



CHAPTER V

POLYMERIC FOAM VIA POLYMERIZED HIGH INTERNAL PHASE EMULSION (HIPE) FILLED WITH ORGANO-MODIFIED BENTONITE

5.1 Abstract

Polymerized high internal phase emulsion of poly(divinylbenzene); poly(DVB)polyHIPE filled with porous clay has been successfully prepared. The hybrid organic–inorganic porous clay heterostructures (HPCH) were obtained from a reaction of organo-modified bentonite, surfactant, and tetraethoxysilane (TEOS)/methyltetraethoxysilane (MTS) occurring in the galleries of the clay mineral. HPCH loadings of 1, 3, 5, and 10 wt% were added to the highly porous material, formed by the high internal phase emulsion technique (HIPE). The effects of the inorganic filler on surface area, morphology, thermal and mechanical properties were studied by N₂ adsorption–desorption (Autosorb I), scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and a Lloyd universal testing machine, respectively. Results showed that the presence of layered silicate in the polymer matrix resulted in the enhancement of physical properties of the foams filled with hybrid organic–inorganic porous clay heterostructures (HPCH). Mechanical properties, including compressive stress and Young's modulus, were found to be improved by up to 84% and 137%, respectively, when compared with neat poly(DVB)polyHIPE foams. The best mechanical properties of the obtained material were achieved at 5 wt% loading of HPCH. The use of HPCH as the reinforcing agent of poly(DVB)polyHIPE was also capable of improving the surface area of the obtained materials by 35–97%. It was clearly demonstrated in this study that the suitable content of HPCH, i.e. 5 wt%, has an effect on improving the mechanical properties and surface area of the poly(DVB)polyHIPE foams.

Key words: High internal phase emulsion, PolyHIPE foams, Clay, Porous clay heterostructure, Organo-modified clay, Composite.

5.2 Introduction

Highly open, porous polymeric foams obtained from high internal phase emulsions (HIPE) have recently attracted much attention and increasing interest because of their excellent properties (high porosity, high surface area, and high degree of interconnectivity). This microporous material, so-called polyHIPE, has been defined as the polymer from the polymerization of an emulsion where the internal phase occupies more than 70–90% of the total volume.^{1–3} After the polymerization of the continuous phase (continuous phase or organic phase containing monomers, crosslinking comonomers, and suitable surfactants), followed by the removal of the aqueous or droplet phase, the formation of highly porous materials with interconnected pore structure occurs. Due to their unique properties (such as high porosity, high surface area, and high degree of interconnectivity), polyHIPE porous materials are considered attractive for many applications. Poly(S/DVB)polyHIPE, modified with bioactive molecules, for example, is being used as a scaffold for biomedical applications,^{4–7} and poly(GMA/EGDMA)polyHIPE is used as a separation media.⁸ Additionally, polyHIPE porous materials are used as a catalyst support,⁹ a filtration media,¹⁰ and as an ion exchange membrane.¹¹ Wakeman *et al.* (1998) found that the combination of high surface area and permeability of the high internal phase emulsion makes polyHIPE attractive as a potential adsorbent.¹¹ Results from the literature have demonstrated that one of the most important requirements for the success of polyHIPE in applications is its high surface area, i.e. small cell size, and high porosity. At present, there are many works reporting attempts to improve the surface properties of polyHIPE materials, including polyHIPE prepared with an inert porogenic solvent and a three-component surfactant system.^{12–16} The three-component surfactant and porogenic solvent increase the emulsion stability of the system and the high surface properties of the polyHIPE can be obtained when a secondary pore structure is generated within the cell walls of the obtained materials. Not only is high surface area a requirement, good mechanical properties of polyHIPE foams are also another important for polyHIPE to be useful in industrial applications.^{17,18} In 2006, more research was focused on ways to improve the physical properties of polymerized high internal phase emulsions using

inorganic materials as a reinforcing agent. Haibach *et al.* (2006) synthesized highly open, porous low-density polymer foams with greater mechanical properties by adding nanosized silica particles as filler.¹⁷ The Young's modulus of the silica-reinforced polyHIPE foams increased by 280% and the crush strength increased by 218%, as compared to polyHIPE foam without a reinforcement. Attempts were also made to increase the density and pore interconnectivity of polyHIPE porous materials by preparing carbon black-filled polyHIPE foams.¹⁸ The pore interconnectivity was increased by adding *in situ* grafting carbon black into the pore walls of polyHIPE foams; this led to an increase in emulsion stability while its mechanical properties were much improved by increasing the organic phase volume, thus increasing the foam density. Another inorganic filler which may be suitable for use as a reinforcing phase in polyHIPE foam is clay mineral. By the modification of the clay mineral using an ion exchange reaction with quaternary ammonium cations, the layered silicates in the clay would be expanded and the d-spacing of the organo-modified clay increased, which helps in better intercalation and exfoliation.^{19,20} Polymer nanocomposites with nanoclay as a filler results in a significant improvement in mechanical strength, caused by the nanometric dimensions and high aspect ratio. Studies of porous clay heterostructures (PCHs) and hybrid organic-inorganic PCHs (HPCHs) were also carried out.²⁰⁻²² In 1995, Galarneau *et al.* discovered a new porous material called a porous clay heterostructures (PCHs) which had uniform pore diameters in the supermicropore to small mesopore range (1.5–3.0 nm), with high specific surface areas and high thermal stability.²¹ Additionally, in order to improve the chemical properties and hydrophobicity on the porous structure, hybrid organic-inorganic porous clay heterostructures (HPCHs) have been prepared by incorporating organic components via the co-condensation reaction. Organic modification of the mesoporous silica permits precise control over the surface properties and pore size of the mesoporous materials for specific applications.^{22,23} Because of the high performance of PCHs and HPCHs, they have the ability to impart the combination between tremendous surface area and excellent stress transfer to the reinforcement phase, leading to improvement in the mechanical strength and surface area of the resulting materials.

