Conductivity and Atmospheric Aging Studies of Polypyrrole-Coated Cotton Fabrics

Amol J. Patil, Smita C. Deogaonkar

Conductive Textiles Laboratory, The Bombay Textile Research Association (BTRA), Ghatkopar, Mumbai 400086, India

ABSTRACT: Polypyrrole (PPy) is one of the preferred alternatives among the intrinsically conductive polymers (ICPs). In this study, PPy-coated cotton (PPy-CT) fabrics were synthesized by two step *in situ* chemical polymerization. The reaction parameters, such as monomer concentration and temperature, were studied in detail. The surface resistivity of PPy-CT fabrics ranged $\sim$ 15–5000 Ω^{-2}. To assess long-term usage potential, the atmospheric aging of conductivity characteristics of treated fabrics was monitored over a period of 6 months. It was found that the synthesis temperature had a significant impact on conductivity and atmospheric aging of PPy-CT fabrics. Furthermore, various sulfonic acid sodium salts added as external doping agents during polymerization also had a positive effect. The scanning electron microscopy revealed smoother morphology of sulfonic acid salt doped PPy coatings. The overall study addresses the durability aspect of PPy-CT fabrics in potential applications areas. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 125: 844–851, 2012

Key words: polypyrrole; aging; conducting polymers; cotton fabric

INTRODUCTION

Electrically conductive textiles are nowadays desired to possess conductivity along with flexibility, light weight, and comfort properties.[1] The advent of interactive textiles/electronic textiles consisting of an in-built electronic functionality has aggravated the need for conductive clothing with an acceptable level of comfort properties. This has put the role of conventional metal based textiles in jeopardy because of their weight burden, metallic appearance, and inflexible nature.[2] Some of the novel alternatives for imparting conductivity to textiles are intrinsically conductive polymers (ICPs),[3] carbon nanotubes,[4] and quantum tunneling composites.[5] These materials add negligible weight to textiles and retain flexibility and aesthetic properties. Amongst these, ICPs have simple chemistry, lower cost of preparation and are easy for synthesis.[6] They are produced prominently by chemical or electrochemical oxidation route. Polypyrrole (PPy), polyaniline and polythiopene are some of the most studied ICPs for producing electrically conductive textile substrates.[7,8] Interestingly, the chemical polymerization of ICPs on textile substrates has a close resemblance to the conventional textile dyeing and finishing operations providing an opportunity for easy adoption for large scale production for ICP-coated fabrics with existing machinery set-up.

The unprecedented conductivity characteristic of ICPs is marred by their tendency to undergo degradation in ambient atmosphere. This has been a major limiting factor in exploiting ICP for several potential applications.[9] Nevertheless, PPy was reported to have relatively better atmospheric stability and higher conductivity and has been regarded as one of the promising ICPs.[10] The inclusion of PPy on textiles substrates with a special focus on synthesis conditions and polymerization kinetics has been discussed in the literature.[7–9] Moreover, textiles coated with PPy have been produced commercially.[11,12]

Durability is an important aspect in any technology that aims its transition from lab scale to the practical usage. In our opinion, chemically polymerized PPy/textile composites at best could be termed as nondurable due to the fact that the alkaline atmosphere degrades the conductivity of PPy drastically as a result of the deprotonation of the chains accompanied by deintercalation of dopant ions that are responsible for conductivity.[13] Further, since house hold detergents are alkaline in nature, the washing and laundering of PPy-coated textiles may prove to be a contentious issue. The research reports have indicated a drastic decrease in conductivity for PPy coatings on wool[14] and polyester fabrics[15]

subjected to prevalent standard domestic laundering tests. Hurren and Kaynak reported[15] loss of conductivity even with distilled water washing. In this context, studies are essential to ascertain the stability of conductivity of PPy-coated textiles in products where washing/after-care is avoidable/not necessary, e.g., disposable electrostatic discharge (ESD) protection products in clean rooms, smart textiles, heat generation textiles, indoor electromagnetic interference (EMI) shielding, and microwave attenuating fabrics.

