A Comparative Study Of Mixing Technique For Nanocomposite Filled Geopolymer

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Abstract— The effects of mixing approach on compressive properties, water absorption and morphology of the epoxy nanocomposites incorporating 0 and 3 wt% fly ash-based geopolymer were investigated. The mechanical stirrer with two different type of propeller; four blade (type A) and dissolver (type B) was used to blend the epoxy with the nanoclay and the fly ash-based geopolymer filler. It was found that the epoxy nanocomposite prepared using dissolver propeller (type B) performed higher compressive strength as compared to the one prepared using four blade propeller (type A). We postulate that this was due to an improved mixing efficiency and dispersion quality as observed through Field Emission Scanning Electron Microscopy (FESEM)

Keywords— epoxy, compressive, montmorillonite, fly ash, geopolymer, nanocomposites, dissolver stirrer

I. INTRODUCTION

Epoxy resins have been widely employed as matrix material due to their ease of processing as well as versatility for numerous applications [1]. Many of researchers reported epoxy has successfully enhanced mechanical properties when incorporating with nanoparticles such as montmorillonite organoclay, nanosilica, carbon nanotubes and nanofibres [2]. However, the structure and properties of nanocomposites are influenced by processing method to disperse the filler or nanoparticles in an epoxy matrix. Numerous methods, such as mechanical stirring, high shear mixing, ultrasonic; ball milling and solvent mixing have been practiced by many authors in an attempt to obtain a homogeneous dispersion of nanofiller in the nanocomposite [3-5]. Among these processes, high speed shear mixing reported to result in better mechanical properties of the polymer-based nanocomposites as compared to other techniques such as ultrasonic mixing [6].

In this study, epoxy-layered silicate nanocomposites were developed based on Digilcydyl ether of bisphenol A (DGEBA), and contained montmorillonite as the nanofillers with the addition of 3% optimum fly ash geopolymer filling. The aim of this work is to study the effect of two different mixing propeller, which are four blades (type A) and dissolve stirrer (type B) on the mixing efficiency, filler dispersion quality and mechanical performance of the epoxy nanocomposites incorporating MMT and the fly ash-based geopolymer.

II. MATERIALS AND METHOD

The epoxy resin which is based on Digilcydyl ether of bisphenol A (DGEBA) chemical compound was supplied by Euro Pharma and employed as the matrix material. Isophorondiamine (IPDA) was used as curing agent to harden the nanocomposites, and it was supplied by DrRahmatullah Sdn. Bhd. The nanoclay used was montmorillonite surface modified with 35-45 wt.% dimethyl dialky(14-18) amine, which was supplied by Sigma Aldrich. The raw fly ash-based geopolymer filler with sizes range 50-100µm was obtained from Arab Saudi.

A series of nanocomposite with 3% nanoclay content with 0% and 3% of fly ash based geopolymer were prepared by mixing and compounding using mechanical stirrer with two different shear mixing propeller; four blades (type A) and dissolve stirrer (type B). The mixture then was deggased in a vacuum chamber to remove entrapped air, which after blended with the appropriate stoichiometric amount of IPDA hardener. The nanomodified resin afterwards poured into release coated silicon mold and cure at room temperature.

III. TESTING AND CHARACTERIZATION

A. Compression Testing

The cured resins with dimension 10x10x14 mm³ were fabricated for the uniaxial compression test using the Instron test machine. Compression test was conducted on cube specimen according to ASTM D695 with a crosshead speed 1mm/min.
B. Water Absorption Test

The water absorption tests were carried out according to ASTM D570 by total immersion of three specimens in distilled water at room temperature. At regular intervals, the specimens were taken out from the water and wiped with filter paper to remove surface water and weighed with digital scale. The percentage of water absorption (W %) was calculated using the following equation:

\[
W(\%) = \left( \frac{W_2 - W_1}{W_1} \right) \times 100\%
\]

where \( W_2 \) is the weight of the sample after immersed in water and \( W_1 \) is the initial weight of the sample respectively.

C. FESEM Characterization

The microstructure observation were carried out using field emission scanning electron microscope (FESEM) Nova NanoSEM-450 working to 5 kV to allow the observation with more resolution. In order to facilitate the observations under FESEM microscope, the cross section nanocomposite samples were previously cut and covered with a thin platinum layer using pump sputter coater (Quarum Q 150R-S) machine.

IV. RESULTS AND DISCUSSION

A. Compression Properties

The effectiveness of two mixing methods was quantified by comparing the values of the compressive properties for the nanocomposites filled 0% and 3% fly ash geopolymer as illustrated in Fig. 1. The differences between the resulting composite properties can be explained by method of mixing for nanocomposites preparation that assimilate shear mixing four blades (type A) and dissolver stirrer (type B).

