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A Study on Water Absorption and its Effects on Strength of Nano Organoclay-epoxy Composites

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Abstract: Nanoclay-epoxy composites have been applied widely since they have excellent properties which resulted from the incorporation of the nanoclay material in epoxy matrix. However, the improved properties can degrade by presence of water in the epoxy matrix, leading to limitation of service life of the material. Diffusion of water having 40 and 60°C in the nanoclay-epoxy composites made by direct mixing process was investigated through immersion test. The strength of the materials was also measured by using the three point bending test. The results showed that the two-stage diffusion process occurred when the composites immersed in water at 60°C whereas the Fickian diffusion mode was observed in case of water at 40°C. The saturation and the diffusion rate are influenced by organoclay loading and by temperature. Increase of clay loading resulted in an increase of the water saturation and reduction of diffusion rate of water. The results also showed the significant influence of temperature on water absorption of the composites. Water saturation, increased about 20% and water diffusion rate was 5 time-higher when temperature of water shifted from 40 to 60°C. Investigation of flexural strength was carried out by use of the three point bending test. The results showed decrease of flexural strength with an increase of clay loading for the composites. However, there was no significant change of flexural strength during the immersion. Results for the neat resin showed to the contrary.

Key words: Diffusion, nanoclay-epoxy composites, flexural strength, saturation, water immersion

INTRODUCTION

Nanocomposites are a new class of materials in which the ultrafine phase dimensions ranges from 1 to 100 nm (Komarneni, 1992). Much investigation has proven that these materials have new and improved properties in comparison to their micro- and macro-composite counterparts. In general, polymer nanocomposites, a branch of the nanocomposite material show an improvement in terms of properties to conventional filled polymer. The polymer-layered silicate nanocomposites is a special example for the improvement, in which markedly improved properties such as modulus and strength (Kojima *et al.*, 1993a), permeability (Yano *et al.*, 1993; Kojima *et al.*, 1993b), shrinkage (Kelly *et al.*, 1994; Haque and Armeniades, 1986), heat resistance and flammability (Giannelis, 1996; Lee *et al.*, 1997) is attributed nanometer-size dispersion in the base polymer.

Epoxyes are considered as the best polymeric materials for many applications and widely used in industry as matrix materials of fiber-reinforced composites due to their superior characteristics such as good mechanical properties and good resistance to chemicals. Thus, this resin is significantly utilized in industry as a matrix material, especially in the aerospace and automotive

industry (Kshirsagar *et al.*, 2000; Becker *et al.*, 2003). However, the service life of epoxy based composites materials can be decreased due to the degradation of epoxy matrix caused by working environment such as temperature, moisture and corrosive elements (Kshirsagar *et al.*, 2000; Kim *et al.*, 2005). With dispersion of nanoclay in the epoxy matrix to perform a nanocomposite, the material is expected to not only overcome the disadvantages of the base polymer and also to obtain new characteristics.

The clay-epoxy nanocomposite materials can be produced by two common methods: the direct mixing and the solution mixing (Jiankun *et al.*, 2001; Zerda and Lesser, 2001; LeBaron *et al.*, 1999; Kornmann *et al.*, 2001). These techniques produce intercalated or intercalated/exfoliated composites rather than fully exfoliated composites (Zerda and Lesser, 2001). It is generally believed that the diffusion performance of clay nanocomposites normally influenced by the clay content, the aspect ratio of the barrier (ratio of width over length of the barrier) and the dispersion of the silicate layers in which the reduction of water permeability was attributed to the high aspect ratio of the clay platelets and the high degree of exfoliation (Ke and Yongping, 2005; Gain *et al.*, 2005; Picard *et al.*, 2007;

Gusev and Lusti, 2001; Fredrickson and Bicerano, 1999; Nielsen, 1967; Bharadwaj, 2001). This reduction is one of the most excellent properties that made the material to be applied widely in industry recently.