The atmospheric degradation kinetics of PPy in the form of powder and films has been studied in the literature.[9,16–20] These reports suggest that the degradation of PPy is a result of action of atmospheric oxygen, moisture, and harmful radiations distorting chain continuity irreversibly. Among these the oxygen attack has been reported to be the major source of degradation,[17] which was further substantiated by the studies showing better stability of PPy in inert atmosphere.[21] The loss in conductivity of PPy caused by atmospheric aging was found to be arrested to a remarkable extent by the use of arylsulfonate salts as dopants.[22–24] Further, the work so far suggests that the atmospheric degradation is a function of type and concentration of dopant ions, conditions and duration of aging.[24]. The issue of stability of PPy/textile composites has been dealt in greater detail in the context of thermal aging.[14,25,26] Although, atmospheric aging finds mention as a general observation[14,27–29] rather than specific studies, e.g., Acqua et al.[27] mentioned a loss of 70–80% in two weeks and Tao and coworkers[29] observed 10-fold increase after storage of several months.

This work aims at precise analysis and quantification of the atmospheric aging of PPy polymerized on cotton fabrics with more emphasis on the effect of synthesis conditions. For this purpose an innovative two–step polymerization approach has been adopted. The novelty of this approach lies in the controlled addition of oxidant and substrate motion to achieve slower reaction and uniform deposition respectively. So far, use of stationary baths was reported in the literature[30,31] to carry out two-step polymerization of PPy onto textiles which might result into nonuniform deposition.

EXPERIMENTAL

Materials

Scoured and bleached cotton fabric (plain weave, 130 g/m^2) was purchased from RSR Mohta mills, India. Pyrrole (97%) monomer was procured from Spectrochem, India. Iron (III) chloride hexahydrate was used as oxidant and procured from Loba Chemie. Anthraquinone-2-sulfonic acid sodium salt (AQSA, 97%), Naphthalene-2-sulfonic acid sodium salt (NSA, 98%); 2,6-disulfonic acid disodium salt (N_2D_2SA, 99%), Dodecyl benzene sulfonic acid (DBSA, 98%) were procured from Sigma Aldrich and used as additional dopants. Pyrrole was used after distillation and stored at 4°C. All other chemicals were used as received.

Methods

Synthesis of PPy on cotton fabrics

In situ chemical polymerization of PPy on textile substrate was carried out as a two step dynamic synthesis process. For this purpose a laboratory grade jigger machine (basically used for textile dyeing) was suitably modified. The details of the experimental set up were given in our previous work.[32]

Cotton fabric samples were cleaned before polymerization to remove left-over impurities. The fabrics were treated with monomer and oxidant sequentially to yield PPy-coated cotton (PPy-CT) fabrics. For polymerization, the monomer concentration was varied from 10 to 30% based on the weight of fabric (w/w). Fabric samples were cut along warp way (7 × 110 cm^2) direction with a weight of 10 ± 0.05 g. The molar ratio of monomer: oxidant was fixed at 1 : 2.33 because two electrons are required for oxidation of each pyrrole unit and the remaining 0.33 electrons are used for oxidative doping of neutral PPy chains.[33] The fabric sample was first treated in a monomer solution for 1 h allowing diffusion of monomer into the fibrous structure. It was followed by drop wise addition of oxidant solution for 1 h. Further, the treatment was continued for specific time which was counted as duration of polymerization. For each polymerization experiment, monomer treatment and oxidation addition time of 1 h was fixed, whereas the duration of polymerization was varied at 2 and 3 h. The color of the treated fabric changed gradually from white to black after the addition of oxidant evidencing the deposition of PPy. The polymerization was carried out at two temperatures: 4–5°C (low temperature) and 29–30°C (room temperature). The final material to liquor ratio was 1 : 40. The fabric samples were washed in distilled water after the completion of specified duration of polymerization.

Characterization

The electrical properties of fabric samples were measured in terms of electrical surface resistivity according to AATCC-76:2007 method.

Figure 1 shows the measurement set-up designed according to AATCC 76 method. Two gold plated copper rectangular electrodes (30 × 20 mm^2) were pressed on specimen surface (30 × 60 mm^2) with a
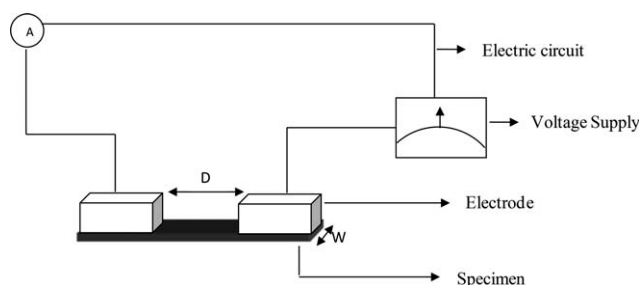
Figure 1 Schematic diagram of electrical surface resistivity measurement set-up.

