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Investigation of the Electrical Properties and Electromagnetic Shielding Effectiveness of Polypyrrole Coated Cotton Yarns

Abstract

Polypyrrole (PPy) coated cotton yarns were prepared by the vapour phase polymerisation technique at various initiator concentrations. Iron (III) chloride ($FeCl_3$) was used as the initiator at four different concentrations (0.2, 0.4, 0.6 & 0.8 mol/l) respectively. In this work, the effects of initiator concentration on the tensile, electrical and morphological properties of the cotton yarns were investigated. PPy coated cotton yarns were woven on a handloom for the investigation of electromagnetic shielding effectiveness. The fabric samples prepared were evaluated in terms of shielding behavior. Scanning electron microscopy (SEM) analysis showed that the yarn treated with 0.4 mol/l initiator concentration gave the most uniform PPy layer and highest shielding value. The existence of PPy was proved by using Fourier transform infrared spectroscopy (FTIR) analysis.

Key words: polypyrrole, vapour phase polymerization, electrical property, electromagnetic shielding effectiveness.

Introduction

In the last two decades, conductive textile materials have gained a great deal of attention due to the wide application area, such as in electromagnetic shielding [1 - 3], sensors [4 - 6], antistatic applications [7, 8], electrocardiograms [9], capacitors [10], wearable health monitoring systems [11], field effect transistors [12] etc. In general, textile materials are known as insulator substances. In order to introduce conductivity into textile materials, various methods can be used such as coating the textile surface with a conductive polymer [13, 14], embedding metal particles in the textile layer [15 - 17] etc.

As a result of the increment electronic device usage, the electromagnetic interference (EMI) problem has been emerged. Electromagnetic waves emitted from electronic equipment affect human health and other devices negatively. Thus this undesirable radiation should be inhibited by shields. A number of researches have been carried out to design textile based shielding material [18, 19]. The usage of conductive polymers in electromagnetic shielding applications is widespread. Mostly PPy [20 - 22] and polyaniline (PANI) [23, 24] are used in conductive textile composites for EMI shielding.

In this paper, cotton yarn was coated with PPy by the vapour phase polymerisation technique. During the polymerization process, $FeCl_3$ was used as an initiator. The effects of the initiator concentration on the tensile strength, electrical and morphological properties, and electro-

magnetic shielding effectiveness were investigated. Four different concentration values were chosen for the experimental process. Following the coating process, in order to measure the shielding value, sample yarns were processed into woven fabrics on a handloom in a plain pattern.

Experimental

Materials

Carded single cotton yarns (with a count of linear density 19.7 tex) were used in the experimental process. All glass wares used in the experimental set-up were purchased from Boru-cam Co. Pyrrole of reagent grade was purchased from Sigma-Aldrich, hexahydrate $FeCl_3$ and methanol from Merck, respectively. Stock solutions of the initiator in methanol at four different concentrations (0.2, 0.4, 0.6 and 0.8 mol/l) were prepared.

Coating process

The experimental set-up for the continuous vapour phase polymerisation method can be seen in **Figure 1**. At the beginning of the process, cotton yarn is unwound from a bobbin (A) at a constant speed of 2 m/min and directed to a glass flask (B) with the initiator in methanol situated inside it. The yarn samples are immersed in this initiator solution and then directed to a hot air stream (C). After the drying process, samples are sent to the polymerisation chamber (E). Pyrrole monomer is situated in a flask (D) and continuously carried to the polymerization chamber with the help of nitrogen gas. At the end of the polymerization chamber there is another flask (F) filled with distilled water which helps to hold the un-reacted pyrrole monomer after the polymerisation process. The coated yarn is wound on a bobbin with the help of the