In order to enhance the polyHIPE performance in ever-broadening applications, the objectives of this research were to elevate the surface properties along with the thermal and mechanical properties of poly(DVB)polyHIPE above their inherent values by using hybrid organic–inorganic porous clay heterostructures (HPCH) derived from Na-Bentonite as the inorganic filler. Because of the desirable properties of HPCH, which impart both tremendous surface area and excellent stress transfer to the reinforcement phase, this material could be offered as an alternative inorganic filler for nanocomposite materials. By incorporating the HPCH porous clay into the continuous phase of polyHIPE foams, it would not only improve the mechanical properties of polyHIPE foams but also would increase the surface area of the resulting materials when compared to the unfilled materials.

5.3 Experimental

5.3.1 Materials

Divinylbenzene (DVB; Merck) was used as a monomer to produce polyHIPE materials. The surfactant, sorbitan monooleate (Span80; S80), was purchased from Sigma-Aldrich Chemical. The initiator and stabilizer used in the experiments were potassium persulfate ($K_2S_2O_8$, purity $\geq 98\%$ (RT), Fluka Chemie) and calcium chloride ($CaCl_2$, purity $\geq 97\%$ (KT), Fluka Chemie), respectively. Toluene (T) was employed as a porogenic solvent. Na-Bentonite (BTN) (Mac-Gel[®] GRADE SAC, Thai-Nippon Chemical Industry Co., Ltd.) was ion exchanged with cetyltrimethylammonium bromide (CTAB) to yield CTAB-modified bentonite. The organo-modified bentonite (hybrid organic–inorganic porous clay heterostructures; HPCH) was prepared in our laboratory using the procedure described by Prakobna *et al.* (2006).²⁴

5.3.2 Preparation of poly HIPE-HPCH Nanocomposites

Poly(DVB)polyHIPE materials were prepared from high internal phase emulsion. The continuous phase (10% vol of total volume) of the emulsion consisted of DVB, toluene (monomer: porogen; 1:1 ratio by volume), Span80 (S80; 2

ml), and HPCH (1, 3, 5, and 10 wt% relative to monomer, see Table 5.1). The aqueous phase contained water (90 ml), $K_2S_2O_8$ (0.2 g) as initiator, and an electrolyte, $CaCl_2$ (1.0 g). PolyHIPE was formed by adding the aqueous phase dropwise to the organic mixture with mechanical stirring. The resulting materials were placed in a water bath at $60^\circ C$ for 24 h for polymerization. After that, the obtained solid materials were extracted with isopropanol for 6 h to remove any residual materials from the pore structures of the polyHIPE and then they were placed in a vacuum oven at $60^\circ C$ until a constant weight was obtained.

5.3.3 Characterizations

5.3.3.1 Surface Properties

N_2 adsorption–desorption isotherms were carried out at $-196^\circ C$ on a Quantachrome Autosorb I using a BET model. The samples were degassed at $100^\circ C$ for 12 hours in a vacuum furnace prior to analysis. Surface areas were calculated using the BET equation (see equation 5.1) and pore volumes were determined by the t-plot method of De Boer.²⁵ The resulting BET equation is expressed by equation (5.1):

$$\frac{1}{W\left(\frac{P_0}{P}-1\right)} = \frac{1}{W_m C} + \frac{C-1}{W_m C} \cdot \frac{P}{P_0} \quad (5.1)$$

where W is the weight of gas adsorbed at a relative pressure (P/P_0), W_m is the weight of adsorbate constituting a monolayer of surface coverage and C , a constant that is related to the heat of adsorption. Equation (5.1) is an adsorption isotherm and can be plotted as a straight line with $1/W[(P_0/P)-1]$ and P/P_0 according to the experimental results. A linear relationship between $1/W[(P_0/P)-1]$ on the y-axis and P/P_0 on the x-axis is required to obtain the quantity of nitrogen absorbed. The slope A and intercept I were used to determine the quantity of nitrogen adsorbed in the monolayer (W_m) and used to calculate the total surface area. The following equations were used:

$$A = \frac{C - I}{W_m C} \quad (5.2)$$

$$I = \frac{I}{W_m C} \quad (5.3)$$

$$W_m = \frac{I}{A + I} \quad (5.4)$$

the weight of a monolayer W_m was obtained from equation (5.4) by combining equation (5.2) and (5.3). A total surface (S) area was evaluated by the following equation:

$$S = \frac{W_m N A_{cs}}{M} \quad (5.5)$$

where N is Avogadro's number (6.023×10^{23} molecules/mol), M is the molecular weight of the adsorbate and the cross sectional area (A_{cs}) for N_2 at 77 K (-196°C) is 16.2 \AA^2 .

5.3.3.2 Morphology and Cell Density

Scanning electron microscopy was done with a JEOL scanning electron microscope (MP 152001 Model), operating at 15 kV and 1500× and 15000× magnification. All specimens were coated with gold under vacuum before analysis. The average cell size of poly(DVB)polyHIPE foams was determined by using SemAfore program (version 5.0) on the obtained SEM micrographs. Cell density measurement for all poly(DVB)polyHIPE foams were determined using pycnometer (Quantachrom, Ultrapycnometer 1000) under helium purge at pressure of 17 Psi. At least five polyHIPE foam samples were analyzed to obtain statistically significant data.