load of 5 kg. The electric current values were acquired by applying DC voltage (0–10 V). The surface resistivity was calculated by following formula:

Surface resistivity (Ω/square)

$$= \frac{\text{Resistance}(R) \times \text{Width of specimen}(W)}{\text{Distance between electrodes}(D)}$$

Since each experiment was performed in triplicate and three specimens were selected from each sample, electrical surface resistivity value for a particular treatment was an average of nine readings. All measurements were performed in a conditioned atmosphere at a temperature of 20°C $\pm$ 2°C and relative humidity of 65% $\pm$ 5%. To assess atmospheric aging, these specimens were stored in ambient indoor conditions. The surface resistivity of these specimens was measured after every month for a period of 6 months. The morphology of PPy deposition on cotton fabrics was studied by scanning electron micrographs (SEM) on JEOL-JSM 5400 model at different magnifications. The weight add-on (%) of the PPy onto cotton fabric was estimated by calculating the difference in conditioned weights of treated and untreated fabric.

RESULTS AND DISCUSSION

Conductivity characteristics of PPy-CT fabrics

The polymerization of PPy is characterized by simultaneous chain growth and doping, the partial oxida-tion of chains thereby creation of positive charges on polymer backbone.[9] The Cl$^-$ anions released by the oxidant get settled near positive charges to maintain the neutrality of PPy polymeric backbone. The presence of anions (termed generally as dopants) is essential as the charge carriers only move in the vicinity of negative anions. The large surface area of cotton fabric in the reaction bath ensures adsorption of monomer and subsequent growth of polymeric chain on the fabric surface. The polymer growth initiates inside the amorphous region of cotton fiber accompanied by deposition of PPy into interfiber and interyarn spaces.[30]

Table I reports the electrical surface resistivity and weight gain values of experimental trials performed by varying monomer concentration, synthesis temperature, and duration of polymerization. As seen from the Table I, the surface resistivity of cotton fabric after treatment was drastically reduced in the range of $\sim$ 15–5000 Ω/square as a result of deposition of PPy. It is worth noting that the black color of the fabric was uniform evidencing good distribution of polymer onto the fabric surface.

For low temperature synthesis, with increasing duration of polymerization from 2 to 3 h, the surface resistivity decreased to almost half the value to that of the value at 2 h. This can be explained by the increase in weigh add-on of PPy-CT fabrics with increased duration (Table I). The weight increase ranges from about 5–24% depending upon the experimental conditions. It can also be pointed that the surface resistivity of untreated cotton fabric is around 10^9 Ω/square.[34] In this study, the weight add-on at the lowest pyrrole concentration (i.e., 10%) was mere 4–6%, whereas the corresponding reduction in resistivity of treated cotton fabrics was of 8–9 orders of magnitude. It typified the ability of ICP coatings to impart conductivity with a negligible weight burden onto textile substrate.

The surface resistivity of PPy-CT fabrics prepared at room temperature was higher compared with the low temperature synthesis for the same monomer concentrations at all three levels. This behavior is in agreement with the results reported for synthesis of

TABLE I
Electrical Surface Resistivity and Weight Gain Properties of PPy-CT Fabrics

	Low temperature synthesis			Room temperature synthesis	
Sample details	Surface resistivity (ohm/sqr)	Weight gain (%)	Sample details	Surface resistivity (ohm/sqr)	Weight gain (%)
Py/10/L/2h	2717 $\pm$ 229	4.6	Py/10/R/2h	5354 $\pm$ 967	5.1
Py/10/L/3h	1012 $\pm$ 110	6.0	Py/10/R/3h	4049 $\pm$ 150	6.5
Py/20/L/2h	86 $\pm$ 14	13.1	Py/20/R/2h	215 $\pm$ 37	12.5
Py/20/L/3h	44 $\pm$ 2	14.9	Py/20/R/3h	255 $\pm$ 24	12.5
Py/30/L/2h	27 $\pm$ 1	21.4	Py/30/R/2h	85 $\pm$ 9	17.2
Py/30/L/3h	17 $\pm$ 1	23.5	Py/30/R/3h	138 $\pm$ 16	16.6
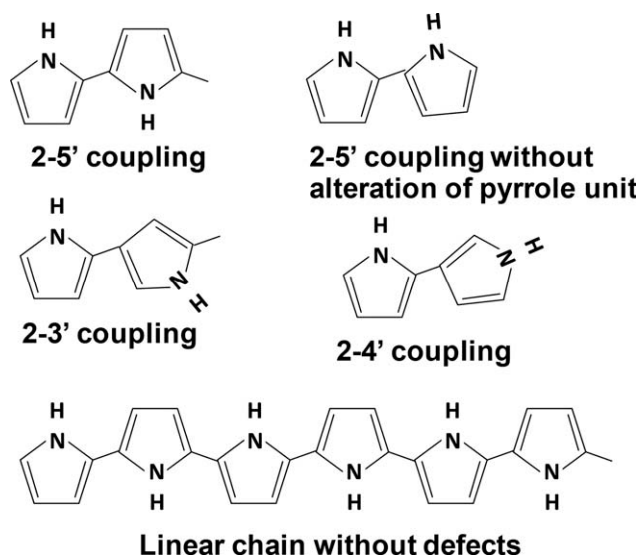
Figure 2 Prominent defects during PPy polymerization.

