**Morphology and Mechanical Properties of an Elastomeric Poly(HIPE) Nanocomposite Foam Prepared via an Emulsion Template**

Mohammad Reza Moghbeli* and Mercedeh Shahabi

School of Chemical Engineering, Iran University of Science and Technology (IUST), P.O. Box: 16844/13114, Tehran, Iran

Received 21 June 2010; accepted 29 January 2011

Open-cellular elastomeric nanocomposite foams were obtained by polymerization of water-in-oil high internal phase emulsions (HIPEs) containing 2-ethylhexylacrylate, styrene, divinylbenzene, and organo-modified montmorillonite (org-MMT)s. The effects of various org-MMT contents on the emulsion viscosity and microcellular structure and mechanical properties of the resulting elastomeric foams were investigated. The results showed that the incorporation of modified clay (organoclay) led to lower emulsion torque value, approximately to a characteristic value of the emulsion viscosity. Scanning electron microscopy (SEM) micrographs revealed that the presence of modified clays, 1-5% (by weight), considerably influenced the microstructure of the copolymer solid foam. For instance, the introduction of 1% (by weight) organoclay (Cloisite® 15A) decreased the mean void diameter from 5.47 to 4.45 μm and the intercellular pore size from 1.39 to 0.86 μm. At above this content of organoclay, the mean void diameter and intercellular pore size increased. The presence of organoclay may influence the separation phase behaviour within the polymerizing monomeric organic phase and it may act as a co-surfactant to vary the sizes of voids and intercellular pores. X-Ray diffraction (XRD) patterns and transmission electron microscopy (TEM) micrographs showed an intercalated nanocomposite structure for the elastomeric composite foams containing Cloisite® 30B organoclay. TEM Micrographs showed that less hydrophobic Cloisite® 30B organoclay tends to be located at the interface between aqueous and organic phases of emulsion, while in case of more hydrophobic organoclay, i.e., Cloisite® 15A, it finds its position away from the interface; inside the continuous organic phase of concentrated emulsion.

Surprisingly, adding the organoclays to the emulsions lowers the compressive mechanical properties of the reinforced solid rubbery foams compared with the copolymer foam without any reinforcement. On the whole significant improvement on the compression set was observed.

**INTRODUCTION**

Highly open-cell emulsion-derived template foams with unique properties such as high porosity and intercellular pore interconnectivity have attracted the attention of engineers, material scientists and technologists. This kind of porous material is prepared by polymerizing the continuous phase of a high internal phase emulsion (HIPE).

The emulsion consists of a large volume of internal liquid phase, at least 74% (by volume), which is dispersed in a small volume of monomeric continuous phase [1-4]. The polymerization of the HIPE...
template and the subsequent removal of the locked internal phase from the solidified foam emulsion provide an open-cellular solid porous material, termed as poly(HIPE). The cells or voids in the solid foam are interconnected with small intercellular pores [5,6]. The voids usually have a mean diameter between 5 and 100 μm, while the average size of the pores varies within 1-10 μm range. Poly(HIPE) materials have been used as biological scaffolds [7], catalyst support [8], membranes [9], separation media [10], ion-exchange resins [11,12], absorbents [13] and electrochemical sensors [14].

Most poly(HIPE) polymers are prepared based on a styrene/divinylbenzene monomer mixture and exhibit a rather chalky and brittle nature [15]. The introduction of a sufficient amount of an elastomeric monomer to styrenic monomers would improve the brittleness of rigid emulsion-template foams. A poly(HIPE) material with a low glass transition temperature (Tg) below room temperature and very low Young's modulus and crush strength is referred to as elastomeric foam [16-18]. Cameron et al. [15] prepared elastomeric poly(HIPE) materials from HIPEs containing higher levels of low Tg comonomers such as 2-ethylhexylacrylate (2EHA) and 2-ethylhexylmethacrylate (EHMA). They compared the Tg values of the resultant foams with the values determined from some theoretical models. It was found that the styrene/comonomer unit sequencing had a crucial role in determining the Tg value of the emulsion copolymer foam prepared.

Tai et al. [19] prepared a sequential interpenetrating network foam with a broad damping peak based on a P(St/EHA/DVB) poly(HIPE) foam and styrene. Menner et al. [20] have synthesized very resilient hybrid poly(HIPE) foams using poly(ethylene glycol) dimethacrylate (PEGDMA) as the main cross-linker, which substituted for divinylbenzene (DVB). They have successfully improved the mechanical performance of the foams by adding 200 nm silica particles along with a methacryloxypropyl trimethoxysilane (MPS) coupling agent to the concentrated emulsions that contain 60% (by volume) internal aqueous phase. Nevertheless, as compared with the hybrid foam without any reinforcement, a drastic change of morphology involving a great void diameter and wall thickening was observed. This kind of foam did not possess the highly open porous structure.

