

## EFFECT OF NANOFILLERS DISPERSION ON MECHANICAL PROPERTIES OF CLAY/EPOXY AND SILICA/EPOXY NANOCOMPOSITES

Napisah Sapiai, Aidah Jumahat\*, Norhashidah Manap, Mohd Aidil Idham Usoff

Faculty of Mechanical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

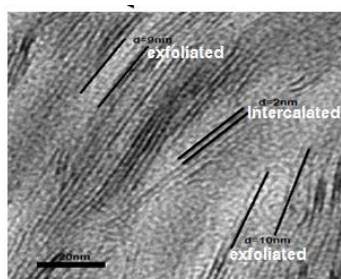
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\*Corresponding author

aidahjumahat@salam.uitm.edu.my

### Graphical abstract



### Abstract

A homogeneous dispersion of nanofillers in epoxy polymer still remains as one of the biggest challenges in advanced nanocomposites research. In this paper, the effects of nanoclay content (1wt%, 3wt%, 5wt%) and nanosilica content (5wt%, 13wt%, 25wt%) on tensile and compressive properties of epoxy polymer were studied. The nanoclay and nanosilica were dispersed in epoxy using the three-roll milling and mechanical stirrer machine, respectively. The compressive and tensile properties were evaluated using Instron universal tester machine according to BS standards. The Transmission Electron Microscopy (TEM) was used to evaluate the degree of dispersion of nanofillers in epoxy. The results showed that the intercalated structure of clay/epoxy nanocomposites gave detrimental effect on the tensile and compressive strength of the polymer. Whereas, the results of nanosilica/epoxy nanocomposite system showed that a well-dispersed nanosilica contributes to the improvement of tensile and compressive strength and Young's modulus of the polymer.

Keywords: Nanoclay, nanosilica, nanocomposite, tensile, compression

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## 1.0 INTRODUCTION

The use of epoxy resins as a matrix in automotive, aerospace and ship building has gained much interest due to their outstanding mechanical properties; good flexibility to strength, good adhesion strength, good heat resistant and high electrical resistance[1-3]. However, cured epoxies are brittle thus this behaviour is a big constraint to high end engineering applications. In order to improve the properties of epoxy, the addition of different types of nanofillers (such as nanosilica, nanoclay and carbon nanotubes) has gain great attention to many researchers[4-5]. The final properties of the nanocomposite not only depend on types of fillers but also the size, quality of particles dispersion, volume percentage of nanofiller and the compatibility between nanofiller and polymer matrix

[6-8]. The dispersion of nanoclay still remains as the biggest challenge in order to get exfoliated and homogeneous dispersion. Nanoclay platelets tend to retain their stacked-layer structure even though the d-spacing has been expanded through milling process. The dispersion of nanoclay either tactoids, intercalated or exfoliated structure is usually indentified using Transmission electron microscopy (TEM) or X-ray diffraction (XRD). An Intercalated structure has a d-spacing between 2.37-8 nm and an exfoliated structure has d-spacing more than 8nm. While, for tactoid structure, clay platelets are still stacked together of less than 2.37nm clay inter galleries [9]. In this study, the effect of nanofillers distribution and content on tensile and compressive properties of clay/epoxy and silica/epoxy nanocomposites is investigated.

## 2.0 EXPERIMENTAL

### 2.1 Preparation of Epoxy/Silica and Epoxy/Clay Nanocomposites

The epoxy resin and hardener used for this experiment were supplied by Miracon (M) Sdn Bhd. Nanoclay (I30) and Nanosilica (Nanopox F400) were used as filler where these materials supplied by Nanocor Inc.USA and nanoresin AG, Geesthacht, Germany, respectively. Nanoclay I.30E is an onium surface modified montmorillonite mineral. Nanopox F400 consists of surface-modified syntetic SiO<sub>2</sub> nanosphere which has an average diameter of 20-50 nm.

In order to produce a series of clay/epoxy nanocomposites with 1wt%, 3wt%, 5wt% nanoclay content, the I.30 E nanomer has been mixed with epoxy resin using high shear three roll mill machines at 10 rpm with temperature of 60 °C. While, a series of silica/epoxy nanocomposite (addition of 5wt%, 13wt%, 25wt%) was prepared using a mechanical stirrer at 400rpm for 1 hour. The mixtures were degassed in a vacuum oven for 1 hour to remove entrapped air. After that, the mixtures of nanocomposites were poured into silicone mould according the standard size for tensile and compression. Finally the samples were post cured at 60°C for 2 hours, followed by 60°C for 2 hours, 80°C for 2 hours, 100°C for 2 hours and 120°C for 2 hours.