Degradation process, such as chemical degradation, reduction of mechanical properties etc., occur when the composite materials contacted to the environment. The environment penetration into a polymer composite is influenced by the composite's characteristics such as density and the order of pore, defects or contamination and the polymer-environment affinity (Wong and Broutman, 1985; Diamant *et al.*, 1981; Adamson, 1980; Moy and Karasz, 1980). In the studies of liquid molecular transport into a polymer membrane, diffusion has been classified as Case 1 (Fickian type), Case 2 (relaxation-controlled) and non-Fickian (anomalous) (Thomas and Windle, 1980; Frisch, 1980; Peterlin, 1980; Hansen, 1980; Astaluta and Sarti, 1978). Penetrant molecules diffuse into the membrane until the concentration is equal over the whole of the membrane (saturation state). When Fickian diffusion is assumed, the time-dependent relative concentration of the liquids into the polymer membrane can be expressed by the following equation (Vergnaud, 1991):

$$\frac{C_{(t,x)}}{C_{\infty}} = 1 - \frac{4}{\pi} \sum_{m=0}^{\infty} \frac{1}{(2m+1)} \exp\left[-\frac{D(2m+1)^2 \pi^2 t}{l^2}\right] * \sin\left[\frac{(2m+1)\pi x}{h}\right] \quad (1)$$

in which $C_{(t,x)}$ and C_{∞} are the concentrations of the liquid inside the membrane at time t , position x and saturate state, D is diffusivity which depends on the nature of liquid-polymer interactions and h is the thickness of the membrane. The boundary conditions for solving Eq. 1 are: $C = 0$ when $t = 0, 0 \leq x \leq h$; $C = C_{\infty}$ when $t > 0, x = 0, x = h$ and $\partial C / \partial x = 0$ when $x = 0, t > 0$. The diffusion coefficient has been calculated by Crank (1975):

$$\frac{M_t}{M_{\infty}} = \frac{4}{h} \sqrt{\frac{Dt}{\pi}} \quad (2)$$

where, M_t and M_{∞} are the mass uptake at time t and saturation state, respectively. The diffusivity can be calculated from the initial slope of the environmental uptake M_t/M_{∞} versus time ($t^{1/2}/h$) as:

$$D = \frac{\pi}{16} \left[\frac{M_t/M_{\infty}}{\sqrt{t/h}} \right] \quad (3)$$

The water content may predict by using the equation as follow (Shen and Springer, 1976):

$$\frac{M_t}{M_{\infty}} = 1 - \exp\left[-7.3\left(\frac{Dt}{h^2}\right)^{0.75}\right] \quad (4)$$

The high order absorption of water is a major disadvantage of epoxy resins. Furthermore, the absorbed water is considered as a main factor resulted in degradation of functional, structural and mechanical properties of the composites (Lee and Neville, 1957; Lu *et al.*, 2001; Nunez *et al.*, 1999; Yano *et al.*, 1993). Therefore, understanding of diffusion behavior of water in particular clay epoxy nanocomposite system is need for application of the material.

In this study, the water absorption behavior of nano organoclay epoxy composites was investigated. Effects of clay loading, environment temperature on the absorption water barrier characteristic are discussed. Mechanical properties such as flexural strength, flexural modulus during environmental exposure, were also studied and discussed in the study.

MATERIALS AND METHODS

Materials and sample preparation: Three components are need to form the clay-epoxy nanocomposites using for the investigation: the Bisphenol A Epoxy Epomik R140 made by Mitsui Chemical Co., Ltd used as the matrix component of the composites; the diamine having commercial name as Jeffamine D230 made by Huntsman Co. used as curing agent and the organoclays received from Nanocor Inc. named as Nanomer I.28E.

