

The Optimum Reaction Condition of Synthesize Epoxy-organoclay Nanocomposites

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ABSTRACT: The aim of this study is to obtain the optimum reaction of synthesize epoxy-organoclay nanocomposit. The optimum conditions of synthesize epoxy-organoclay nanocomposite are determined by the effect of duration, speed and temperature of premixing of epoxy-organoclay. The optimum reaction of nanocomposites is confirmed by tensile test and optical microscopy result. In situ polymerization method is used to synthesize epoxy-organoclay nanocomposite. The tensile test results indicate that the optimum conditions to synthesis of epoxy-3.1 wt.% organoclay nanocomposite by using 210 rpm of mixing speed, 90 minutes of mixing time and 80°C of mixing temperature. The elastic modulus of nanocomposite material is 16% higher than epoxy matrix. Meanwhile, the maximum stress is 3% lower than epoxy matrix due to the existence of voids. According to the fracture surface analysis, the nanocomposite material formed coarse material, bumpy and a lot of voids and clumps of filler particles that indicated the nanocomposite material to be brittle. The surface morphology of epoxy matrix is smooth and flat, and the characteristic of amorphous materials can also be seen with a glowing surface and smooth like glass.

Keywords - Nanocomposite, epoxy, organo clay, process condition, tensile strength.

I. INTRODUCTION

Nanotechnology, especially polymer nanocomposites, has been used in a wide range of advanced plastic products such as biomedical engineering, electronics, energy, automotive, sports gear, packaging as well as aircraft and aerospace engineering. Polymer nanocomposites have become a new class of materials that could replace conventional composite materials for better performance with excellent properties in terms of increased strength and modulus, improved heat resistance, decreased gas permeability and flame retardance at a very low loading nanofillers [1-2].

Several researchers have undertaken the study of the clay dispersed epoxy polymer composite materials. In the 1990's, Toyota Research who was the first to introduce this concept had discovered the possibility to build a nanocomposite from 6-nylon and organophilic clay. Their new material showed dramatic improvement in mechanical and physical properties. The addition of organophilic clay can simultaneously improve the tensile strength, tensile modulus, flextural strength and flextural modulus of theses polymer matrix of higher functionalities [3-4]. Kornmann et al. [2] have reported the modified MMT clay that incorporate epoxy resins cured with different amines. Haipan Salam et al. [5] has studied the effect of organo-clay amount to mechanical properties of epoxy-organoclay nanocomposites.

R. Kotsilkova in "Thermoset Nanocomposites for engineering application" said that the best performance of an epoxy nanocomposite system with nanofillers would result from the homogeneous dispersion of the nanofillers and strong interfacial adhesion between the epoxy matrix and the nanofillers. Furthermore, shear premixing is an important process for the dispersion of nanoclays in polymeric resins, the effect of temperature, duration, speed of premixing, and also the interlamellar spacing of clay platelets on the dispersion of organoclay in epoxy by using a high speed premixing technique which can generate high shearing mechanism. The following major problems may be derived from the compatibilisation between nanofiller and matrix polymer as an underlying critical success factor that must be highlighted [6-7].

The aim of this study is to obtain the optimum reaction of synthesize epoxy-organoclay nanocomposit. The optimum conditions of synthesize epoxy-organoclay nanocomposite are determined by the effect of duration, speed and temperature of premixing of epoxy-organoclay. The optimum reaction of nanocomposites is confirmed by tensile test and optical microscopy result. In situ polymerization method is used to synthesize epoxy-organoclay nanocomposit.

II. EXPERIMENTAL

In this study, the epoxy resin was DGEBA (EPR 174; Bakelit Korea Co. Ltd.) processed with the reactive diluent polyamide (Versamide 140; COGNIS Corporation). The EPR174/ Versamid 140 mixing ratio was 50:50 (w/w). The nanocomposites were made with Nanomer I.30E Nanoclay (Nanocore Inc,USA).

