Electromagnetic and Electrical Properties of Coated Cotton Fabric with Barium Ferrite Doped Polyaniline Film

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ABSTRACT: In this study, we aimed to produce fabrics with microwave absorbing properties in broad band. For this purpose, the cotton fabrics were coated with polyaniline (PAni) by using chemical oxidative polymerization method. Firstly, the type of protonic acid used, the polymerization time, the type and concentration of dopant, and the number of coating layer were varied as parameters. The effect of these parameters on resistance values of coated fabrics was investigated. We determined the most appropriate process conditions to provide the lowest resistance values. Secondly, the fabric was coated with PAni by adding barium ferrite powder as filler with different ratios. Morphological properties, electrical, and electromagnetic properties of coated fabrics were determined. As a result, we obtained 350 Ω of the minimum resistance values of coated fabric using 1M HCl, 0.5M aniline and 0.5M ammonium persulphate by chemical oxidative polymerization method for totally 4 h of polymerization time. The results of electromagnetic parameters showed that the absorption values of the fabric coated PAni was average 48% in the frequency range of 6–14 GHz. It was concluded that microwave absorber for a textile material can be successfully produced by this process. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 358–366, 2009

Key words: conducting polymers; coatings; monomers

INTRODUCTION

Conductive polymers show the electrical properties due to their conjugated double bonded chain structures.[1] In recent years, electrically conductive polymers have been investigated for various industrial applications, including rechargeable batteries, electrochromic 'smart windows, composite structures, and electrodes.[2] π-electron conjugate polymers are polythiophene, polyaniline and polypyrrole etc. Among these polymers, PAni widely attracts interests because of the resistance to environmental, thermal and chemical action, unique electrochemical property, reversibility in the doping/dedoping process, ready availability of raw materials, and simplicity and low cost of synthesis. Therefore, it was the first commercially produced polyconjugated polymer.[3–6] Nonetheless, its industrial application has some drawbacks, such as poor processibility and poor mechanical properties. Improvement of mechanical properties could be accomplished by forming composites of PAni with conventional nonconductive polymers or textile substrates.[5] There are various methods to prepare PAni coated fabrics such as electrochemical polymerization,[7,8] chemical polymerization,[9,10] block or graft polymerization,[11,12] plasma polymerization.[6] To prepare the conductive composite fabrics, many scientists have focused on chemical oxidative polymerization, because this method does not require the destruction of the substrate and provides reasonably good conductivity. Furthermore, chemical oxidative polymerization is expected to be one of the most convenient methods, because it is a relatively simple and easy method to control the conductivity by maintaining the high strength of substrate fabric.[13] Electrically conductive textiles can be used as electromagnetic interference (EMI) shielding materials for personal computers and home electronic devices, flooring and ceiling materials, heating elements, deelectrifying cloths, dissipation of electrostatic charge, microwave absorber, radar cross section reducing protective fabrics for stealth technology.[5,14] Many studies were performed to investigate the electromagnetic wave absorption properties of conductive polymer coated fabrics, but the microwave absorbing properties of magnetic powder doped conductive polymer coated fabrics was not studied beforehand.
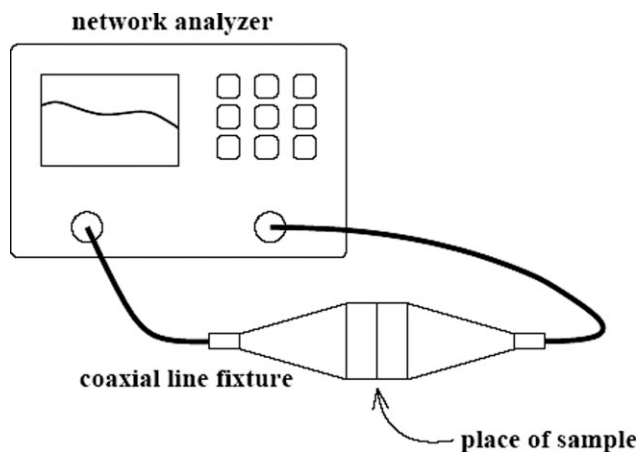
Figure 1 Measurement setup with network analyzer and coaxial line fixture.

Dhawan et al. obtained the results of −3 to −11 dB of shielding efficiency values in 8–12 GHz region.[15] The same researchers also reported a shielding effectiveness of about 35 and 21 dB in a PAni coated silica cloth and polyester fabric at 101 GHz, respectively, using a phase-log oscillator measurement technique.[16] Marchant et al. reported the free-space microwave reflectivity properties of PPy coated E-glass fabric in 2–18 GHz region. The highest microwave loss can be achieved about −13 dB.[17] Hakansson et al. measured the microwave properties of PPy coated polyester by free space measurement method in 1–18 GHz of frequency range. The transmission loss of the samples was determined as high as −8.68 dB, corresponding to a maximum total shielding effectiveness of around 86% with an absorption dominant loss.[18] Hakansson et al. provided a maximum shielding effectiveness of 89.9% at 18 GHz of PPy coated Nylon-Lycra fabric.[19] Kim et al. investigated the electromagnetic interference shielding effectiveness of PPy coated polyester fabric by chemical and electrochemical polymerization and showed that the fabrics achieved 36 dB of shielding

efficiency value over a wide frequency range up to 1.5 GHz.[20]