In another piece of work, poly(HIPE) foams with highly interconnected void network structure and desirable mechanical properties were prepared by polymerizing emulsion templates containing a higher level of DVB, silica particles and MPS coupling agent [21]. Hybrid poly(HIPE) solid foams with inorganic-organic interconnected network structure and higher thermal stability were prepared by copolymerizing MPS with styrene and DVB [22]. In this case, the inorganic poly(silsesquioxane) network inside the organic polymer network was formed through the condensation polymerization of hydrolyzed methoxyxilane groups of MPS coupling agent molecules.

Menner et al. [23] synthesized porous nano-composite foams using carbon nanotubes (CNT)s. CNT as an intrinsically hydrophobic nano-fibre is a convenient tool for stabilizing water-in-oil (w/o) concentrated emulsions. The addition of the CNTs not only eliminated the need for traditional non-ionic surfactants but also increased the mechanical and electrical properties of the poly(HIPE) foams.

Recently, layered silicate particles, such as natural and organically modified montmorillonites (org-MMT)s, have been introduced at different concentrations to control the microstructure of rigid P(St/DVB)-based poly(HIPE) solid foams [24]. It was believed that the open cell contents would increase with higher level of organoclay because of the lowering of the viscosity ratio of dispersed to continuous phases in the HIPEs.

In another research, the organic phase of SAN/DVB copolymer foam was reinforced by using different levels of organoclays to investigate the effects of organoclay on the microstructure and mechanical properties of the resulting rigid foams [25]. The X-ray diffraction (XRD) patterns of the reinforced SAN/DVB copolymer foams revealed an intercalated layered silicate nanocomposite structure for all emulsion foams.

Although many scientists have investigated the effect of emulsion composition on poly(HIPE) microstructure and properties, less attention has been focused on the role of modified clays in significant changes of microcellular structure of the rubbery
emulsion-derived foams. According to authors' finding, there is no publication on the influence of organoclays on the characteristics of elastometric poly(HIPE) foam. In this work, the effects of organically modified montmorillonites (MMT)s on the morphology and compressive properties of the P(EHA/St/DVB)-based poly(HIPE) foams have been investigated.

EXPERIMENTAL

Materials
All reagents were purchased from Merck, Germany, unless otherwise stated. Styrene (St), 2-ethylhexylacrylate (2EHA) and divinylbenzene (DVB) as cross-linking agents were purchased and distilled under vacuum to remove trace amounts of inhibitors. Sorbitan monooleate (Span80) as emulsifier was used. Potassium persulphate (K$_2$S$_2$O$_8$) and calcium chloride (CaCl$_2$.4H$_2$O) were used as water-soluble initiator and electrolyte, respectively. Toluene was used as swelling agent. Organically modified sodium montmorillonite, Cloisite® 15A (C15A) and Cloisite® 30B (C30B) organoclays purchased from Southern Clay Co., USA, were used to reinforce the polymer foams. The clay's modifier of C15A and C30B are dimethyl dihydrogenated tallow (2M2HT) and methyl tallow bis-2-hydroxyethyl ammonium (MT2EtOH), respectively [26]. Distilled de-ionized water was prepared and used.

Poly(HIPE) Synthesis
Table 1 shows the procedures in preparing HIPEs or the concentrated emulsions. The organic phase of HIPEs 0-1 consisted of monomers and sorbitan monooleate and it was poured into a 100-mL flat-bottomed glass reactor equipped with an overhead stirrer, a nitrogen inlet and a condenser. The reactor was placed in a bath with thermostatic control. In all emulsion samples, the monomer/DVB weight ratio was constant at 80/20 (wt/wt), and the sorbitan monooleate content was 15% (by weight) based on total organic phase. The aqueous phase was added drop-wise to the continuous phase through a feeder at a constant rate of 1.3 mL.min$^{-1}$. The stirring speed was held at 1000 rpm. After the addition of the internal phase, stirring speed was held constant for a further 5 min to achieve a concentrated emulsion for the homogeneous foam.