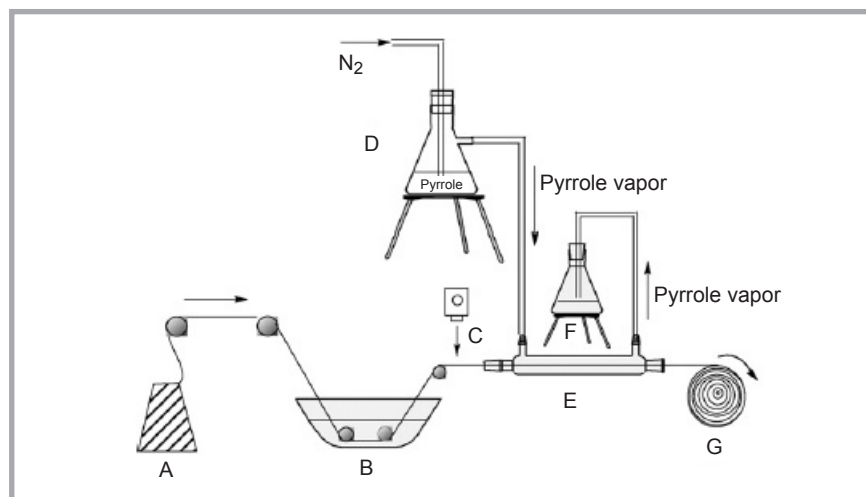


Figure 1. Experimental set-up [22].

winding machine (G) at a constant speed of 2 m/min.

Characterisation

Before characterisation, the polypyrrole coated cotton yarn samples were conditioned for 24 h under laboratory conditions of 20 ± 2 °C and $65 \pm 2\%$ relative humidity. The tensile strength of the yarns were measured with an Instron 4411 instrument according to the TS 245 EN ISO 2062 standard [25]. To ensure the reliability of the results, each test was repeated 5 times. The electrical resistance of the coated yarn samples was measured with a multimeter DL MULT3999D by applying 2 g/tex pre-tension to the yarn. The electrical resistance results were given in two different approaches: First is the difference in electrical resistance depending on the distance between two probes. The second is the mass specific resistance, as mentioned by Kaynak et al. [26], in terms of the initiator concentration. Morphological characterisation of the yarn samples was performed with SEM (JEOL Ltd., JSM-5910LV) and FTIR (Perkin Elmer Spectrum 100 ATR-FTIR spectrophotometer) analyses. The thickness of the PPy layer on the coated yarns was measured with the ImageJ programme using SEM cross-section images. Coating thickness measurement was repeated five times for each cross-section image. PPy coated yarns were woven on a handloom in order to measure the surface resistivity and electromagnetic shielding effectiveness. A Keithley 6517A Electrometer/High Resistance Meter instrument was used to determine the surface resistivity of the fabrics according to the ASTM D257-07 standard [27]. For the ASTM D4935-10 standard [28], a Network Analyzer instrument (ROHDE&SCHWARZ) was used for the measurement of the electromagnetic shielding effectiveness of the fabrics. The coaxial transmission line method was used to measure the shielding effectiveness [29].

Results and discussion

Effects of FeCl₃ concentration and PPy on the tensile properties of cotton yarns

The effects of initiator concentration on tensile strength and elongation at break values for both pre-treatment with an ini-

Table 1. Average (Avg.) and standard deviation (SD) of yarn tenacity and elongation values for both pre-treatment with FeCl₃ and after PPy coating in terms of initiator concentration.

FeCl ₃ concentration, mol/l	Pre-treatment with FeCl ₃				After PPy Coating			
	Yarn tenacity, cN/tex		Yarn elongation, %		Yarn tenacity, cN/tex		Yarn elongation, %	
	Avg.	SD	Avg.	SD	Avg.	SD	Avg.	SD
0	11.59	0.66	4.79	0.34	11.59	0.66	4.79	0.34
0.2	12.18	0.91	3.96	0.24	13.49	1.91	4.71	0.83
0.4	10.52	0.45	3.24	0.27	14.03	2.14	2.89	0.70
0.6	10.92	0.84	3.99	0.30	12.48	0.94	3.78	0.49
0.8	8.35	0.75	2.65	0.27	9.59	0.80	1.74	0.37

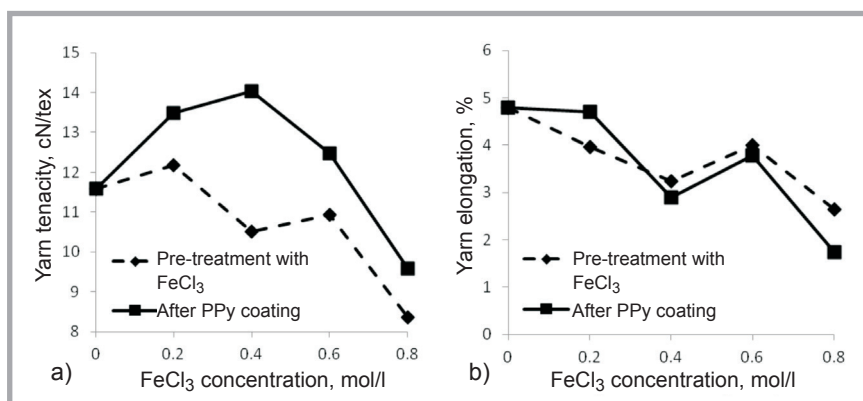


Figure 2. Pre-treatment with FeCl₃ and after PPy coating: a) Tensile strength, b) Elongation values.

