Improving The Properties of a Polyester-Based Composite Using Nanoparticle Fillers

Zainab K. Younus\textsuperscript{a}\textsuperscript{*}, Laith K. Abbas\textsuperscript{b}, Abbas K. Hussein\textsuperscript{c}

\textsuperscript{a}\textsuperscript{*}Materials Engineering Dept., University of Technology-Iraq, Alsina’a street, 10066 Baghdad, Iraq.
\textsuperscript{b}Nanotechnology and Advanced Materials Research Centre., University of Technology-Iraq, Alsina’a street, 10066 Baghdad, Iraq.
\textsuperscript{c}Corresponding author Email: mae.19.28@grad.uotechnology.edu.iq

\textbf{HIGHLIGHTS}

- The tensile test revealed that the latest mix has the better modulus of elasticity at (3\% wt).
- This nanocomposite material showed very good roughness, hardness, flexural, and impact resistance.
- The good dispersion of the nanoparticles had a great role in having this material good properties as aggregation may lead to a decrease in the quality of the properties.

\textbf{ABSTRACT}

In this work, unsaturated polyester, resin, matrix reinforced, with fiberglass mat chopped were developed using TiO\textsubscript{2} and SiO\textsubscript{2} Nanoparticles as filler materials. The individual and combined effects of nanoparticles on the nanocomposite and material properties were examined. An improvement in Young’s modulus (39.3\%) using combined weight of TiO\textsubscript{2} + SiO\textsubscript{2} was observed to be 3\%. Examined nanoparticles have shown normal distribution in grain size indicated to be within standard limit, therefore boosting nanocomposite material to have excellent hardness, roughness, impact, and flexural resistance. DSC was characterized by transitions in this material. SEM and EDS techniques were used to confirm the formation of TiO\textsubscript{2} and SiO\textsubscript{2}. The nanocomposites can be used in light-shielding, applications as well as in the industrial sectors.

\textbf{ARTICLE INFO}

Handling editor: Jawad K. Oleiwi

Keywords: Nanoparticles
Properties
Polymer nanocomposites
Unsaturated polyester resin
SiO\textsubscript{2}
TiO\textsubscript{2}

1. Introduction

Composite materials can be characterized as heterogeneous materials comprised of at least two solid phases which are firmly interconnected. Specifically, nanocomposite materials are structured with nanoscale materials embedded in a ceramic, or polymer matrix at scales of 0-D, 1-D, and, 2-D.

