CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Polymer material

1. Styrene, Fluka
2. Divinylbenzene, Merck
3. Ethylene glycol dimethacrylate, Sigma

3.1.2 Solvent

1. Tetrahydrofuran (THF), Sigma
2. Deionized Water

3.1.3 Others

1. Sorbitan monooleate (Span 80), Sigma
2. Potassium persulphate ($K_2S_2O_8$), Fluka
3. Calcium chloride ($CaCl_2$), Fluka
4. Sodium Chloride (NaCl), Suksapan

3.2 Equipment

1. Scanning Electron Microscope (SEM) (Hitachi/S-4800)
2. Autosorb-iQ
3. Universal testing machine (Lloyd)
4. Water bath
5. Convection Oven
6. Analytical Balance
7. Overhead stirrer
8. Propeller stirrer
9. Glassware

3.3 Software

1. SemAfore
2. Sigma Plot

3.4 Methodology

3.4.1 Preparation of poly(S/DVB)HIPEs

The HIPEs emulsion was formed by adding the aqueous phase (about 90% of the total volume; deionized water, potassium persulphate as initiator, and stabilizing salt) slowly, and drop-wise to the organic phase (about10% of the total volume; styrene/divinylbenzene: 80/20 monomers, and 20% span80 of organic phase as emulsifier). The mixture was stirred constantly with a mechanical stirrer at 300 rpm, until all aqueous phase had been added. The dosing time was 32 minutes and homogenizing time was another 3 minutes. After that, the resulting emulsion was then poured into glass vials and then placed in a water bath for polymerization at 60 °C for 48 hours. The cured material was removed from the vials and further dried in convection oven at 60 °C until constant weight was obtained.

3.4.2 Investigation of porous structure of poly(S/DVB)HIPEs by varying synthesizing conditions

1. Changing the type of the stabilizer salt: $CaCl_2$ and NaCl.

2. Increasing the percentage of the droplet phase with oil:aqueous phase ratio; 10:90, 8:92, 6:94 and 4:96.

3. Increasing the degree of divinylbenzene comonomer with S:DVB ratio; 80:20, 70:30 and 60:40.

3.4.3 Testing the water adsorption

Water adsorption capacity of polyHIPEs: was determined according to Burke *et al.* (2010), with sample disks 24 mm in diameter and 4 mm thickness. The disks, obtained from different systems, were fully dried and weighted before being immersed into deionized water at 30°C for varying periods of time, until the maximum adsorption has been achieved.

The dry weight was recorded first, then each sample was immersed into deionized water, dabbed dry with tissue paper to remove water from the surface, and reweighed. The water adsorption was determined by the following equation:

$$\text{Water adsorption capacity} = (W_s - W_d)/W_d \quad (1)$$

Where W_s and W_d are the weights of the soaked and dry polyHIPEs, respectively.

3.4.4 Preparation of poly(S/EGDMA)HIPEs

Poly(S/EGDMA)HIPEs were formed same as in 3.4.1 but, use ethylene glycol dimethacrylate instead of divinylbenzene.

3.4.5 Synthesis of poly(S/EGDMA)HIPEs with varying monomer composition

The composition of styrene/ethylene glycol dimethacrylate was varied from 80:20, to 70:30 and to 60:40.

3.4.6 Comparing water adsorption capacity between poly(S/DVB)HIPEs and poly(S/EGDMA)HIPEs

First, the effect of the amount of EGDMA was studied, by using the same procedure described in 3.4.3. The results of the water adsorption capacity were then compared to poly(S/DVB)HIPEs.

3.5 Characterization of polyHIPEs

3.5.1 Scanning Electron Microscope (SEM)

Scanning electron microscopy was performed on Hitachi S-4800 with an accelerating voltage of 10 kV to observe surface morphology of polyHIPE. The specimens were coated with platinum under vacuum before testing to behave them electrically conductive.

3.5.2 Autosorb-iQ

N_2 adsorption-desorption isotherms were obtained at -196°C on a Quantachrome Autosorb-iQ. Samples were degassed at 100°C during 12 hours in a vacuum furnace prior to analysis. Surface areas were calculated using the BET equation.

3.5.3 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.