### 2.2 Morphology Evaluation of Epoxy/Silica and Epoxy/Clay Nanocomposites

Transmission electron microscopy (TEM) FEI TECNAI 120KV BIOTWIN was used to evaluate the dispersion of the nanosilica particles and nanoclay platelet in the epoxy resin. The images were captured at magnifications of 43000x and 300000x for silica/epoxy and clay/epoxy, respectively.

### 2.3 Tensile and Compression Tests

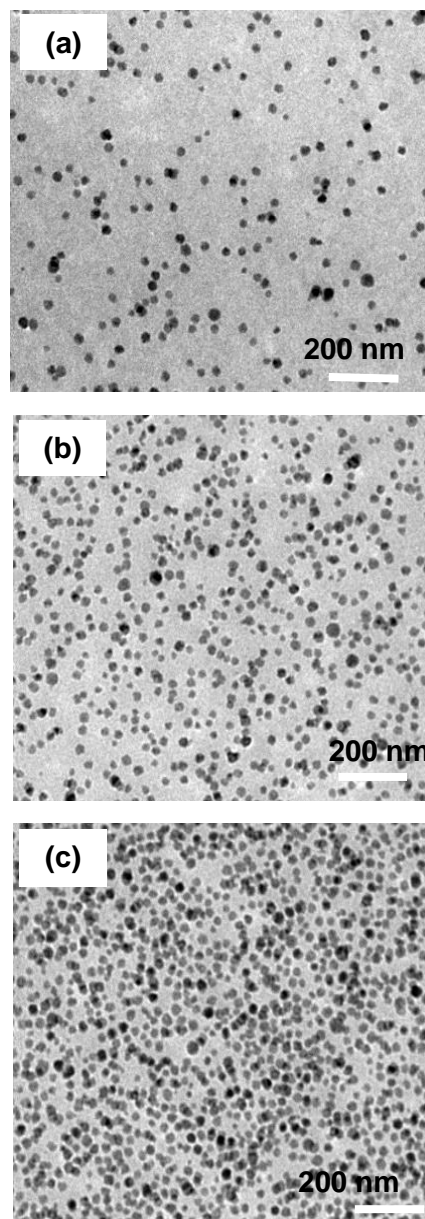
Tensile and compression tests were carried out using Instron universal testing machine. Tensile specimens with a dimension of 33mm x 50mm x 2mm were adhered with a tab of 1.5mm thick glass fiber, which follows the BS EN ISO 572-1. The cylindrical specimens with dimension of 1:1 length to diameter (L/D) ratio (10mm/10mm) were used for compression test according to BS EN ISO 604:2003. Both types of specimens were tested at a crosshead speed of 1mm/min.

## 3.0 RESULTS AND DISCUSSION

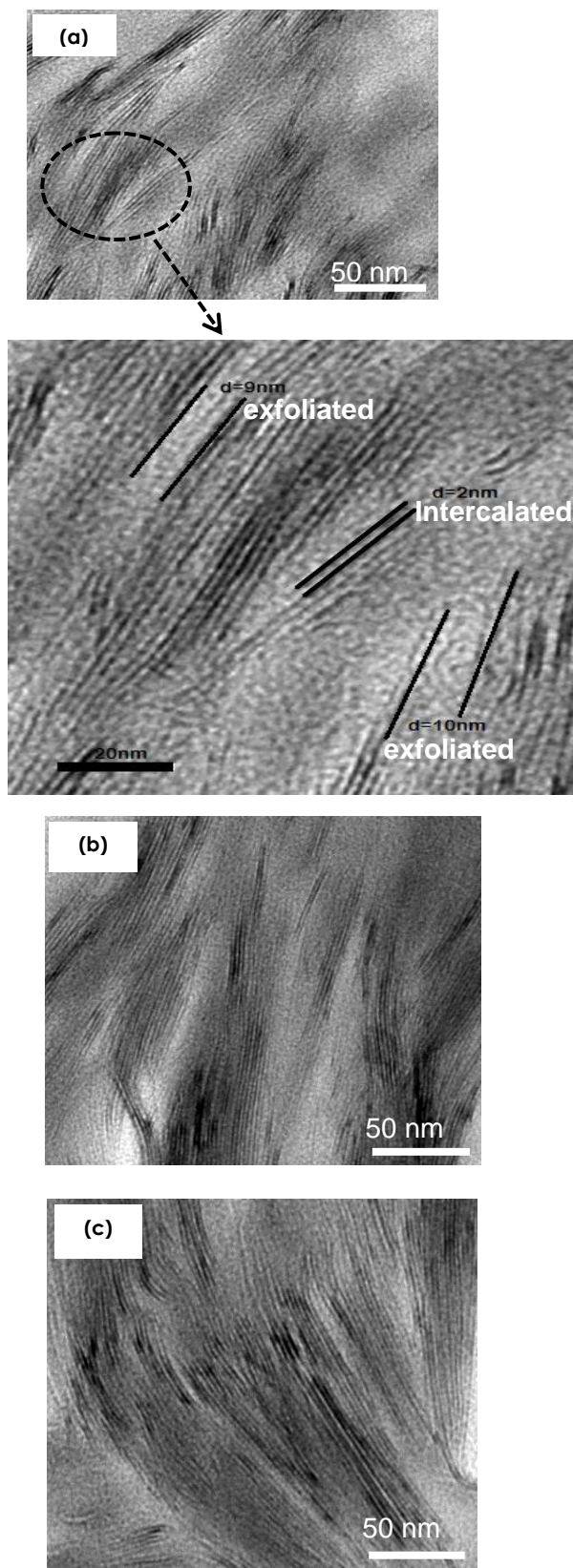
### 3.1 Morphology of Clay/Epoxy and Silica/Epoxy Nanocomposites