Figure 3 Effect of atmospheric aging on the surface resistivity of PPy-CT fabrics at 10% pyrrole concentration.

PPy[35] and PPy/polyester fabrics.[36] The low temperature of the reaction lowers the rate of reaction. The slower progression of reaction ensures joining of pyrrole units to growing chain preferably at 2-5′ position with 180° rotation.[33] PPy thus produced has a planarity and linearity of chains yielding higher conductivity characteristics. The increased reaction temperature results into higher rate of reaction and give rise to defective sites. Figure 2 shows some of the chemical defects occurring in PPy polymerization. Accordingly, the undesired couplings of pyrrole units could occur at 2-3′ or 2-4′ positions as well as at 2-5′ position without 180° rotation.[10] These defect sites restrict the mobility by breaking the planarity and linearity in chains which reflects into lower conductivity of PPy. It is worth to realize the importance of the controlled oxidant addition especially in the context of room temperature synthesis. The altogether oxidant addition could accelerate the reaction rate further worsening the conductivity of PPy. Furthermore, in case of room temperature synthesis, there has been no substantial decrease in surface resistivity values with the increase in duration

of polymerization from 2 h to 3 h as was observed in case of low temperature synthesis. The effect of higher reaction rate seemed to have dominated the polymerization kinetics nullifying the role of increased duration.

Aging studies

PPy possessing superior conductivity is characterized by an uninterrupted linear chain—termed as conjugation length—with fewer defects. It ensures hopping of charge carriers along the length aided by interchain transfer of charges.[21] In our studies, the aging characteristics of PPy-CT fabrics were analyzed by storage of samples in ambient atmosphere with due care to avoid any foreign matter interaction.

TABLE II
S_R Factor for PPy-CT Fabrics

Sample details	S_R factor	Sample details	S_R factor
Py/10/L/2h	10.7	Py/10/R/2h	34.9
Py/10/L/3h	9.9	Py/10/R/3h	42.3
Py/20/L/2h	8.2	Py/20/R/2h	15.7
Py/20/L/3h	6.7	Py/20/R/3h	19.1
Py/30/L/2h	7.3	Py/30/R/2h	13.3
Py/30/L/3h	7.7	Py/30/R/3h	11.7

Resistivity rise factor (S_R factor) = (Surface resistivity after 6 months- Initial surface resistivity)/Initial surface resistivity.
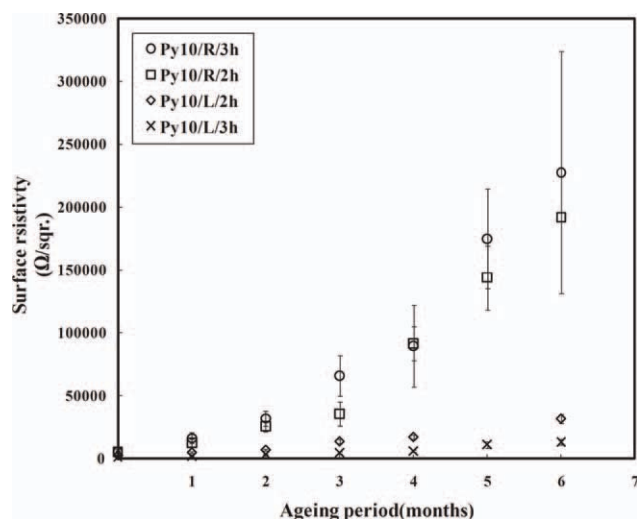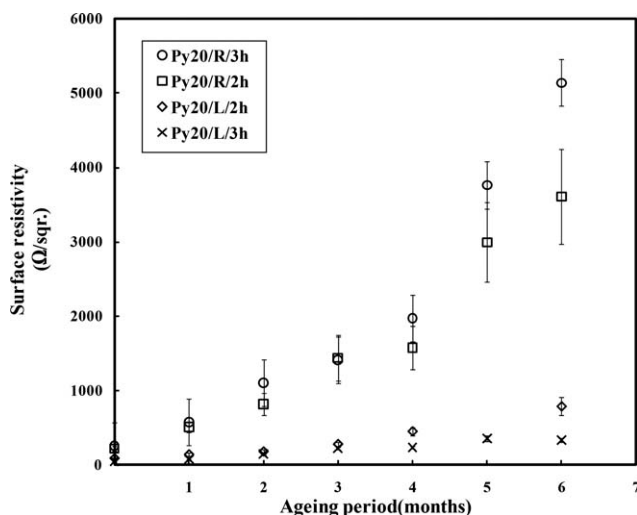

