

Structural, Electrical, and Electromagnetic Properties of Cotton Fabrics Coated with Polyaniline and Polypyrrole

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ABSTRACT: In this study, the structural, electrical, and electromagnetic properties of cotton fabrics coated with polyaniline (PAni) and polypyrrole (PPy) were investigated and compared. For the aims, anilin and pyrrole were used as monomers, and *in situ* polymerization on cotton fabric by chemical oxidative polymerization was performed. After production, the structural properties of the fabrics were determined with Fourier transform infrared spectroscopy and X-ray diffraction. In addition, ultraviolet (UV) permeability, tensile strength, colorfastness, and electrical and electromagnetic measurements of the fabric samples were carried out. The resistance values of the cotton fabrics coated with PAni and PPy were found to be 350 and 512 Ω , respectively. The average electromagnetic shielding efficiency and average absorption values of

the cotton fabrics coated with PAni were determined to be 3.8 dB and 48%, respectively, and these values for the cotton fabrics coated with PPy were 6 dB and 50%, respectively. Consequently, a significant difference was not observed between the resistance values and electromagnetic parameters of the fabrics coated with PAni and PPy, although the intact textile characteristics of the fabric coated with PPy were protected and improved, whereas the characteristics of the fabric coated with PAni were inferior. Moreover, we first report that the fabrics coated with conductive polymers had excellent UV-protection properties. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 2003–2010, 2009

Key words: coatings; conducting polymers; FT-IR

INTRODUCTION

The 2000 Nobel laureates Hideki Shirakawa, Alan MacDiarmid, and Alan Heeger¹ were rewarded for their revolutionary discovery of conductive polymers. Intensive studies of conductive polymers have been carried out since then. Among the conductive polymers, polyaniline (PAni), polypyrrole (PPy), and polythiophene have attracted much interest. PAni and PPy have some advantages, such as unique electrochemical properties, good conductivity, easy synthesis, and thermal, environmental, and chemical stability. On the other hand, their low processability and low mechanical properties cause some problems in industrial applications. Although the conductivity values of insulator materials are 10^{-12} S/cm, the values of conductive polymers are in the range 10^{-5} to 10^2 S/cm.² There are some methods for coating with conductive polymers, for instance, electrochemical polymerization, chemical polymerization, graft poly-

merization, and plasma polymerization. Conductive polymers have potential for some applications, for example, as conductive coatings, rechargeable batteries, light-emitting diodes, gas sensors, electrochromic smart glass, electromagnetic shielding materials, and antistatic paints.³ Harmful electromagnetic interferences have increased in this decade while space technology, navigation, telecommunication, plane technology, electronic devices, and wireless systems have rapidly improved.⁴ Electromagnetic waves also have harmful effects on health. The World Health Organization proposed that intensive electromagnetic waves in the environment give rise to biological effects.⁵ Hence, conductive polymers gain importance for use in electromagnetic interference shielding, electromagnetic charge dissipation, and stealth technology.⁴ Moreover, the use of conductive polymers together with textile materials has some advantages, such as flexibility with the elimination of the low mechanical properties of the conductive polymers.

Dhawan et al.⁶ reported that fabrics coated with PAni had -3 to -11 dB of electromagnetic shielding efficiency in the frequency range 8–12 GHz. In their other study,⁷ these authors determined that the shielding efficiency values of silica and polyester

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fabrics coated with PANi were 35 and 21 dB at 101 GHz, respectively. Marchant et al.⁸ found a -13 dB of reflection loss of E-glass fabric coated with PPy at 2–18 GHz. Hakansson et al.⁹ reported -8.68 dB shielding efficiency values for cotton fabric coated with PPy at 1–18 GHz. These other authors measured 36 dB shielding efficiency values for polyester fabric coated with PPy with chemical and electrochemical methods up to a 1.5-GHz frequency with a wide frequency range.¹⁰ Until now, the electrical and electromagnetic properties of fabrics coated with PANi and PPy have not been compared, whereas a lot of researchers have studied the electrical and electromagnetic properties of fabrics coated with PANi or PPy. Furthermore, there is some opening for the textile characteristics of fabrics coated with conductive polymers in the literature.

In our study, cotton fabric was coated with PANi and PPy polymers with the chemical oxidative polymerization method. After the production process, the structural properties of the fabrics were determined with Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD). Moreover, ultraviolet (UV) permeability, tensile strength, colorfastness, and electrical and electromagnetic measurements of the fabric samples were carried out. Thus, we aimed to characterize and compare the structural, electrical, and electromagnetic properties of the cotton fabrics coated with PANi and PPy in this study.