The properties of nanocomposites depend on different factors, particularly grid material which can show measurements of the Nanoscale, the level of scattering, size, stacking, shape, direction of the Nanoscale second phase and interactions between the subsequent phase and the lattice. Nanomaterials have an ordinary grain size of under 100nm, while micromaterials are recognized by grain size of about 500nm. The unsaturated polyester resins are recently used in different applications like manufacturing the thermosetting polymers vital. Structurally, this resin fortiﬁes with different ﬁbers and ﬁllers to improve mechanical and physical properties. Additionally, the chemical properties of this polymer rely upon the cross-linking agents, initiators, diced, and other additives. Many researchers have investigated this ﬁeld over the last few years, M.T. Rahman et al., considered unsaturated polyester resin UPR, matrix-based polymer nanocomposites which were made by blending sol-gel Fe\textsubscript{2}O\textsubscript{3}, TiO\textsubscript{2} and co-precipitation synthesized NiFe\textsubscript{2}O\textsubscript{4} nanoparticles. The mechanical, electrical, and optical properties of the fabricated polymer, nanocomposites were estimated. Their results indicated that both tensile and strength properties have increased in NiFe\textsubscript{2}O\textsubscript{4} UPR and Fe\textsubscript{3}O\textsubscript{4}+TiO\textsubscript{2}+NiFe\textsubscript{2}O\textsubscript{4}/UPR. Nanocomposite and Young modulus improved in NiFe2O4/UPR composite only [1]. Sudad I. Younis et. al. have examined the effect of Nano, SiO2, particles on wear demeanor test and other
properties like ultimate tensile, strength, impact, strength, fracture, and hardness shore D of specimen’s composite material. The best results were observed from unsaturated polyester resin matrix reinforcement with 4% weight fraction glass fiber, and 1%, 3% and 5% weight fraction of Nano SiO$_2$ particle. The results showed that the specimen UP+4% woven glass fiber+5% nano, SiO$_2$ gives preferable mechanical properties [2]. Suresh J.S et al. have investigated the effect of filler material like titanium oxide TiO$_2$, and silicon, carbide SiC, particulates on mechanical properties of Glass fiber. They found that the mechanical, properties like tensile, strength, flexural, strength, impact, strength, and hardness of the glass-reinforced composites are modified with the joining, of fillers [3]. T. Ahmed. et al. Examined SiO$_2$ Nanoparticles and glass fibers, in unsaturated, polyester. Mechanical tests like tensile, impact and micro-hardness were implemented on the obtained polymer hybrid, composites. The results of hybrid polymer matrix composites showed good development in mechanical properties [4]. Sudirman et al. Investigated, the effects of silica, as filler. The investigation appeared that the mechanical properties like tensile, strength, elongation, and Young’s modulus, increase with the addendum of SiO$_2$ nanoparticle up to 1.0 wt%, and then decrease, over 1.0 wt% [5]. In this study, the main objective is to improve the properties of a polyester-based composite using nanoparticle fillers. The (Unsaturated Polyester, Resin) (UPR), is used as the matrix which was tested alone and then mixed with other materials to select the one that has better mechanical properties. The unsaturated polyester resin was reinforced with mat glass fiber. The Titanium Dioxide TiO$_2$ and Silicon Dioxide SiO$_2$ Nanoparticles are utilized as fillers in the planning of test polymer composite materials provided by Hongwu International, Group (Ltd). The purity of TiO$_2$ and SiO$_2$ are (99.9% and 99.8%) respectively. While the particle size of TiO$_2$ and SiO$_2$ are 30-502.5 nm [6] and 20-302.5 nm [7] respectively.

2. Materials and Methods

2.1 Materials

Unsaturated Polyester Resin, glass fiber, methyl ethyl, ketone per Oxide, TiO$_2$ and nanoparticles SiO$_2$ Nanoparticles were used as filler.

2.2 Methods

in order to synthesize composite materials, several specimens were prepared with different materials as follows:

1. Pure polyester: Unsaturated polyester resin which is a clear liquid had been used for casting. It has a density equal to 1.15gm/cm$^3$ and a viscosity of 450 mPa.s. It is elastic with medium reactivity. The most common usage is the production of marbles by adding up filling materials in the proportion of 70-80%. Furthermore, it can be safely used in the production of cuvettes, sinks, kitchen counters, table edges-legs, buttons, ornaments, etc. [8]. As UPR is viscous and liquid, 20 wt% methyl, ethyl ketone peroxide, (MEKP) [9] it was added as a curing agent hardener and blended very well at ambient temperature.

2. UPR reinforced with mat glass fiber: The mat glass fiber (chopped) with a density equal to 2g/cm$^3$ was added to the mix of (UPR) and hardener where the mold should be covered with wax matrix to avoid abrasive and insure flattening. Then roller and brush were used to apply pressure for distributing the mix resin and hardener through the fiber then another layer of the mix was added on the fiber then for ensuring air removal and wet out, a torch is passed near the mixed surface.

3. UPR reinforced with glass fiber (mat) with TiO$_2$ Nanoparticles as filler: Unsaturated polyester resin-based polymer nanocomposites were manufactured by using 1 wt%, 2 wt%, and 3 wt% of Titanium Dioxide Nanoparticles respectively. Where the nanoparticles were added to the UPR in a plastic vessel and the mix were blended using a blending device mixer drill with an angular velocity of 750 cycle/minute, then the hardener was added to the mix and blended in the same blending device for 5 minutes to make the mixture ready for casting and the reinforcing fiber was added in the same way that previously mentioned.