Figure 4 Effect of atmospheric aging on the surface resistivity of PPy-CT fabrics at 20% pyrrole concentration.
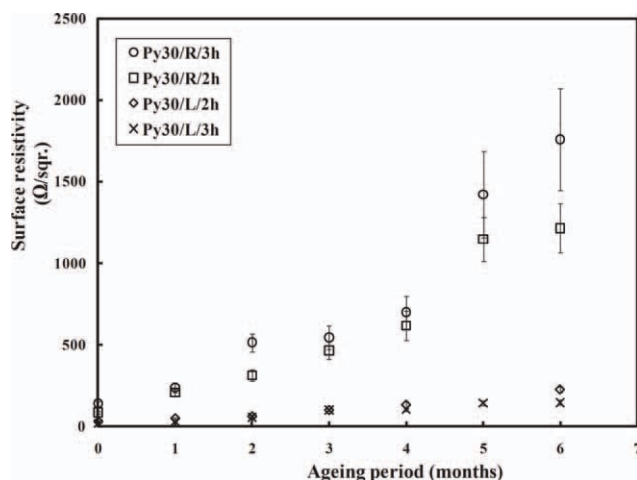
Figure 5 Effect of atmospheric aging on the surface resistivity of PPy-CT fabrics at 30% pyrrole concentration.

Table II lists the "S_R factor" for each experimental reaction condition. It was calculated by taking the ratio of increased surface resistivity in a span of 6 months to the initial surface resistivity in each case. The role of synthesis temperature is evident from the higher S_R factor observed for room temperature synthesis revealing higher conductivity degradation for all the three monomer concentrations with 10% showing maximal loss. It appears that higher the initial value of surface resistivity, the poorer is the stability of the structure toward degradation factors. Additionally, the surface resistivity values obtained after each month for a period of 6 months were studied graphically for further elaboration on the aging pattern. Figure 3 shows the progression of surface resistivity in 6 months for 10% pyrrole concentration.

The difference in rise of surface resistivity of Py10/R and Py10/L samples is evident. The absence of regioregular polymer structure for room temperature synthesis was the reason for rapid rate of rise of surface resistivity. This is also supported by the higher initial surface resistivity values (Table I). The atmospheric oxidation causes chemical degradation of PPy by disrupting the continuity in conjugation length and subsequently hinders the charge movement as well as charge hopping at defect sites. The reaction of oxygen on PPy backbone leads to formation of α, β-unsaturated carbonyl groups which are believed to act as an electron trap.[16] The low temperature synthesis has also resulted in atmospheric aging; nevertheless, the rate of rise is much slower with lower magnitude of increase in resistivity in the span of 6 months. The trend of higher rate of degradation of room temperature synthesis was also observed for 20 and 30% pyrrole concentrations (Figs. 4 and 5). For low temperature synthesis, aging of fabrics prepared with 3 h duration was found to be slightly less than the fabrics prepared with 2 h

duration for all the three pyrrole concentrations. This can be explained by the fact that there was a drop of initial surface resistivity values to almost half for each pyrrole concentration by increasing duration from 2 to 3 h. By contrast, no such relation was evolved between the aging characteristics and duration of polymerization for room temperature synthesis. Here, the surface resistivity values for 10% were statistically not significant for both durations (i.e., 2 and 3 h) in the entire period of 6 months. Further, for 20% and 30% concentration the values were found to be not significantly different except for the 6th month. Here, the increased value was noted for 3 h than for 2 h duration of polymerization. This behavior may be attributed largely to the similar initial surface resistivity values for both the durations of polymerization as against the lower surface resistivity values obtained for higher duration at low temperature synthesis. Further, for room temperature synthesis, the progression of curves suggests an exponential increase in aging at all the three concentrations. This is in good accordance with the aging behavior for PPy films reported by Kaynak et al.[24] They noted two regions of degradation that consisted of rapid rise followed by an exponential increase.

