CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Monomers

Aniline was purchased from Merck and thiophene was purchased from Aldrich.

3.1.2 Oxidizing Agents

Ammonium peroxydisulfate (APS) was purchased from sigma and ferric chloride ($FeCl_3$) was purchased from Aldrich.

3.1.3 Surfactant

Dodecylbenzene sulfonic acid, sodium salt, tech. (DBSA) was purchased from Aldrich.

3.1.4 Dopants

Dodecylbenzene sulfonic acid, sodium salt, tech. (DBSA) and benzene sulfonic acid (BSA) were purchased from Aldrich. 5-sulfosalicyclic acids dehydrate (5-SCA) and p-toluenesulfonic acid monohydrate (PTSA) were purchased from Sigma. (+)-camphor-10-sulfonic acid (β) (CSA) was purchased from Fluka.

3.1.5 Fabric

Plain weave polyester fabric (fabric weight = 161.0 g/m^2.) was used. The fabric was washed in a washing machine at 95°C several times until it was free from any remaining surfactant before use.

3.2 Equipment

Air circulating oven

Dyeing Machine (Daelim Starlet Model DL-6000)

Scanning Electron Microsope JEOL, model JSM 2590+

(Joel Ltd., Tokyo, Japan)

Electrometer/high resistance meter KEITHLEY Model 6517A

Resistivity test fixture KEITHLEY Model 8009

3.3 Experimental Procedures

3.3.1 Monomer Purification

Aniline monomer was purified by vacuum distillation at 50-70°C several times. It was stored at 0-5°C and covered with aluminium foil to keep away from light. Thiophene monomer was used as received.

3.3.2 Admicellar Polymerization

The conditions used for admicellar polymerization were summarized in Table 3.1. The pH of the solution was adjusted to 4 using HCl. The fabric (8×8 cm^2) was immersed into the dyeing pot containing solution of 1.2 mM DBSA, 10 mM monomer, and 0.5 M NaCl. Total volume of the solution was equal to 60 mL. The pots were placed in the dyeing machine using a turning speed of 45 rpm. The temperature was set at 30°C for 15 h for the combined admicellar formation and monomer adsolubilization steps. After that, the oxidant was added into the pots and polymerization was allowed to take place at 30°C for 4 h. Then, the treated fabric was washed in water at 80°C for several times until the absorbance of the washing liquid is less than 0.015 at the wavelength of 225 nm. The fabric was then dried in an oven at 65° C overnight.

Table 3.1: Admicellar polymerization conditions

	Type of Monomer	
	Aniline	Thiophene
Oxidant	APS	FeCl$_3$
Monomer concentration (mM)	10	10
Oxidant: monomer ratio	1:1	1:1
NaCl concentration (M)	0.5	0.5

3.3.3 Doping of the Coated Fabric

Two methods of doping coated fabric were studied to find the best method in terms of the resistivity of the doped fabric.

a) Doping after the final step of admicellar polymerization

The first method of doping was doping after the final step. A solution of dopant was prepared by using distilled water. The coated fabric was immersed in an aqueous dopant solution for 2 h. After this, the doped fabric was taken out and dried in the oven at 65 °C for 2 h.

b) Doping during the first step of admicellar polymerization

The second method was doping during the first step by mixing the solution of dopant, monomer, surfactant and salt together. The fabric was treated in the mixed solution at 30°C for 15 h. After that, the oxidant was added and the polymerization was allowed to take place at 30°C for 4 h. The doped fabric was then rinsed with water and dried at 65°C for 2 h.

3.3.4 Testing and Characterization

3.3.4.1 *Resistivity measurements*

The resistivity of the fabric sample was measured by using Electrical Resistance Meter at 25±1 °C with 60±2% relative humidity. The specimens were kept in this condition for more than 24 h before testing. The fabric sample (8×8 cm) was placed between two electrodes (resistivity test fixture KEITHLEY Model8009). According to the ASTM standard D-257, a DC voltage of 500 V was applied for 60 seconds. For measuring the surface resistivity, voltage was applied across the surface of sample. The resulting current is measured and the resistivity is calculated from the following equation:

$$\text{Surface resistivity,} \qquad \rho_s = \frac{53.4V}{I} \qquad \text{ohm/ sq}$$

For each sample, the surface resistivity of both sides of the fabric was measured and the average was taken. For measuring the volume resistivity, the voltage was applied across the sample and the resulting current was measured. The volume resistivity value was calculated from the geometry of the electrodes and the thickness of sample using the following equation:

$$\text{Volume resistivity,} \qquad \rho_v = \frac{22.9V}{It} \qquad \text{ohm-cm}$$

where t = fabric thickness = 0.4 mm

For each experimental condition, at least 3 samples were prepared and the average value was reported.

3.3.4.2 *Surface morphology of the coated fabric*

Scanning Electron Microscope (SEM) was used to study the surface morphology of the coated fabric. Specimens were sampled at random from different fabric locations and sputter coated with gold prior to observation.