

CHAPTER III EXPERIMENTAL

3.1 Materials

Divinylbenzene (DVB; merck) was used as crosslink-comonomer polyHIPE materials. Styrene was supplied from Sigma-Aldrich chemical. Surfactant were used sorbitant monolate (Span 80), dodecylbenzolsulfonic acid, sodium salt (DDBSS), purchased from Sigma-Aldrich chemical and cetyltrimethylammonium bromide (CTAB) supplied from Fluka. Initiator and stabilizer used in experiment 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) and Polyethyleneimine (PEI, MW 2500) were supplied Sigma-Aldrich.

3.2 Equipments

3.2.1 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) was performed on Hitachi S-4800 (JEOL/JAM 5200 Model to observe surface morphology and pore size of poly(S/DVB)HIPE *with $\times 500$ magnifications*. Before observation the specimens was cut a small pieces which coated with gold under vacuum to make them electrically conductive.

3.2.2 Autosorb-1MP

N_2 adsorption-desorption isotherms were obtained at $-196^\circ C$ on a Quantachrome Autosorb-1. The samples were degassed at $110^\circ C$ for 12 hours in a vacuum furnace before analysis. Surface areas were calculated using the BET equation. Surface area was measured using the dynamic seven points and the isotherm 24 points.

3.2.3 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 \times 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.

3.2.4 Thermogravimetric Analysis (TGA)

Thermo-gravimetric analysis (TGA) was performed to measure the thermal stability of the poly(DVB)HIPE under nitrogen gas atmosphere. The sample was cut into small pieces weigh about 2-5 mg. Then the sample was put into the platinum pan and heated from 30°C to 800°C at a heating rate 10°C/min. The degradation temperature was determined at weight loss from the weight loss vs. temperature curve.

3.2.5 Fourier Transform Infrared Spectroscopy (FT-IR)

The incorporation of organic group into silicate was investigated by using FTIR. The FTIR was obtained in the frequency range of 400-4000 cm^{-1} with 32 scans at resolution of 2 cm^{-1} . KBr pellet technique was applied in the preparation of powder samples.

3.2.6 Carbon Dioxide(CO₂) Adsorption

Study of CO₂ gas adsorption capacities of poly(S/DVB)HIPEs at room temperature. The samples were load into sample tube 2 \times 15 cm and were cut a diameter about 2 cm with 1-2 g. CO₂ 15 mL/min and N₂ 85 mL/min were flowed through at room temperature. The amount of CO₂ gas adsorbed by the polyHIPE was determined by Gas Chromatography instrument with Rt®-Q-BOND column with 0.53 mm id \times 20 μm film thickness \times 30 m length was used to operate at an isothermal temperature of 40 °C. The GC-TCD injection port was heated to 100 °C with a split flow of 8 mL/min and helium as a carrier gas.

3.2.7 Contact Angle Measurement

Contact angle measurement was performed by using a Kruss (model DSA 10) contact angle was determined wettability of the surface of poly(S/DVB)

polyHIPE. A 10 μL droplet of deionized water was dropped with a micro-syringe onto the polyHIPE surface. Drop shape analysis was explained hydrophilic and hydrophobic for this work.

3.3 Experimental Procedures

3.3.1. Preparation of Poly(S/DVB)polyHIPE

HIPE were comprised of aqueous phase and organic phase. HIPE was contained highly pore volume part of aqueous phase (about 90% of total volume) ; deionize water(DI), potassium persulphate ($\text{K}_2\text{S}_2\text{O}_8$) as initiator and stabilizer salt (CaCl_2). Organic phase containing styrene as monomer divinylbenzene (DVB) as crosslink, porogenic solvent as toluene and mix surfactant (Span 80, CTAB, DDBSS, 6.3 wt%, 0.3 wt%, 0.4 wt%). Preparation poly(S/DVB) was carried out slowly by added aqueous phase into organic phase by stirring approximately 60 minutes with mechanical stirrer at 350 rpm and homogenous time about 10 minutes. The ratio of poly(S/DVB) was showed 0:100, 80:20, 20:80. After finish was taken a material into a glass mold cavity and bring into a water bath at 60 °C for 48 h for polymerization. After all, materials dried in oven at 60 °C for 48 hours or until constant waight. The material was extracted with isopropanol for 6 h and dried in oven at 60 °C unit a constant weight.

3.3.2 Poly(S/DVB)HIPEs Modify Surface

Surface modification by the layer-by-layer polyelectrostatic self-assembly leading to polyelectrolyte multilayers (PEMs) technique. The LbL technique carry out by alternated adsorption between polycation and polyanion. This technique could improve any charged surface and created a hydrophilicity which used for CO_2 adsorption. PEM technique there are 2 type coating consisted of the primary coating and secondary coating. The primary coating was coated between polystyrenesulfonate (PSS) and poly(diallyldimethyl-ammonium chloride) (PDADMAC). Modification, material was cut a thickness about 2 mm and deposition time of each solution was 2 min. The primer layer was adsorb onto surface by an aqueous solution of PDADMAC concentrated 10 mM in 1 M NaCl and PSS concentrated 10 mM in 1 M NaCl which between them rinsed with DI water.

The number of layer in this layer was showed 8 layers. Then the secondary coating was coated between polyethylene imine (PEI) and polystyrenesulfonate (PSS). Deposition was performed an aqueous solution of PEI concentrated 1M in 1 M NaCl and PSS concentrated 10 mM in 1 M NaCl which between them rinsed with DI water and then them dried in the air. All solution in secondary layer had a charge density in pH 4. The number of layer was exposed 5 layers.

3.3.3 Characterization of Poly(S/DVB)HIPE

Morphology of poly(S/DVB) polyHIPEs was studied by using scanning electron microscope (SEM).

Surface area analysis of poly(S/DVB) polyHIPEs was measured by using Autosorb-1MP (BET).

Wettability of poly(S/DVB) polyHIPE was investigated by using contact angle.

Identification of chemical property on surface of poly(S/DVB)polyHIPE was determined by fourier transform infrared spectroscopy (FT-IR).

Thermal properties characterizing of Poly(S/DVB)HIPEs was studied by using TG/DTA.

Mechanical properties analysis of Poly(S/DVB)HIPEs was studied by using a Universal Testing Machine (LLOYD).

CO₂ adsorption of poly(S/DVB) polyHIPE was detected by using CO₂ adsorption testing unit.