The organoclay was swelled with the curing agent and mixed with a mechanical stirrer at 2500 rpm for 1.5 h at 60°C, followed by ultra-sonication for an additional 1 h. When the swelling process had been finished, the epoxy resin was added. It should notice that before the curing process started, degassing process under vacuum was applied. The curing was done in two stages: the first stage was carried out at 70°C for 6 h while the second was at 110°C for 6 h. The load of the clay varied from 0 to 6 phr (parts per hundred resin). Table 1 shows code and formula of the prepared samples.

The test pieces having dimension of 60×25×1 mm were cut out from the prepared sample sheets. The pieces were then dried up at 50°C for 72 h to ensure that the remaining moisture/gas was removed before the immersion test was carried out.

Table 1: Sample used for investigation

Code	Organoclay (phr)	Curing agent (phr)
CE0	0	32
CE2	2	32
CE4	4	32
CE6	6	32

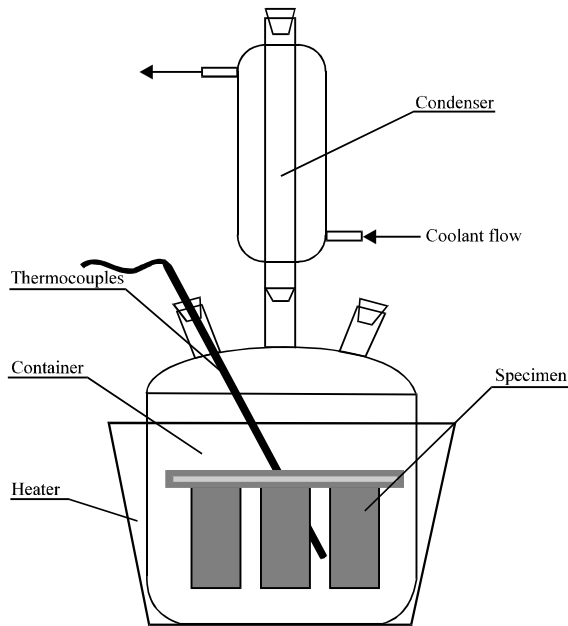


Fig. 1: Schematic of the immersion test apparatus

Immersion test: Immersion experiment was carried out by simply immersing the test pieces in de-ionized water. The test pieces were fixed in a polytetrafluoroethylene holder to avoid contact surface each other and immersed in separable flash bottles which filled with the water. Temperature of the water was constantly set at 40 and 60°C. Figure 1 shows a schematic of the immersion test apparatus.

At interval time, the specimens were taken out, wiped by filter paper to remove attached water on their surfaces and weighed by a balance with 0.1 mg accuracy. A change in mass was measured related to initial mass.

Flexural test: Specimens, taken out from the immersion chamber at interval time of immersion, were measured the flexural modulus and strength by the three point bending test. The test was done regarding to ASTM D790 (American Society for Testing and Materials ASTM, 2005) with Shimadzu Autograph AGS-1KNJ machine. Speed of the crosshead was set at 15 mm min⁻¹ constantly.

RESULTS AND DISCUSSION

The water uptake during immersion was measured by comparing the test specimen's weight to its initial weight. The time-dependent uptake amount for the different organoclay loadings is shown in Fig. 2 to 5. It can be seen that the uptake increased linearly with increasing time at first, then leveled off. The leveling off is considered as