The first step was to heat the clay in the furnace at 50°C for 30 min. To fabricate the nanocomposites, the epoxy resin was mixed mechanically in a beaker glass with 3.1 wt % of clay. Duration of mixing varied from 30 min to 120 min and also mixing speed varied from 210 rpm to 1480 rpm. Moreover, temperature mixing varied from 70°C to 90°C. Later, the mixture was cooled to room temperature for 15 min. Then, a stoichiometric amount of the curing agent (curing agent : epoxy resin ratio = 1:1) was added. After mixing it mechanically for 15 min, the mixture was poured into aluminum molds. The composites were cured for 2 h at 150°C.

Tensile testing was conducted to measure modulus elastic and maximum stress for both imides-cured neat epoxy and its clay nanocomposites with different mixing process variable. The experiments were performed at a crosshead velocity of 2 mm/min. At least five specimens for each composition were tested. The fracture surface morphology was investigated by optic microscope.

III. RESULT AND DISCUSSION

3.1 The Optimum Premixing Variable of Synthesize Epoxy-organoclay Nanocomposites

3.1.1 The Effect of premixing duration on tensile properties

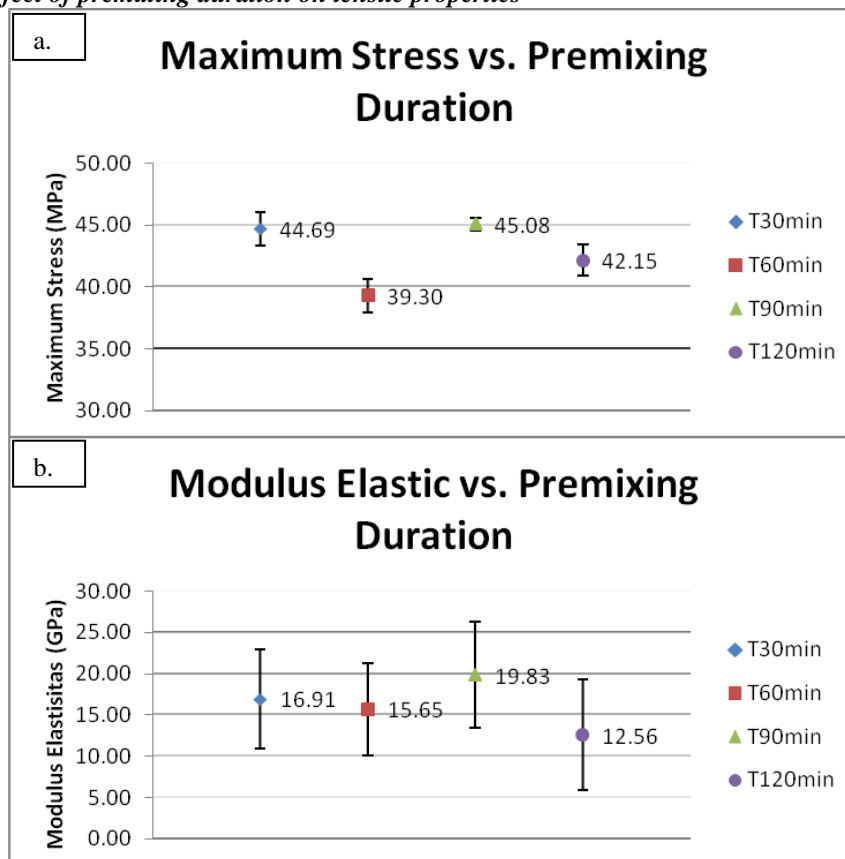


Figure 1 Effect premixing duration on tensile properties of epoxy-organoclay nanocomposit, a.

Maximum stress vs. premixing duration, b. Modulus elastic vs. premixing duration.

Tensile tests were conducted to measure the maximum stress and modulus elastic of nanocomposites presented with different duration of premixing in Figure 1. It can be seen that the maximum stress fluctuated with the increasing of duration of premixing. However, 90 minute of premixing showed the highest maximum stress with 45.08 MPa.

