00	0.74	0.13	0.13	9.35	0.70	0.18	0.12	9.28	0.79	0.09	0.12	9.30	0.85	0.03	0.12
1.5	9.94	0.65	0.24	0.11	10.49	0.60	0.31	0.09	10.49	0.74	0.17	0.09	10.61	0.83	0.09	0.08
2	10.50	0.73	0.18	0.09	10.61	0.68	0.23	0.09	10.48	0.79	0.12	0.09	10.68	0.86	0.05	0.09
2.5	9.62	0.72	0.17	0.11	10.27	0.68	0.23	0.09	10.36	0.79	0.12	0.09	10.64	0.85	0.07	0.08

frequencies emitting from electronic devices in daily life. As mentioned, conductive polymer coated materials are intended to absorb the electromagnetic waves instead of reflect or transmit them ^[14]. The highest electromagnetic wave absorption value of 0.86 was obtained in the 2 h alkali treated sample. Increasing the alkali treatment duration caused a small decline in shielding efficiency and absorption values, due to the agglomeration of PPy layer on fabric surface.

3.6 Abrasion and Tensile Testing

Abrasion test was employed in order to observe the effect of alkali treatment on PPy deposition. The abrasion test was performed based on the mass loss percentage after 100 cycle abrasion on a standard wool fabric ^[31]. Accordingly, PPy coated fabrics without alkali treatment and with 2 h alkali treatment showed 13.2 % and 4.5 % weight loss values after 100 cycle abrasion, respectively. This result shows that alkali treatment increased the adhesion of PPy layer on fabric surfaces. In other words, in the alkali treated fabrics, PPy deposition penetrated more deeply on fabric surface whereas in PPy coated fabric without alkali treatment, the weight loss was high, the PPy layer agglomerated on fabric surface and it was easily removed from the surface with abrasion.

Tensile strength and elongation at break values of both warp and weft yarns before and after PPy coating are given in Figure 5 and Figure 6, respectively. In general, alkali treatment and PPy coating processes both caused decline in strength and elongation for all samples because of the brittle nature of PPy structure and degradation/hydrolysis of polyester polymer chains exposed to the alkali solution. In Figure 5, it can be seen that the tensile strength value of the untreated warp yarn is about 37 cN/tex, and after PPy coating it decreased to 35 cN/tex. Considering the alkali treatment process, up to 65 % tensile strength loss was observed in 2.5 h alkali treated sample. In the 2 h alkali treated samples, the PPy coating process caused an increase in tensile strength for both warp and weft yarns in comparison with the uncoated samples because of the uniform PPy formation on the yarn surface.



Figure 5. Tensile strength values of the polyester warp/

weft yarns before and after PPy coating

The elongation at break values of weft and warp yarns before and after the PPy coating process can be seen in Figure 6. For all the samples, the elongation behavior was decreased with increasing alkali treatment time. An average 52 % elongation loss was observed in 2.5h alkali treated sample. The brittleness of PPy layer on the yarn surface prevents the elongation extension of the yarn, thus the elongation value decreased after PPy coating. Alkali treatment resulted in the deformation of polymeric chains and a decrease in the chain length. Therefore the elongation of the yarn decreases after alkali treatment.



Figure 6. Elongation at break values of the polyester warp/weft yarns before and after PPy coating

4. Conclusions

Chemical oxidative polymerization of Py monomer on woven polyester fabric surfaces was performed. Fabrics were treated by alkaline solution prior to the PPy coating process. The effects of the alkali treatment time on EMSE, surface electrical resistivity, tensile properties, and abrasion resistance were investigated. The formation of PPy layer on the fabric surface was proven by FTIR and XRD analysis. Light microscopy images showed that a much more uniform PPy layer was obtained on the alkali treated fabric surface. Weight uptake value after PPy coating process increased with increasing alkali treatment time. The least surface electrical resistivity of 4.8×10^2 ohm/sq and the highest EMSE value of 12.06 dB were obtained in the 2 h alkali treated sample. Although the highest PPy amount was obtained in the 2.5 h alkali treated sample, the EMSE and conductivity values were not the highest in this sample because of the agglomeration of PPy on the surface. Due to the brittle nature of PPy as well as the deformation of the polymeric chains with alkali treatment, both tensile strength and elongation at break values were all decreased with the introduction of the PPy coating and increasing alkali treatment time. A greater weight loss against abrasion was recorded on the PPy coated fabric without alkali treatment. All results are in a good agreement that is alkali treatment caused a better penetration and bonding property of PPy layer to the polyester fabric surface due to the formation of strong hydrogen bonding between newly formed carboxyl groups on polyester and NH groups of PPy.

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