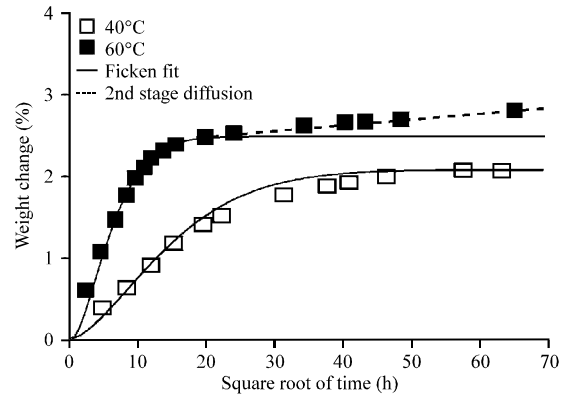


Fig. 2: Weight change vs. time of CE0 sample under exposure of de-ionized water

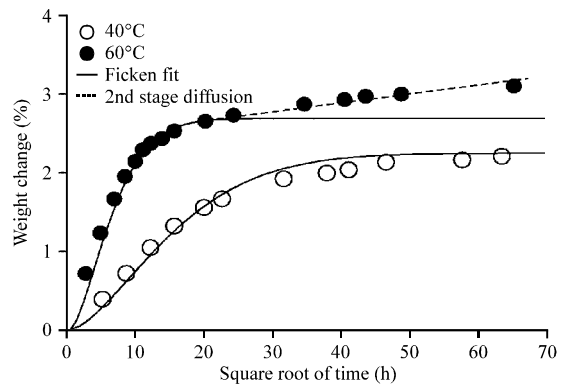


Fig. 3: Weight change vs. time of CE2 sample under exposure of de-ionized water

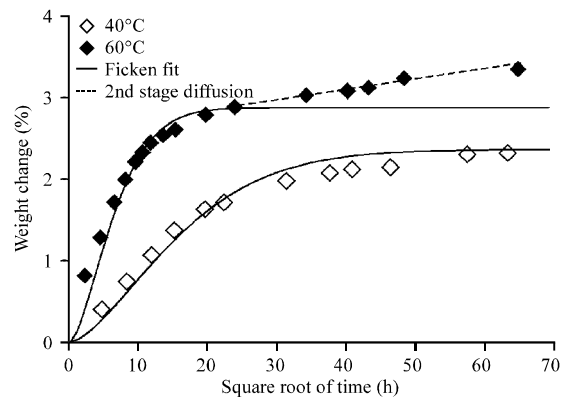


Fig. 4: Weight change vs. time of CE4 sample under exposure of de-ionized water

saturation state of the absorption process. The prediction curves based on Eq. 3 and 4 are also superimposed in Fig. 2-5. The water diffusion characteristic into the samples was shown in Table 2.

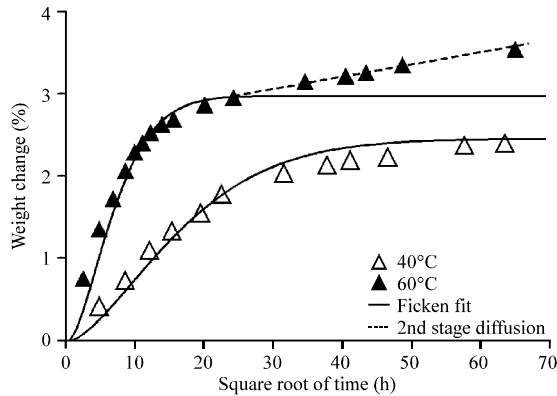


Fig. 5: Weight change vs. time of CE6 sample under exposure of de-ionized water

Table 2: Water diffusion characteristics of samples

Sample	Saturation		1st stage diffusivity, 10^{-4} (mm ² h ⁻¹)
	Value (%)	Time (h)	
40°C water			
CE0	2.091	2078	2.96
CE2	2.270	3364	2.70
CE4	2.407	3969	2.47
CE6	2.496	4356	2.27
60°C water			
CE0	2.499	1764	14.95
CE2	2.699	2025	13.44
CE4	2.899	2209	12.56
CE6	3.001	2401	11.39