On the other hand, the modulus elastic of the nanocomposites increased continuously with increasing premixing duration (Fig. 1). Using 120 minute premixing duration, it showed the lowest modulus elastic compared to other duration. Meanwhile, 90 minute premixing duration resulted the highest modulus elastic which is around 19.83 GPa. Modulus elastic is a measure of the stiffness of materials. The increasing of modulus elastic values has influenced the strain failure of materials. It would make nanocomposit material become rigid. This response is characteristic for materials reinforced with stiff fillers.

3.1.2 Effect of premixing speed on tensile properties

The optimum duration of premixing shown above was used to synthesize epoxy-organoclay nanocomposites with variation of premixing speed. It was found that premixing speed influenced tensile properties as showed in Fig. 2. The maximum stress values gradually decreased with the increasing of premixing speed. Premixing with 210 rpm of speed has the highest maximum stress which is 50.31 MPa. However, premixing with 1040 rpm of speed was 50% lower than 210 rpm of premixing speed. The trend of reducing the maximum stress may be related to air bubble issues of higher concentration of clay in polymer system.

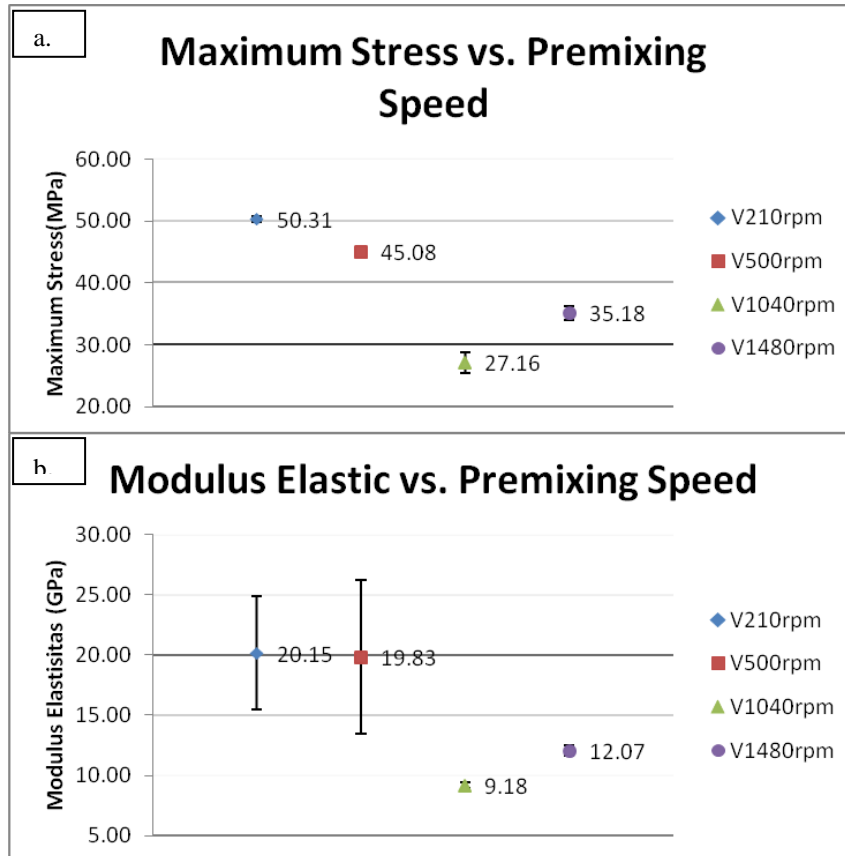


Figure 2 The Effect of premixing speed on tensile properties of epoxy-organoclay nanocomposit, a.

Maximum stress vs. premixing speed, b. Modulus elatic vs premixing speed.

On the other hand, it showed that modulus elastic values dramatically decreased with the increasing of premixing speed (Fig. 2). As well as maximum stress, premixing with 210 rpm has the highest modulus elastic value which was 20.15 GPa. Its values closed to 500 rpm of those which was 19.83. Moreover, modulus elastic with 1040 rpm of speed had the lowest maximum stress value.