4. UPR reinforced with mat glass fiber with SiO$_2$ Nanoparticles as filler: The UPR based polymer nanocomposites were fabricated by using 1 wt%, 2 wt%, and 3 wt% of Silicon Dioxide and the method of preparation was the same as the preview method with using SiO$_2$ instead of TiO$_2$.

5. UPR reinforced with glass fiber (mat) and both TiO$_2$ and SiO$_2$ Nanoparticles are used as filler and the reinforcing fiber was added in the same way that previously mentioned. UPR based polymer nanocomposites were fabricated by using 1wt%, 2 wt%, and 3 wt% of both Titanium Dioxide and Silicon Dioxide nanoparticles respectively, and in the same method of mixing used before mixing drill. After completing blending the mix was added to the mix and adding hardener, fiber was added in the same way of fabricating fiberglass/polyester. All the specimens were treated at a temperature 27°C and for 24 hr period of time.

2.3 Tensile Test

Tensile tests were used to determine the modulus of elasticity and the tensile strength for each specimen. All test specimens were cast in a specially designated mold made from steel as shown in Figure 1-(A) according to the standard geometry of ASTM D 638 [10, 11] as shown in Figure 1-(B) and its dimensions in Table1.

Thirty-three tensile test specimens were prepared for the materials previously mentioned which are three tensile test specimens of UPR as shown in Figure 2-(A), three of UPR+ chopped glass fiber as shown in Figure 2-(B), three of UPR+ glass fiber+ 1% TiO$_2$ as shown in Figure 2-(C), three of UPR+ glass fiber+ 2% TiO$_2$ as shown in Figure 2-(D), three of UPR+ glass fiber+ 3% TiO$_2$ as shown in Figure 2-(E), three of UPR+ glass fiber+ 1% SiO$_2$ as shown in Figure 2-(F), three of UPR+ glass fiber+ 2% SiO$_2$ as shown in Figure 2-(G), Three of UPR+ glass fiber+ 3% SiO$_2$ as shown in Figure 2-(H), three of mix include UPR+ glass fiber+ 3% SiO$_2$ as shown in Figure 2-(I), three of mix include UPR+ glass fiber+ 2% TiO$_2$+
SiO₂ as shown in Figure 2-(J), and three of mix include UPR+ glass fiber+ 3% TiO₂+ SiO₂ as shown in Figure 2-(K). The tensile test specimen is mounted vertically in a servo-hydraulic testing machine, and is hydraulically retracted using stroke control with large steel grips. The specimen which is a mix of (UPR+ Fiber+ 3% TiO₂+ SiO₂ revealed the best behavior where it has the highest modulus of elasticity at 1.5 wt% TiO₂+ 1.5 wt% SiO₂ and according to that, It had been chosen to be studied in detail, in addition to the tensile test, other tests have been done for the chosen material to get a sufficient idea of its properties where these tests are:

### 2.4 Flexural Test

The flexural test was carried out at room temperature by utilizing the all-inclusive test instrument that equivalent utilizing in the ductile test relied on a three-point, bending test technique.

![Figure 1](image1.png)

**Figure 1:** (A) Tensile test specimens mold (B) Standard geometry of ASTM (D 638)

![Figure 2](image2.png)

**Figure 2:** Tensile test specimens for (A) UPR, (B) UPR+ chopped glass fiber, (C) UPR+ glass fiber+ 1% TiO₂, (D) UPR+ glass fiber+ 2% TiO₂, (E) UPR+ glass fiber+ 3% TiO₂, (F) UPR+ glass fiber+ 1% SiO₂, (G) UPR+ 2% SiO₂, (H) UPR+ glass fiber+ 3% SiO₂, (I) mix include UPR+ glass fiber+ 1% TiO₂+ SiO₂, (J) mix include UPR+ glass fiber+ 2% TiO₂+ SiO₂, and (K) mix include UPR+ glass fiber+ 3% TiO₂+ SiO₂