5.3.3.3 Thermal Analysis

Thermal properties with DTG curves were collected on a Perkin-Elmer Pyris Diamond TG/DTA instrument, and the experimental procedure used was described by several workers.^{17,24} Sample was loaded on the platinum pan and heated from 30°C to 800°C at a heating rate of 10°C/min under N₂ flow of 100 ml/min. The degradation temperature was determined at a weight loss of 50% from the weight loss versus temperature curve.

5.3.3.4 Mechanical Properties

A Lloyd Universal Testing Machine was used to measure the 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 length 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.

5.4 Results and discussion

In order to improve the properties of polyHIPE foams, hybrid organic–inorganic porous clay heterostructures (HPCH), which were prepared by the surfactant-directed assembly of tetraethoxysilane (TEOS)/methyltetraethoxysilane (MTS) into the galleries of Na-bentonite clay, was used as the inorganic filler in this study. Poly(DVB)polyHIPE foams with hybrid organic–inorganic porous clay heterostructures (HPCH: 1, 3, 5, and 10 wt%) were prepared by high internal phase emulsion technique.^{1–3} From physical observation, with a suitable amount of reinforcement (3–5 wt%) the resulting polyHIPE foams exhibited less chalkiness than HIPE1 and 2 (see Table 5.1).

5.4.1 Morphology

The effect of hybrid organic–inorganic porous clay heterostructures (HPCH) on the phase morphology of the obtained polyHIPE foams was demonstrated in this study. The phase morphology of poly(DVB)polyHIPE filled with various weight percents of HPCH (HPCH; 1, 3, 5, and 10 wt%) are shown in Figure 5.1a–e. Organoclay particles were incorporated into the continuous phase (monomer phase) of poly(DVB)polyHIPE foams. These figures illustrate that both HPCH-containing poly(DVB)polyHIPE foams (HIPE2–5) and pristine poly(DVB)polyHIPE foams (HIPE1) exhibited a similar phase behavior which showed an open cellular morphology with a highly interconnected pore structure. They indicate that the addition of HPCH did not affect the porous structure of polyHIPE foams. Moreover, from SEM micrographs, the primary cell size of poly(DVB)polyHIPE foams was also determined and the values were found to be around 7–8 μm (see Table 5.2). In addition, poly(DVB)polyHIPE foams exhibited cell density in a range 1.2–1.4 g/cm^3 . Cell density of neat Poly(DVB)polyHIPE foams (HIPE1) was not significantly different when compared to poly(DVB)polyHIPE foams filled with HPCH (HIPE2–5) as shown in Table 5.2. Even though the overall cell size and cell density of the poly(DVB)polyHIPE foams are relatively similar, the roughness of the wall surface were differ and depending on the amount of HPCH added in the organic phase, which affects the surface area of the resulting materials. As see from SEM micrographs of the poly(DVB)polyHIPE with higher magnification (see Figure 5.1f–j), which show that the roughness of the wall surface increased. This could be due to the ability of the porogenic solvent to generate the secondary pore structure within the wall surface of the obtained materials and the inherent porous structure of the hybrid organic–inorganic porous clay heterostructure (HPCH). Consequently, to study the effect of HPCH on the rough surface of the obtained materials, poly(DVB)polyHIPE without added porogenic solvent and HPCH, which was denoted as reference, was prepared. The wall surface of the reference (Figure 5.2d) appears flat, and the roughness of the wall surface seems to increase when toluene was used as the porogenic solvent (see Figure 5.2e; HIPE1). This observation was also made by Hainey *et al.* (1991), who indicated that the porogenic solvent has the ability to generate a secondary pore

structure within the cell wall of polyHIPE foams.² In addition, Figure 5.2f also illustrates that by adding porous clay (HPCH) into poly(DVB)polyHIPE foams, roughness of the wall surface seems to increase when compared to HIPE1 and the roughness of the surface was further increased with the percentage of added HPCH increased (Figure 5.1f–j). These observations were consistent with the results from qualitative analysis carried out by elemental analysis (EDX), which indicated different chemical element compositions of poly(DVB)polyHIPE foam with 1, 3, 5, and 10 wt% HPCH, as presented in Table 5.3 and Figure 5.3; when the content of HPCH increased, the amount of silica (Si) particle also increased from 0.32–3.35%. From the EDX images (see Figure 5.4), uniform dispersion of Si particle throughout the poly(DVB)polyHIPE foams was observed, suggesting that the porous clays (HPCH) were evenly dispersed in the continuous phase of the polyHIPE foams. However, when the content of HPCH was more than 5 wt%, agglomerations of clay particles occurred as small aggregates in the poly(DVB)polyHIPE foams, as observed in Figure 5.4d.

5.4.2 Surface Properties

From the BET results in Figure 5.5, all of the polyHIPE foams showed an increase in nitrogen adsorption at low partial pressure and gradually increased to medium partial pressure (P/P_0 :0.05–0.3). The BET surface area of filled poly(DVB)polyHIPE foam (HIPE2–5) was improved by 35–97% (178–260 m²/g) and pore volume was increased by 0.09–0.3 cc/g when compared to HIPE1. Improvement in the surface areas and specific pore volume of the obtained materials was clearly due to the incorporation of hybrid organic–inorganic porous clay heterostructures (HPCH) into the continuous organic phase of polyHIPE foams, resulting in intensified occupation of the porous structures. Additionally, an enhancement of surface areas might also result from the increased roughness of the wall surface of poly(DVB)polyHIPE foams (see Figure 5.1f–j).