Moreover, there have been many investigations on the microwave absorption properties of barium ferrite powders. Ferrites are widely used in many industrial applications because of their spontaneous magnetization. Therefore, the development of new and cost-effective techniques for fabricating nanostructures based on ferrites is of great commercial and scientific interest.[21] Barium ferrite powders are ideal fillers for the development of electromagnetic attenuation materials at microwave, because of their low cost, low density, high stability, large electrical resistivity and high microwave magnetic loss.[22–24] The absorption and reflection loss of various doped barium ferrites demonstrated that the materials may be used as electromagnetic materials with low reflectivity at microwave frequency.[25–36] Many studies were performed to investigate the electromagnetic wave absorption properties of magnetic powders, but the microwave absorbing properties of magnetic powder doped conductive polymer coated fabrics was not studied previously. We firstly investigated the microwave absorbing properties of cotton fabric coated with PAni doped various amount of barium ferrite.

In this study, we coated the cotton fabrics with PAni by using chemical oxidative polymerization method. Firstly, we varied the type of used protonic acid, the polymerization time, the type and concentration of dopant and the number of coating layer. The effect of these parameters on resistance values of coated fabrics was investigated. The most appropriate process conditions were determined to provide the lowest resistance values. Secondly, the fabric was coated with PAni by adding barium ferrite powder as filler with different ratios. Morphological properties, electrical and electromagnetic properties of coated fabrics were determined. In conclusion, we obtained 350 Ω of the minimum resistance values of coated fabric using 1M HCl, 0.5M
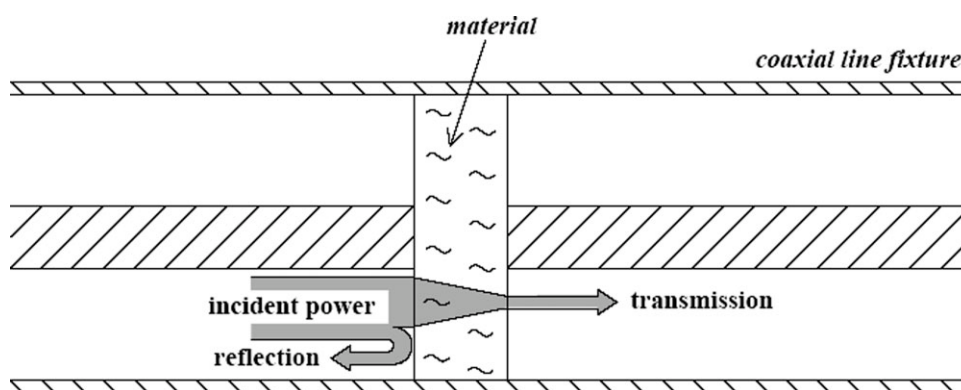

Figure 2 Incident, reflected, and transmitted power at the sample material.

1.Step

Dissolve 1 M Anilin in acid solution

Adding of dopant agent (if required)

Adding of the fabric and treatment at 0 °C for various times (1 h, 3 h and 4 h)

2. Step

Droping of IM APS solution in acid solution at 0 °C to prepared aniline solution in first step

Polimerization step : Treatment for various times (1 and 4 h)

Removing fabric

Washing with acidic solution

Washing with distilled water

Drying in room temperature
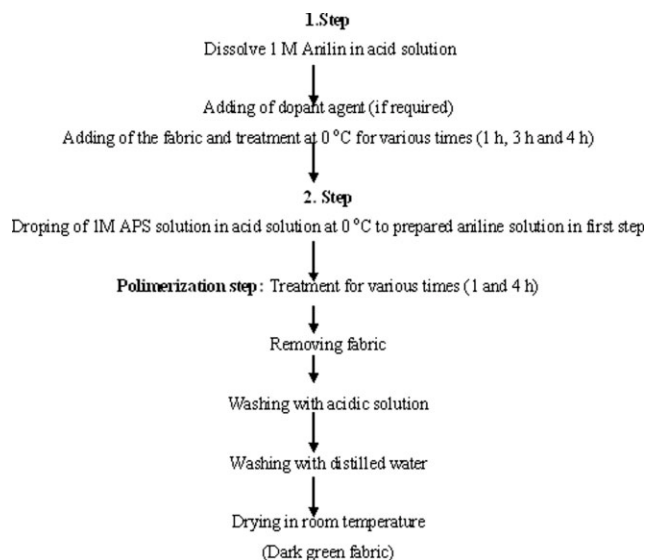
(Dark green fabric)

Figure 3 The flow chart for PAni coating of the fabrics by chemical oxidative polymerization.

aniline and 0.5M ammonium persulphate by chemical oxidative polymerization method for 4 h of totally polymerization time. The results of electromagnetic parameters showed that the reflection loss of the fabric samples coated with PAni, 10% barium ferrite doped PAni and 30% barium ferrite doped PAni were 11, 11, and 17 dB, respectively. The average absorption values of the fabric coated with PAni, 10% barium ferrite doped PAni and 30% barium ferrite doped PAni were 48, 48, and 27% in the frequency range of 6–14 GHz. It was concluded that microwave absorber textile material can be successfully produced by this cost-effective process.