Effect of sulfonic acid dopants

The chloride (Cl⁻) ions released by the oxidant act as doping agents for PPy. After analyzing the degradation behavior of Cl⁻ doped PPy, it was decided to study the effect of sulfonic acid as dopants on PPy-CT fabrics. The addition of aromatic sulfonic acids makes significant difference in terms of improvement in conductivity and stability of PPy as reported in the literature. Accordingly, sulfonic acid salts such as AQSA, N_2D_2SA, NSA and DBSA were selected for doping during polymerization with dopant/monomer molar ratio of 0.2 and reaction conditions corresponding to treatment conditions: Py/10/L/3 h—"10" % pyrrole with low temperature (4–5°C) synthesis and "3" hours of polymerization (h). Figure 6 shows the effect of sulfonic acid dopants on the surface resistivity of PPy-CT fabrics. It is worth noting that the inclusion of sulfonic acid dopants resulted in the decrease in surface resistivity to the extent of 1/2–1/3 of the value corresponding to only Cl⁻ doped fabrics with the exception of DBSA. On the basis of the observed surface resistivity values, the order of dopant performance is: AQSA > NSA > N_2D_2SA > DBSA, Cl. Similar results have been reported for PPy-coated wool yarns whereupon the replacement of *p*-toluene sulfonate dopant with AQSA yielded a large reduction in the resistance.[37]

The more planer structure of the sulfonic acid dopants was believed to be responsible for
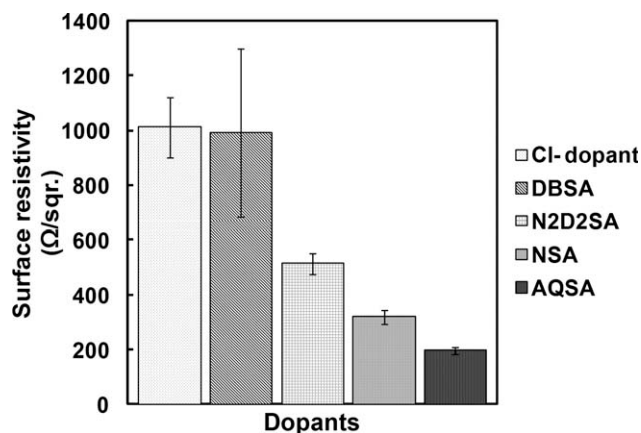
Figure 6 Effect of addition of sulfonic acid dopants on the surface resistivity of PPy-CT fabrics.

improvement in conductivity as it helps in interplanar stacking of dopant molecules.[38,39] This peculiar positioning of dopants between PPy chains facilitates interchain charge hopping more readily than small dopants such as Cl⁻ ions. Hence, the increased hopping opportunities contribute toward higher conductivity. Furthermore, the variation in the performance of the individual sulfonic acid dopants can be attributed to the two aspects, molecular size and concentration of dopants. The size of dopant molecules in terms of bulkiness alters the charge transfer in a remarkable way. The peculiar structure of DBSA, a bulky anion containing long alkyl chain with benzene ring, could have resulted in separation of PPy molecular chains eventually restricting the additional opportunities of charge hopping as compared with other sulfonic acid salts. Versano and Tonin[40] reported gelatinous precipitation in polymerization bath for PPy/wool substrate with DBSA as dopant. Although, we have not come across such observation even after using the same oxidant, it can be noted that DBSA is primarily a surfactant in nature. Hence, its behavior has to be studied in the context of "critical micelle concentration" (CMC), the amount above which any addition likely results in precipitation.

As regards the concentration, there have been different opinions about optimum amount of sulfonic acid salts, e.g., Varsano and Tonin reported dopant/monomer ratio of 0.8 as optimum in case of N_2D_2SA dopant for PPy/Wool fibers.[40] Further, an optimum ratio of 0.4 was deduced by Kaynak and Beltran based on the experiments with the use of AQSA for PPy/PET fabric.[36] These results suggest that for PPy coatings on textile, the optimization of dopant concentration is highly dependent upon the nature of textile substrate and its interaction with PPy along with the various other synthesis conditions. Finally, the effect of sulfonic acid salts on the atmospheric aging of PPy-CT fabrics was studied by varying the

concentration of NSA at three dopant/monomer ratios (0.1, 0.2, 0.3).