5.4.3 Thermal Analysis

The thermal behaviors of poly(DVB)polyHIPE filled with hybrid organic–inorganic porous clay heterostructures (HPCH) were investigated in this study. The differential thermograms (DTG) of polyHIPE foams are delineated in Figure 5.6 and the thermal properties are listed in Table 5.2. The incorporation of HPCH into poly(DVB)polyHIPE improved the degradation temperature (T_d) of the resulting materials. Results from the thermograms indicate that the thermal stability of filled poly(DVB)polyHIPE (HIPE2–5) was slightly increased when compared to that of unfilled poly(DVB)polyHIPE; HIPE1. The improvement in the thermal properties of polyHIPE foams, shifting considerably towards higher temperature, might be attributed to the formation of a high-performance carbonaceous–silicate char building up on the surface of the poly(DVB)polyHIPE foams when the materials were subjected to high temperature.²⁶

5.4.4 Mechanical Properties

The effect of hybrid organic–inorganic porous clay heterostructures (HPCH) on the mechanical properties of the obtained polyHIPE foams is shown in Table 5.2 and Figures 5.7 and 5.8. All of the mechanical properties including compressive stress, percentage strain, and Young's modulus of the poly(DVB)polyHIPE foams containing HPCH exhibit higher values than those of the unfilled one; this reveals the reinforcement effect induced by the incorporation of HPCH into the continuous phase of the obtained materials. The effect of HPCH loading on both compressive stress and percentage strain was also investigated. From Figure 5.7 and Table 5.2, we can see that, in the presence of 1 wt% HPCH, the compressive stress and percentage strain increased by 44% (0.0771–0.1108 MPa) and 25% (12.7913–16.0829), respectively. With the inclusion of 5 wt% HPCH, a maximum improvement in both compressive stress and percentage strain was observed and rapidly decreased as HPCH content was further increased. The increased compressive stress of poly(DVB)polyHIPE foam with 5 wt% HPCH (0.0771–0.1424 MPa) is 84%, whereas strain increased by 52% higher than that of neat poly(DVB)polyHIPE foam. This observation was quite similar to the result of Young's modulus (see Figure 5.8); it is revealed that the highest Young's modulus

(increased to 137%) was observed when HPCH loading was 5 wt%. The incorporation of hybrid organic–inorganic porous clay heterostructures (HPCH) with good dispersion into the continuous phase (organic phase) of poly(DVB)polyHIPE foams contributed to the enhancement of the mechanical properties of the obtained materials. This could be due to the high aspect ratio of the organoclay, which gives a large surface area for improving adhesion between phases and provides better stress transfer to the reinforcing phase.²⁷ Similar results were reported by Jo *et al.* (2007), who showed that significant improvements in tensile strength and Young's modulus were observed by incorporating clay mineral as reinforcement into composite materials.²⁸ However, the mechanical performance of poly(DVB)polyHIPE foams was reduced as HPCH loading increased up to 10% by weight. This reducing phenomenon suggested the agglomeration of organoclay particles, which acted as stress concentrator points in the continuous phase of the poly(DVB)polyHIPE foams (see Figure 5.4d). This was further supported by EDX images, which showed that the agglomeration of clay particles occurred when a high loading of HPCH was used.

5.5 Conclusions

The effect of highly porous clay materials (hybrid organic–inorganic porous clay heterostructures: HPCH) on phase morphology, surface properties, thermal behavior, and mechanical properties of poly(DVB)polyHIPE foams was investigated in this study. The incorporation of HPCH into poly(DVB)polyHIPE foams caused an improvement of both mechanical properties and surface areas of the resulting poly(DVB)polyHIPE foams. An increase in the surface areas was observed (from 132 to 260 m²/g) with HPCH content increase (from 0–10 wt%). This phenomenon occurred due to the increasing roughness of the wall surface when HPCH was added into the continuous phase and to the inherent characteristics of HPCH, which exhibited an open-framework of silica and high surface areas with uniform and specific pore sizes in the clay galleries. Both compressive stress and Young's modulus were found to increase by 84% and by 137%, respectively. The addition of 5 wt% porous clay (HPCH) yields foams with higher mechanical properties (compressive stress and Young's modulus) than the neat poly(DVB)polyHIPE foams.