<table>
<thead>
<tr>
<th>Symbols</th>
<th>Description</th>
<th>Dimension (mm)</th>
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<tbody>
<tr>
<td>W</td>
<td>Width of narrow section</td>
<td>12.5</td>
</tr>
<tr>
<td>L</td>
<td>Length of narrow section</td>
<td>75</td>
</tr>
<tr>
<td>WO</td>
<td>Width overall</td>
<td>20</td>
</tr>
<tr>
<td>LO</td>
<td>Length overall</td>
<td>165</td>
</tr>
<tr>
<td>G</td>
<td>Gage length</td>
<td>50</td>
</tr>
<tr>
<td>D</td>
<td>Distance between grips</td>
<td>115</td>
</tr>
<tr>
<td>R</td>
<td>Radius of fillet</td>
<td>20</td>
</tr>
<tr>
<td>R2</td>
<td>Radius of fillet</td>
<td>6.5</td>
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<tr>
<td>T</td>
<td>Thickness</td>
<td>3 or under</td>
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</table>
In this strategy the perpendicular load continuously at the center of the Nanocomposite samples at strain rate, 2mm/min. until a fracture happens to acquire the (load-displacement) curvature for the composite specimen. The flexural modulus and flexural strength are the properties acquired from this test for composite example was proposed in this examination as per the global norm ASTM D-790, [11, 13] as in the schematic shown in Figure 3A. Figure 3B shows the experimental flexural test specimen.

2.5 The Impact Test

Impact testing is carried out according to international standards (ISO-180), [11, 12, 14] by utilizing (Izod) Impact test machine type is (XJU)series pendulum (Izod/Charpy) impact testing machine. For (Izod test): the samples were clipped toward one side and held upward cantilevered. beam. Impact test samples may be with or without, snick. Figure 4. (A, B, C) shows the standard sample of the impact test.

2.6 Hardness Test

Hardness is the most popular calculated property of the surface. The shore hardness is measured with an apparatus recognized as a “Durometer hardness”. The value of hardness is specified by the penetration of the Durometer, indenter foot into the Specimen. Due to the versatility of Rubbers, and plastics, the indentation, reading may change, over time so the indentation, time is sometimes recorded along with the hardness number.

Shore (D) Hardness is a standardized- test consisting in measuring, the depth of penetration of a specific, indenter. Test methods used to measure Shore (D) Hardness are ASTM D2240 and (ISO 868) [15, 16].

2.7 Roughness Test

Surface roughness is characterized as the anomalies which are inherent in the production process (for example, cutting instrument or abrasive grit). Since the individual, roughness inconsistencies are very, small to see with the bare, eye and a roughness estimating, the instrument is required. A little pointer is drawn across the surface at a consistent, speed for a set distance. An electrical, signal is gotten and intensified to create a much-amplified vertical amplification. This sign might be shown, on both chart and screen, outputs, together, with mathematical qualities that give details of the surface as shown in Figure 5.

2.8 Density calculation

Density is the mass per unit volume of a material. The total density (ρ) was determined by taking a piece of the laminate and measuring its volume and mass as shown in Figure 6.

2.9 Water Absorption Test

Different polymeric materials are powerless to water retention during their life exposures which cause dimensional insecurity, as per that this test is essential to do. The test of water absorption is achieved according to (ASTM D570) [17, 18]. After putting the specimen in distilled water for 24 hr, The water absorption can be calculated according to the following equation [17]:

\[
\text{Water absorption percentage} = \left(\frac{W_s - W_d}{W_d}\right) \times 100
\]

Where: Wd: Mass of the sample before immersion dry.
Ws: after immersion for 24 hr. in distilled water.

\[\text{(1)}\]
To arrive at the correct temperature, they will have acquired sufficient energy to move into exceptionally high states. This drop appears in the heat flow as a major plunge in the plot of heat flow versus temperature. Energy Dispersive Spectroscopy (EDS) microanalysis system collects the X-rays, sorts and plots them by energy, and automatically, identifies and labels the elements responsible for the peaks in this energy distribution.