EXPERIMENTAL

Materials

Scoured canvas fabric (panama-weave, 239 g/m², 22 ends/cm, and 22 picks/cm) was used in this research. Aniline (≥99, Fluka) was distilled under vacuum before use. The other chemicals included ammonium peroxydisulfate (APS, 98+%, A.C.S. reagent, Sigma-Aldrich), dodecylbenzene sulfonic acid (DBSA, 70 wt % solution, in 2-propanol, Aldrich), anthraquinone-2-sulfonic acid sodium salt (ASA, ≥98%, Fluka), 2-naphtalenesulfonic acid (NSA, technical grade, 70%, Aldrich), hydrochloric acid (%37, Riedel-de Haen), sulfuric acid (95–97%, Fluka), nitric acid (65%, Riedel de Haen) and phosphoric acid (85%, Riedel de Haen,) all of reagent grade. APS was selected as an oxidant. Hydrochloric acid, sulfuric acid, nitric acid, phosphoric acid used as a protonic acid. DBSA, ASA and NSA were used as a dopant. Barium ferrite nanopowders (<100 nm parti-

TABLE I
The Resistance and Add-On Values of Coated Fabric with Respect to Acid Type

Acid type	Resistance, Ω	Add-on, %
HCl (1M)	350	9.03
H$_2$SO$_4$	358	20.28
H$_3$PO$_4$	375	16.12
HNO$_3$	2051	13.84

cle size (BET), 99.5%, Aldrich) were also used as a filler in thin film on fabric.

Characterization

The add-on values of the fabric samples were calculated according to eq. (1):

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

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 at standard atmosphere of 20 ± 2°C and 65 ± 2% relative humidity before weighting. Thus the dry weights of fabrics were determined.[37]

The resistance values of the fabric samples and the pellets produced from PAni powders were measured by an ohmmeter (Brymen BM 805 Model Digital Multimeter) by using two probe resistance measurement method with 1-cm distance.

The pH meter (J.P. Selecta, Spain) was used to measure the pH values of acidic solutions and polymerization solutions.

The electromagnetic parameters of the fabrics were measured with transmission/reflection method in the region of 6–14 GHz. All samples are characterized by the reflection parameter S_{11} and the transmission parameter S_{21} measured with a Network Analyzer HP8720D. S_{11} and S_{21} parameters (S-

TABLE II
The pH Values of Polymerization Solution in Different Steps by Using of Various Protonic Acid Solutions

Acid type and process step	pH value
HCl solution	0.15
HCl+Aniline	3.62
HCl+Aniline+APS	0.15
H$_3$PO$_4$ solution	0.15
H$_3$PO$_4$+Aniline	2.12
H$_3$PO$_4$+Aniline+APS	0.69
H$_2$SO$_4$ solution	0.15
H$_2$SO$_4$+Aniline	5.02
H$_2$SO$_4$+Aniline+APS	0.69
HNO$_3$ solution	0.15
HNO$_3$+Aniline	5.09
HNO$_3$+Aniline+APS	0.69

TABLE III
The Resistance and Add-On Values of Coated Fabrics with Different Polymerization Times by Using 1M HCl Solution

Time, hour : hour	Resistance, Ω	Add-on, %
4 : 4	701	9.46
3 : 1	350	9.03
1 : 1	1152	8.62

parameters) represent reflection and transmission coefficient, respectively. Figure 1 shows schematically the setup. The network analyzer and coaxial line fixture have been calibrated. In Figure 2, the incident power, delivered by the network analyzer, and the reflected and transmitted power, measured by the network analyzer, are depicted.

According to the analysis of S-parameters, transmittance (T), reflectance (R) and absorbance (A) can be described as

$$R = \left|\frac{E_r}{E_i}\right|^2 = |S_{11}|^2 \qquad (2)$$

$$T = \left|\frac{E_t}{E_i}\right|^2 = |S_{21}|^2 \qquad (3)$$

$$A + T + R = 1 \qquad (4)$$

Whereas E_i, E_r, and E_t are the incident electric field, reflected electric field and transmitted electric field, respectively. A, R and T can have values between 0 and 1.

EMI SE value expressed in dB was calculated from the ratio of the incident to transmitted power of the electromagnetic wave as following eq. (5):

$$SE = 10 \log \left|\frac{P_i}{P_t}\right| = 20 \log \left|\frac{E_i}{E_t}\right| \text{ (decibels, dB)} \qquad (5)$$

Whereas P_i and P_t are the incident power and transmitted power, respectively.[20,38]

The surface morphology of PAni coated fabrics was investigated with a JEOL JJM 6060 scanning electron microscope attached to an energy-dispersive spectroscopy apparatus (JEOL, Tokyo, Japan).