EXPERIMENTAL

Materials

Scoured canvas fabric (panama weave, 239 g/m², 22 ends/cm, and 22 picks/cm) was used in this research. Aniline ($\geq 99\%$, Fluka, Steinheim, Germany) and pyrrole (98%, Aldrich, Steinheim, Germany) as monomers were distilled *in vacuo* before use. The other chemicals included hydrochloric acid (37%, Riedel-de Haen, Steinheim, Germany), ammonium peroxydisulfate (APS; 98%, American Chemical Society reagent, Sigma-Aldrich, Steinheim, Germany), and iron(III) chloride hexahydrate ($\geq 99\%$, Riedel-de Haen, Steinheim, Germany); all were reagent grade. Hydrochloric acid as a protonic acid and APS and iron(III) chloride hexahydrate as an oxidant were used for the coating process.

Coating process

The polymerization of aniline on the fabrics samples was carried out by a chemical oxidative polymerization process. A 1M hydrochloride solution was prepared. The pH meter (J. P. Selecta, Barcelona, Spain) was used to measure the pH values of acidic solu-

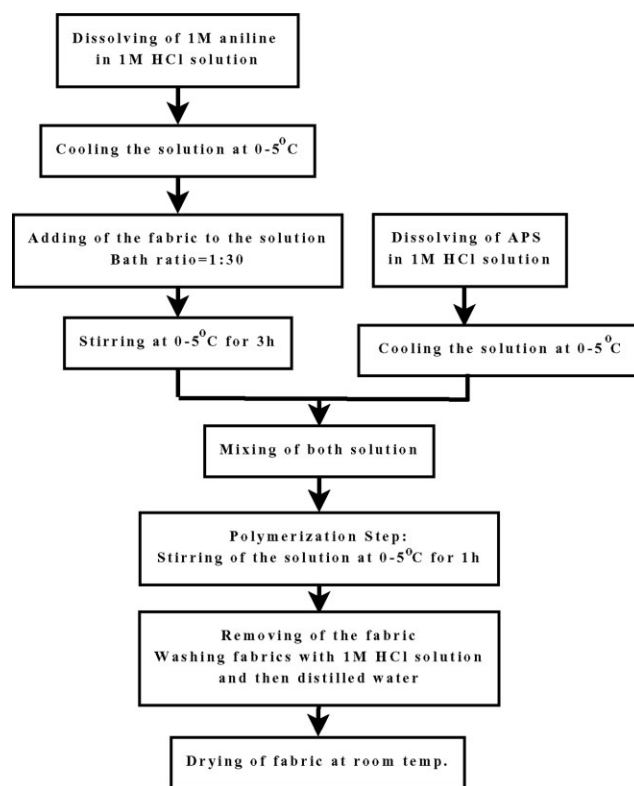


Figure 1 Flow chart of the PANi-coating process on cotton fabric.

tions and polymerization solutions. The pH value of the acidic solution was determined to be 0.15. Aniline (1M) was added to the acidic solution. The pH value of the aniline solution was measured as 3.62. The solution was cooled at 0–5°C. After that, the fabric samples were soaked in the solution for 3 h at 0–5°C. APS (1M) was separately added to a precooled (0–5°C) 1M hydrochloride solution (pH 0.15), and subsequently, the APS solution was gradually added to the aniline solution to polymerize the aniline. The oxidant-to-aniline ratio was maintained at 1 : 1. The pH value of the polymerization solution was determined to be 0.15. Polymerization occurred with continuous mild stirring, and after the addition of all of the oxidant solution, the mixture was further stirred for 1 h at 0–5°C to complete the polymerization. These samples were then thoroughly washed with a sufficient quantity of a 1M hydrochloride solution in many portions to remove any unreacted monomer and excess APS and with an equal volume of distilled water to remove excess acid molecules. The fabric samples were various shades of emeraldine green, which resembled PANi and thus proved that PANi deposition had indeed occurred. The final substrate-to-bath ratio was maintained at 1 : 30, and the final concentrations of aniline and oxidant were 0.5M. Figure 1 schematically shows the steps of process of PANi coating on the fabrics.^{11,12}

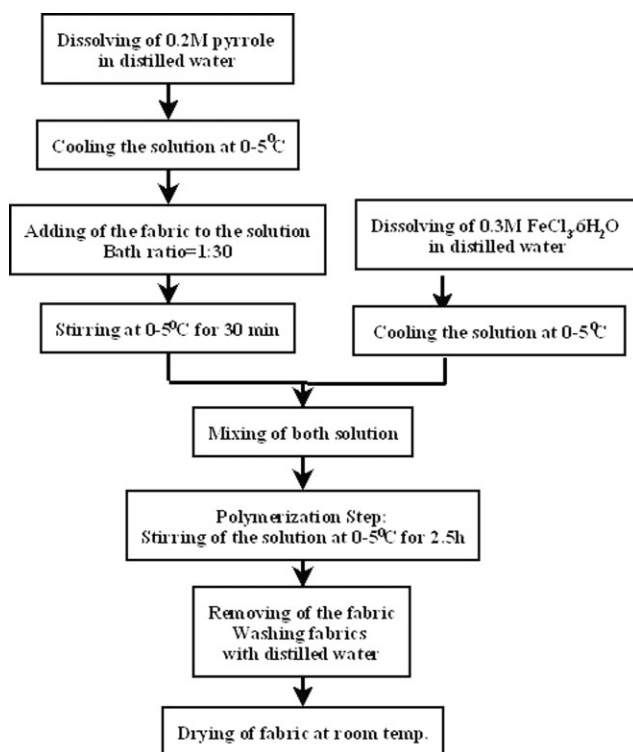


Figure 2 Flow chart of the PPy-coating process on cotton fabric.

