

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Materials

Divinylbenzene (DVB) and styrene (S) were purchased from Merck and Sigma-Aldrich, respectively. Surfactant, sorbitan monolaurate (Span 80), dodecylbenzenesulfonic acid, sodium salt (DDBSS), were purchased from Sigma-Aldrich chemical and cetyltrimethylammonium bromide (CTAB) was supplied from Fluka. The initiator and stabilizer were potassium persulphate ( $K_2S_2O_8$ , purity  $\geq 98\%$  (RT), Fluka) and calcium chloride ( $CaCl_2$ , purity  $\geq 97\%$  (KT), Fluka). Toluene (T) was supplied by Lab scan. Polystyrenesulfonate (PSS, sodium salt, MW 70,000), poly(diallyldimethylammonium chloride) (PDADMAC, MW 350,000), polyethyleneimine (PEI, MW 2000), and tetraethylenepentamine (TEPA, MW 198) were purchased Sigma-Aldrich. All the chemicals were used as received.

#### 3.2 Experimental Procedures

##### 3.2.1 Preparation of Poly(S/DVB)HIPE

Poly(S/DVB)HIPE was prepared by styrene, divinylbenzene, mix surfactant (Span 80, CTAB, DDBSS, 6.3 wt%, 0.3 wt%, 0.4 wt%), and 5 mL of solvent as toluene mixed together as an organic phase. The ratio of Styrene:Divinylbenzene used in this study were 0:100, 20:80, and 80:20. Next, aqueous phase contained 90 mL of deionized water, 0.2 g of potassium persulphate as initiator, and 1 g of stabilizer salt ( $CaCl_2$ ). The mixture of aqueous phase was slowly dropped to the organic phase with mechanical stirring at 360 rpm. Then, the emulsion was poured into glass mold and polymerization of polymer was carried into a water bath at  $60\text{ }^\circ\text{C}$  for 48 hours. Next, poly(S/DVB)HIPE was removed from glass mold and dried in oven at  $60\text{ }^\circ\text{C}$  until for 24 hours. Unreacted styrene and divinylbenzene were extracted by Soxhlet extraction for 6 hours using 2-propanol. Finally, the materials were returned to dry in oven at  $60\text{ }^\circ\text{C}$  until a constant weight.

### 3.2.2 Preparation Poly(S/DVB)HIPE Surface Modification

Layer-by-Layer polyelectrolyte multilayers (PEM) technique was used to prepare multilayered polyHIPE. Poly(S/DVB)HIPE was cut into 2 cm thick sample. Then a vacuum pump was applied to run solutions through poly(S/DVB)HIPE. PEM technique is composing 2 coating, the primary coating and secondary coating. The primary coating, a positively charged solution of PDADMAC was run through poly(S/DVB)HIPEs for 2 min, then the poly(S/DVB)HIPE was rinsed with DI water 2 times. Next, a negatively charged solution of PSS was run through poly(S/DVB)HIPE for 2 min, then the poly(S/DVB)HIPE was rinsed with DI. The deposition of primary layer contains 6 layers. The secondary coating, tetraethylenepentamine (TEPA) and polyethylenimine (PEI) polymer solution were used as alternative polymer and operated as primary coating. Finally, sample was dried in the air.

## 3.3 **Characterization**

### 3.3.1 Scanning Electron Microscope (SEM)

Scan Electron Microscope was used to observe surface morphology of poly(S/DVB)HIPE. The specimens were cut to a small pieces, and coated with platinum under vacuum before observation to make them electrically conductive.

### 3.3.2 Autosorb-1MP

N<sub>2</sub> adsorption-desorption isotherms were obtained at -196 °C on a Quantachrome Autosorb-1MP. Samples were degassed at 100 °C during 12 hours in a vacuum furnace prior to analysis. Surface areas were calculated using the BET equation.

### 3.3.3 Mechanical Properties

Lloyd Universal testing machine was used to measure mechanical properties of all samples in compression mode, according to ASTM D822. Test specimens in a cylinder shape 2.54 cm in diameter × 2.54 cm in height were prepared. A speed of 0.127 cm/min and 500 N load cells were used for all measurements. The value of the compression stress and the Young's modulus were determined from an average of five samples.

#### 3.3.4 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was performed to measure the thermal stability of the poly(S/DVB)HIPE under N<sub>2</sub> flow of 100 ml/min. The sample was cut into small pieces weigh about 2-5 mg. Then the sample was heated from 30 °C to 800 °C, heating rate 10 °C/min. The degradation temperature was determined at weight loss 50 % from the weight loss vs. temperature thermogram.

#### 3.3.5 Fourier Transform Infrared Spectroscopy (FT-IR)

FTIR was used to confirm the existence of amine and PSS on surface of the polyHIPE. KBr pellet technique was applied in the preparation of powder samples.

#### 3.3.6 UV-Vis Spectroscopy

UV-Vis Spectroscopy was used to monitor and confirm the coating on surface of materials which is characteristic of layer-by-layer process.

#### 3.3.7 Adsorption of Carbon Dioxide (CO<sub>2</sub>)

The amount of CO<sub>2</sub> gas adsorbed of polyHIPE was determined by Gas Chromatography instrument. The samples were load into sample tube 2×25 cm. Before doing the adsorption, the polyHIPE sample in the reactor was pretreated with N<sub>2</sub> (80 mL/min). Then, the gas was switched to 15 vol% of CO<sub>2</sub> (15mL/min) at room temperature. Finally, desorption was applied with N<sub>2</sub> (80 mL/min)