Effect of clay loading: The results showing in Fig. 2 to 5 as well as in Table 2 confirmed the influence of the clay loading on water diffusion behavior into the materials. The saturation degree and saturation time increased with an increase of the clay content, whereas the diffusivity decreased with an increase of the clay loading. For the neat epoxy exposed to the 40°C water (Fig. 2, Table 2), the saturation obtained as 2.091% after about 2078 h of the immersion and the diffusivity was as $2.96 \times 10^{-4} \text{ mm}^2 \text{ h}^{-1}$. It is also seen that the water diffusion behavior was fitted well with the Fickian model. Exposure to the same water environment, the nanocomposites of 2, 4 and 6 phr clay loading became saturated at 2.27, 2.407 and 2.496% within 3364, 3969 and 4356 h and the diffusivity decreased to 2.70×10^{-4} , 2.47×10^{-4} and $2.27 \times 10^{-4} \text{ mm}^2 \text{ h}^{-1}$, respectively (Fig. 3-5). The similar results were also obtained for the samples exposed to the 60°C water environment. As seen in these figures, saturation value was lowest as about 2.499% for the neat epoxy, higher as 2.699 and 2.899% for the 2 and 4 phr clay nanocomposites then highest as 3.001% for the 6 phr clay nanocomposite, whereas, the diffusion rate was the highest for the neat epoxy then the lower for the 2 and 4 phr clay nanocomposites and the lowest for the 6 phr clay nanocomposite. These results are similar to that reported

by Abacha *et al.* (2009), Okada *et al.* (1990), Lan and Pinnavaia (1994) and Messersmith and Giannelis (1995). Thus, presence of incorporated nanoclay particles in the epoxy matrix resulted in the increase of saturation degree and the decrease of water diffusion rate. As discussed by Popineau *et al.* (2005), the water molecules after diffused in the composites can only stay in micro-voids and form clusters. If all micro-voids/free volumes were filled by diffused water molecules, the saturation is attained. The number of micro-voids/free volume may increase when more organoclay was added and that is considered as a reason for the increase of saturation order with an increase of clay loading. The decrease of the diffusion rate with an increase of clay loading is attributed by dispersion of clay particles within the epoxy matrix and by exfoliation order of the composites (Okada *et al.*, 1990; Lan and Pinnavaia, 1994; Messersmith and Giannelis, 1995). Under the same preparation conditions, the higher clay loading composite may result in higher order of the exfoliation and that contribute to the better barrier characteristic of the composite.

Effect of temperature: Temperature affected significantly the diffusion behavior of water into the materials. As seen in Fig. 2-5, the saturation value and the diffusion rate increased when the temperature of water environment shifted from 40 to 60°C. For instance, for the neat epoxy resin (Fig. 2), the water saturation was higher as about 12.5% at 60°C than those at 40°C. For the 2, 4 and 6 phr clay nanocomposite (Fig. 3-5) this difference is about 18.89, 20.44 and 20.23%, respectively. Remarkable 5 time increase of the diffusivity was observed when water temperature shifted to 60 from 40°C. Because the difference between saturation values as well as diffusion rates at 40 and 60°C is the same among the composite systems then it may conclude that the effect of temperature was mainly acted on the epoxy matrix. An explanation for the effect of temperature would be an expansion/creation of micro-voids/free volume inside the material by thermal relaxation of polymer chain of the epoxy matrix.

An interesting was found when observation effect of temperature on the water diffusion behavior of the material. The two stage diffusion (non-Fickian mode) occurred when the samples immersed in water at 60°C. This phenomenon is attributed to chemical interaction of diffused water molecules to the functional groups of the epoxy matrix and the thermal relaxation effect (Nguyen and Martin, 1996).

Mechanical properties: The result of three-point bending test under wet condition is shown in Fig. 6 and 7.