To reinforce the EHA/St/DVB copolymer foam, the commercial C15A and C30B organoclays, 1-5% (by weight) based on total monomers, were mixed with the monomeric phase before the emulsification process. For this purpose, the monomer/organoclay dispersion was mechanically stirred at 1000 rpm overnight and then homogenized by an ultrasonic homogenizer (Banedlin, Germany) for 30 min. Although the C30B organoclay is more hydrophilic than C15A, a fine dispersion in the aqueous phase was not achievable. Other emulsification process conditions were similar to those applied for the emulsions prepared without any reinforcement. The prepared

<table>
<thead>
<tr>
<th>Poly(HIPE) code</th>
<th>Oil phase (g)</th>
<th>Aqueous phase (g)</th>
<th>Clay (% by weight)</th>
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<td>EHA</td>
<td>DVB</td>
</tr>
<tr>
<td>0</td>
<td>8.3</td>
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<td>1.7</td>
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<td>1.7</td>
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</table>
HIPE samples were transferred into glass moulds and sealed to prevent water evaporation. The emulsions were polymerized at 60°C in a circulating oven for 24 h. The poly(HIPE) solid foam materials were then dried at 70°C to achieve a constant weight.

The polymerization conversion (X) of the resultant solid foams was determined by gravimetrical measurements according to the following equation [25]:

$$X = \frac{m_d (m_a - m_b)}{m_a m_b} \times 100$$  \hspace{1cm} (1)

where \(m_a\), \(m_d\), \(m_b\) and \(m_c\) are the masses of: monomers in the concentrated emulsion; dried solid foam; sample before Soxhlet extraction and sample after extraction, respectively. In addition, \(m_c\) is the mass of the organoclay inside the sample prepared after extraction. The Soxhlet extraction was carried out for 24 h in de-ionized water followed by 24 h in methanol to remove the residual monomers and emulsifier [14].

**HIPE Torque Measurement**

The torque value of concentrated emulsion as an approximate characterization of the viscosity variations and emulsion stability during HIPE preparation was measured. The software was equipped with an overhead stirrer (RTZ Hidolf, Germany) for recording the torque of the emulsions.

**Foam Morphology**

The morphology of the open-cellular foam materials was studied by means of scanning electron microscopy (SEM). In this case, the porous materials were frozen in liquid nitrogen, and then the fractured cross-section was imaged. The fracture surface of the samples was gold-sputtered before microscopy. The mean diameter of the voids, as well as the intercellular pore size in the emulsion solid foams, was approximately measured from the SEM images based on the size of 350 voids or pores for each sample [27].

The nanostructure of the organoclay-reinforced foams was studied by means of a Zeiss CEM 902A transmission electron microscope (TEM). For this purpose, ultrathin sections of the solid foam filled with epoxy resin were prepared and transferred onto copper grids and dried in open air for 2 h before microscopy. Morphology observation was carried out at an acceleration voltage of 120 kV.

**Mechanical Properties**

Comression tests on the emulsion foams were carried out according to standard ASTM D1621-94. The samples were compressed to 75% of their initial height. Young’s modulus was determined from the initial linear slope of the stress-strain curve. In addition, the stress at yield was recorded to give the crush or compression strength. Compression set of elastomeric foams under constant deflection was measured using the ASTM D395 protocol test method.

**RESULTS AND DISCUSSION**

The volume fraction of the internal phase of all HIPEs was held at 80% (by volume), while the continuous organic phase occupied 20% (by volume) of the total liquid phase. The continuous phase of HIPE 0 contained St, DVB and the surfactant (Table 1). In contrast, HIPE 1 consisted of a high level of 2EHA (2EHA/St: 70/30 by weight) as well as a cross-linker. Poly(HIPE) materials having more than 45% (by weight) 2EHA are elastomeric foams because of their low glass transition temperature \(T_g\) below room temperature [15].

In addition, HIPEs 2-9, with organic composition similar to HIPE 1, contain 1-5% (by weight) of the organoclays based on total monomer. In fact, HIPEs 0-1 without any reinforcement were prepared as reference emulsions to investigate the effects of both the 2EHA elastomeric monomer and organoclays on the microcellular structure and mechanical properties of the resulting emulsion foams. The conversions of all the neat and reinforced copolymer foams measured by gravimetrical measurements were in the range of 95-99%. The incorporation of organoclays decreased the emulsion foam conversion to some extent.