The polymerization of pyrrole on the fabrics samples was also carried out by a chemical oxidative polymerization process. Pyrrole (0.2M) was dissolved in distilled water. The pH value of the pyrrole solution was measured as 5. The solution was cooled at 0–5°C. After that, the fabric samples were soaked in the solution for 30 min at 0–5°C. Iron chloride hexahydrate (0.3M) was separately dissolved in distilled water at 0–5°C. Both solutions were mixed. The pH value of the polymerization solution was determined to be 1.37. The mixed solution was stirred for 2.5 h at 0–5°C to complete the polymerization. The final substrate-to-bath ratio was maintained at 1 : 30, and the final concentrations of pyrrole and oxidant were 0.1 and 0.15M, respectively. The fabric samples were removed from the solution and then washed with distilled water. The color of the fabric samples was black.¹³ Figure 2 schematically denotes the process steps of PPy coating on the fabrics.

Characterization

FTIR (PerkinElmer, Inc., Beaconsfields, United Kingdom) absorption spectra of the fabric samples were measured over the range 4000–400 cm^{-1} at room temperature in attenuated total reflectance mode at a resolution of 2 cm^{-1} . The XRD patterns of the fabric samples were obtained by with a Rigaku D (Max-2200/PC model XRD, Tokyo, Japan) X-ray diffractometer at 40 kV and 20 mA with monochromatic

Cu K α irradiation ($\lambda = 0.15418$ nm) by both the θ – 2θ mode and the 2θ scan mode with a scan speed of 8°/min. Thin-film XRD geometry, where the incident angle was fixed at 1°, was used to collect data from only the thin films.

The UV-protection characteristics of the coated fabrics were determined according to the Australian/New Zealand standard AS/NZS 4399 : 1996 with a Camspec M350 ultraviolet–visible spectrophotometer.¹⁴ The washing-fastnesses of the fabric samples were determined according to the BS EN ISO 105-C06-A1S standard (without balls) with a Linitest Plus apparatus (Atlas, Gelnhausen, Germany).¹⁵ The colorfastness values to the rubbing of the fabric samples were determined according to the BS EN ISO 105-X12 standard with a crockmeter (AATCC, Atlas Electric Devices Co., Chicago, IL).¹⁶ The colorfastness values to light of the fabric samples were determined according to the BS EN ISO 105-B02 standard with an Atlas Xenotest Alpha Light Exposure and Weathering Test Instrument (Etki Corp., Istanbul, Turkey).¹⁷ The tensile properties (in the warp direction) of the fabric samples were determined with an Instron (USA) 4411 tester according to ASTM D 5035-90 (strip test) for three repetitions at room temperature. The tensile strength and extension of the fabrics were evaluated in this respect.¹⁸

The add-on ($W_{\text{add-on}}$) values of the fabric samples were calculated as follows:

$$W_{\text{add-on}} (\%) = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

where W_1 is the dry weight of the untreated fabric and W_2 is the dry weight of the treated fabric. The treated and untreated fabrics were conditioned in a standard atmosphere of $20 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ relative humidity before weighing. Thus, the dry weights of fabrics were determined.¹⁹

The resistance values of the fabric samples and the pellets produced from PANi powders were measured with an ohmmeter (Brymen BM 805 model digital multimeter, Taipei, Taiwan) by a two-probe resistance measurement method for a 1-cm distance. The electromagnetic parameters of the fabrics were measured with the transmission/reflection method in the region 6–14 GHz, 5–6 GHz, and 50 MHz–4 GHz with a HP8720D network analyzer (Agilent Technologies, Santa Clara, CA) and a coaxial line fixture.^{10,12,20}

RESULTS AND DISCUSSION

FTIR spectroscopy

The FTIR spectra of the cotton fabric coated with PANi and PPy and bare fabric are illustrated in