Figure 7 shows the surface resistivity profile of PPy-CT fabrics for NSA and Cl⁻ dopants. It is worth noting that the initial surface resistivity values of NSA doped fabrics were lower than only Cl⁻ dopant at all the three ratios. As regards aging, the increase in surface resistivity was found to be 13-fold in case of Cl⁻ doped fabrics within a period of 6 months, whereas NSA doped fabrics showed an increase of 2–3 times in the same period [Fig. 7(B)]. This shows that the NSA dopant had contributed in retardation of PPy degradation. SEM images of the Cl⁻ and NSA doped PPy-CT fabrics were evaluated (Fig. 8). The images obtained at lower magnifications [Fig. 8(a,b)] show no appreciable difference in the surface morphology of PPy coatings for both dopants. However, higher magnifications [Fig. 8(c,d)] reveal smoother surface of coatings for NSA doped fabrics, whereas Cl⁻ doped fabrics show surface with irregular serrations evidencing cracks. The microvoids and cracks on PPy surface could lead to easier oxygen ingress
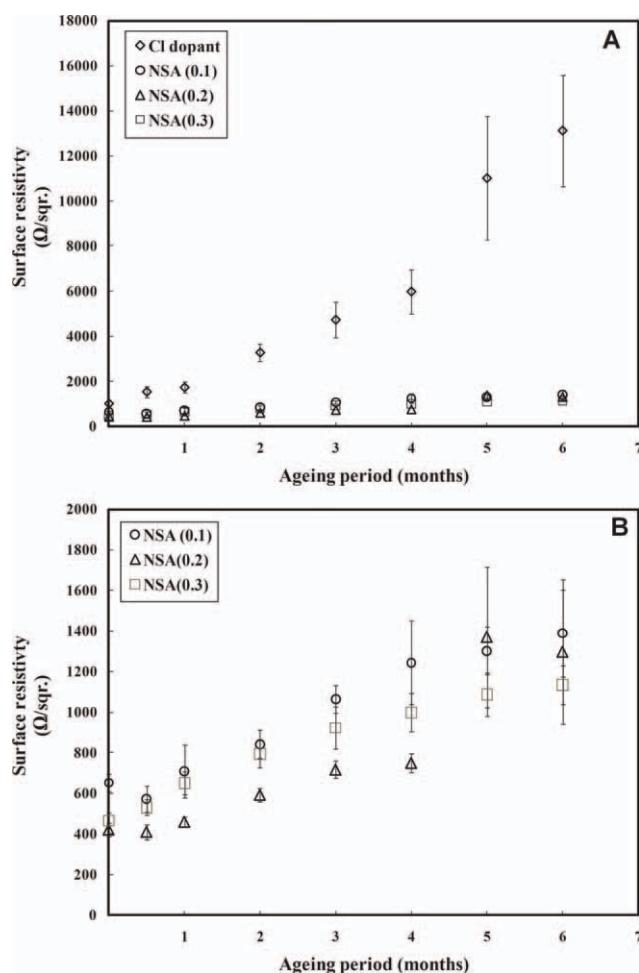