tiator and after PPy coating with standard deviation values can be seen in **Table 1** and **Figure 2**. Accordingly pre-treatment with an initiator increased the yarn tenacity at a concentration of 0.2 and 0.6 mol/l. After the PPy coating process, the tensile strength of the yarns increased to 0.4 mol/l FeCl₃, and then decreased. The lowest tensile strength value was obtained at a 0.8 mol/l FeCl₃ concentration, caused by excess Fe ions, which do not participate in the polymerisation process.

The reason for the decrease in yarn elongation percentage after PPy coating is that PPy is a brittle polymer. As can be seen in **Table 1** and **Figure 2**, yarn elongation decreased generally with the increment of the initiator concentration.

Electrical resistance of yarn samples

Figure 3 shows the mass specific electrical resistivity of the PPy coated yarn samples depending on the initiator concentrations. The electrical resistivity value is lowest when the initiator amount is 0.4 mol/l. The lowest resistivity means the highest conductivity. Thus the highest conductivity value was obtained with the addition of 0.4 mol/l FeCl₃. Over-oxidation on the yarn surface at 0.6 and 0.8 mol/l initiator concentrations cause the formation of carbonyl defects on the PPy backbone. These carbonyl defects cause interruptions in the π conjugation and act as electron withdrawing groups, inhibiting charge transfer [30, 31]. Thus conductivity decreases while resistivity

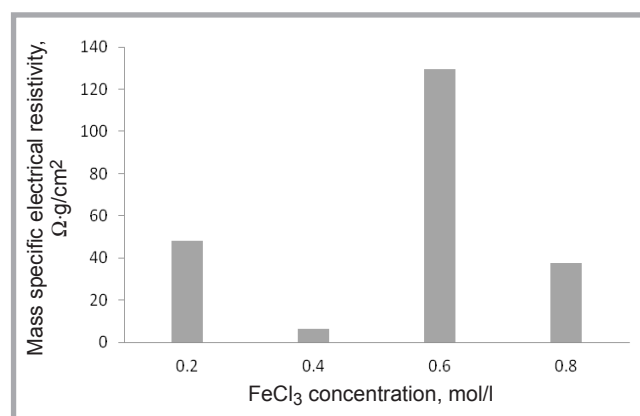


Figure 3. Effect of initiator concentration on the PPy coated cotton yarns' electrical resistivity.