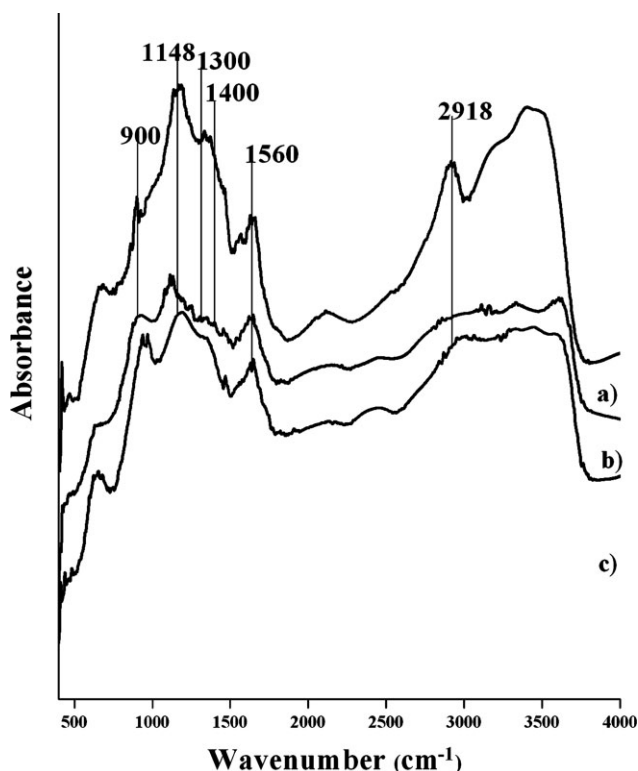


Figure 3 FTIR spectra of (a) bare fabric and (b,c) cotton fabrics coated with PPy and PANi, respectively.

Figure 3. The characteristic bands of PPy at 1560, 1400, 1300, 1170, and 900 cm^{-1} were observed in the fabric coated with PPy. The peaks overlapped the peaks of the cotton.^{21,22} Furthermore, the bands at 1562 and 1293 cm^{-1} for the fabric coated with PANi were attributed to the C=N stretching modes of quinoid rings and C-N stretching modes of benzenoid rings.²³ The N=Q=N stretching of the quinonoid units of PANi due to electron delocalization of the fabric coated with PANi were observed as peaks at 1148 cm^{-1} .¹¹ The peak at 2918 cm^{-1} of the bare fabric was due to the CH_2 antisymmetric stretching vibration of secondary CH_2OH groups in the glucose units of cellulose. The intensity of the peak was significantly reduced in the spectra of the fabrics coated with PANi and PPy because of the interaction of PANi and PPy with CH_2OH groups in the glucose units of cellulose. Hence, we deduced that PANi and PPy were attached to cellulose by hydrogen bridges over OH of CH_2OH ; this was in agreement with Bhat et al.¹¹ However, all of the CH_2OH groups could not completely interact with the conductive polymers because there was still a small peak of the fabric coated with conductive polymers at the wave number shown in Figure 3. Some CH_2OH groups in the glucose units of cellulose were kept without interaction.

XRD analysis

XRD patterns of the cotton fabrics coated with PANi and PPy are given in Figure 4. The PANi displayed a few broad peaks at 15, 21, and 26°. The peaks of PANi on the coated fabric at 15 and 21° overlapped with the peaks of cotton, whereas the broad peak at 26° of the fabric coated with PANi confirmed the presence of PANi and its amorphous form. The PPy peaks on the coated fabric were also superimposed on the peaks of the cotton.²⁷

UV protection

The ultraviolet protection factor (UPF), mean UVA, and mean UVB values of the cotton fabrics coated with PANi and PPy and the bare fabric are given in Table I. Optical whitening agents and various dye-stuffs as UV absorbers have been reported.¹⁹ It was first determined that the cotton fabrics coated with PANi and PPy had excellent UV-protection properties with a 50+ UPF.

Tensile properties

In Table I, the tensile strength and elongation values of the cotton fabrics coated with PANi and PPy and the bare fabric are given. After coating with PPy, the tensile strength values of the fabric samples increased from 94.23 to 106.8 kgf, whereas the values decreased from 94.23 to 60.47 kgf after coating with PANi. The elongation values to break decreased from 23.71 to 18.39 and 14.85% of the cotton fabric coated with PPy and PANi, respectively. The significant decrease in the tensile strength values of the

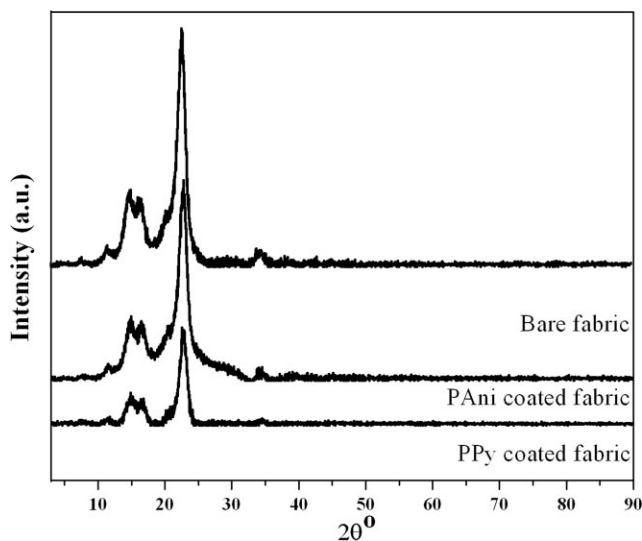


Figure 4 XRD patterns of bare fabric and cotton fabrics coated with PANi and PPy.