The dispersion of nanofillers (clay and silica) in epoxy resin is one of the important factors that influence

the mechanical properties of the nanocomposites. In order to improve the mechanical properties of polymer, nanofillers need to be well-dispersed in epoxy resin. Some researchers claimed the nanoparticles have higher tendency to agglomerate due to higher aspect ratio and smaller particles size[6]. The agglomerated nanofillers will introduce local stress concentration and poor matrix adhesion. This leads to a premature failure and thus will reduce the strength of the nanocomposites. Figure 1 shows the TEM images for silica/epoxy nanocomposite. From the result, a uniform distribution of nanosilica in epoxy resin was observed even at 25 wt% volume fraction.



**Figure 1** TEM images for dispersion of silica/epoxy nanocomposites (a) 5wt% (b) 13wt% and (c) 25wt% with magnification of 43000x



**Figure 2** TEM images for dispersion of clay/epoxy nanocomposites (a) 1wt% (b) 3wt% and (c) 5wt% with magnification of 300000x

Figure 2 shows the TEM images for clay/epoxy nanocomposite. The morphology of 1 wt%, and 3wt% clay/epoxy nanocomposite is predominantly intercalated with some exfoliated structures. At high clay content (5wt%), the image present of intercalated structure. The images showed that the d-spacing of nanoclay is about 2-3 nm which indicates that the intercalated structure was obtained.

### 3.2 Tensile Properties

Figure 3(a) shows the tensile properties of the clay/epoxy nanocomposites. The addition of small amount nanoclay of 1wt% increased the tensile strength up to 20.1% but the addition of 5wt% nanoclay decreased the tensile strength about 12%. TEM image (as shown in Figure 1) shows the evidence of intercalated structure rich regions in 5wt%. This may contribute to high localized stress during tension and caused a premature failure in nanocomposites properties.

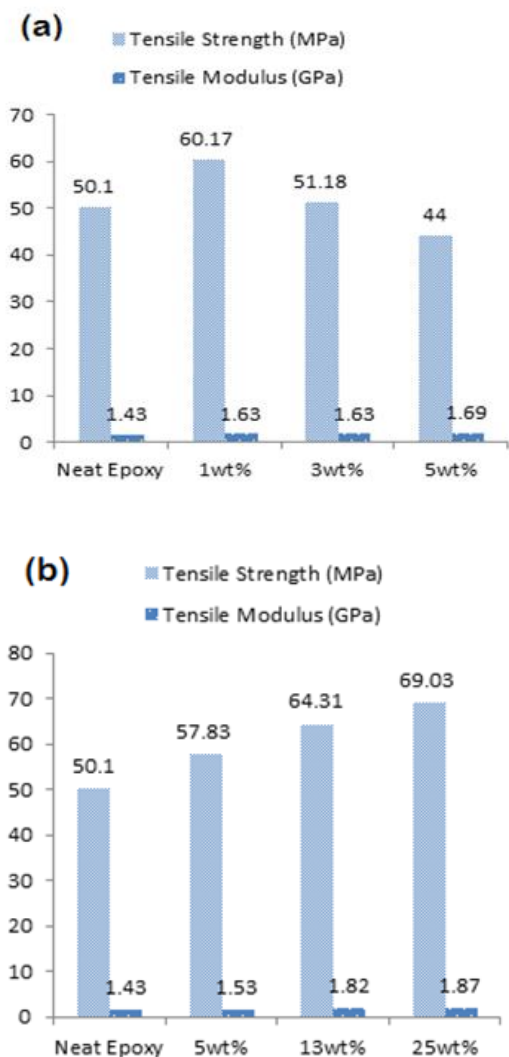
Figure 3(b) shows that the tensile strength improves with increasing percentage of nanosilica addition. The addition of 5wt% of nanosilica enhanced tensile modulus of about 6.6% and tensile strength of about 15.4%, while 13wt% nanosilica improved tensile modulus by 2.67% and tensile strength by 28.4%. 30% and 37% improvement of tensile modulus and tensile strength, respectively, were found in 25wt% nanosilica. This proves that homogenous dispersion of silica nanofiller as shown in TEM micrographs contributes to the improvement in tensile microproperties of polymer. In addition, the compatible of nanofiller-matrix also contributes to improved strength of nanocomposites.

