CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Monomer

Divinylbenzene (DVB) was supplied by Merck.

3.1.2 Solvents

Toluene (T) was supplied by Lab Scan. Isopropanol was supplied by Etalma.

3.1.3 Surfactants

Sorbitan monooleate (SPAN80) and Dodecylbenzenesulfonic acid, sodium salt (DDBSS) were supplied by Sigma. Cetyltrimethylammonium $[C_{16}H_{31}N^{+}(CH_3)_3]$ bromide (CTAB) was supplied by Fluka.

3.1.4 Clay Minerals

Bentonite (BN) was supplied by Thai Nippon Chemical Industry Co., Ltd. The cation exchange capacity (CEC) of BTN is 43 mmol/100g of clay.

3.1.5 Initiator

Potassium persulfate ($K_2S_2O_8$) was supplied by Fluka.

3.1.6 Stabilizer

Calcium chloride dehydrate ($CaCl_2 \cdot 2H_2O$) was supplied by Fluka.

3.2 Equipment

3.2.1 Surface Area Analyzer (SAA)

N_2 adsorption-desorption isotherms were obtained at -196°C on a Quantachrome Autosorb-1. Samples were degassed at 100°C during 12 h in a vacuum furnace prior to analysis. Surface areas were calculated using the BET equation.

3.2.2 Thermogravimetric Analysis (TGA)

Thermo-gravimetric analysis (TGA) was performed to measure the thermal stability of the poly(DVB)HIPE. TGA of both neat and poly(DVB)HIPE nanocomposites were performed using a Perkin Elmer/Pyris Daimond TG/DTA instrument. Experiments were carried out under nitrogen gas atmosphere. Samples were cut into small pieces weigh about 2–5 mg. Then the samples were loaded on the platinum pan and heated to 800°C from 40°C at a heating rate of 10°C/min. One steps degradation was observed during testing, and the decomposition temperature was recorded corresponding to 50% decomposition of the material.

3.2.3 Scanning Electron Microscope (SEM)

Scanning electron microscopy was performed on Hitachi S-4800 Model to observe surface morphology of poly(DVB)HIPEs. The specimens were coated with platinum under vacuum before observation to make them electrically conductive.

3.2.4 Universal Testing Machine (LLOYD)

A Lloyds Universal Testing Machine (Lloyds/LRX) equipped with a 500 N load cell was used to measure mechanical properties in compression. The samples were loaded at a rate of 1.27 mm/min. Samples of 25.4 mm in diameter and 25.4 mm in height were used for tested of each poly(DVB)HIPEs. The samples were loaded until a displacement of 70 percent of the height of the examined sample was reached.

3.2.5 CO_2 Gas Adsorption

Study of CO_2 gas adsorption capacities of poly(DVB)HIPE filled with acid-treated clay were carried out using a pilot gasification unit at the Chemical Technology Department, Faculty of Science, Chulalongkorn university. Samples were cut into small pieces weigh about 1–2 g. Then the samples were loaded into sample tube 2×25 cm. CO_2 3 mL/min and He 17 mL/min were flowed through the sample at room temperature. The residue of CO_2 was measured by a Gas Chromatography instument, column used Shimadzu 2014, flow rate 35 mL/min.

3.3 Methodology

3.3.1 Preparation of Poly(DVB)HIPE Filled with Acid-treated Clay

The cellular materials were prepared by first dissolving organic phase containing 5 mL of DVB monomer, 5 mL of toluene, required amounts of acid-treated clay, and a mixture of nonionic, anionic, and cationic surfactants (Table 3.1 and 3.2): SPAN80, DDBSS, and CTAB was added to the mixture, stirred for 10 min. While 90 mL of distilled water containing 0.2 g of potassium persulfate and 1 g of calcium chloride dihydrate were added dropwise. After all the water has been added, the emulsion was further stirred for 20 min and placed in a glass bottle. The obtained emulsions were capped and put in a convection oven at 70°C for 24 h to polymerize (Barbetta *et al.*, 2004). After polymerization, the cellular materials were removed from the glass bottles and washed by soxhlet for 6 h with Iso-propanol. Then the cellular materials were returned to vacuum oven to dry at 70°C for 48 h (Pakeyangkoon *et al.*, 2008).

Table 3.1 Neat and filled poly(DVB)HIPE prepared by varying the composition of nonionic surfactant(SPAN80)

SPAN 80 (%wt)	DDBSS (%wt)	CTAB (%wt)	% total surfactant
4.3	0.4	0.3	5
6.3	0.4	0.3	7
7.8	0.4	0.3	8.5
9.3	0.4	0.3	10
11.3	0.4	0.3	12

Table 3.2 Mol ratio of mixed surfactant using for prepare neat and filled poly(DVB)HIPE

SPAN 80	DDBSS	CTAB	% total surfactant
0.8358	0.0956	0.0686	5
0.8818	0.0689	0.0494	7
0.9023	0.0569	0.0408	8.5
0.9167	0.0485	0.0348	10
0.9304	0.0405	0.0291	12

3.3.2 Characteristics of Poly(DVB)HIPE filled Acid-treated Clay:

a. Thermal properties characterizing : The thermal properties of poly(DVB)HIPE filled with acid-treated clay were studied using TG/DTA.

b. Morphology characterizing : The morphology of poly(DVB)HIPE filled with acid-treated clay was studied using SEM.

c. Mechanical properties analysis : The mechanical properties of poly(DVB)HIPE filled with acid-treated clay were studied using LLOYD.

d. Surface area analysis : The surface area of poly(DVB)HIPE filled with acid-treated clay was analyzed using Autosorb-1.

e. Adsorption analysis : Study of CO_2 gas adsorption properties of poly(DVB)HIPE filled with acid-treated clay were carried out using a pilot gasification unit at the Faculty of Chemical Technology Chulalongkorn university.