TABLE I
Color Fastness Against Washing and Rubbing, Tensile Strength and Elongation Values, UPF Values, and Mean UVA and UVB Values (Standard AS/NZS 4399 : 1996) for Fabrics Coated with PPy and PANi and Bare Fabric

	Light fastness	Rubbing fastness		Washing fastness		Tensile strength [kgf (SD)]	Elongation [% (SD)]	UPF	Mean UVA (%)	Mean UVB (%)
		Wet	Dry	Color Staining	Change					
Bare fabric	—	—	—	—	—	94.23 (0.56)	23.71 (0.42)	5	19.7	9.5
PPy-coated fabric	6	3	3	5	3	106.8 (1.55)	18.39 (0.015)	50+	0.1	0.1
PAni-coated fabric	7	2	3	4/5	1 (bluish shade)	60.47 (5.15)	14.85 (0.18)	50+	0.0	0.0

SD = standard deviation.

fabric samples coated with PANi resulted from the very low pH values of the process (pH 0.15). Because cotton is very sensitive to the effect of dilute mineral acids such as hydrochloric acid and is degraded by acid with the hydrolysis of glycosidic linkages, hydrocelluloses are formed. The formation of the hydrocelluloses causes the losses of weight and tensile strength in the cotton.^{28–30}

Colorfastness

The colorfastness values to washing, rubbing, and light of the fabrics coated with PPy and PANi are shown in Table I. After a washing-fastness test, the color of the fabric sample coated with PANi changed to blue, whereas the PANi film on the fabric turned to a leucoemeraldine form, which was an insulating form of PANi because the detergent used for the washing-fastness test had a basic nature that caused a dedoping effect on the PANi film. Hence, the fabric samples coated with PANi lost their conductivity after the washing-fastness test. The resistance values of the fabric samples coated with PPy were also measured after the washing-fastness test and are mentioned in the following section. The fastnesses to color staining of the fabric samples were very high, at 5 and 4/5, whereas the fastnesses to color change were as low as 3 and 1 for the fabrics coated with PPy and PANi, respectively. The colorfastness values to rubbing of both coated fabric samples were as low as 3 gray scale degrees. The fabric samples coated with PPy and PANi had moderate lightfastness values at 6 and 7, respectively.

TABLE II
Resistance and Add-On Values for the Fabrics Coated with PPy and PANi and Bare Fabric

	Resistance (Ω)	Add-on (%)
Bare fabric	Over the range	—
PPy-coated fabric	512	8.3
PAni-coated fabric	350	9.03

Electrical properties

In Table II, the resistance and add-on values of the fabrics coated with PPy and PANi are shown. The resistance values of the fabrics coated with PPy and PANi were 512 and 350 Ω , respectively, whereas the bare fabric had a resistance value higher than 10⁹ Ω . In addition, the fabric samples coated with PANi lost their conductivity after the washing-fastness test. The resistance values of the fabric samples coated with PPy increased from 512 Ω to 15.4 k Ω after the washing-fastness test.

Electromagnetic properties

Figures 5–7 depict the reflection loss, shielding efficiency, and absorption values of the cotton fabrics coated with PANi and PPy and the bare fabric in the 6–14 GHz frequency range. The mean shielding efficiency, reflection loss, and absorption values of the cotton fabric coated with PPy, PANi, and bare fabric

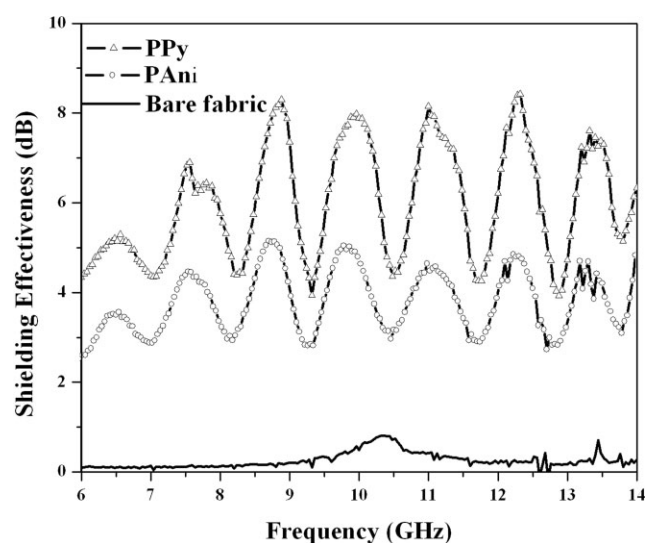


Figure 5 Graph of the shielding efficiency of cotton fabrics coated with PANi and PPy and bare fabric in the frequency range of 6–14 GHz.