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5.7 References

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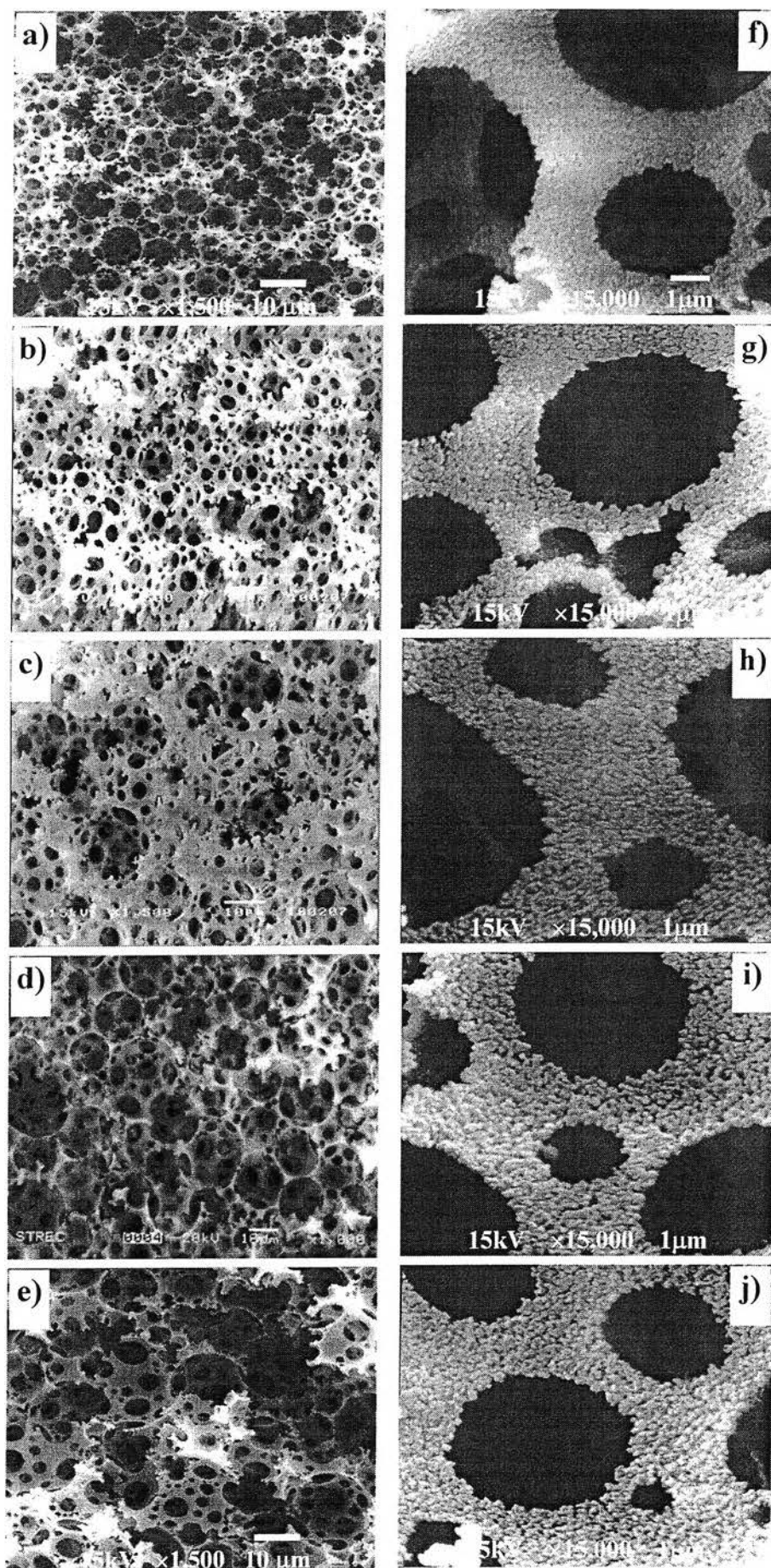


Figure 5.1 Scanning electron micrographs of poly(DVB)polyHIPE: (a–e) HIPE1–HIPE5 (1500 \times); (f–j) HIPE1–HIPE5 (15000 \times).

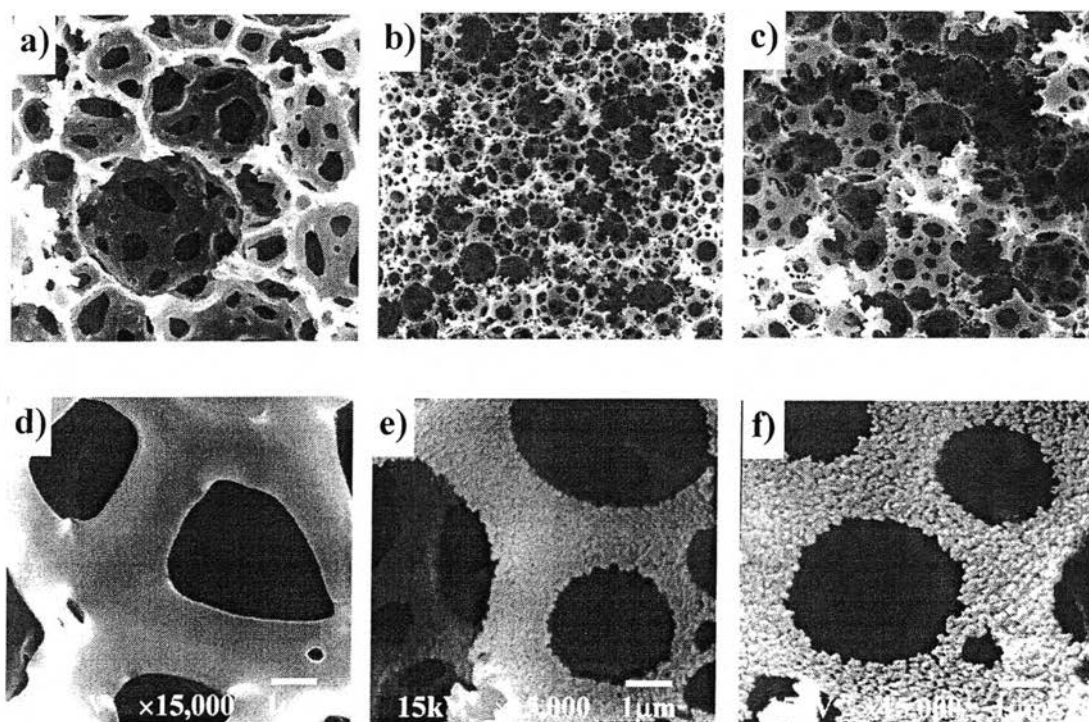


Figure 5.2 Scanning electron micrographs of poly(DVB)polyHIPE: (a–c) reference, HIPE1, and HIPE5, respectively (1500 \times); (d–f) reference, HIPE1, and HIPE5, respectively (15000 \times).