3. Results and Discussion

3.1 Tensile test

The mechanical properties for all specimens obtained from the tensile test are shown in Table 2 below:

From the results above reinforcing UPR with fiberglass could highly increase the modulus of elasticity and the tensile strength with percentage reaches to 47% and 57.96% respectively. On the other hand, adding fillers of nanomaterials like TiO₂ and SiO₂ showed better modulus of elasticity at a weight ratio of 1% and 2% respectively, while the 3wt % revealed decreasing...
in modulus of elasticity value with percentage reaches to 13.9% and 6.86% in TiO$_2$ and SiO$_2$ respectively where this decrease could happen due to some regions of aggregation.

3.2 Flexural test

Figure 9 revealed the load-extension curve obtained from the flexural test. According to the information obtained from this curve, the magnitude of the flexural strength, flexural modulus, and maximum shear stress are gained as shown in Table 3.

3.3 Impact test

The impact results represent the impact strength for the composite specimen prepared in this study which is equal to 5.5 KJ/m$^2$ and it has broken, at an impact energy of 2.2 J of the pendulum, and impact velocity of 3.5 m/s.

3.4 Hardness test

The hardness esteem was dictated by the infiltration, of the Durometer, indenter foot into the example. Shore hardness measures are dimensionless. It goes between 0 and 100. The higher, number represents the harder, material. Hardness Shore-D =86 which is considered extra hard due to the durometer shore hardness scale.

![Figure 7: Dispersion device](image1)

![Figure 8: Grain size analyzer](image2)

Table 2: Experimental mechanical properties of tensile test

<table>
<thead>
<tr>
<th>Material</th>
<th>Mechanical properties</th>
<th>1%</th>
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<th>4%</th>
<th>5%</th>
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<th>11%</th>
<th>12%</th>
<th>13%</th>
<th>14%</th>
<th>15%</th>
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<tbody>
<tr>
<td>Mix of UPR+ Fiber+ TiO2+ SiO2</td>
<td>Young modulus (Mpa)</td>
<td>66.2</td>
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<tr>
<td>Mix of UPR+ Fiber+ TiO2</td>
<td>Tensile strength (Mpa)</td>
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<td>Mix of UPR+ Fiber+ SiO2</td>
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<tr>
<td>Pure UPR</td>
<td>Young modulus (Mpa)</td>
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<td>Pure UPR</td>
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<tr>
<td>Pure UPR + fiber</td>
<td>Young modulus (Mpa)</td>
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<tr>
<td>Pure UPR + fiber</td>
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</table>
3.5 Roughness test

The examination of roughness was carried out in the laboratories of the Department of Materials Engineering at the University of Technology, where three readings were taken on the sample surface and these three readings are 2.316, 2.315 and 2.316 nm respectively, where it’s clear that the suitable value is equal to 2.316 nm.

3.6 Density calculation

According to the dimension of the specimen, and according to the measured mass, the density is obtained is \( \rho = 1.547 \ \text{g/cm}^3 \).

3.7 Water absorption measurement

For measuring the water absorption, after measuring the mass of the sample before immersion dry which is equal to 6.767 and the mass of the sample after immersion for 24 hr in distilled water which is equal to 6.825, the Water absorption could be obtained as follows: Water absorption percentage \( \frac{6.825 - 6.767}{6.767} \times 100 = 0.8571 \% \).

3.8 Antiseptic resistance test

According to the antiseptic resistance test the sample was placed in the substance of propanol with a density of 0.804 g/mL for 24 hours. After the sample was taken out, it was found to be completely resistant to sterilization materials and no effect occurred in it.

3.9 Grain size test

From the grain size test, it became clear that the granular size for TiO\(_2\) is 34.3 nm as revealed from the grain size summary in figure 10. On the other hand, the grain size test summary shown in figure 11 for SiO\(_2\) revealed that the granular size is 28.8nm, where it is within the limits of the certificate of origin previously mentioned.