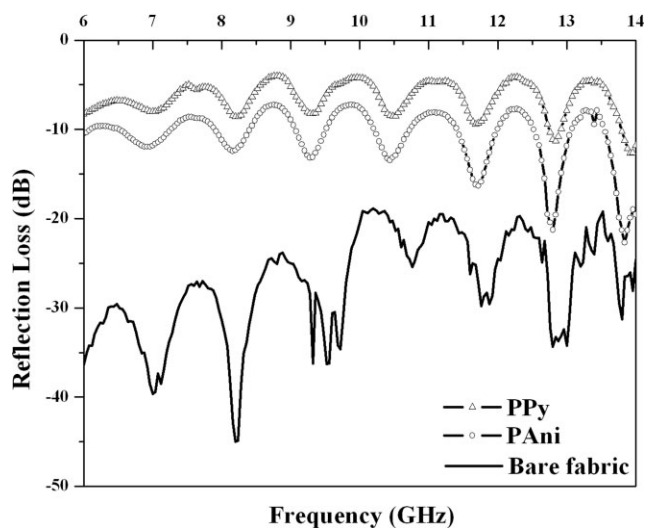


Figure 6 Graph of the reflection loss of cotton fabrics coated with PANi and PPy and bare fabric in the frequency range of 6–14 GHz.

were as follows: 6, 3.8, and 0.26 dB; -6.5 , 27, and -11 dB; and 50, 48, and 2%, respectively.

The reflection loss, shielding efficiency, and absorption values of the cotton fabric coated with PANi are illustrated in different frequency ranges, such as 6–14 GHz, 5–6 GHz, and 50 MHz–4 GHz, in Figures 8–10. Mean absorbance values of the fabric samples of 48, 43, and 42%, mean shielding efficiency values of the samples of 3.8, 12, and 12 dB, and mean reflection loss values of the samples of -11 , 2.88, and -2.86 dB were determined at 6–14 GHz, 5–6 GHz, and 50 MHz–4 GHz, respectively. We deduced that the shielding efficiency values of the samples increased, their reflection loss values (a

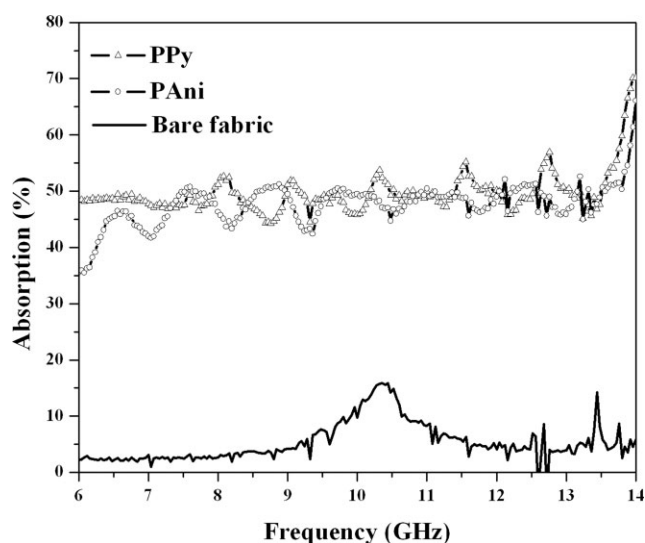


Figure 7 Graph of the absorption of cotton fabrics coated with PANi and PPy and bare fabric in the frequency range of 6–14 GHz.

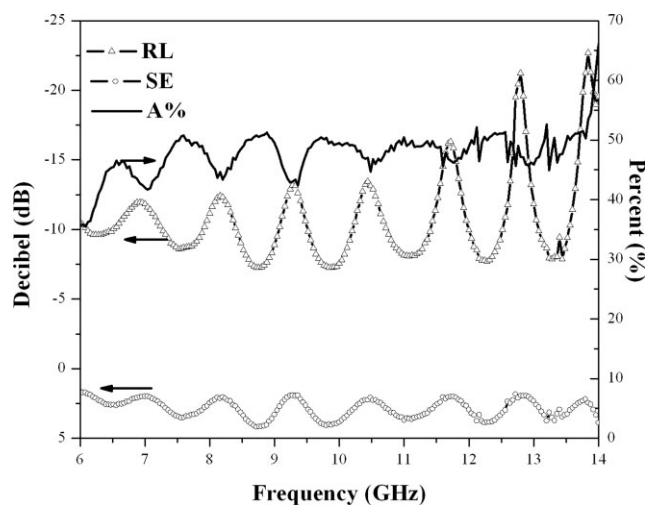


Figure 8 Graph of the reflection loss (RL), shielding efficiency (SE), and absorption (A%) of cotton fabric coated with PANi in the frequency range of 6–14 GHz.

minus sign shows the loss values) decreased, and their absorption values decreased, whereas the frequency values decreased.