Moreover glass substrates were coated with PAni as one time by using chemical oxidative polymerization method under the same optimum condition

TABLE IV
The Resistance Values of the Pellets by Produced PAni Powders During Various Polymerization Times

Time, hour : hour	Resistance, Ω
4 : 4	13.5
3 : 1	13.3
1 : 1	8

TABLE V
The Resistance and Add-On Values of Coated Fabrics with Different Type of Dopant Agents and Different Dopant Concentrations by Using 1M HCl Solution and During 3 : 1 h Polymerization Time

Dopant type	Dopant concentration	Resistance, Ω	Add-on, %
NSA	0.1M	592	9.6
	0.01M	440	9.13
	0.001M	699	8.75
ASA	0.1M	1728	8.82
	0.01M	496	8.91
	0.001M	962	8.88
DBSA	0.1M	669	14.49
	0.01M	387	10.82
	0.001M	1026	8.62

with that used for coating on the fabric samples. Glass substrates coated were washed with distilled water and dried at room temperature. The film thickness of the PAni films produced on the glass substrate was evaluated with a refractometer and a spectrophotometer. The refractive indices of the thin films were measured in the visible region with an Abbe high-accuracy refractometer (Bioiberica, Barcelona, Spain) at room temperature. The refractive indices were used to determine the thickness and band gap of the film on the glass substrate by a Jasco V-530 ultraviolet-visible spectrophotometer (Jasco, Tokyo, Japan) in the range of 190–800 nm.[37]

It was determined that the refractive index of the film was 2.5186 nD, the thicknesses were 483 nm, and band gap of the film was 3.96 eV (Base line).

TABLE VI
The Resistance And Add-On Values of the Fabric Samples Coated with 10 and 30% wof Barium Ferrite Doped and Undoped PAni During 3 : 1 h without Adding Doping Agent with Different Layer Number

Layer	Process	Resistance, Ω	Add-on, %
	Ba-A[a]	–	7.46
	Ba-B[b]	–	13.95
One layer	PAni1[c]	350	9.03
Two layers	PAni2[d]	104	22.3
Three layers	PAni3[e]	64	36.10
One layer	Ba-PAni-A1[f]	440	15.75
Two layers	Ba-PAni-A2[g]	105	30.23
Three layers	Ba-PAni-A3[h]	58	41.10
One layer	Ba-PAni-B1[i]	1829	16.07
Two layers	Ba-PAni-B2[j]	375	26.69
Three layers	Ba-PAni-B3[k]	135	37

[a] Treated fabric with 10% Ba-ferrite.
[b] Treated fabric with 30% Ba-ferrite.
[c,d,e] Coated fabric with PAni as one layer, two layers, and three layers, respectively.
[f,g,h] Coated fabric with 10% Ba-ferrite+PAni as one layer, two layers, and three layers, respectively.
[i,j,k] Coated fabric with 30% Ba-ferrite+PAni as one layer, two layers, and three layers, respectively.