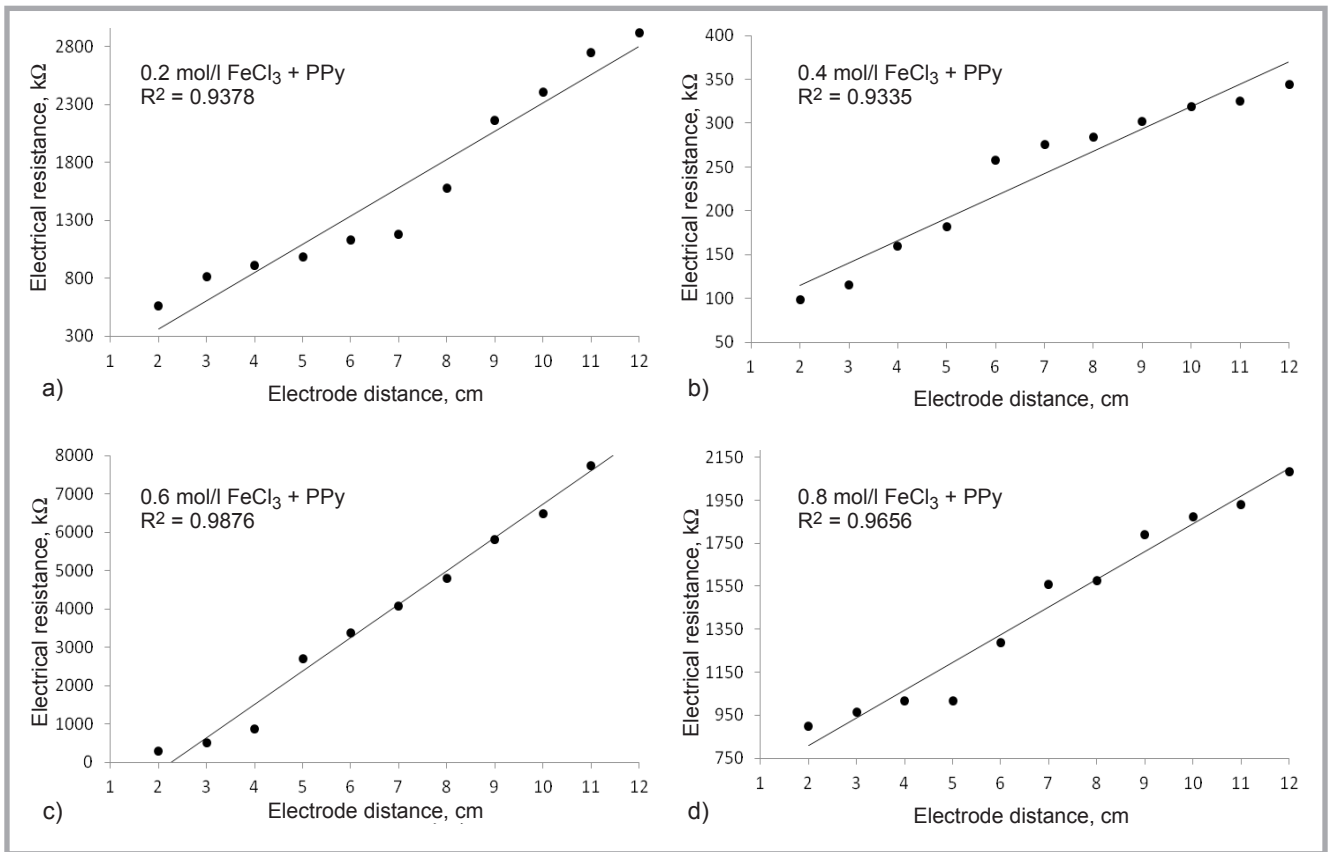


Figure 4. Electrical resistance depending on the electrode distance at a concentration of a) 0.2 mol/l, b) 0.4 mol/l, c) 0.6 mol/l, d) 0.8 mol/l FeCl₃ after the PPy coating process.

increases after a 0.4 mol/l initiator concentration.

In **Figure 4**, electrical resistance values depending on the electrode distance are shown for each initiator concentration. According to the figure, the correlation coefficients between the electrode distance and electrical resistance are extremely close to 1, meaning the PPy layer along the yarn surface is homogeneous.

In other words, PPy was coated uniformly along the yarn surface, thus any point of the yarn surface shows the same electrical property.

Morphological characterisation

Figure 5 presents the FTIR spectra of PPy coated cotton yarn samples. This spectrum shows characteristic bands of PPy like N-H bonds (3200 cm⁻¹ & 617 cm⁻¹) and C-N stretching (1480 cm⁻¹). In pure cot-

ton spectra, there is an acute peak which refers to C-O-C bonding (1100 cm⁻¹) due to the cotton substrate. After PPy coating, the intensity of this sharp hydroxyl peak decreased substantially. The peaks between 1600 - 1700 cm⁻¹ in pure cotton spectra refer to the H₂O absorbed from the environment. The peak in 2800 cm⁻¹ is due to C-H stretching.

Figure 6 shows the morphology of cotton fibres after pre-treatment with an initiator. According to the SEM images, the FeCl₃ was well penetrated in the cotton fibres, especially at a concentration of 0.4 mol/l (**Figure 6.b**) and 0.6 mol/l (**Figure 6.c**).

The morphology of PPy coated cotton fibres can be seen in **Figure 7**. According to this figure, the most uniform PPy layer was obtained at a 0.4 mol/l FeCl₃ concentration (**Figure 7.b**). Also a fairly uniform PPy layer was obtained at a concentration of 0.6 mol/l (**Figure 7.c**). In the case of 0.8 mol/l FeCl₃, the polymerisation process was not efficient enough to obtain an uniform PPy layer (**Figure 7.d**). This affect can be attributed to the existence of excessive Fe⁺ ions.

