Investigation of nano-alumina on some mechanical and morphological properties of unsaturated polyester

Zahraa T. Khamees, Khalid M. Oweed
College of Engineering, Al-Mustansiriya University, Baghdad, Iraq.

Received 9 Jan. 2017; Received in revised form 10 March 2017; Accepted 13 March 2017; Available online 1 Nov. 2017

Abstract
The objective of this research to investigate the influence of adding nano (Al$_2$O$_3$) on some mechanical properties of unsaturated polyester based resin (UP) by adopting two different mixing methods. Different weight fractions (0, 0.5, 1, 1.5 and 2) wt% of nano (Al$_2$O$_3$) was used to reinforce the UP by using two mixing methods; shaker mixing method (SM) and mechanical mixing method (MM). The mechanical properties of both control and polymeric nanocomposites (compressive strength, and flexural strength) were evaluated. The results showed that higher enhancement in these characteristics were demonstrated for samples with 2 wt% and 1.5wt% of nano (Al$_2$O$_3$), these enhancements were 26.84% and 23.33%, when SM and MM were used, respectively. Regarding flexural strength, an increment of 93.22% was recorded for samples with 2 wt% for SM when compared with MM and pure UP samples. Finally, SEM micrographs approve that equal dispersion of nanoparticles progress the mechanical features of PNCs.

Copyright © 2017 International Energy and Environment Foundation - All rights reserved.

Keywords: Polymeric nanocomposites; Unsaturated polyester [UP]; Nano Alumina.

1. Introduction
A nanocomposite is a new class of materials frequently know as a multiphase solid material where one of the possess one, two or three dimensions of less than 100 nanometers (nm), or structures having nanoscale replication spaces between the phases dissimilar that make up the material [1]. Nanocomposites own superior mechanical and physical properties compared to conservative composites because of their nanoscale features maximize interfacial adhesion [2]. The outstanding strengthening of nanocomposites is chiefly attributed to the large interfacial area per unit volume or weight of the sparse phase [3]. The basic challenge in order to get suitable PNCs features to disseminate the nanoparticles like separate particles in the polymer matrix. In this method, industrially accessible particle powders fuse accumulation, where the nanoparticles stick firmly with each other as the result of the adhesive forces, the latter was increased within decreasing the particle size [4, 5]. Due to the attractive features of nanomaterials, many researchers concentrated the addition of different kinds of nanomaterial and used different mixing methods to polymer matrix composite such as thermoplastics and thermosets [6, 7]. Yinghong et. al., [8] investigated the influence of different types of nano TiO$_2$ (27 nm) at various weight portions (1, 2, 3, 4, 6) wt% on the mechanical features of unsaturated polyester sap by utilizing ultrasonic wave washer. The results showed that the extreme development of bending strength, tensile strength, impact strength and elongation at standoff were (173%, 47%, 60% and 48%) correspondingly, when...
nano TiO2 was added with (4 wt%), and the maximum improvement of tensile elasticity modulus and bending elasticity modulus were 22% and 12%, respectively at (3-4% wt).

Linj et al., [9] stated the effect of nanoparticles (nanoclay and Titanium dioxide) full of epoxy in percent from (2 to 10% vol) on the compression and wear behavior prepared by utilizing hand mixing. The experimental results showed that the compression strength, for both reinforced PNCs is much higher than those of pure epoxy matrix. The improvement of the wear and compression strength proportions were [(13.7% and 47%), (30% and 58.6%)] for the titanium dioxide and clay nanocomposites respectively. Amer et al., [10] studied the effect of nanocarbon black particles (N220) on some substantial mechanical characteristic of epoxy strengthened with it [carbon black nanoparticles]. The PNCs were intended with (1 to 10 wt. %) of carbon black nanoparticles by means of ultrasonic wave bath machine dispersion method. The results had indicated that the tensile strength, impact strength and flexural strength are enhanced by (24.02%, 6% and 17.3%) respectively at 2wt %. The hardness and compressive strength are improved by (12% and 44.4%) respectively at 4wt%

In present work, nano-Alumina was chosen to reinforce a polymeric matrix UP with different concentrations of nano Alumina using two different mixing methods. Nanoform Al2O3 powder are utilized entomb alia as part of scratch- and scrap area resistance in coatings, as grating atoms in slurriesfor cleaning semiconductors and exactness optical segments in covering of light and fluorescent tubes, as fire resistant, as fillers for polymers and tires., etc., [11]. The main aim of this work is to study the effect of two mixing methods. The outcomes will help to identify the practical mixing method that leads to highest enhancement in the characteristics.