a) Bare fabric

b) PAni1*   c) PAni3

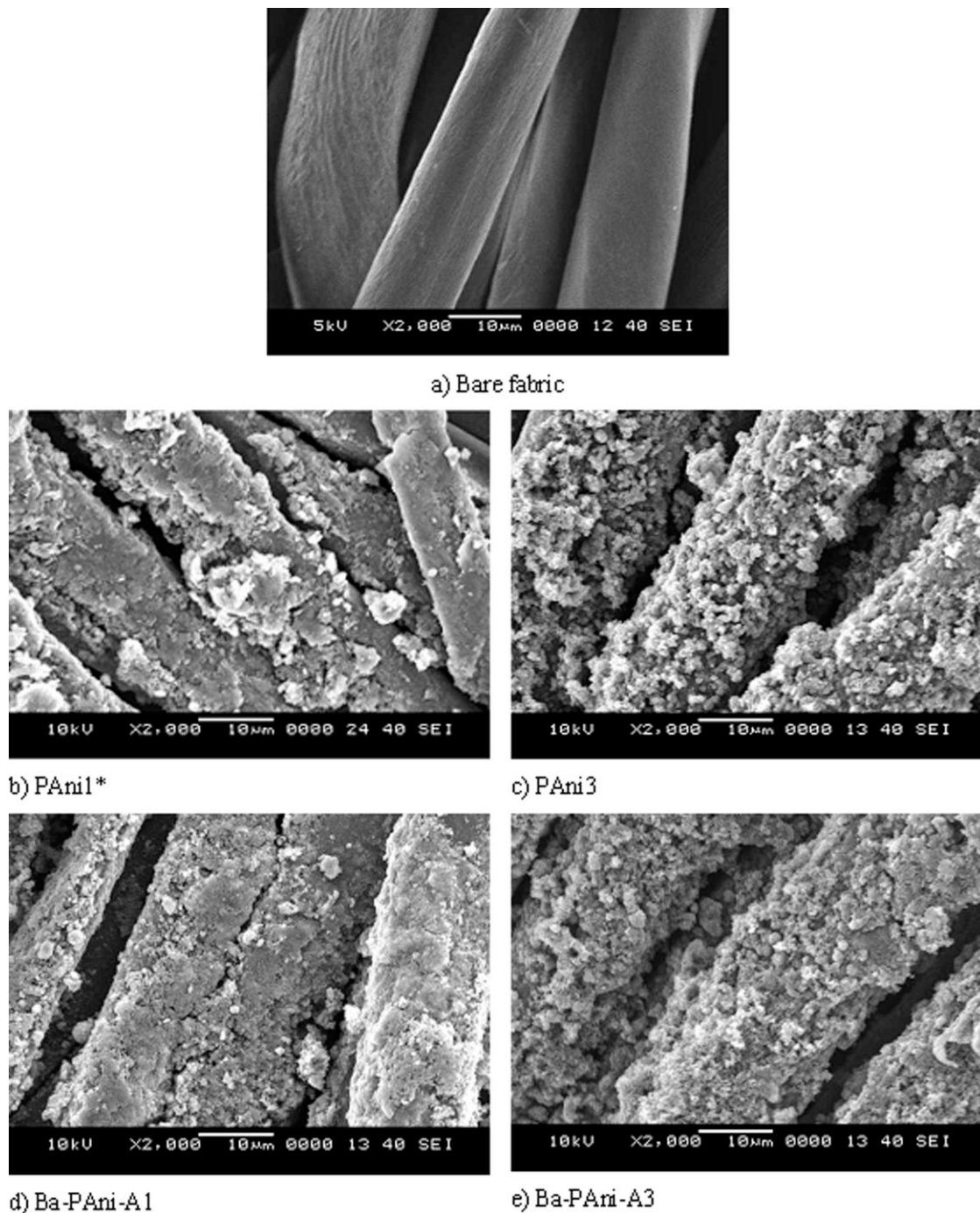d) Ba-PAni-A1   e) Ba-PAni-A3

Figure 4   The longitudinal SEM images of coated and bare fabrics.

Stejskal et al. reported that the thickness of PAni coating by chemical oxidative polymerization was 100–200 nm.[39] The thickness of PAni film on fabric was estimated as the thickness of PAni film on glass substrate.

Fabric treatment

Barium ferrite was dispersed in distilled water. Firstly, the fabric samples were treated with 10% wof (weight of fabric) and 30% wof of barium ferrite solu-tion in distilled water at 80°C for 10 min. The sub-strate-to-bath ratio was maintained at 1 : 10. WB 14 Model-Memmert water bath machine was used for the treatment of the fabrics. After the treatment, the fabrics were removed from the beaker and squeezed by a Rapid Fulard (Model P-A1, Labortex, Taiwan) for 100% A_F (take up) at a nip pressure of 0.5 kg/cm^2. The fabrics were dried at room temperature.

In the second step, the polymerization of aniline on the treated and untreated fabrics with barium fer-rite solution was performed by chemical oxidative
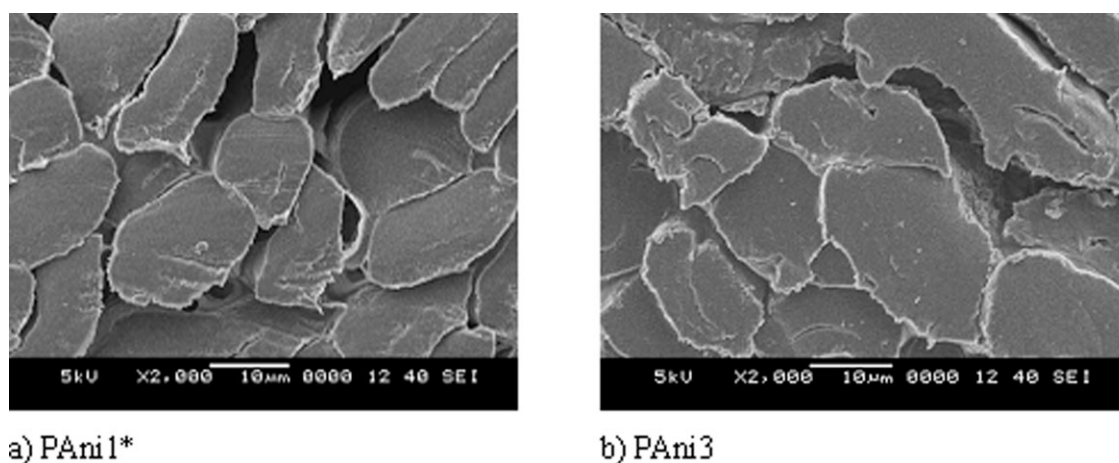
Figure 5 The cross-sectional SEM images of coated and bare fabrics.