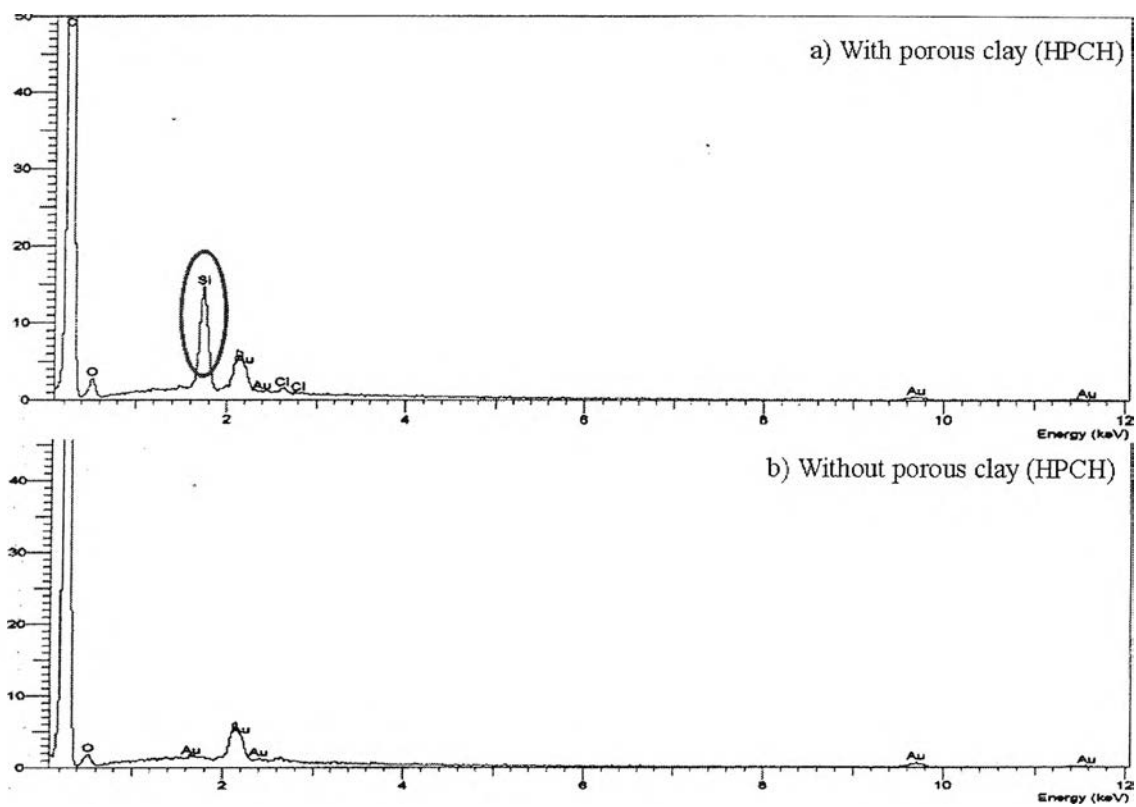


Figure 5.3 EDX spectra of poly(DVB)polyHIPE: a) with porous clay (HPCH) and b) without porous clay (HPCH).

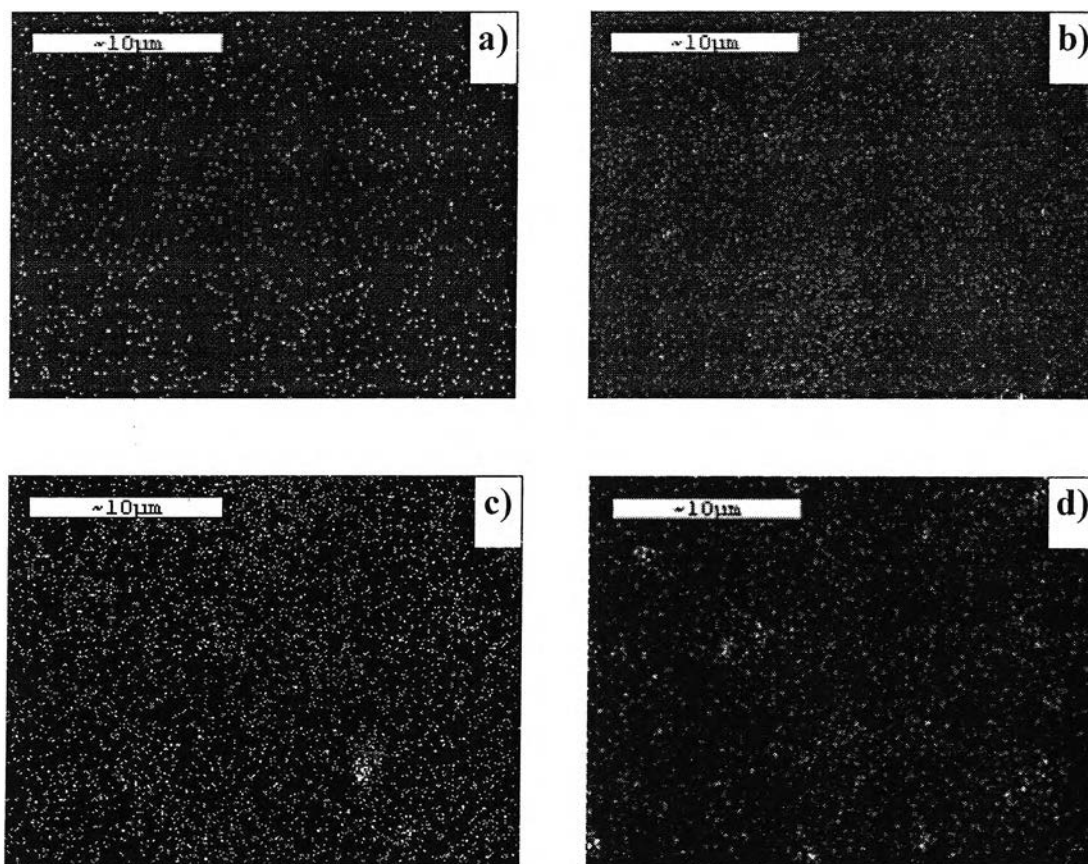


Figure 5.4 Spectra Si mapping of scanning electron micrographs of poly(DVB)polyHIPE: a) 1 wt% HPCH; b) 3 wt% HPCH; c) 5 wt% HPCH; and d) 10 wt% HPCH.

