CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Monomer

Aniline was purchased from Merck. Pyrrole, thiophene and *N*-methylpyrrole were purchased from Aldrich.

3.1.2 Oxidizing agent

Ammonium peroxydisulfate (APS) was purchased from Sigma. Ferric chloride was purchased from Aldrich.

3.1.3 Surfactant

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

3.1.4 Fabrics

Plain weave cotton and polyester fabrics were used. The fabrics were washed in a washing machine at 95°C several times until they were free from any remaining surfactant before use.

3.2 Equipment

- Air circulating oven
- Shaken water bath
- Scanning Electron Microscope JEOL, model JSM 2590+ (Joel Ltd., Tokyo, Japan)
- Programmable Electrometer/Source KEITHLEY Model 617 (Keithley Instrument, Inc., Cleveland, Ohio, USA)
- Picoammeter/Voltage Source KEITHLEY Model 487 (Keithley Instrument, Inc., Cleveland, Ohio, USA)

- Resistivity Chamber KEITHLEY Model 6105 (Keithley Instrument, Inc., Cleveland, Ohio, USA)

3.3 Experimental Procedures

3.3.1 Purification of monomer

Aniline (Merck) and pyrrole (98%, Aldrich) was purified by vacuum distillation and kept cool in the dark prior to use. Thiophene (99+%, Aldrich) was used as received. N-methylpyrrole (99%, Aldrich) was filtered through basic alumina (Sigma) several times.

3.3.2 Admicellar polymerization

The admicellar polymerization of monomer on fabrics was carried out using 1.0, 1.2 and 1.4 mM aqueous DBSA solution at pH 4 with different types of monomer, monomer concentrations, amounts of salt, and oxidant:monomer ratios. The 8×8 cm^2 fabric was placed in the test tube containing surfactant and monomer solution. Then the test tube was placed in the shaking bath at 30°C for 15 hours to allow time for admicellar formation and monomer adsolubilization into the admicelle. Total volume of the solution was equal to 80 ml. Then oxidant was added into the test tube and polymerization was allowed to take place at 30°C for 4 hours. For the polymerization of aniline on fabrics, ammonium peroxydisulfate was used as oxidant. The oxidant FeCl$_3$ was used for pyrrole, N-methylpyrrole and thiophene monomers. Conditions used in this part were summarized in Table 3.1. Following polymerization, the treated fabric was taken out from the test tube and washed by stirring in tap water in a beaker two times and dried at 65°C in an oven overnight prior to use.

Table 3.1 Admicellar polymerization conditions

	Type of Monomer			
	Aniline	Pyrrole	Thiophene	N-methylpyrrole
Oxidant	APS	FeCl$_3$	FeCl$_3$	FeCl$_3$
Surfactant concentration (mM)	1.0, 1.2, 1.4	1.0, 1.2, 1.4	1.0, 1.2, 1.4	1.2
Monomer concentration (mM)	5,10,15	5,10,15	5,10,15	5,10,15
Oxidant:monomer ratio	1:1, 2:1	1:1, 2:1	1:1, 2:1	1:1, 2:1
NaCl concentration (M)	0, 0.15, 0.5, 1, 1.5	0, 0.15, 0.5, 1, 1.5	0, 0.15, 0.5, 1, 1.5	0, 0.15, 0.5, 1, 1.5

3.3.3 Testing and characterization

3.3.3.1 Surface morphology of the treated fabric

Surface morphology of the treated fabric was examined by Scanning Electron Microscope (SEM). Specimens were sampled at random from different sections of the fabric and sputter coated with gold prior to imaging.

3.3.3.2 Volume and surface resistivity of the treated fabric

The surface resistivity of the treated fabric was measured at 70±5 % relative humidity, 22±2°C (ASTM D257). The specimens were kept in this condition for more than 24 hours before testing. The treated fabric was placed between two electrodes (resistivity Chamber KEITHLEY Model 6105). The measurement is dependent upon both the surface and volume resistance of the sample. According to the ASTM standard, a DC voltage of 500 V was applied by Picoammeter/Voltage Source KEITHLEY Model 487 for 60 seconds and the resulting current was then measured by Programmable Electrometer/Source KEITHLEY Model 617. The

volume resistivity is measured by applying a voltage across the sample and measuring the resulting current. The resistivity is calculated from the geometry of the electrodes and the thickness of the sample. The surface resistivity is measured by applying a voltage across the surface of the sample, between the bottom two electrodes. The resulting current is measured and the resistivity is calculated from the following equation:

Volume resistivity,

$$\rho_v = \frac{22.9V}{tI} \quad \text{ohm-centimeter} \tag{3.1}$$

Surface resistivity,

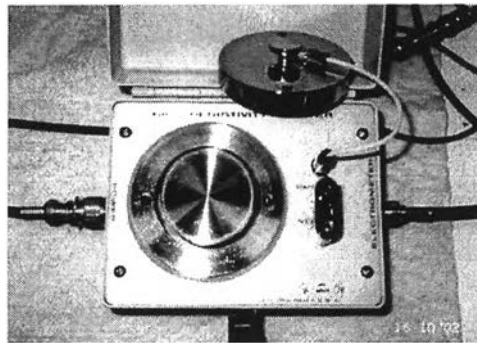$$\rho_s = \frac{53.4V}{I} \quad \text{ohms} \tag{3.2}$$
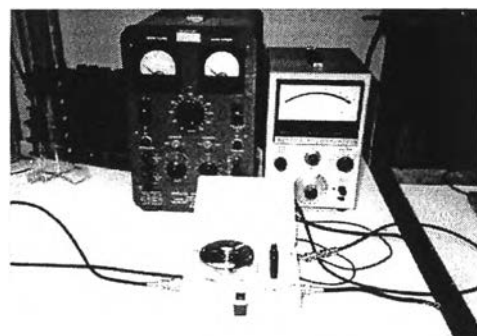

Figure 3.1 Electrode of resistivity chamber.

Figure 3.2 Electrometer for volume and surface resistivity measurements.