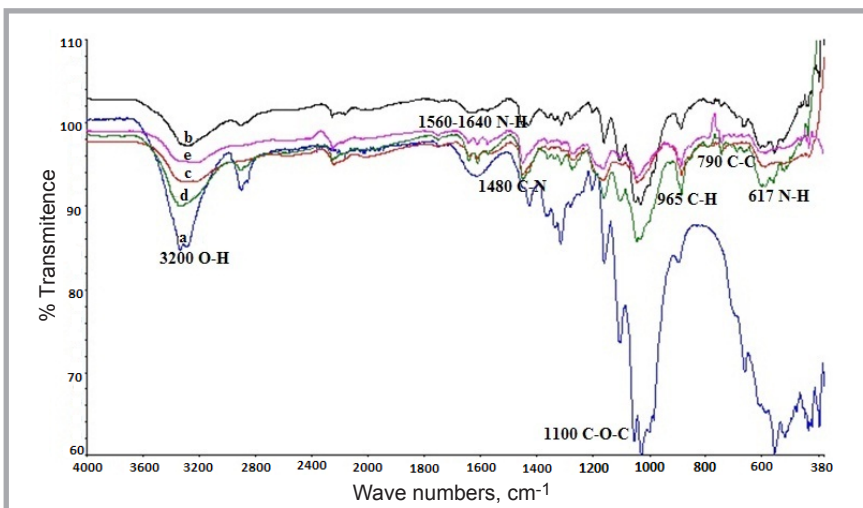


Figure 5. FTIR spectra of PPy coated cotton yarns at different FeCl₃ concentrations a) Pure cotton, b) 0.2 mol/l, c) 0.4 mol/l, d) 0.6 mol/l, e) 0.8 mol/l.

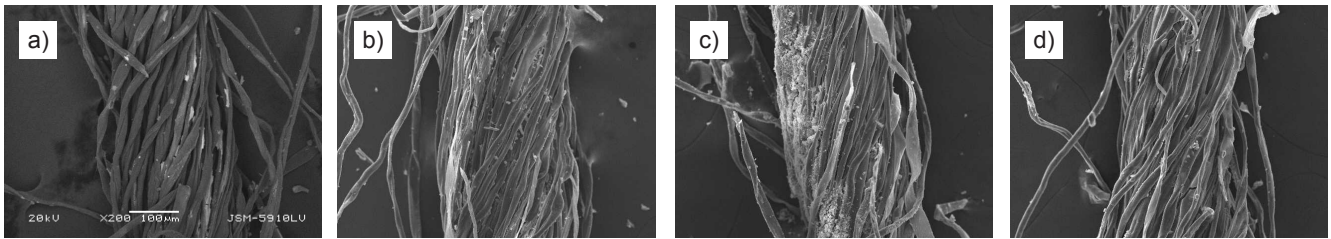


Figure 6. SEM images of cotton fibres after pre-treatment with FeCl_3 at various concentrations: a) 0.2 mol/l, b) 0.4 mol/l, c) 0.6 mol/l, d) 0.8 mol/l).

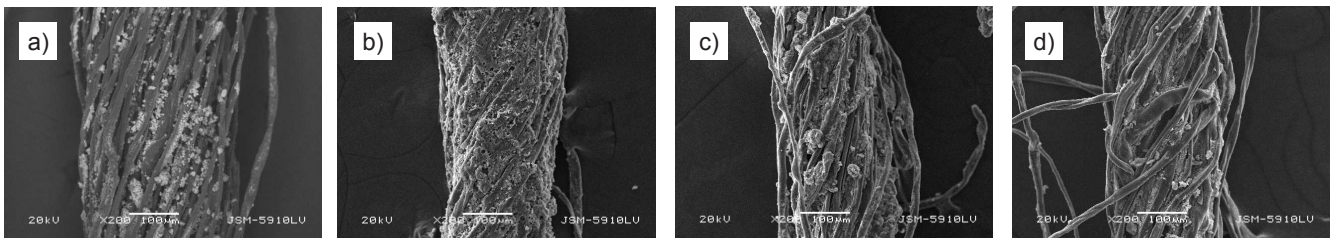


Figure 7. SEM images of PPy coated cotton fibres at various FeCl_3 concentrations: a) 0.2 mol/l, b) 0.4 mol/l, c) 0.6 mol/l, d) 0.8 mol/l).