Figure 7 Aging behavior of PPy-CT fabrics: A: doped with Cl⁻ dopants and NSA dopants, B: NSA dopants (with enlarged scale).
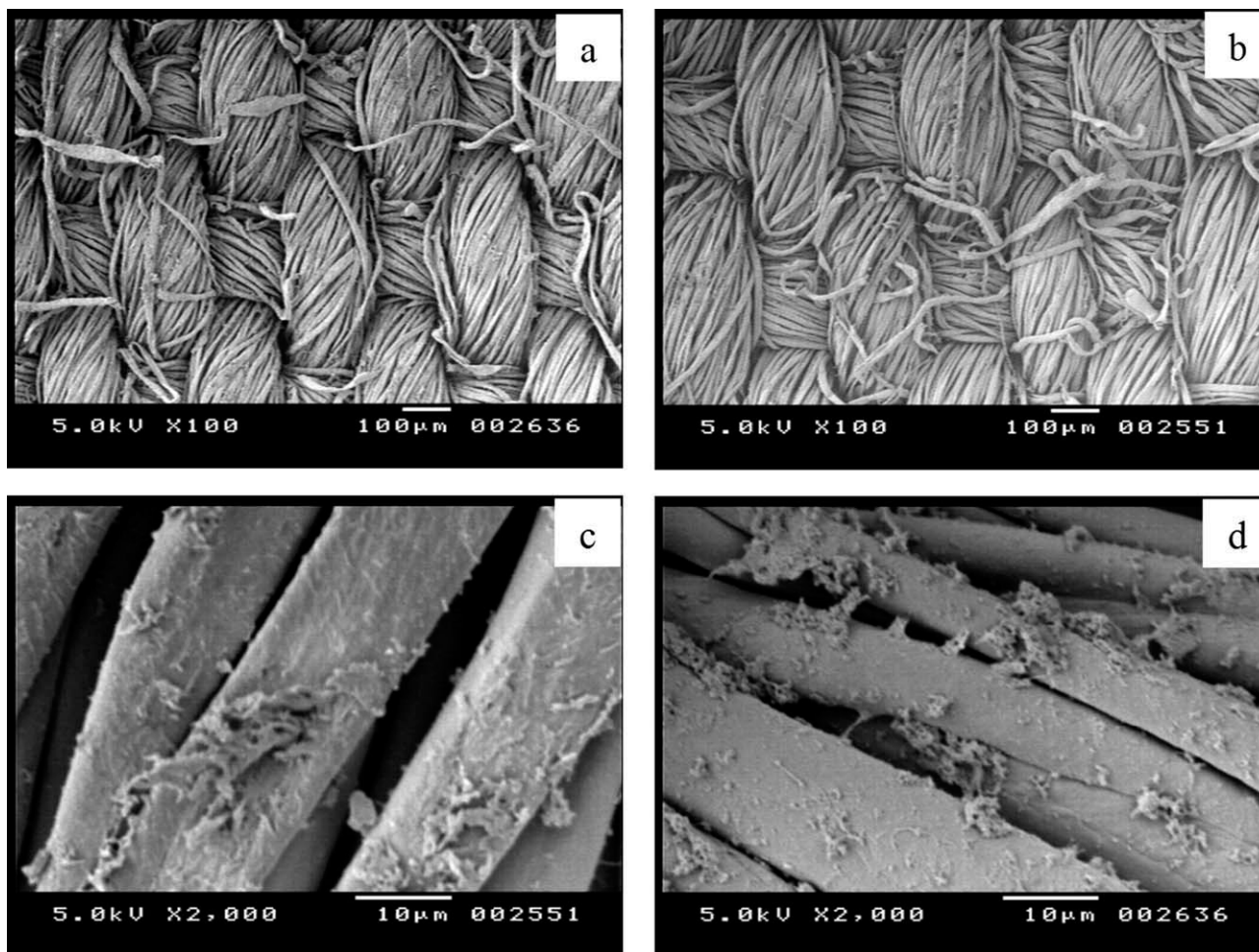
Figure 8 SEM of PPy-CT fabrics at 10% pyrrole concentration: (a) Cl^- doped (×100); (b) NSA doped (×100); (c) Cl^- doped (×2000); (d) NSA doped (×2000).

and thus deteriorate conductivity.[21] Kuhn et al. observed smoother morphological features of PPy films prepared with AQSA than Cl^- dopants which exhibited rough and porous surface.[25] It can be inferred that the smoother surface contributes toward reducing penetration of oxygen and subsequent attacks on the conjugation backbone.

CONCLUSIONS

The two step *in situ* polymerization has produced uniform coatings of PPy onto cotton fabrics. The surface resistivity obtained was in the range of 15–5000 Ω^{-2} by varying the reactant concentration and reaction parameters. It is evident from the study that synthesis temperature plays a pivotal role in conductivity and atmospheric stability of PPy-CT fabrics. The low temperature synthesis (4–5°C) produces PPy coatings with higher conductivity and superior resistance against atmospheric aging when compared with room temperature synthesis. Hence, although low temperature synthesis may seem to be a burden for

prospective commercial process, it can be justified in the context of excellent performance of PPy coatings. The addition of sulfonic acid performs the dual role of improvement in conductivity and atmospheric stability in the studied dopant concentration range. The atmospheric aging was controlled substantially with the addition of NSA dopants during polymerization which can be attributed to the smoother morphology of PPy coatings. The PPy-CT fabrics thus produced needs to be analyzed further to satisfy performance criteria in actual applications.

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