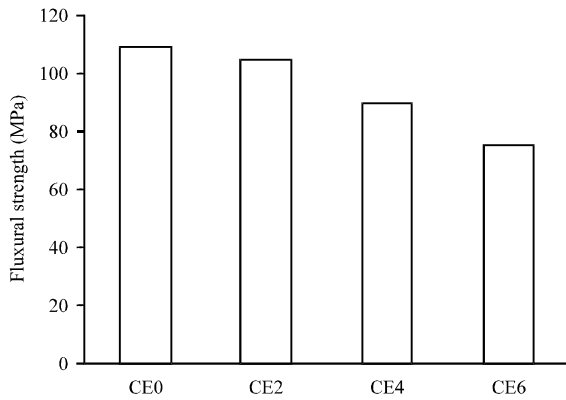


Fig. 6: Flexural strength of samples

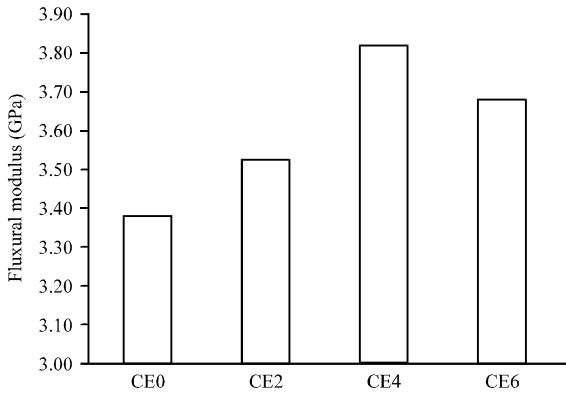


Fig. 7: Flexural modulus of samples

Figure 6, the initial flexural strength decreased with the increase of the nanoclay loading. It may result from agglomeration of the nanoclay in epoxy matrix. The adhesion strength and interfacial stiffness of interface between the nanoclay particles and the epoxy matrix are the important factors which contribute flexural strength of the composites due to they are significant to stress transfer and elastic deformation from the epoxy matrix to the fillers. The result of flexural modulus showed in Fig. 7 is accordance with the manner showed in Fig. 6.

Retention of properties as same as at initial, during service time, is an important criteria to evaluate application potential of any materials. In terms of mechanical properties, the flexural strength of the test pieces was measured during the immersion test and its result is shown in Fig. 8. An evaluable information, as seen in this figure, is that there are not much change of the strength of nanoclay-epoxy composites samples while the strength of the neat epoxy reduced about 20% after immersed 100 h, then maintaining as long as the immersion time. It can be explained by distortion produced around the amine salt resulted the decrease of the C-N bond of

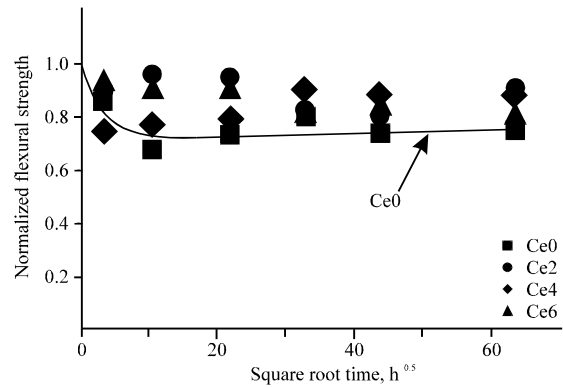


Fig. 8: Normalized flexural strength (wet condition) vs. Immersion time in the 40°C de-ionized water

the epoxy matrix. When the organoclay is added, this distortion may be hindered, therefore, the strength is retention.

CONCLUSIONS

This study concluded that diffusion behavior of water into the organoclay-epoxy composites is influenced by the clay loading and temperature of environment. The water situation increased with an increase of the clay content whereas the diffusion rate decreased with an increase of the clay loading. The uniform dispersion of clay particles in the epoxy matrix and higher order of exfoliation in the composites are considered as a main reason resulted in the effect. Temperature effect on diffusion behavior of water into the materials is significant. The saturation value increased about 20% when temperature shifted from 40 to 60°C. The diffusion rate increased about 5 times when temperature changed in these values. The effect of the temperature is attributed only by the thermal effect of epoxy matrix. Two-stage diffusion occurred for the polymers immersed in water at 60°C.

Flexural strength of the samples decreased with increasing the organoclay loading for the composite samples. However, the retention of this property during immersion test is the same as those at initial which did not obtain for the neat resin sample.

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