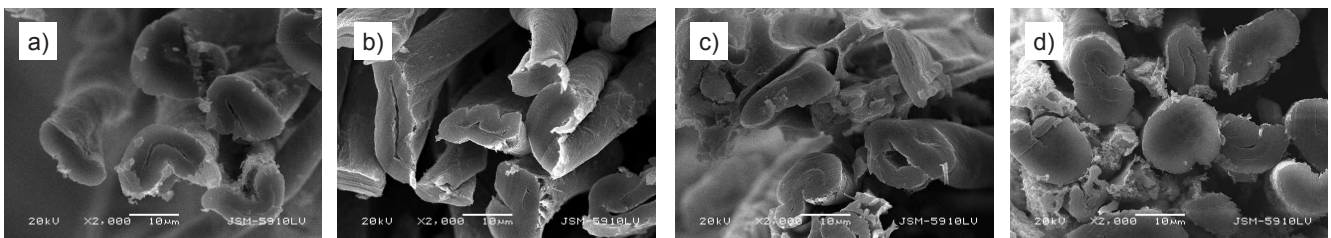


Figure 8. Cross-sections of PPy coated cotton fibres at various initiator concentrations; a) 0.2 mol/l, b) 0.4 mol/l, c) 0.6 mol/l, d) 0.8 mol/l).

Figure 8 shows cross-sections of PPy coated cotton fibres. With the addition of 0.2 mol/l FeCl_3 (**Figure 8.a**), the PPy layer did not coat the yarn surface uniformly. In **Figure 8.b**, it is obvious that the PPy layer uniformly coated the fibre surface and good penetration was obtained. On the other hand, as seen in **Figure 8.c**, due to the highly poor penetration a non-uniform PPy coating was seen. Higher concentration of FeCl_3 also gave poor uniformity. Excessive Fe^+ ions can be seen in **Figure 8.d**.

Coating thickness measurement on PPy coated cotton yarns

By using the SEM cross-section images of the PPy coated cotton yarn samples, the coating thickness was measured. The best coating layer, with a value of 2.05 μm , was obtained at a concentration of 0.4 mol/l FeCl_3 . Coating thickness values with standard deviation can be seen in **Table 2**.

Surface resistivity of the fabrics

The surface resistivity of the fabrics which were produced with PPy coated cotton yarns can be seen in **Table 3**. Accordingly it can be said that the lowest

surface resistivity value, in other words the highest conductivity, was obtained at a concentration of 0.4 mol/l FeCl_3 .

Electromagnetic shielding effectiveness (EMSE) of the fabrics

EMSE values of the PPy coated cotton fabrics at different FeCl_3 concentrations can be seen in **Figure 9** (see page 36). Accordingly the highest shielding is given in a decreasing order for concentrations of 0.4, 0.6, 0.2 and 0.8 mol/l, respectively. For each concentration the highest value was at a frequency of 200 MHz. A remarkable decline in shielding values can be seen in the frequency range of 200 - 800 MHz. The shielding behaviour of the samples showed almost a linear shielding effect between 800 and 1800 MHz. Afterwards a slight increase to 1800 - 2500 MHz and then a slight decrease to 300 MHz were observed.

Table 4 (see page 36) shows shielding effectiveness (SE), absorption (A), reflection (R) and transmission (T) values at different FeCl_3 concentrations for PPy coated cotton fabrics. The values were obtained at a frequency of 1000, 1500, 1800 and 2500 MHz, respectively. These

frequencies were chosen because they are the most exposed electromagnetic waves, produced by electronic devices, mobile phones etc. According to **Table 4** the highest electromagnetic wave absorption value was observed at a concentration of 0.4 mol/l FeCl_3 . At the 0.8 mol/l concentration, absorption decreased a little, being a result of excessive Fe^+

Table 2. Average coating thickness values of the PPy layer on cotton yarns with standard deviation.

FeCl_3 concentration, mol/l	Average coating thickness, μm	Standard deviation
0.2	1.16	0.27
0.4	2.05	0.45
0.6	1.16	0.19
0.8	1.56	0.39

Table 3. Surface resistivity of fabrics woven with PPy coated cotton yarns.

FeCl_3 concentration, mol/l	Surface resistivity, $10^7 \Omega/\text{sq}$
0	61.00
0.2	0.250
0.4	0.118
0.6	0.182
0.8	1.310

Table 4. Shielding effectiveness (SE), absorption (A), reflection (R) and transmission (T) values of the PPy coated cotton fabrics with various initiator concentrations.

