

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Polymer materials

1. Styrene, Fluka
2. Divinylbenzene, Merck
3. Ethylene glycol dimethacrylate, Sigma
4. polystyrenesulfonate (PSS, sodium salt, MW 70,000), Sigma Aldrich
5. poly(diallyldimethylammonium chloride) (PDAD, MW 350,000), Sigma Aldrich
6. poly(acrylic acid) (PAA, MW 1,800), Sigma Aldrich
7. Alginic acid (ALG, sodium salt), Sigma Aldrich
8. Chitosan ((CTS, poly(D-glucosamine) deacetylated chitin, low MW), Sigma Aldrich

3.1.2 Solvents

1. Tetrahydrofuran (THF), Lab-Scan
2. Deionized Water

3.1.3 Others

1. 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 Equipments

1. Scanning electron microscope (SEM) (Hitachi/S-4800)
2. Autosorb-1MP (Quanta chrome)
3. Universal testing machine (Lloyd)
4. Water bath

5. Convection Oven
6. Analytical balance
7. Glassware
8. Mechanical stirrer

3.3 Methodology

3.3.1 Preparation of Poly(S/DVB) and Poly(S/EGDMA)polyHIPE

The HIPEs emulsion will be 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 (about 10% of the total volume; styrene/divinylbenzene: 80/20 monomers, and 20% span80 of organic phase as emulsifier). The mixture will be stirred constantly with a mechanical stirrer at 300 rpm, until all aqueous phase has been added. The dosing time was 32 minutes and homogenizing time was another 3 minutes. When this process finished, the resulting emulsion will be poured into glass vials and then placed in a water bath for polymerization at 60 °C for 48 hours. The cured material will be removed from the vials and extracted with methanol for 6 h and water for 6 h further dried in convection oven at 60 °C for 48 hours. Poly(S/EGDMA)HIPEs will be formed as in step 1 by used EGDMA instead of DVB.

3.3.2 PEM Surface Modification

Surface modification by the layer-by-layer polyelectrolyte multilayers (PEM) technique leads polyHIPE to increase hydrophilicity, thus water absorption. PolyHIPEs were cut to 3 mm thick and 1 inch diameter, were dried in conventional oven at 60 °C for 24 h. PEM coating was performed by injected polymer solution through polyHIPE disk. There are two coating consisted of the primary coating and the secondary coating. Primary coating, polyHIPE disk were alternate deposit to two polymer solutions and between them rinse with DI water. Volume of polymer solution and DI water were 10 mL each. The deposit time was 3 minutes for each solution. Then follow by the secondary coating, it also do as primary coating but different in polymer solutions. Then let them dried in air. The experimental condition

for the primary coating and the secondary coating onto polyHIPE are given in table 1.

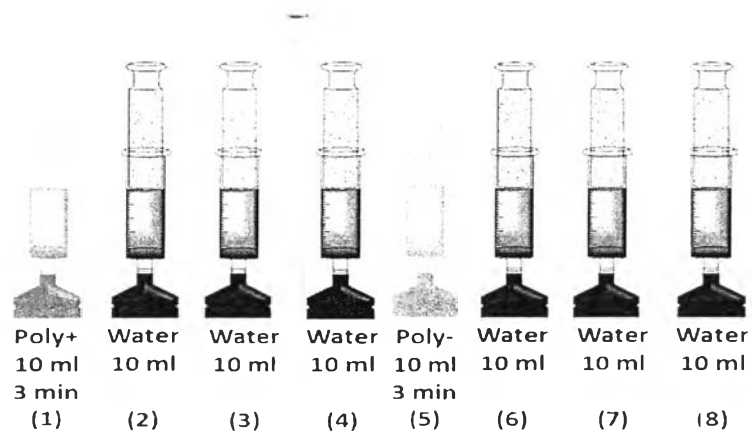


Figure 3.1 Step of coating both primer and secondary layer.

Table 3.1 Experimental Condition for the Primary Coating and the Secondary Coating onto PolyHIPE

Entry	PolyHIPE	Primary Coating			Secondary Coating		
		Solution 1	Solution 2	# of bi-layer	Solution 1	Solution 2	# of bi-layer
1	S/DVB	10 mM PDAD/ 1 M NaCl	10 mM PSS/ 1 M NaCl	5	10 mM PDAD/ 0.2 M NaCl (pH 9)	0.1% Humic acid/ 0.2 M NaCl (pH 9)	1, 2, 3, 4, 5
2	S/DVB	10 mM PDADMA C/ 1 M NaCl	10 mM PSS/ 1 M NaCl	1, 2, 3, 4, 5	-	-	-
3	S/DVB	10 mM PDAD/ 5 M NaCl	10 mM PSS/ 5 M NaCl	4	10 mM PDAD/ 5 M NaCl	10 mM PSS/ 5 M NaCl	10
4	S/DVB	10 mM PDAD/ 5 M NaCl	10 mM PSS/ 5 M NaCl	4	10 mM PDAD/ 5 M NaCl (pH 9)	10 mM PAA/ 5 M NaCl (pH 9)	10
5	S/DVB	10 mM PDAD/ 5 M NaCl	10 mM PSS/ 5 M NaCl	4	10 mM CTS/ 5 M NaCl (pH 5.5)	10 mM ALG/ 5 M NaCl (pH 5.5)	10
6	S/EGDMA	10 mM PDAD/ 5 M NaCl	10 mM PSS/ 5 M NaCl	4	10 mM PDAD/ 5 M NaCl (pH 9)	10 mM PAA/ 5 M NaCl (pH 9)	10

3.3.3 Physical Characterization

3.3.3.1 *Scanning Electron Microscopy*

Pore diameter was determined using scanning electron microscopy (FE-SEM, Hitachi S-4800). The specimens were coated with platinum under vacuum, viewed using accelerating voltage of 10kV.

3.3.3.2 *Surface Area Measurement*

N₂ adsorption-desorption isotherms were obtained at -196°C on a Quantachrome Autosorb-1. Samples were degassed at 100°C during 12 hours in a vacuum furnace prior to analysis. Surface areas were calculated using the BET equation.

3.3.3.3 *Compression Strength*

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 Wetting Ability

The shapes of droplets recorded by sessile drop (Krüss, model DSA 10) at the surface of the porous material clearly indicate hydrophilicity.

3.3.5 Reflectance of 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.

3.3.6 water adsorption

Water adsorption capacity of polyHIPEs: will be determined according to Burke *et al.* (2010), with sample disks 24 mm in diameter and 4 mm thickness. The disks, obtained from the different systems, will be 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 will be recorded first, then each sample will be immersed into deionized water, dabbed dry with tissue paper to remove water from the surface, and reweighed. The water adsorption will be 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.