3.10 DSC test

DSC test gives the curve related to the heat flow to the temperature as shown in Figure 12. The step appears in the plot when the polymer was heated, past its glass transition temperature at 70°C. Then it showed a major, plunge when the polymer reached its crystallization temperature at 159°C. Then finally it revealed a big peak when the polymer, reached its melting temperature at 184°C.

<table>
<thead>
<tr>
<th>Table 3: Flexural test results</th>
<th>magnitude</th>
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</thead>
<tbody>
<tr>
<td>Flexural strength (MPa)</td>
<td>19.212</td>
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<tr>
<td>Flexural modulus (MPa)</td>
<td>607.15</td>
</tr>
<tr>
<td>Maximum shear stress (MPa)</td>
<td>1.729</td>
</tr>
</tbody>
</table>

Figure 9: Load-extension curve of flexural test

![Figure 9: Load-extension curve of flexural test](image)

Figure 10: grain size test summary for (TiO\(_2\))

![Figure 10: grain size test summary for (TiO\(_2\))](image)

Figure 11: grain size test summary for (SiO\(_2\))

![Figure 11: grain size test summary for (SiO\(_2\))](image)
3.11 SEM test

Figure 13 shows the SEM test. This figure shows the presence of glass fibers embedded in polymer matrix and these fibers provide strengthening mechanism to polymer in all directions. SEM micrographs were done to the mix of nanocomposite samples at different magnification 700x, 1600x, 2200x and 2400x were recorded in Figure 13 A, B, C and D respectively. As shown from this figure, in most cases, the system appears to have a semi-continuous morphology, observed for different magnifications. The fibers were buried inside the polymeric matrix material, and that most of the fiber became part of the matrix material which seems to indicate better interfacial adhesion between fibers materials and components of composite materials.

The filler materials Nanoparticles appeared as the white molecule, where the presence, of bigger particles, is because of the molecule, accumulation. Huge district of conglomeration could be feeble, communication between the nanoparticles and the polymer, phase causes a few voids, on the network surface. This condition altogether diminished the mechanical properties of the composite, henceforth to stay away from that, great scattering in low convergence of nanoparticles, particularly, silica in the UPR framework plays a fundamental, job to give all-around shaped, interpenetrating, network IPN structures.

3.12 EDS test

To affirm the arrangement SiO$_2$-TiO$_2$, EDS analysis was executed successfully. During the EDS measurement different areas were focused and the corresponding peaks are shown in Figure 14. Both SiO$_2$ and TiO$_2$ can be seen in the synthesized composite nanostructure in the EDS spectrum. In this spectrum, the weight composition of Ti, Si, and O were 4.17, 4.86, and 90.97 respectively. Details of the EDS spectra of the nanocomposite worth, measured in atomic and weight % are listed in Table 4.
4. Conclusions

In this study, UPR, UPR reinforced with fiberglass mat chopped, UPR reinforced with fiberglass and SiO$_2$ or TiO$_2$ with 1%, 2%, and 3% weight percentage used as a nanoparticles filler, and UPR reinforced with fiberglass and SiO$_2$ and TiO$_2$ with 1%, 2%, and 3% weight percentage used as a nanoparticles filler nanocomposites were prepared by casting technique. The tensile test revealed that the latest mix has the better modulus of elasticity at 3% wt and according to that other mechanical properties were gained and other tests were done to examine the resistance and the structure of this nanocomposite material. This nanocomposite material showed very good roughness, hardness, flexural, and impact resistance. In addition, it showed not affected by sterilization materials, up to 100%. On the other hand, the good dispersion of the nanoparticles had a great role in having this material good properties as aggregation may lead to a decrease in the quality of the properties. On the premise, of the acquired, result, the created, nanocomposites can be utilized to deliver differently, items in (medical, structures development, auto, and so forth areas with better, properties. These nanocomposites can likewise be utilized as light safeguarding, applications, and covering material.

Acknowledgment

Acknowledgments may be directed to individuals or institutions that have contributed to the research or a government agency.

Author contribution

All authors contributed equally to this work.

Funding

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

References