polymerization process. Acidic solution was prepared as 0.15 pH values. One molar aniline solution and then dopant agent (if required) were added the acidic solution. After that, the fabric samples were soaked in the solution for various durations (1, 3, and 4 h). The bath containing the fabric was then cooled to 0–5°C. One molar ammonium persulphate was separately added to a precooled (0–5°C) the acidic solution (pH 0.15) and subsequently the APS solution was added gradually aniline solution to polymerize the aniline. The oxidant-to-aniline ratio was maintained at 1 : 1. Polymerization occurred with continuous mild stirring, and after the addition of all the oxidant solution, it was further stirred for different times including 1 and 4 h so as to complete the polymerization. These samples were then thoroughly washed with a sufficient quantity of acid solution in many portions so as to remove any unreacted monomer and excess ammonium persulphate, and with an equal volume of distilled water

to remove excess acid molecules. The fabric samples were various shades of emeraldine green, resembling PAni, thus providing PAni deposition had indeed occurred. The final substrate-to-bath ratio in both cases was maintained at 1 : 30, and the final concentration of aniline and oxidant was 0.5M. This deposition process was performed as one time, two times, and three times. Figure 3 schematically shows the steps of process of PAni coating on the fabrics.[39] Moreover, the accompanying PAni precipitates in the solution were washed repeatedly in the filtering funnel with acid solution and then methanol until filtrate became transparent. The powder was obtained by drying in Nüve KD400 Oven (Turkey) at 50°C for 24 h. Powders (0.2 gr) were pressed by Camilla 95 OL 57 (Manfredi S.p.A, S. Second DI Pinerolo (Torino), Italy) under 200 bar of pressure. The prepared pellets have the 1 cm of radius and 1 mm of thickness.[40]
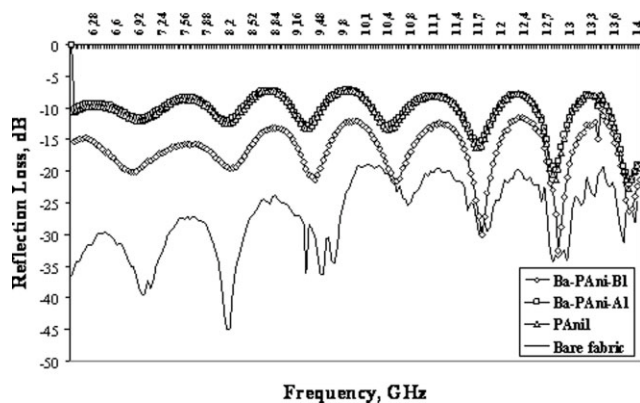

Figure 6 The reflection loss (dB) versus the frequency range of 6–14 GHz of the fabric coated with 10, 30% wof barium ferrite added PAni and only coated with PAni and bare fabric.
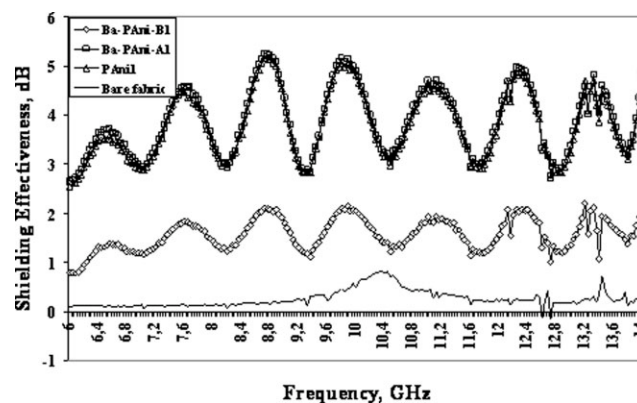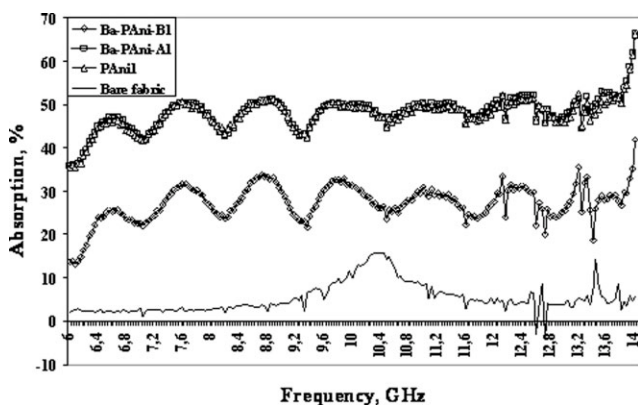

Figure 7 The shielding effectiveness (dB) versus the frequency range of 6–14 GHz of the fabric coated with 10, 30% wof barium ferrite added PAni and only coated with PAni and bare fabric.

Figure 8 The absorption values (%) versus the frequency range of 6–14 GHz of the fabric coated with 10, 30% wof barium ferrite added PAni and only coated with PAni and bare fabric.

RESULTS AND DISCUSSION

The effect of acid type on resistance

The pH values of acid solution prepared by using HCl, H_2SO_4, H_3PO_4, and HNO_3 to polymerization solution were adjusted as 0.15. The effect of acid type on resistance and add-on values of coated fabric was investigated. The resistance and add-on values of coated fabric with respect to acid type were given in Table I. Table II shows the pH values of polymerization solution in different steps by using of various protonic acid solutions.