FeCl ₃ concentration, mol/l	Frequency, MHz															
	1000				1500				1800				2500			
	SE, dB	A	R	T	SE, dB	A	R	T	SE, dB	A	R	T	SE, dB	A	R	T
0.2	0.16	0.03	0.003	0.96	0.19	0.03	0.014	0.96	0.20	0.01	0.036	0.95	0.23	0.05	0.002	0.95
0.4	0.26	0.06	0.000	0.94	0.24	0.05	0.008	0.95	0.22	0.00	0.050	0.95	0.26	0.05	0.004	0.94
0.6	0.20	0.05	0.000	0.95	0.22	0.04	0.010	0.95	0.20	0.00	0.040	0.95	0.27	0.06	0.004	0.94
0.8	0.20	0.04	0.000	0.96	0.18	0.03	0.007	0.96	0.16	0.01	0.050	0.96	0.21	0.04	0.004	0.95

ions. The reflection values are lower than for the absorption because conductive polymers tend to absorb electromagnetic waves instead of reflecting them. Absorbing radiation is the most important property distinguishing conductive polymers from metal particles. In terms of the transmission values of the fabrics, it can be easily said that about 95% of the waves can transmit to the other side, which is related to the coating thickness of the PPy layer. The coating thicknesses were obtained at μm levels.

Conclusions

Vapour phase polymerisation of pyrrole on a cotton yarn surface was performed in order to obtain conductive textile substrates. PPy coated cotton yarns were transformed into fabric by the weaving process for measurement of the surface resistivity and EMSE. The effect of the initiator concentration on the polymerization process was investigated. For the PPy coated cotton yarn surface, the tensile properties, electrical resistivities, FTIR spectrum, longitudinal and cross-sectional views, and coating thicknesses were investigated. From the fabric sur-

face, resistivity and EMSE were measured.

PPy coated cotton yarns exhibited higher tenacity than un-coated yarns till a 0.6 mol/l FeCl₃ concentration. After that concentration, the tenacity decreased because of excess Fe ions, which do not participate in the polymerisation. However, the elongation values gradually decreased with an increase in the initiator concentration because PPy is a brittle polymer. Electrical resistivity measurement showed that there is a high correlation coefficient between the electrode distance and electrical resistance, which means a homogeneous PPy layer was obtained through the yarn surface. The FTIR spectrum proved PPy existence. SEM images showed that the most uniform PPy layer was obtained with the addition of 0.4 mol/l FeCl₃. The coating thickness was obtained up to 2.05 μm . In terms of the EMSE values it can be concluded that the best value (about 1 dB) was obtained at the 0.4 mol/l FeCl₃ concentration. Also the lowest surface resistivity of $1.18\text{E}+06 \Omega/\text{sq}$ was obtained at this concentration, because of carbonyl defects on the PPy backbone formed by over-oxidation on the yarn surface. These carbonyl defects inhibit charge transfer

by acting as an electron withdrawing group.

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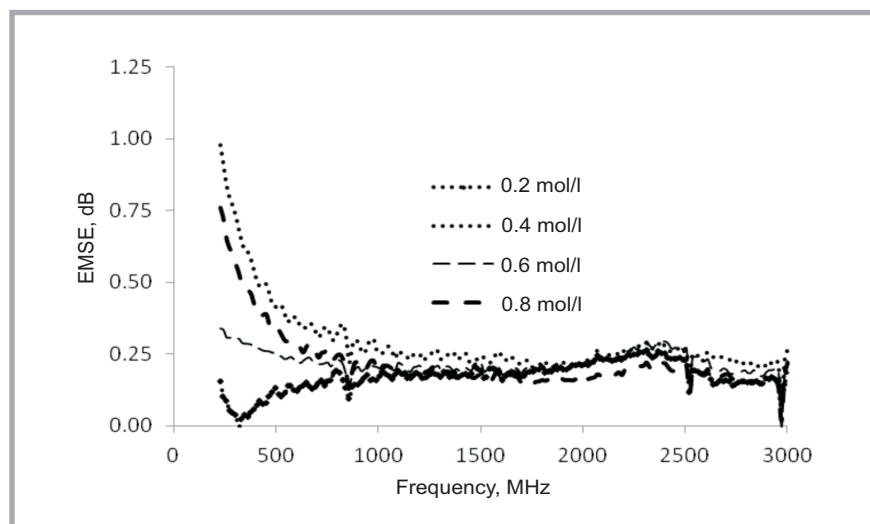


Figure 9. EMSE of PPy coated fabrics with various FeCl₃ concentrations.

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