**Effect of Organoclay on the Emulsion Torque**

The rheological behaviour of the emulsions during the emulsification process with continuous addition
of the aqueous droplet phase was approximately evaluated by the torque values, which were measured automatically (Figure 1). According to this figure, the torque value of the continuous phase of emulsion at the onset of emulsification process is termed as the initial torque value and the torque at the end of emulsification process is termed as equilibrium torque.

Figure 2 shows the effect of C15A and C30B organoclay content levels on the initial and equilibrium torque values of the concentrated emulsion. The value of equilibrium torque can represent the stability of the concentrated emulsion. As shown, the incorporation of the C15A organoclay decreased the torque values up to 2% (by weight), while at above this limit, the torque increased to reach a constant value (Figure 1). This behaviour can be connected to the different performances of the organoclay as a co-surfactant when its level increases. At the lower levels, a desirable organoclay dispersion and orientation at the interface of two immiscible phases effectively lower the interfacial tension and likewise, the torque value of the concentrated emulsion.

An increased amount of organoclay above 2% (by weight) makes it to agglomerate and lowers its performance as a co-surfactant. An increase in torque value was observed for the emulsion containing 3% (by weight) C15A. In contrast, small changes in torque values were observed in emulsions containing lower or higher levels of organoclay. In fact, the additional clay by itself seems to act as a co-surfactant in lowering the interfacial tension between the two immiscible phases and compensate for the torque enhancement upon increasing the organoclay level.

However, the chemical nature of clay's modifier would influence its performance as a co-surfactant. Organoclays, depending upon their hydrophobicity, migrate and at equilibrium they are located in a thermodynamically favourable phase. It seems that less hydrophobic organoclay tends to find its location at the interface between the aqueous and organic phases of emulsions, while the hydrophobic organoclay is located away from the interface inside the continuous organic phase of concentrated emulsion.

A rather similar torque variation was observed for the concentrated emulsions containing various amounts of C30B. Nevertheless, the emulsions with C30B organoclay showed higher torque values than those containing C15A organoclay (Figure 2). This difference may arise from more hydrophilic nature of C30B, which may depress the performance of Span80 used with a low hydrophilic-lipophilic balance (HLB) number.
Foam Morphology

SEM Micrographs (Figure 3) show a typical highly open porous network structure for the P(St/DVB) poly(HIPE) prepared by polymerizing the continuous organic phase of its corresponding emulsion. For this brittle emulsion foam, the weight-average cell diameter and intercellular pore size were 7.82 and 2.45 μm, respectively. The addition of 2EHA monomer to the St/DVB organic phase (2EHA/St: 70/30) lowered the cell diameter to 6.57 μm and intercellular pore size to 1.90 μm. This behaviour can be attributed to the lowering of interfacial tension between two immiscible phases within the concentrated emulsion upon addition of 2EHA ingredient (Table 2).

Table 2. Characteristics of the elastomeric poly(HIPE) foams reinforced by various levels of organoclays.

<table>
<thead>
<tr>
<th>Poly(HIPE) code</th>
<th>Void</th>
<th>Pore</th>
<th>dD/Dn</th>
<th>ρ×10² (g.cm⁻³)</th>
<th>E×10⁶ (MPa)</th>
<th>σcr×10² (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Dn (μm)</td>
<td>PDI</td>
<td>d₀ (μm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>5.47±0.68</td>
<td>1.21</td>
<td>1.39±0.20</td>
<td>0.254</td>
<td>11.0±0.88</td>
<td>7.54±0.90</td>
</tr>
<tr>
<td>2</td>
<td>4.45±0.51</td>
<td>1.16</td>
<td>0.86±0.11</td>
<td>0.193</td>
<td>9.8±0.59</td>
<td>3.67±0.55</td>
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<tr>
<td>3</td>
<td>4.68±0.70</td>
<td>1.26</td>
<td>0.90±0.10</td>
<td>0.192</td>
<td>10.6±0.05</td>
<td>2.71±0.27</td>
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<tr>
<td>4</td>
<td>4.85±0.47</td>
<td>1.10</td>
<td>1.09±0.19</td>
<td>0.224</td>
<td>10.8±0.97</td>
<td>2.78±0.39</td>
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<tr>
<td>5</td>
<td>3.66±0.43</td>
<td>1.16</td>
<td>1.08±0.11</td>
<td>0.295</td>
<td>11.2±0.90</td>
<td>4.30±0.40</td>
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<td>9</td>
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<td>0.247</td>
<td>10.9±1.09</td>
<td>3.71±0.48</td>
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</table>

Figure 4. SEM Micrographs of elastomeric poly(HIPE) nanocomposite foams containing various levels of C15A: (a) 1%, (b) 2%, (c) 3% and (d) 5% (all by weights); C30B: (e) 1%, (f) 2%, (g) 3% and (h) 5% (all by weights) (mag.: ×2000).
Figure 5. Effect of organoclay levels on mean void diameter and intercellular size of resultant composite foams.