The effect of polymerization times on resistance

The resistance and add-on values of coated fabrics with different polymerization times by using 1M HCl solution as protonic acid solution were shown in

Table III. The results show that the resistance values of coated fabric during 3 : 1 h (first step: second step) were lower than that of coated fabric during other polimerization times. Therefore, we determined 3 : 1 h as optimum polymerization time for the process.

Moreover the PAni precipitates in the polymerization solution accompanied with PAni coating on the fabric. The precipitates were washed and filtrated. The filtrate powder was dried at 50°C for 24 h. The obtained powders were pressed. The prepared pellets have the 1 cm of radius and 1 mm of thickness.[40] The resistance values of the pellets were shown in Table IV. The resistance values of the pellets were about 10 Ω and significantly lower than the values of coated fabrics.

The effect of dopant type and concentration on resistance

The resistance and add-on values of coated fabrics with different type of dopant agents and different dopant concentrations by using 1M HCl solution as protonic acid solution and during 3 : 1 h polymerization time were shown in Table V.

As explained in Ref. 13, the addition of doping agents up to certain concentrations decreased the resistance values. However the addition of higher concentration of the certain level of doping agent increased resistance values. The present results were in accordance with the Ref. 13. As a result, we performed lower resistance of coated fabric by using only HCl solution as protonic acid without doping agent as 350 Ω than that with doping agent as 387 Ω. Moreover the results of pH measurement exhibited that dopant addition significantly didn't affect the pH values of polymerization solution. Hence, it was determined
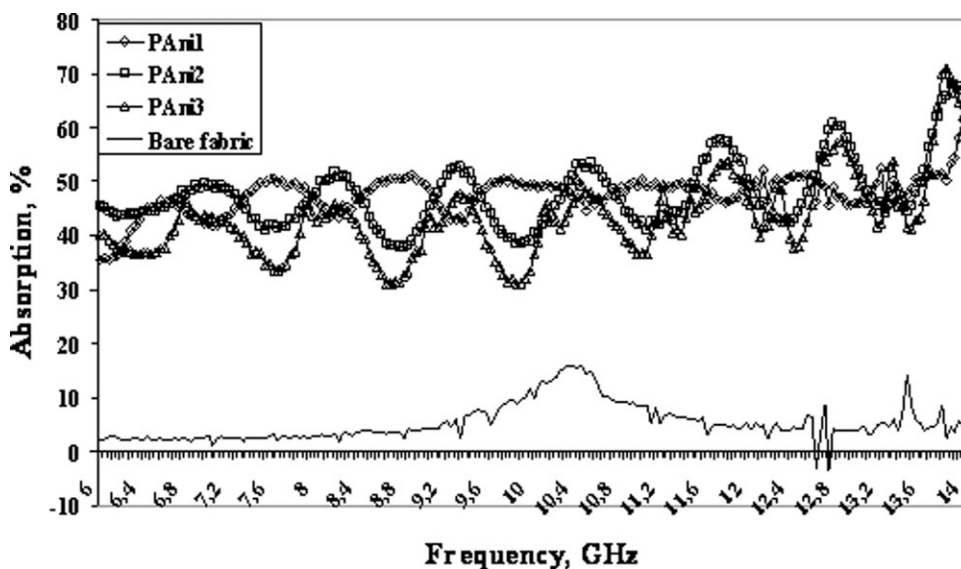

Figure 9 The absorption values of the fabric coated PAni as one, two, and three layer(s).
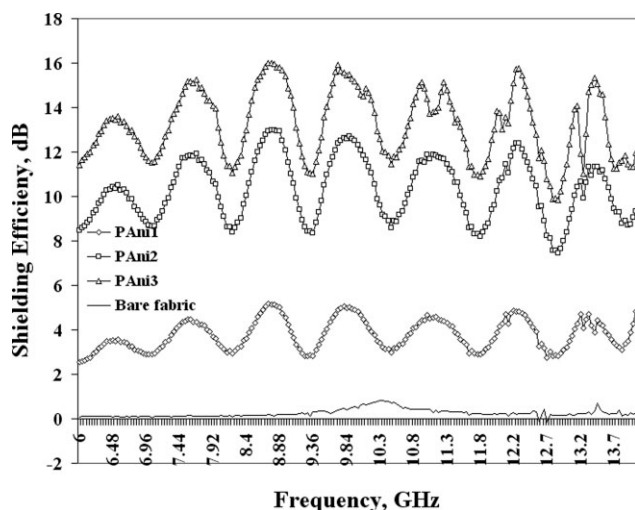
Figure 10 The shielding efficiency values of the fabric coated PAni as one, two, and three layer(s).

that the most appropriate process conditions to obtain the fabric samples with the lowest resistance values were using HCl as protonic acid, 4 h of totally polymerization time and without using doping agents.

