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Characterization of electro-conductive fabrics prepared by *in situ* chemical and electrochemical polymerization of pyrrole onto polyester fabric

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ABSTRACT

This paper reports a study on electro-conductive fabrics prepared by a combined *in situ* chemical and electrochemical polymerization of pyrrole. Specific observations are made to establish the roles of add-on and surface roughness on the surface resistivity of the electro-conductive fabrics. The performance characteristics of the fabrics are reported in terms of electrical conductivity, voltage–current and voltage–temperature characteristics and electromagnetic interference (EMI) shielding capability. The surface resistivity of the fabric was found to be as low as 11.79 Ω. The voltage–current profile of the fabric is observed to be non-ohmic as well as the voltage–temperature curve is found to be exponential. The EMI shielding efficiency of the fabric was found to be about 98%.

1. Introduction

The award of the Nobel Prize in chemistry on the discovery of electro-conductive polyacetylene for the year 2000 [1] excited many researchers all over the world that resulted in quick development of many more electro-conductive polymers such as polyaniline, polypyrrole, polythiophene and their derivatives. The conjugation in the polymers is responsible for the electrical conductivity which could come very close to that of metals; in fact, sometimes these are termed as synthetic metals. Subsequently it was realized that these polymers have some limitations, in that they have poor processability and environmental stability [2,3]. Despite these challenges, there have been notable successes in converting these electro-conductive polymers into fibres, yarns, fabrics and other flexible products.

Polypyrrole is established as one of the most promising electro-conductive polymers. It has received more attention due to its high conductivity, ease of preparation, and environmental stability and shown potential for a wide range of applications in sensors, actuators and electronic and electrical devices [4–7]. Pyrrole can be polymerized by many techniques such as chemical polymerization, vapor phase polymerization and electrochemical polymerization. Gregory et al. [8] demonstrated the process for preparing electro-conductive textiles by *in situ* polymerization of either aniline or pyrrole on the surface of polyester or nylon fabrics. Diaz et al. [9] reported that polymerization of pyrrole by electrochemical polymerization on platinum electrode produced a stable polymer film with good electrical properties. Since then, Park et al. [10], Chen et al. [11], Hwang et al. [12], Bhadani et al. [13], Maiti et al. [14] and Sen et al. [15] made significant contributions to this field. Subianto et al. [16] prepared electro-conductive cotton fabric by electrochemical polymerization of pyrrole. They reported that the conductivity decreased with an increase in current density, however, at a fixed current density, the conductivity increased with an increase in the dopant concentration. Kim et al. [17] prepared a stretchable electro-conductive fabric by electrochemical polymerization of pyrrole onto nylon/spandex stretchable fabric. They observed that the electro-conductivity of the fabric first increased and then decreased with the increase in the concentrations of monomer and dopant and the time of polymerization. This observation, however, did not exactly match with those reported by Park et al. [10] and Hwang et al. [12]. Molina and co-researchers [18] developed electro-conductive textile by the combination of chemical and electrochemical polymerization and also found that change in counter ion during the process changed the conductivity of the fabric. Babu et al. [19] produced electro-conductive cotton fabric by the combination of chemical and electrochemical polymerization at a constant current density (2 mA cm^{-2}) at room temperature for 4 h. They reported that the conductivity and weight gain were directly proportional to monomer concentration as well as

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polymerization time. Maiti et al. [20] reported that the polymerization time and temperature played a significant role in determining the electrical conductivity of cotton yarn. Apart from these studies, a few researchers also characterized the electro-conductive fabrics in terms of voltage–current and voltage–temperature characteristics. Acqua et al. [21] reported linear voltage–current characteristics of viscose and lyocell fabrics treated with polypyrrole however, Bhat [22] and Cucchi [23] observed non-linear voltage–current characteristics of polyaniline treated cotton fabric and polypyrrole treated silk fabric, respectively. Das et al. [24], however, reported non-linear voltage temperature behaviour of electro-conductive fabrics.

While from the above, it is established that *in situ* chemical or electrochemical polymerization can produces flexible electro-conducting textiles, many questions still need clear answers, such as, (a) is the add-on related to conductivity [17–19], (b) does the surface morphology of deposited polymer plays any role, or (c) are the voltage–current or voltage–temperature characteristics [20–24] of these products linear or non linear. Clarity on these can enhance their potential for some interesting applications in future.

In the present study, an attempt has been made to get answers to the above questions and also to examine the electromagnetic shielding behaviour of the polyester electroconductive fabrics prepared by *in situ* chemical and electrochemical polymerization process.