Polymerization of HIPEs 2-9 with various levels of C15A and C30B organoclays resulted in a characteristic open-porous foam structure (Figure 4). Overall, the incorporation of both organoclays within the elastomeric foams seems to considerably decrease the size of the voids and intercellular pores when compared with the copolymer foam without any reinforcement.

Figure 5 indicates the changes in the mean size of the voids and pores with an increase in organoclay level. Based on SEM micrographs (Figures 3d, 4a and 4b) it is clear that by addition of just 1% (by weight) of organoclay to the foam emulsion i.e., HIPE 1, the void size is changed and very small intercellular pores appear in the reinforced solid foam. Nevertheless, the statistical analysis was carefully followed to investigate quantitatively the effect of organoclay on the foam morphology and properties (Table 2 and Figure 5). As it is shown, the experimental error was below 15%. The incorporation of 1% (by weight) organoclay into HIPE 1 foam makes very small intercellular pores to appear with lower mean pore size as well as the mean void diameter (Figures 4a and 4b).

As it is well-known, the size of intercellular pore could be supported by the intensity of phase separation and shrinkage occurring in the polymerizing continuous phase [5,6]. Thus, the introduction of a small amount of organoclay may influence the separation phase behaviour within the polymerizing organic phase for small intercellular pores to emerge. The very small intercellular pores start to disappear as the organoclay level increases (Figures 4g and 4h). The tendency of organoclays to agglomerate at higher levels could be the reason for the granular dispersion of organoclay and pore size enlargement in the foam polymer matrix. According to Figure 5, the mean void diameters and intercellular pore sizes of all reinforced foams were decreased up to 1% (by weight) organoclays, while above this limit the sizes did increase.

The foams with 3% (by weight) organoclays showed the largest void diameter and intercellular pore size as compared with all the other reinforced emulsion foams. Probable organoclay particle agglomeration and subsequent viscosity enhancement for the concentrated emulsions with higher organoclay levels may result in reinforced foams with larger voids and intercellular pores (Table 2). Nevertheless, the poly(HIPE) foams reinforced by using various levels of C30B had a larger void diameter than those containing the C15A organoclay.

Using less hydrophobic organoclay, i.e. C30B, seems to have a lower efficiency in decreasing the interfacial tension between two immiscible phases as compared to a more hydrophobic C15A. A decrease in cell wall thickness and cell interconnectivity (d_n/D_n) was observed for the reinforced foams as compared with the neat copolymer foam (Table 2). On the other hand, the size distribution of voids and intercellular pores decreased upon incorporating the organoclays, especially for the solid foams reinforced by lower organoclay levels.

Foam Micro- and Nanostructure

X-Ray diffraction (XRD) patterns of the P(EHA/St/DVB) poly(HIPE) foams reinforced by using various organoclay contents revealed an intercalate structure only for the layered silicate nanocomposite samples containing C30B organoclay (Figure 6). As shown, an increase in the interlayer spacing of C30B organoclay is observed after polymerizing the emulsions due to the shifts of XRD peaks to lower diffraction angles. This behaviour can be attributed to the probable formation of the copolymer chains within the montmorillonite galleries. In
contrast, the characteristic peaks of the organoclay in nanocomposite samples containing 1%, 2%, 3% and 5% (all by weights) C15A are shifted to higher 2θ values which are indications of no intercalation or exfoliation and for that the clay interlayer distances are decreased. This behaviour could be due to weak thermodynamic interactions between C30B modifier molecules and copolymer chains in the nanocomposite samples.

TEM Micrographs were used to investigate the dispersion state of the organoclay inside the copolymer matrix of the reinforced foam. For this purpose, the open-cell foam was filled with epoxy resin (Figure 7) to provide sufficient structural integrity and prevent the probable brittleness or folding of ultrathin sections during microtomy. The white and grey regions in the TEM micrographs correspond to the copolymer matrix and the epoxy resin, respectively (Figure 8). In contrast, the organoclay can be visualized as dark stacks of silicate layers. At lower magnification, the cellular morphology showed the spherical voids merging together to a great extent to form an interconnected network structure (Figure 8a). The micrographs exhibit the dispersion state of stack roads within the organic copolymer matrix (Figures 8b to 8d). However, the stacks tend to be located at the interface region between continuous and dispersed internal phases.