Moreover, the cotton fabrics were coated with polyurethane (Tubicoat PU80, CHT R. Beitlich GmbH, Tuebingen, Germany) after they were coated with PANi. The fabric samples were dipped in the polyurethane, squeezed at 0.5 kg/cm^2 of nip pressure, and dried at 150°C for 5 min. The electromagnetic properties of these fabric samples are shown in Figure 11. The cotton fabrics coated with onyl polyurethane and polyurethane and PANi had 4 and 8% mean absorbance values in a 6–14 GHz frequency range, respectively. The values were very low compared to

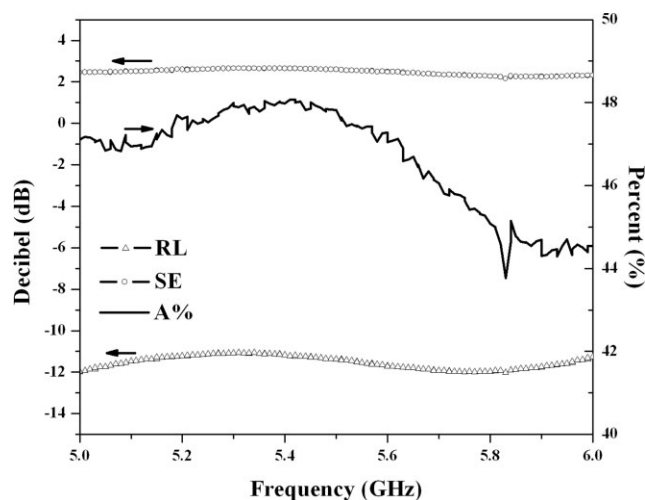


Figure 9 Graph of the reflection loss (RL), shielding efficiency (SE), and absorption (A%) of cotton fabric coated with PANi in the frequency range of 5–6 GHz.

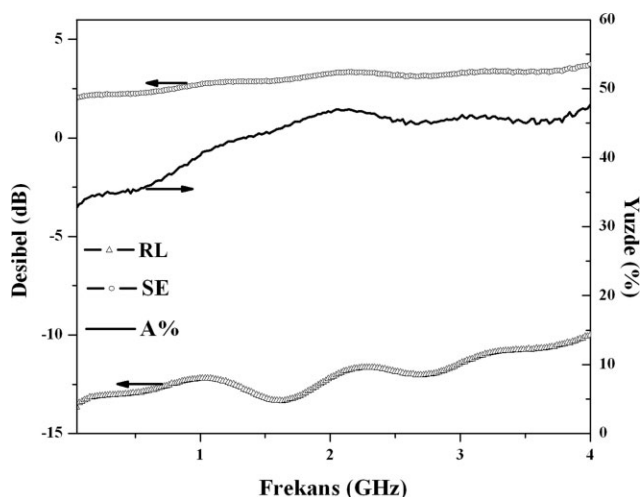


Figure 10 Graph of the reflection loss (RL), shielding efficiency (SE), and absorption (A%) of cotton fabric coated with PANi in the frequency range of 50 MHz to 4 GHz.

that of the cotton fabric coated only with PANi (48%). The electromagnetic properties of the cotton samples coated with only polyurethane were similar to that of cotton samples coated with polyurethane and PANi. We deduced that the top coating on the fabric significantly affected the electromagnetic properties of the fabric.

In addition, the electromagnetic properties of the samples were determined when fabrics with different conductivities and absorbance properties were folded, and a metal plate [perfectly electrical conductor (PEC)] was put back into the fabric. Figures 12 and 13 show the different sample designs and

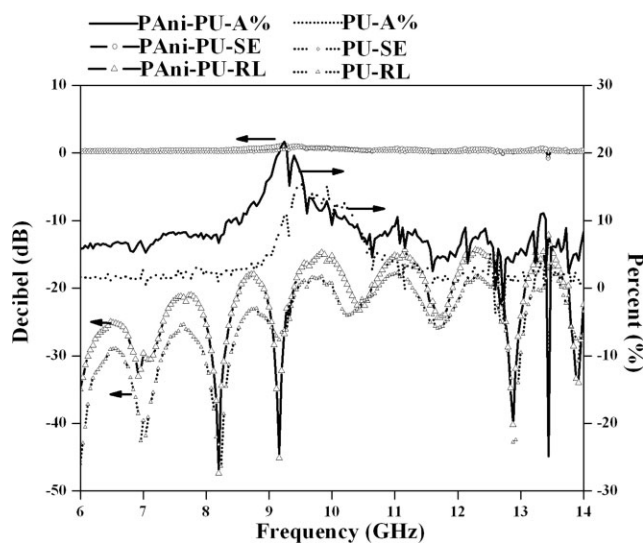


Figure 11 Graph of the reflection loss (RL), shielding efficiency (SE), and absorption (A%) of cotton fabric coated with polyurethane (PU) and/or PANi in the frequency range of 6–14 GHz.