3.1.3 The Effect of premixing temperature on tensile properties

Similar to the previous one, the optimum premixing duration and speed variables are used to obtain the optimum premixing temperature in synthesize epoxy-organoclay nanocomposites. The temperature varied from 70°C to 90°C. The tensile test result is shown in Fig. 3. From this figure, premixing temperature has much more influenced tensile properties of nanocomposit materials. The maximum stress of 80°C premixing temperature increased 25% compared to 70°C premixing temperature. However, the maximum stress of 90°C premixing temperature was slightly lower than 80°C while it's higher than 70°C premixing temperature.

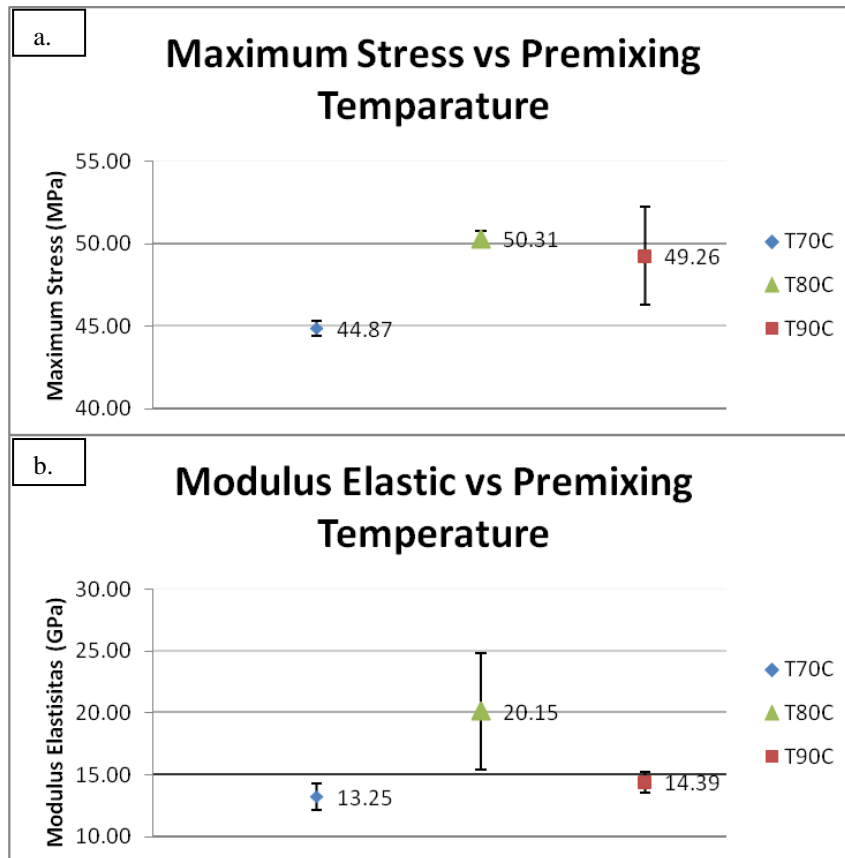


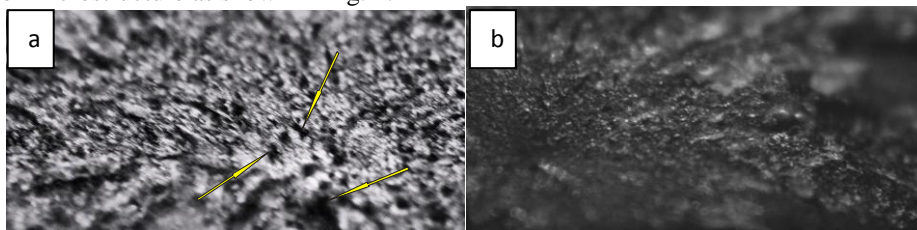
Figure 3 The Effect of premixing temperature on tensile properties of epoxy-organoclay nanocomposit, a. Maximum stress vs. premixing speed, b. Modulus elatic vs premixing speed.