For the nanocomposite foams with highest organoclay content, 5% (by weight) C15A, the stacks tend to aggregate because of the intensive phase separation occurring in the organic matrix (Figures 8c and 8d). Although no intercalation or exfoliation is observed for the C15A nanocomposite samples (Figure 6), the TEM micrographs at higher magnifications show a partial intercalated structure for the C15A nanocomposite sample (Figures 8e and 8f).

**Foam Properties**

A rather constant apparent foam density close to 0.1 g.cm\(^{-3}\) was observed for the copolymer emulsion foams with different organoclay contents. The incorporation of organoclays lowers the compressive
mechanical properties of the reinforced solid foams when compared with the copolymer foam without any reinforcement (Table 2 and Figure 9). This behaviour can be attributed to more intense microcellular heterogeneity and the diminishing void wall thickness of the resulting reinforced foams. Nevertheless, the organoclay particle agglomeration inside the thin void wall seems to decrease the foam compressive strength under loading due to the stress concentration developed around the large agglomerates. Although with the introduction of organoclay particles of a high modulus crystalline mineral material is expected to increase the glass storage modulus of polymers, at the same time it tends to lower the rubbery modulus to some extent [28].

Swelling of the reinforced foams in toluene resulted in higher swelling ratio in comparison to the copolymer foam without any reinforcement. Thus, the

Figure 8. TEM Micrographs of nanocomposite foam reinforced with 5% (by weight) C15A: (a) the cellular morphology of the nanocomposite foam, (b) to (d) the dispersion state of organoclay stacks within the organic copolymer matrix, and (e), (f) partial intercalated structure.
Figure 9. Compressive stress-strain curves of elastomeric nanocomposite foams reinforced with various levels of: (a) C15A and (b) C30B.

The introduction of organoclay seems to decrease the cross-linking density within the continuous phase, and consequently, to reduce the Young’s modulus of the resulting reinforced foam. In fact, the stress-strain curves confirmed that addition of the commercial C15A and C30B organoclays to the neat copolymer foam led to foams with more elastomeric properties (Figure 8). However, the strong chemical covalent bond between copolymer chains and silicate layers within the copolymer matrix could improve the reinforced foam’s mechanical properties.

The ability of elastomeric poly(HIPE) foams to maintain elastic properties after prolonged compressive loading was evaluated by the compression set values. For the nanocomposite rubber foams exposed to compressive stress for a given time, a small permanent deformation dependent upon its elastic property is observed (Table 3). As shown, the lowest compression set values were obtained for elastomeric emulsion foams reinforced with 2% (by weight) organoclays. The lower Young’s modulus and narrow void size distribution of the aforementioned reinforced foams may result in lower compression set values.

### CONCLUSION

Nanocomposite elastomeric foams have been prepared through the copolymerization of continuous phase of HIPEs containing 2EHA, St, DVB and organoclays. The 2EHA/St (70/30 wt/wt) ratio was constant for all the emulsion foams, while the C15A and C30B levels varied from 0 to 5% (by weight) based on total monomer content. Incorporating the organoclays lowered the foam emulsion viscosity to some extent. It seems that the organoclay acts as a co-surfactant in weakening the water/monomer interfacial interactions and consequently in lowering the viscosity of foam emulsion. A decrease in the mean size of the voids and intercellular pores was observed for the nanocomposite copolymer foams as compared with the copolymer foam without any reinforcement.

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**Table 3. Compression set values for the prepared poly(HIPE) nanocomposite foams.**

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<th>Poly(HIPE) code</th>
<th>25°C t = 0 min</th>
<th>25°C t = 30 min</th>
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<td>0.7</td>
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<td>1.4</td>
<td>0.3</td>
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<td>0.2</td>
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Nevertheless, the voids and pore sizes in the foams reinforced by using C15A were smaller than those in the foams containing C30B organoclay. This behaviour can be attributed to the effect of organoclay's nature, which may influence the phase separation behaviour in continuous phase polymerization. Furthermore, increasing the organoclay level increased the mean void diameter and intercellular pore size due to the organoclay particle agglomeration and emulsion viscosity enhancement. The XRD and TEM results confirmed the dispersion of intercalated clay stacks within the copolymer matrix. However, the presence of unexfoliated organoclays did not improve the foam compressive mechanical properties.

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