2. Experimental

2.1. Materials and chemicals

Polyester woven fabric of 58 g/m^2 weight, 0.19 mm thickness, 38 ends per cm, and 32 picks per cm was chosen as a substrate for this study. The chemicals used were sodium carbonate, sodium hydroxide, non-ionic detergent lissapol N (HR Chemical, India), pyrrole (Spectrochem, India), ferric chloride and p-toluene sulphonic acid (Lobal Chemie, India). All these chemicals were of laboratory grade and they were used as received.

2.2. Preparation of electro-conductive fabric

The electro-conductive fabrics were prepared by a combined *in situ* chemical and electrochemical polymerization of pyrrole onto the polyester fabric. The fabric was first saponified with 100 g/L sodium hydroxide at 90 °C for 20 min, keeping the material-to-liquor ratio as 1:30. A two-step polymerization of pyrrole was carried out. In the first step, the *in situ* chemical polymerization of pyrrole was carried out. The hydrolyzed fabric was immersed into pyrrole solution containing 0.5 M pyrrole at 25 °C for 20 min. The pyrrole-enriched fabric was then immersed into 0.75 M ferric chloride solution so as to initiate polymerization onto the fabric at 5 °C for 10 min. After that, the fabric was washed thoroughly with deionised water and dried in an oven at 60 °C for 40 min. These conditions were chosen based on our previous studies [25,26]. In the second step, the *in situ* electrochemical polymerization of pyrrole was carried out onto the fabric in a potentiostat using a regulated DC power supply (ESCORP). The stainless steel electrodes of grade AISI 304 were kept vertically parallel to each other. The fabric was suitably fixed on the anode surface during the electrochemical polymerization The electrolyte solution was prepared in aqueous medium containing 0.3 M pyrrole along with 0.05 M p-toluenesulfonic acid. The voltage, time and temperature of polymerization were varied to produce a wide range of conducting fabrics.

Three process factors (applied voltage, polymerization time, and polymerization temperature) were further optimized by means of a 3^3 Box–Behnken design of experiments along with response surface methodology of analysis. Table 1 displays the levels of the process factors chosen for Box–Behnken design.

Table 1
Process factors and their levels according to Box–Behnken design.

Factors	Levels		
	−1	0	+1
Polymerization time (min)	30	60	90
Polymerization temperature (°C)	15	25	35
Applied voltage (V)	2.0	2.5	3.0

2.3. Measurement of surface resistivity of electro-conductive fabric

The surface resistivity of the electro-conductive fabric was measured by concentric ring-disc electrode configuration with 10-mm outer radius of the inner disc and 25-mm inner radius of the outer ring in a controlled atmosphere at 27 ± 2 °C temperature and 65 ± 2% relative humidity. The testing was carried out as per ASTM standard D-257. The surface resistivity was determined as follows:

$$\rho_s = 2\pi \ \ln\left(\frac{r_1}{r_2}\right) R_s \quad (1)$$

where ρ_s denotes surface resistivity, R_s indicates surface resistance, r_1 stands for the radius of the disc and r_2 refers to the inner radius of the outer ring electrode.

2.4. Measurement of surface roughness of electro-conductive fabric

The surface roughness of the fabric was measured with an instrument Form Talysurf Intra supplied by Taylor Hobson Precision, UK. The stylus, attached to the device, was moved on the substrate from one end to the other and the vibrations were recorded. The rougher is the surface the more is the vibration of the stylus.

2.5. Scanning electron microscopy of electro-conductive fabric

The surface morphology of the surface was studied using scanning electron microscope, Zeiss EVO 50. As the treated fabric samples had enough electrical conductivity, coating of the sample was not carried out.

2.6. Measurement of voltage–current characteristic of electro-conductive fabric

The voltage–current behaviour of the electro-conductive fabrics was characterized using regulated DC power supply equipment (ESCORP). The conducting fabric sample (50 mm × 25 mm) was fixed between two clamps and direct voltage, ranging from 0 V to 8 V, was applied to the fabric and the current in the circuit was recorded.

2.7. Measurement of voltage–temperature characteristic of electro-conductive fabric

The voltage–temperature characteristics of the electro-conductive fabric were characterized by applying direct voltage to a strip of electro-conductive fabric (50 mm × 25 mm), which was placed between two clamps. The temperature rise in the fabric sample was recorded (using IR thermometer) at different applied voltages (2 V, 4 V, 6 V and 8 V) for different time periods (2 min, 4 min, 6 min and 8 min).