CHAPTER III
EXPERIMENTAL

3.1 Materials

O-anisidine (Grade AR, Aldrich) was used as monomer. Ammonium persulfate (APS) (Grade AR, Sigma-Aldrich) and 0.1 M hydrochloric solution (ACI Labscan) was used as an oxidant and a catalyst. Sodium dodecyl lsulfate, SDS (Grade AR, LobaChemie) was used as a surfactant. Deionized water, acetone (ACI Labscan), ethanol(Grade AR, QRëC) were used as solvent and used without purification.

3.2 Experimental

3.2.1 Synthesis of Poly *o*-anisidine (POA)

POA was synthesized via chemical oxidation polymerization (Mazrouaa*et al.*, 2012). A 0.49 g of APS was dissolved in 0.1 M HCl solution (10 ml). An *o*-anisidine (OA) 0.52 g (0.0043M) and SDS were dissolved in 0.1 M HCl (100 ml) at various mole ratios (mole SDS/mole OA) from 0.008 to 16 mole ratio. Then APS solution was dropped into the monomer solution at 1.0 ml/min. The monomer solution and the APS (oxidizer) were mixed at 3, 25, and 60 °C for 18, 24, 48 and 72 h. The product was terminated and precipitated with methanol. The dark green of POA was filtered and washed with methanol, DI water, and acetone and dried at 50 °C for 72 h.

3.3 Characterization

3.3.1 Fourier transforms infrared spectrometer (FT-IR)

FT-IR spectrometer (Nicolet, Nexus 670) was used to characterize the functional groups of synthesized POA. The FT-IR absorption was taken for 64 scans at the wavenumber between 400-4000 cm^{-1} with a resolution of 4 cm^{-1}. The sample

was prepared by glided POA with potassium bromide (dried at 100 °C for 24 h) then compressed to a pellet and inserted in a sample holder.

3.3.2 Ultraviolet-visible spectrophotometer (UV-VIS)

A UV-VIS spectrophotometer (UV-Tecan, Infinite M200) was used to determine the spectra peak of POA structure at wavelengths from 230 nm to 1200 nm.

3.3.3 Proton Nuclear Magnetic Resonance Spectroscopy (H-NMR)

H-NMR (Bruker, Avance) was used to characterize the functional groups and determine the structure of POA.POA was dissolved in 1 ml of DMSO-d_6 and measured in 30 min to prevent the oxidation of the polymer structure.

3.3.4 Scanning Eelectron Microscope (SEM)

SEM (JEOL, model JSM-5410LV) was used to observe the morphology of POA. POA particles were placed on a carbon tape and coated with a thin layer of gold prior measurement. Magnifications used were 20000x and 35000x with 15 kV of electrons beam.

3.3.5 Thermo Gravimetric Analyzer (TG-TGA)

Thermo gravimetric (Perkin Elmer, Pyris Diamond) analyzer was used to investigate the weight losses of volatile molecules, the amount of residual water, and the degradation temperatures of POA.The samples were weighed in the range of 4-10 mg and loaded into an alumina pans. The thermograms of POA were performed with a temperature scan from 25 °Cto 600 °C and with a heating rate 10 °C/min under nitrogen atmosphere.

3.3.6 Tensiometer

Tensiometer (Kruss, Easydyne) was used to investigate the surface tension and critical micelles point (CMC) of SDS. The SDS was dissolved in DI-water at various concentrations from 0.5-16 M. A solution was placed in a water bath to measure surface tension values.

3.3.7 Two-point Probe Meter

The two-point probe meter was used to measure the specific resistivity (ρ) of the material. The specific resistivity indicates the ability of material which resists electrical charge movement. This meter consists of two-point probe, the one probe was connected to a voltmeter for measuring the voltage and the other

probe was connected to a constant current source. The specific conductivity (σ) was calculated from the specific resistivity by using this Eq. (1).

$$\sigma = 1/\rho = 1/(R_s t) = I/(KVt) \qquad (1)$$

Where σ is the specific conductivity (S/cm.), ρ is the specific resistivity (Ω.cm.), R_s is the sheet resistivity (Ω), I is the applied current (A), K is the geometric correction factor, V is the voltage drop (V), and t is the film thickness (cm.). The sheet resistance (R_s) was obtained by introducing a current (I) through the outer two pins and determining the voltage drop (V) across the inner two pins. The sheet resistance was calculated from Eq. (2) as follow:

$$R_s = K \times (V_a / I) \qquad (2)$$

The geometric correction factor was taken into account geometric effects, depending on the configuration and probe tip spacing. The geometric correction factor was determined by a standard material with a known specific resistivity. The sheet resistivity obtained from the two-point probe meter was used to calculate the geometric correction factor by the following Eq. (3).

$$K = \rho_{ref} / (R_s t) \qquad (3)$$

where ρ_{ref} is the known specific resistivity which was 107.373 (Ω.cm.).