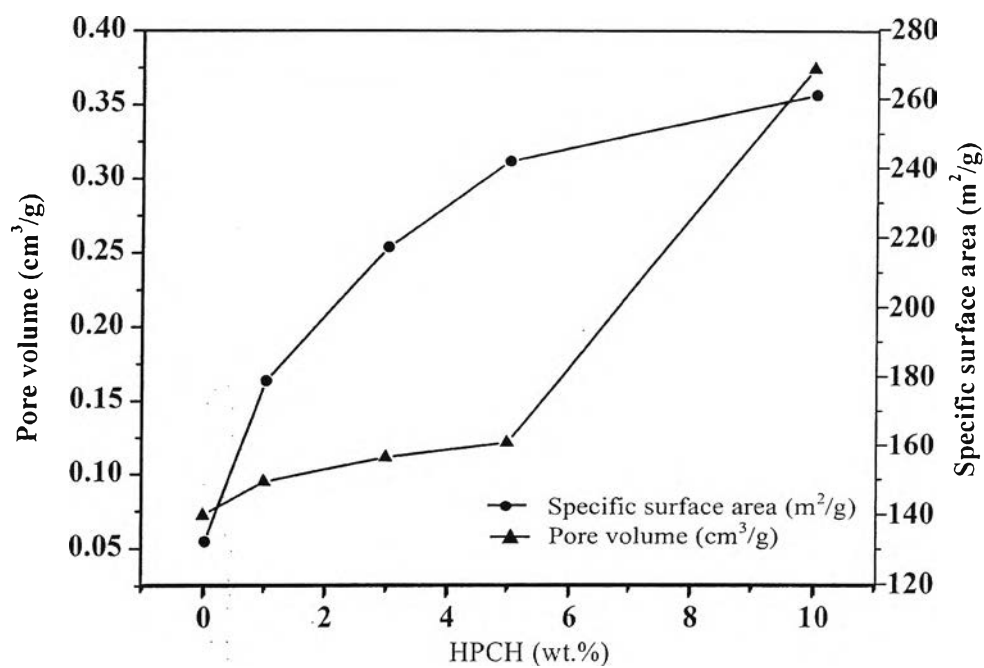


Figure 5.5 Specific surface area and pore volume of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).

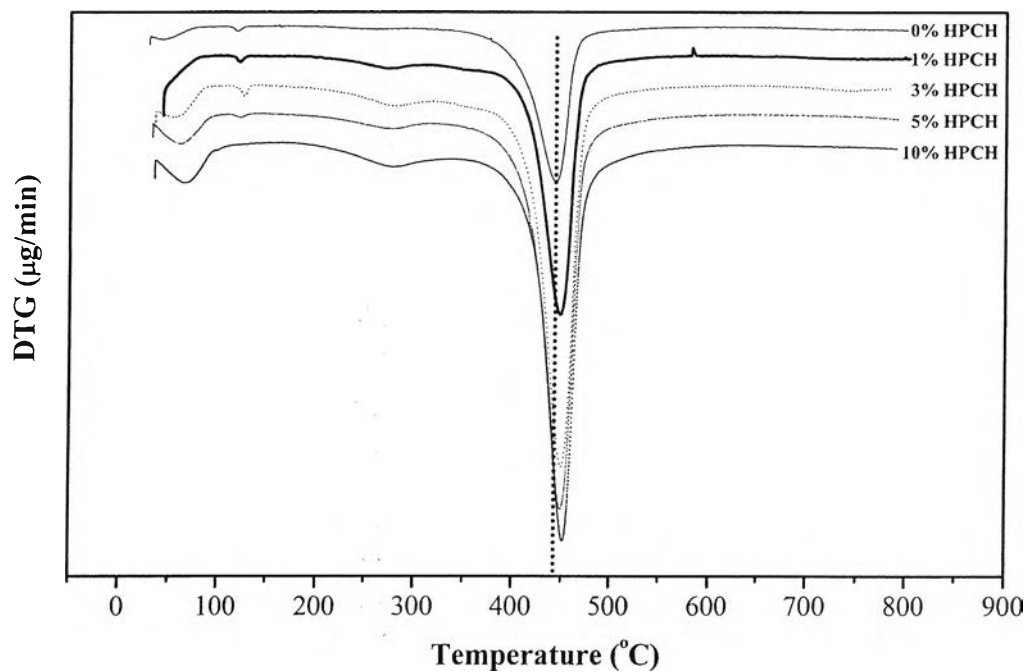


Figure 5.6 DTG curves of poly(DVB)polyHIPE with various weight percents of hybrid organic-inorganic porous clay heterostructures (HPCH).