Those trends also found in the modulus elastic curve (Fig. 3). The modulus elastic of 80°C temperature was one and a half times higher than two other premixing temperature. So, based on those results, the optimum conditions of synthesize epoxy-3.1 wt.% organoclay nanocomposite were used 210 rpm of premixing speed, 90 minutes of premixing duration and 80°C of premixing temperature.

Tabel 1 Tensile properties of epoxy-organoclay nanocomposit and Neat epoxy.

SAMPPEL	Filler	Maximum Stress	Modulus Elastic
	(wt.%)	(MPa)	(GPa)
Neat epoxy	0	51,94±2,58	17,31±3,60
Nanokomposit	3,1	50,31±0,44	20,15±4,69

If it is compared to neat epoxy, the comparisons are shown in table 1. The modulus elastic was 16 % higher than neat epoxy. Meanwhile, the maximum stress was 3% lower than neat epoxy. The trend of reducing the tensile strength may be related to air bubble issues in polymer system which could be found from morphology of microstructure as shown in Fig. 4.



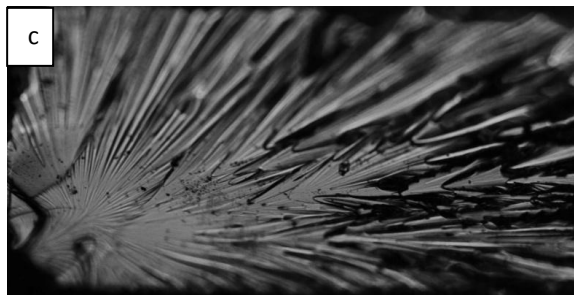


Figure 4 Morphology structure of a. Nanocomposite with 90 minute of premixing duration and 500 rpm premixing speed; b. Nanocomposit with 90 minute of premixing duration and 210 rpm premixing speed; c. Neat Epoxy

The surface morphology of nanocomposit could be seen in Fig. 4 a-b. Based on the result from optical microscopy, it can be seen the surface morphology of soft material with coarse-grain and chevrons or fan-like pattern that indicates the location of the breaking in the surface. It is caused by air bubble as shown in Figure 4 a-b. It is indicated that the nanocomposites are brittle. It is also expected that the mechanical strength is low due to the existence of voids. Elimination of aggregates and tiny voids reduced interfacial debonding which are the important issues to improve the tensile properties. Effective lengths, orientation of the silicate layer, as well as polymer chains with respect to the loading direction are also important in the reinforcement. It is well known that particle aggregation are weak points in the materials and is the primary reason of reducing the strength of materials even though the agglomeration may be strong enough to increase the initial modulus [6]. The surface morphology of epoxy matrix is smooth and flat, and it's also seen the initial cracks that occur during a tensile test. The initial cracks might be formed due to the void that causes crosslinking in the epoxy molecules that are weak. Moreover, it can also be seen that the characteristic of amorphous materials posses a glowing surface and smooth like glass.

IV. CONCLUSION

The present work deals with the effect of reaction condition of synthesize epoxy-organoclay nanocomposites prepared using a variation of duration, speed and temperature of premixing epoxy-organoclay nanoxomposites material. Epoxy-organoclay nanocomposites were prepared using DGEBA epoxy, 3.1 wt.% organoclay and polyamide as hardener by in situ polymerization method. The optimum conditions of synthesis epoxy-3.1 wt.% organoclay nanocomposite utilized 210 rpm of premixing speed, 90 minutes of premixing duration and 80°C of premixing temperature. The tensile test result indicated that elastic modulus of nanocomposite material was 16% higher than epoxy matrix. Meanwhile, the maximum stress was 3% lower than epoxy matrix due to the existence of voids. According to the surface analysis, the nanocomposite material formed coarse material, bumpy and a lot of voids and clumps of filler particles that indicated brittle materials. The surface morphology of epoxy matrix is smooth and flat, and it shows initial cracks that occur during a tensile test. The initial cracks might be formed due to the void that causes crosslinking in the epoxy molecules that are weak.

V. Acknowledgements

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