2. Experimental work
2.1 Materials
The materials used to prepare the PNCs are unsaturated polyester resin with (2%wt), the hardener MEKP in addition to an accelerator cobalt naphthenate.

The nano-Alumina that used in this labor was bought from Sigma-Aldrich Chemical Ltd. located in USA, CAS Number: 1344-28-1. The properties of nano (Al2O3) are exposed in Table 1 as gave by the provider.

<table>
<thead>
<tr>
<th>Appearance (Color)</th>
<th>Purity</th>
<th>MP</th>
<th>Particle Size</th>
<th>Surface Area</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>White</td>
<td>99.5% Based on Trace Metals Analysis</td>
<td>2040 °C</td>
<td>&lt;50 nm (TEM)</td>
<td>&gt;40 m2/g (BET)</td>
<td>gamma phase</td>
</tr>
</tbody>
</table>

2.2 Samples preparation
2.2.1 Pure samples unsaturated polyester preparation
A computed measure of UP was mixed physically with 2% of the hardener for about 10 minutes at surrounding circumstances up to uniform blend was accomplished. The blend was filled the distinctive molds were produced using silicon elastic. The mixed was left in the form for a period of (24) hour in order to cure at encompassing conditions. The specimens then were removed from the mould and put inside oven for final treatment at 50°C and for 2 hours.

2.2.2 Polymeric nanocomposites specimens
Different percentages of nano (Al2O3) (0.5, 1, 1.5 and 2 wt%) were added to UP using two different mixing methods, details about these methods are shown below.

2.2.2.1 Shaker mixing method (SM)
A calculated amount of nano (Al2O3) was added to UP, which was placed inside a container, and mixed by hand. The whole container then was placed inside a shaker machine SM,Heidolph, No.549-59000-0.0,Germany as shown in Figure 1, for exactly (30) minute at maximum speed 354rpm to disperse the nanoparticles uniformly. Then the hardener (2%wt) was applied to the mixture within manually mixing, and then poured into mould, following the same procedure that mentioned in section (2.2.1).
2.2.2.2 Mechanical mixing method (MM)

Ascertained measure of nano (Al₂O₃) was included to UP, which was put in a compartment, and mixed by hand. The whole compartment then was set under mixer machine MM as indicated in Figure 2, for (30) minute at greatest velocity 7 rpm, MODLE NO.:GHM - 818. The mixer possess a most extreme energy with extreme energy 600w, to scatter the nano (Al₂O₃) homogeneously. Then, the hardener was included to the mix with a rate of (2%wt). The method for curing and post curing was accomplished likewise to that specify in portion (2.2.1).

Figure 1. Shaker machine.  
Figure 2. Mechanical mixing method (MM).

3. Mechanical tests

3.1 Compression test

An average of three samples for each mixing method and for each percentage of nano Alumina was tested. The compressive strength of the PNCs were carried out according to ASTM-D695 standard [12], with a speed proportion of about 1 mm/min at ambient temperature. Figure 3 shows the compression samples before and during test. The worldwide machine Model (TINIUS OISENH100KN) was used to obtain compression properties of different samples.

3.2 Bending test (flexural strength)

This test was performed according to ASTM D-790 standard [13] with a speed proportion of about 1.5 mm/min at ambient temperature.

Figure 4 shows the bending samples before and during test. The worldwide machine Model (TINIUS OISENH100KN) was used to obtain flexural strength.

4. Morphology test

4.1 Scanning electron microscope (SEM)

The morphology of the PNCs was revealed by using scanning electron microscope (SEM), the test conducted by means of VEGA3/ Tescan SEM device with vacuum atmosphere (2 kX - 11 kX) magnification. The sample used in the testing requires to cut to small parts to proper into the device, to achieve good conductivity for electric, totally samples are first sputtered with gold has been made from the surface along the edge; with keeping the working voltage at (5 Kv). The image results were analyzed to investigate the distribution of the inorganic filler nano (Al₂O₃) in polymer matrix.