### 3.3 Compressive Properties

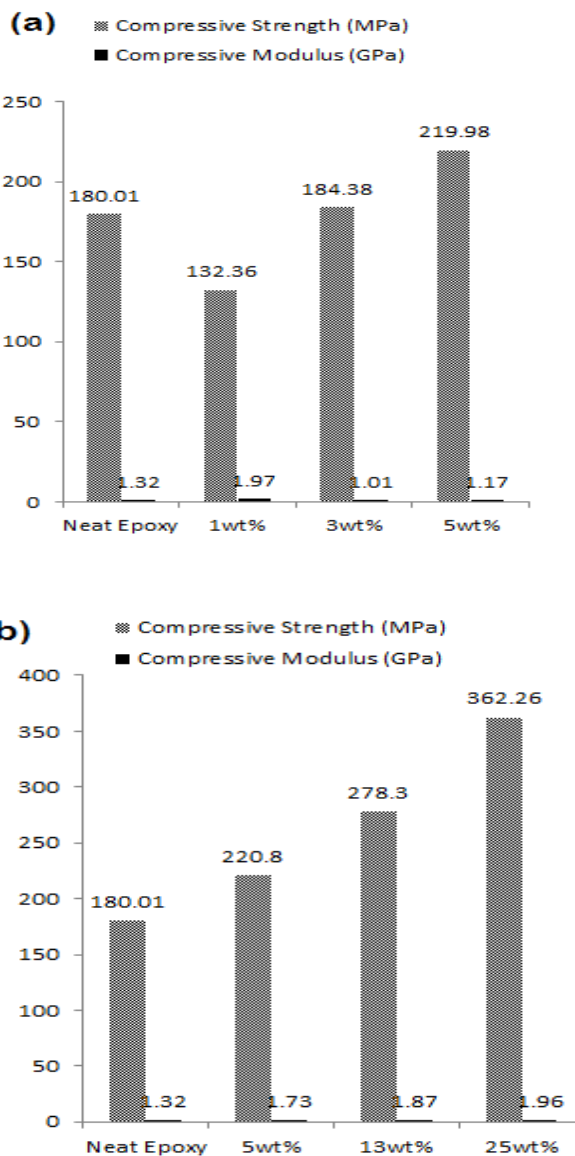
Figure 4(a) shows the compressive properties of the clay/epoxy nanocomposites. The results showed the addition of 1wt% nanoclay reduced the compressive strength by 26.7%. For 3wt% nanoclay addition, the compressive strength increased about 2.4%. As shown in Figure 2, the intercalated structure of clay was found in nanocomposites, therefore this introduces high localised stresses in matrix which leads to premature and brittle failure. For 5wt% nanoclay addition, the compressive strength was increased about 22.2% compared to the pure epoxy resin. Domination of nanoclay platelets was assumed to contribute to this phenomenon as the compressive strength depends more on nanoclay properties rather than epoxy matrix.

The compressive properties of silica/epoxy nanocomposites are displayed in Figure 4(b). It was found that the addition of nanosilica into the epoxy matrix enhanced the compressive properties of nanocomposites. The compressive strength increases up to 22.7% for 5wt% nanosilica addition, 54.6% for 13wt% nanosilica addition and 101.2% for 25wt% nanosilica addition. This suggests that the well

dispersion of nanosilica (as shown in Figure 1) produced good interaction between nanofillers and matrix. Consequently, the stresses were successfully transferred via interfaces thus leads to higher strength of nanocomposites.



**Figure 3** (a) Tensile strength and tensile modulus of clay/epoxy nanocomposites and (b) Tensile strength and tensile modulus of silica/epoxy nanocomposites



**Figure 4** (a) Compressive strength and compressive modulus of clay/epoxy nanocomposites and (b) Compressive strength and compressive modulus of silica/epoxy nanocomposites

## 4.0 CONCLUSION

In this study, a good quality of clay/epoxy nanocomposites and silica/epoxy nanocomposites samples were successfully fabricated. The effects of nanofillers dispersion on tensile and compressive properties were investigated. The TEM was conducted to evaluate the dispersion of nanofillers. The results showed that intercalated structure of nanoclay reduced the tensile and compressive strength of epoxy polymer but a well-dispersed nanosilica in silica/epoxy nanocomposites consistently enhanced the tensile and compressive strength. It was found that the highest percentage of

nanosilica (25wt%) increased the tensile and compressive strength of about 37.8% and 101.2%, respectively.

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