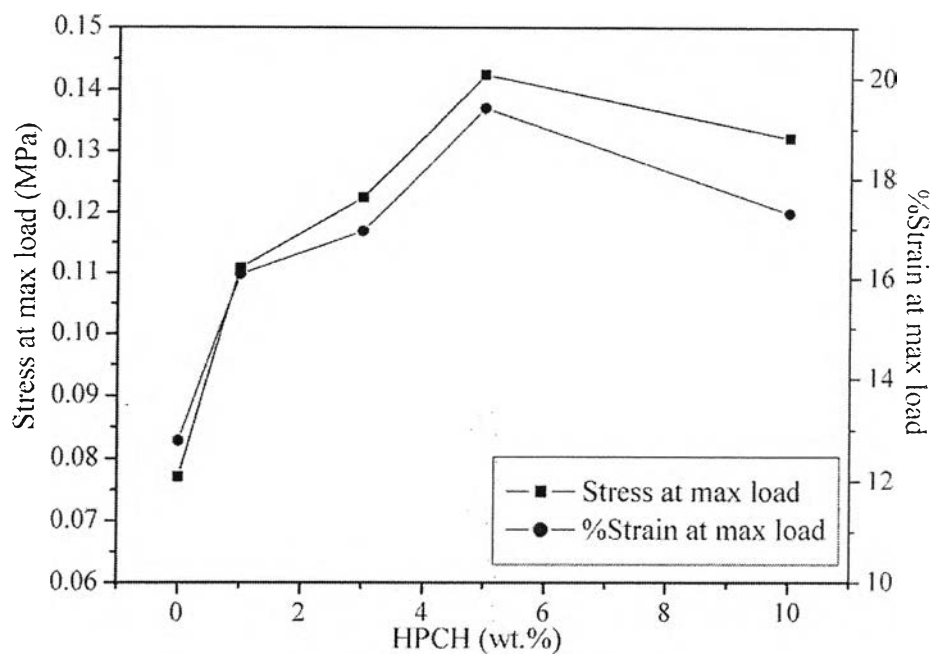


Figure 5.7 Compressive stress and percentage strain of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).

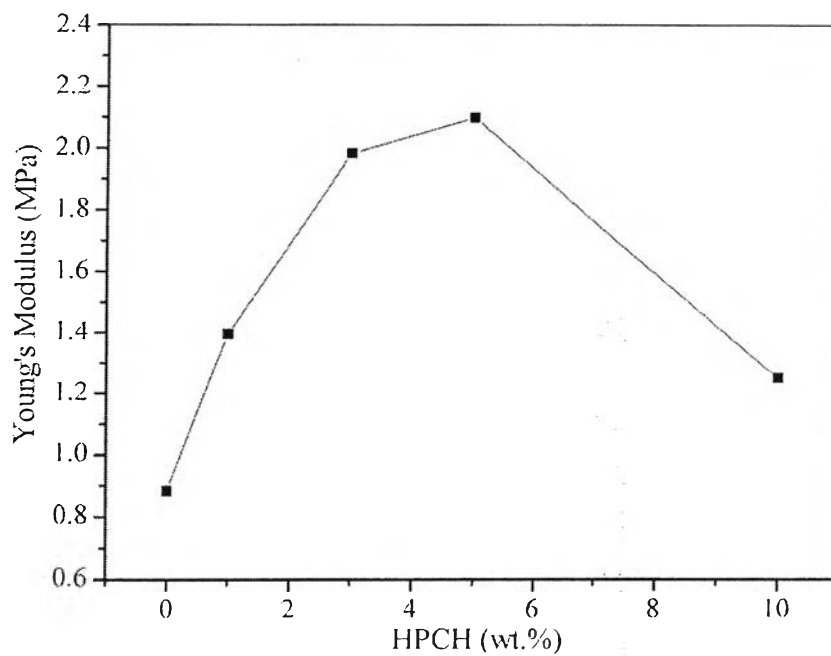


Figure 5.8 Young's modulus of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).

Table 5.1 Composition of poly(DVB)polyHIPE with hybrid organic–inorganic porous clay heterostructures (HPCH)

Sample code	Porogenic solvent	% HPCH added (wt%)	Physical impression of polyHIPE foams
Reference	—	—	brittle
HIPE1	toluene	—	chalky
HIPE2	toluene	1	chalky
HIPE3	toluene	3	less chalky
HIPE4	toluene	5	less chalky
HIPE5	toluene	10	chalky

Table 5.2 Properties of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).

Sample poly(DVB)polyHIPE With S80_T	T_d (°C)	On set (°C)	Cell Size (μm)	Cell density (g/cm³)	Compressive stress (MPa)	Young's modulus (MPa)	Percentage strain
0% HPCH; HIPE1	438.05	443.25	7.75 ± 3.8	1.2382 ± 0.0438	0.0771 ± 0.0064	0.8840 ± 0.1319	12.7913 ± 1.7618
1%HPCH; HIPE2	441.70	448.20	7.73 ± 3.9	1.2745 ± 0.0191	0.1108 ± 0.0028	1.3959 ± 0.1477	16.0829 ± 0.8219
3%HPCH; HIPE3	443.50	448.95	8.00 ± 4.3	1.3170 ± 0.0434	0.1224 ± 0.0092	1.9827 ± 0.2700	16.9475 ± 0.8552
5%HPCH; HIPE4	445.88	449.20	7.96 ± 4.3	1.4562 ± 0.1278	0.1424 ± 0.0015	2.0979 ± 0.0979	19.4126 ± 0.2747
10%HPCH; HIPE5	451.65	450.90	7.99 ± 4.1	1.4972 ± 0.1549	0.1321 ± 0.0030	1.2507 ± 0.2208	17.3091 ± 2.2528

Table 5.3 Elemental percentages of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).

Sample	% Element		
	C	O	Si
poly(DVB)polyHIPE with S80_T			
poly(DVB)polyHIPE without HPCH	93.48	1.49	—
poly(DVB)polyHIPE with 1% HPCH	79.25	2.39	0.32
poly(DVB)polyHIPE with 3% HPCH	91.98	3.16	1.15
poly(DVB)polyHIPE with 5% HPCH	90.70	3.88	1.64
poly(DVB)polyHIPE with 10% HPCH	87.84	4.19	3.35