Figure 3. The compression test: (a) the specimens before the inspection, (b) the sample during the inspection in the test machine.
5. Results and discussions

5.1 Compression test

The compressive strength of nano \((\text{Al}_2\text{O}_3)\) reinforced UP is shown in Figure 5. In this figure, the (SM) curve represents the compressive strength of pure and PNCs using shaker mixing method (SM). The incorporation of nano \((\text{Al}_2\text{O}_3)\) in the UP matrix results in an increase in compressive strength, reaches a maximum at 2wt\% concentration of nano \((\text{Al}_2\text{O}_3)\), with improvement in compression by (26.48\%). In general, the compressive strength was increased with increasing concentration of nano \((\text{Al}_2\text{O}_3)\).

In terms of (MM) curve, a uniformity enhancement in the compressive strength was observed when nano \((\text{Al}_2\text{O}_3)\) was added from (0.5 to 1.5) wt\%. Highest enhancement in the compressive strength (22.33\%) was obtained for samples with 1.5 wt\% of nano \((\text{Al}_2\text{O}_3)\) when compared with pure samples (0 wt\%). Reduction in compressive strength after 1.5 wt\% was observed. This may be attributed to the high concentrations of nano \((\text{Al}_2\text{O}_3)\) particles that might forms accumulation when using (MM) method.

5.2 Bending test (flexural strength)

The flexural strength of nano \((\text{Al}_2\text{O}_3)\) reinforced UP with and without nano \((\text{Al}_2\text{O}_3)\) is shown in Figure 6. The SM curve represents the F.S of control and PNC. The ultimate F.S at weight fraction 2wt\% of nano \((\text{Al}_2\text{O}_3)\) for UP, highest enhancement in the F.S (93.22\%) when compared with control samples. This may be attributed to the improved interfacial communication presented between the filler and matrix, which allowed the transition of stress from UP to nano \((\text{Al}_2\text{O}_3)\) there by improving the stiffness of the nano \((\text{Al}_2\text{O}_3)\), filled UP composite.

In term (MM), the flexural strength of nano \((\text{Al}_2\text{O}_3)\) was noticed uniformity increment. The highest value was reached at 1.5wt\% of nano \((\text{Al}_2\text{O}_3)\) with improvement (101\%). This may be attributed to preferable dispersion and well interfacial bonding. Then slightly decrease in F.S.
5.3 Scanning electron microscope (SEM)

Scanning electron microscopy (SEM) was gave important information on the microstructures of the samples. SEM images show the appearance of control UP and PNCs. Figure (7) represents the SEM image of the sample containing (100%) UP, it can be seen that brittle fracture due to natural of the UP as a matrix, and some impurities were detected.

While Figure 8 show the SEM images of the samples that contain 2wt% of nano (Al₂O₃) and 1.5wt%of nano (Al₂O₃), when using SM and MM mixing methods, respectively. There was observed clearly that the small particles uniformly dispersed in the UP matrix and were well connected into other. As might be seen nanoparticles aggregates are observed as white spots with a wide particle size distribution. When using MM mixing method. There was exhibited agglomeration of nanoparticles in the composites with the appearance of pigmentation. Increased filler content attributed to excessive accumulations and accordingly resulted in the creation small cavities noticed at the fracture surface. The rough fracture surface is due to the presence of nanoparticles, which makes the fracture path more tortuous. Addition of nanoparticles into UP matrix improved the characteristics of the PNCs and hence enhanced the strength of it, and the interfacial bonding between matrix and nanoparticles was very good. At the same time, ductility behavior totally reduced.

Figure 6. Influence of nanoparticles (wt %) on F.S of [UP] matrix by various mixing processes.

Figure 7. Micrographs of UP matrix.

Figure 8. (a) Micrographs of UP+2wt%of Al₂O₃ by SM method, (b) Micrographs of UP+1.5wt%of Al₂O₃ by MM method.
6. Conclusions
The main conclusions for this work can be summarized as follows:

1. From the addition of nano (Al$_2$O$_3$) at different concentrations (0, 0.5, 1, 1.5 and 2) %wt by using two mixing methods (SM and MM), it can be noticed that the mechanical properties (compressive strength and flexural strength) of UP increased with adding different concentrations of nano(Al$_2$O$_3$), better enhancements were found for the samples modified with 2 wt% and 1.5 wt% of nano(Al$_2$O$_3$) for the (SM and MM) methods, respectively.

2. SEM results confirmed that the nanoparticles can be used to modify the moderate fiber strengthened composite materials. Thus, SEM micrographs approve that uniform expansion and best loading of nanoparticles develop the mechanical properties of composites.

3. In general, it can be notice that mixing using (SM) was more practical and provided more homogeneous mixture than (MM) method.

References