The effect of adding of filling agent and filling concentration on resistance

Table VI showed the resistance and add-on values of 10 and 30% wof barium ferrite doped and undoped PAni coated fabrics during 3 : 1 h without adding doping agent with different layer number. As a result, the resistance values of the coated fabrics decreased, whereas the layer number increased in accordance with Ref. 15. The lower resistance values of the fabric coated with PAni as three layers and the fabric treated with aq. Barium ferrite solution

(10%wof) and then coated with PAni as three layers were 64 and 58 Ω, respectively. Furthermore, the resistance values of coated fabric increased although the barium ferrite content was increasing. That's why the barium ferrite has insulation properties. The add-on values of the fabric treated with 10 and 30% wof barium ferrite solution were found to be 7.46, 13.95%, respectively, whereas that of fabric coated PAni was 9.03%.

SEM analysis

Figures 4 and 5 showed the longitudinal and cross-sectional SEM images of coated and bare fabrics, respectively. The images showed the presence of PAni film on fiber as granular form. The addition of barium ferrite distinctly didn't change the surface of fiber as the surface of only PAni coated fiber.

Electromagnetic results

Figures 6, 7, and 8 showed the reflection loss, shielding effectiveness as dB and absorption values as % versus the frequency range of 6–14 GHz of the fabric coated with 10, 30%wof barium ferrite added PAni and only coated with PAni and bare fabric. The highest average absorption values were obtained for the fabric coated with PAni and 10% barium ferrite added PAni as 48%. The absorption value of the fabric coated with 30% barium ferrite added PAni was 27%. Figure 9 showed the absorption values of the fabrics coated PAni as one, two, and three layer(s). The average absorption values of the fabrics did not significantly change while number of coating layer of the fabric was increasing. Figure 10 shown the shielding efficiency of the fabrics coated PAni as
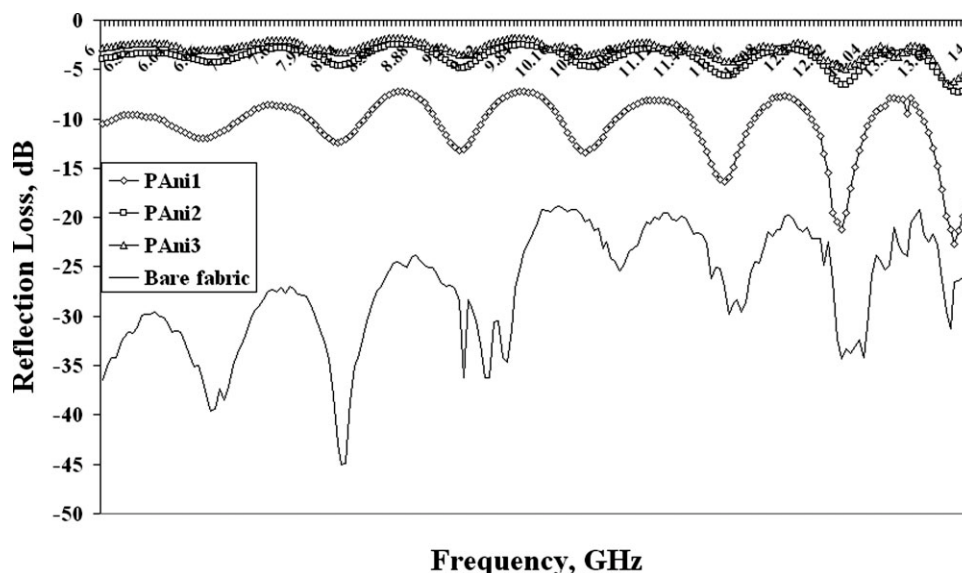

Figure 11 The reflection loss values of the fabric coated PAni as one, two, and three layer(s).

one, two and three layer(s). Mean shielding efficiency values of fabric increased from 3.78 to 13 dB while the number of PAni layer on the fabric was increasing and the resistance values of the fabric samples were decreasing. In Figure 11, the reflection loss values of the fabrics coated PAni as one, two and three layer(s) were illustrated. Mean reflection loss values decreased from −11 to −3 ("−" sign shows loss), whereas the number of PAni layer on the fabric was increasing and the resistance values of the fabric samples were decreasing.

CONCLUSIONS

In summary, the conductive cotton fabric coated with PAni with or without barium ferrite addition was produced. The fabrics have various shades of emeraldine green, resembling PAni, thus providing PAni deposition had indeed occurred. SEM images confirmed the presence of PAni coating on fiber as granular form. We determined 350 Ω of the minimum resistance values of coated fabric by using 1M HCl, 0.5M aniline and 0.5M ammonium persulphate by chemical oxidative polymerization method for totally 4 h of polymerization time. The addition of 10 and 30% wof barium ferrite in the coating certainly didn't affect the resistance and absorption values of coated fabrics in the frequency range of 6–14 GHz. The absorption values of the fabric coated PAni was average 48% in the frequency range of 6–14 GHz. It was deduced that the fabric has potential to use as microwave absorbing materials and electromagnetic shielding materials.

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