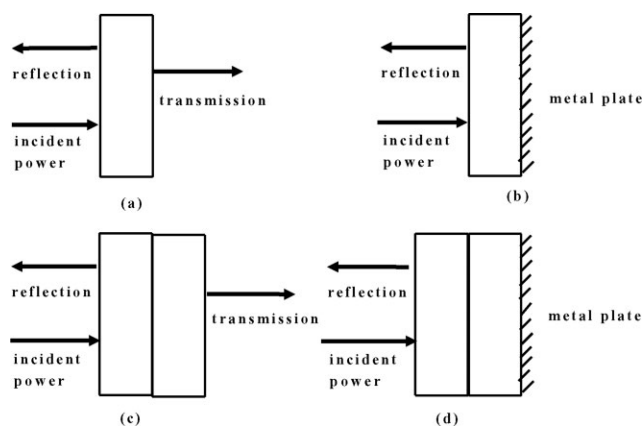


Figure 12 Schematic images of different sample designs for the measurement of electromagnetic parameters in the frequency range of 6–14 GHz: (a) PANi, (b) PANi-PEC, (c) PANi-PANi, and (d) PANi-PANi-PEC.

their absorbance values for the measurement of electromagnetic parameters in a 6–14 GHz frequency range, respectively.

In Table III, mean absorption, reflection loss, and shielding efficiency values for different sample designs in a 6–14 GHz frequency range are given. When a metal plate was put back into the fabrics, their mean absorbance values decreased from 47.66 to 29.27%. The mean absorbance values of the cotton fabric coated with PANi increased from 47.66 to 57.04% with two folds in the fabric. When the metal plate was put back into the twice-folded fabrics coated with PANi, their mean absorbance values decreased from 57.04 to 47.47%. This drop in the mean absorbance values of the fabrics when the

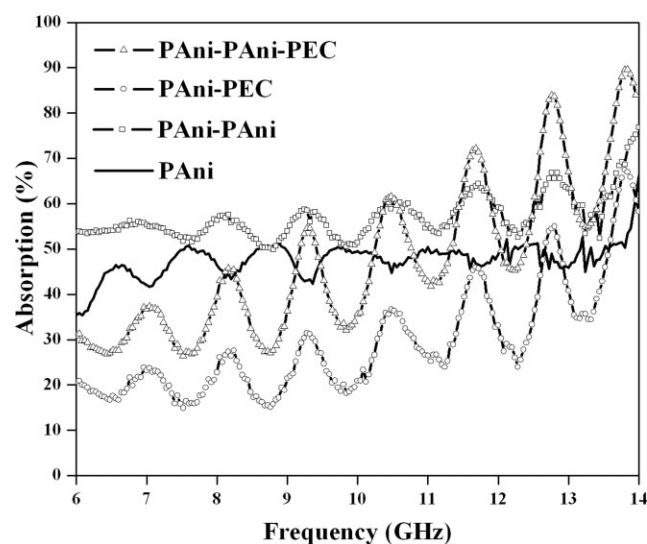


Figure 13 Graph of the absorbance values for different sample designs in the frequency range of 6–14 GHz.

TABLE III
Values of the Mean Absorption, Reflection Loss, and Shielding Efficiency for Different Sample Designs in the Frequency Range of 6–14 GHz

	Mean Absorption (%)	Mean Reflection Loss (dB)	Mean Shielding Efficiency (dB)
PAni	47.66	-10.78	3.78
PAni-PEC	29.27	-1.58	—
PAni-PAni	57.04	-7.11	6.84
PAni-PAni-PEC	47.47	-3.12	—

metal plate was put back decreased when the fold number increased.

CONCLUSIONS

In summary, cotton fabrics were coated with PAni and PPy polymers with a chemical oxidative polymerization method. The structural and tensile properties, UV protection, colorfastness, and electrical and electromagnetic characteristics of the fabric samples were determined. The presence of PAni and PPy films on the cotton fabric by FTIR analysis and the amorphous form of the film by XRD analysis were confirmed. We first reported that the fabric samples coated with the conductive polymers had excellent UV-protection properties. Moreover, the tensile strength properties of the fabric coated with PPy increased, whereas that of the fabrics coated with PAni significantly decreased. Moreover, the colorfastness values of the fabric coated with PPy and PAni displayed the same characteristics, except the colorfastness to color change. After the washing-fastness test, whereas the fabrics coated with PAni completely lost their conductivity, the fabrics coated with PPy kept their conductivity, despite a decrease from 512 Ω to 15.4 k Ω . The electrical resistance values of cotton fabric coated with PAni and PPy were measured as 350 and 512 Ω , respectively. The average electromagnetic shielding efficiency and average absorption values of the cotton fabric coated with PAni and PPy were 3.8 and 6 dB and 48 and 50%, respectively. Furthermore, some special applications of the fabric samples were mentioned. In conclusion, we did not observe a significant difference between the resistance values and electromagnetic parameters of the fabrics coated with PAni and PPy. We determined that the coating of PPy as a conductive polymer for textile materials such as cotton was more suitable with regard to improving and protecting the intact textile characteristics than the coating of PAni.

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