It is conceivable to observe that type A preparation promotes the lowest mechanical properties. Type B shows to be better than type A nanocomposites preparation, promoting values about 72% ultimate stress and 71% higher of modulus for nanocomposites with 0% geopolymer filler and 65% ultimate stress and 77% higher of modulus for nanocomposites with 3% geopolymer filler. The high compressive strength reveals a strong interaction between polymer chains with the filler due to shear mixing effect from the mechanical stirrer [7]. The values of compressive stress achieved by nanocomposites with 0% and 3% geopolymer filler content mixed by type A propeller are 24.26 Mpa and 31.06 Mpa respectively, while the nanocomposites produced by type B propeller exhibit the values of 48.70 MPa and 51.35 MPa respectively. These suggest that the mixing process using type B propeller resulted in greater compressive strength of the epoxy nanocomposites as compared to type A propeller.

Table 1 shows the summarized data for values of compressive stress, strain, yield strength and modulus of nanocomposites filled fly ash geopolymer. This phenomenon can be explained by dissimilar propeller type used during pre-intercalation of montmorillonite and epoxy resin before the addition of geopolymer filler and curing process, where type B propeller promotes a good radial flow for drawing the material to be mixed from the top and the bottom with high turbulence at high shearing forces to lead pre-intercalation process efficiently.

<table>
<thead>
<tr>
<th>Types of Propeller</th>
<th>Geopolymer content(phr)</th>
<th>Compressive Strength (Mpa)</th>
<th>Compressive Strain (%)</th>
<th>Compressive Modulus (Mpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type A</td>
<td>0</td>
<td>24.26</td>
<td>0.43</td>
<td>4259</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>31.06</td>
<td>0.80</td>
<td>4727</td>
</tr>
<tr>
<td>Type B</td>
<td>0</td>
<td>48.70</td>
<td>0.69</td>
<td>6890</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>51.35</td>
<td>0.82</td>
<td>7273</td>
</tr>
</tbody>
</table>
B. Water Absorption Properties

Fig. 2 shows the percentage of water uptake by nanocomposites for two types of processing method type A and type B. It can be seen that the water absorption increase with addition of fly ash based geopolymer filler at 3%. The increase percentage in water absorption is due to hydrophilic nature of nanoclay and geopolymer filler. However, for type B processing method using dissolver stirrer, the water uptake was slightly decrease to 0.09% from 0.10%, 0.12% to 0.10% for 0% and 3% geopolymer content. It shows the processing type B is better than type A in dispersion of nanoclay and geopolymer filler. This phenomenon can be explained by considering the nanoclay possess excellent moisture barrier properties because the penetration direction of water molecule into the polymer structure can be interrupted by their very large aspect ratio if less agglomeration of the filler. As the filler content increase, the formations of agglomeration increase due to difficulties of achieving a homogenous dispersion [9]. The expected agglomeration of filler in the composites due to processing method type A has increase the water absorption of the composite.

![Fig. 2. Water absorption of fly ash based geopolymer nanocomposites column graph for two types of propeller stirrer (type A: four blade; type B: dissolver stirrer).](image)

C. Micrograph study

Fig. 3 and 4 shows FESEM micrograph of cross section morphology for the epoxy nanocomposites with 0% and 3% fly ash geopolymer filler at different method processing type A and B. It can be observed that generally, type A obligate roughness surface which possibly led to lower compressive stress of the material. This might predominantly cause by the micro voids which initiate cracks at the interfaces of nanocomposites, attributed by poor adhesion between the agglomerate filler (nanoclay and geopolymer particles) and the resin [10]. However, high shear mixing produced from dissolver propeller (type B) resulted in greater efficiency of blending and dispersing, hence contributed to smoother surface morphology. This more homogenous mixture of epoxy, MMT and geopolymer particles was believed to contribute to an increased in compressive strength. As can be seen in Fig. 4 (b), the surface of epoxy nanocomposites with geopolymer filler was smooth with the appearance of good dispersion of fly ash particles at 1000x magnification. In contrast, Fig. 4 (a) shows the dispersion of fly ash geopolymer filler are lumped together at the bottom of left corner for type A processing technique, due to restricted filler/fiber load transmission between fly ash and epoxy nanoclay.

![Fig. 3. FESEM micrographs of the nanocomposites with 0% fly ash geopolymer at 1000x magnification (a) Type A: four blade (b) Type B: dissolver stirrer](image)
V. SUMMARY

In this study, the influence of mixing technique on epoxy nanocomposites with 0% and 3% of fly ash geopolymer filler was studied by means of compressive properties, water absorption and morphology. Nanocomposites were fabricated using two different mixing techniques by different propeller; four blade (type A) and dissolver stirrer (type B). The compressive properties were measured and cross sections were characterized using Field Emission Scanning Electron Microscopy. It was observed that mixing of nanocomposites using dissolver propeller (type B) resulted in greater compressive strength and less water uptake when compared to four blade propeller (type A) as well as better dispersion of clay particles and fly ash filler.

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REFERENCES