Evaluation of Mechanical Properties for Epoxy reinforced with palm oil /Zinc oxide composites

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Abstract
In this research, the effect of reinforcing epoxy resin composites with a filler derived from chopped agriculture waste from oil palm (OP). Epoxy/OP composites were formed by dispersing (1, 3, 5, and 10 wt%) OP filler using a high-speed mechanical stirrer utilizing a hand lay-up method. The effect of adding zinc oxide (ZnO) nanoparticles, with an average size of 10-30 nm, with different wt% (1,2,3, and 5wt%) to the epoxy/oil palm composite, on the behavior of an epoxy/oil palm composite was studied with different ratios (1,2,3, and 5wt%) and an average size of 10-30 nm. Fourier Transform Infrared (FTIR) spectrometry and mechanical properties (tensile, impact, hardness, and wear rate) were used to examine the composites. The FTIR results show a strong interaction between ZnO and oil palm fiber and epoxy resin. Tensile strength was reduced from 22.78 MPa to 19.03 MPa for the epoxy/OP composite as the wt% of OP was increased but increased to 29.224MPa for epoxy /oil palm / 5% ZnO samples. Young modulus increased from 1.9 MPa to 4.3 MPa while elongation decreased (9.6 to 6.8 %) with the increase of wt% OP and ZnO. The impact and hardness increased for all composites between (6.94 - 10.8 KJ/m²) and between (80.8- 84.55 KJ/m²) respectively. Also, wear resistance of the epoxy/OP and epoxy/OP/ZnO samples increased with the increase of wt% OP and ZnO. This studied in order to provide a new step in the utilization of green nanoparticle fillers for sustainable and renewable structural products for biodegradability.

1. Introduction
Biodegradable polymer and resins are now gaining popularity in both industrial and research applications. When compared to traditional petroleum-based polymers, such polymers have several benefits. They are biodegradable and may be obtained at a lesser cost from renewable resources [1]. Natural fiber derived from diverse biodegradable natural resources has shown promise as a reinforcing material when compared to organic fiber reinforcement matrix composites. Natural fiber reinforcement has improved due to several advantages of low density, low cost, biocompatible, and superior insulating and thermal resistance [2]. Recently, the qualities of stiffness, elasticity, impact strength, and modulus of elasticity have been attained in applications of industrialized natural fiber reinforcements, such as the use of a flax-sisal fiber mat epoxy composite matrix to create Mercedes E-Class door panels [3].
Epoxy resins are thermosetting engineered synthetic polymers utilized in electrical encapsulating, heavy machinery, dielectric materials, mixing and composites[4]. However, the mechanical qualities of cured epoxy resin (modulus, hardness, and toughness) are insufficient for many end-use applications. These limits stem mostly from delamination, poor impact resistance, intrinsic brittleness, and fracture hardness behavior [5]. As a result, it is necessary to change the resins to enhance its physico - mechanical qualities by inducing diverse fracture modes using just 1% to 10% additions (in mass to resin). Modified epoxies are now being researched in order to broaden their uses in civilian infrastructure and transportation [6]. Epoxy resin as a matrix material is excellent for natural fibers as a reinforcement composite because of its strong adhesive capabilities, superior chemical and heat resistance, superb electrical insulator properties, and exceptional mechanical performance [7].

Zinc oxide (ZnO) nanoparticles drew the attention of all technological features because of their exceptional optimal and electrical characteristics with ZnO nanoparticles by direct mixing. Nano particles are high-performance components known for their high thermal and mechanical stability at room temperature, and they have been used as fillers to improve the properties of polymer materials [8].

Saba et al. [9] used epoxy-based polymer nanocomposites prepared by dispersing 1, 3, and 5 wt. % nano OPEFB filler using a high-speed mechanical stirrer through hand lay-up technique. The mechanical properties (tensile and impact) qualities, as well as morphological features, were examined and compared. The scanning field emission scanning electron microscope (SEM) and electron microscopy (TEM) consider the dispersion of OPEFB in the epoxy matrix, there is a nano-filler. The nano composites' tensile and impact characteristics improved by up to 3% but decreased by more than 3%. The Overall mechanical qualities attained maximum loading levels of 3%, due to the improvement of pressure transfer due to the homogeneous scattering of the (OPEFB) nanofiller within the epoxy matrix.

Ghazilan et al. [10] evaluated the performance of oil palm empty fruit bunch fiber reinforced epoxy composites as a substitute for synthetic or traditional reinforced compounds. It was demonstrated that tensile strength, modulus of elasticity, and Poisson's ratio were lower compared to a normal epoxy structure with reduced performance of 38% for elasticity modulus and 61% for tensile resistance.

Faizi et al. [11] used oil palm empty fruit bunch (OPEFB) composed of single fibers and fortified for untreated and treated fibers with four different methods of alkaline treatments: NaOH (3%), silane (2%), mixed NaOH (3%) + silane (2%), and multistage NaOH (3%) - silane (2%) and the performance of both methods were compared. OPEFB composites were prepared at 90:10, 80:20, 70:30 and 60:40 epoxy: fiber parts. The results showed that the treated fiber composite had better performance. The ultimate tensile strength was increased for the 90:10 epoxy: fiber parts by 145.3%. The highest Young's modulus was increased by about 166.7% for 70:30 parts. The highest hardness was increased by 389.5% for the 30:70 fractions. The treated fibers provided a better crosslinking mechanism between the matrix and the reinforced fibers vehicles.

The aim of this study is to prove that the biomaterial derived from oil palm wastes incorporated in polymeric materials can improve biodegradability; minimize pollution, and lower costs.
2. Experimental work

2.1 Materials and Method

The materials employed in the preparation of the samples are: the polymeric base material epoxy (made in Egypt) and a transparent liquid at room temperature that mixes well with the hardener, which is one of the types of thermosetting polymers. Also, a natural material was used, which is palm oil fruit fibers obtained from its famous homeland in Malaysia after chemical treatment. The oil Palm (OP) fibers contain approximately 50% saturated fatty acids, 44% palmitic acid (C16:0), 5% stearic acid (C18:0), and trace amounts of myristic acid (C14:0). The unsaturated fatty acids are approximately 40% oleic acid (C18:1), 10% polyunsaturated linoleic acid (C18:2) and linolenic acid (C18:3). Zinc oxide nanopowder (ZnO, 99.8%, white to light yellow) was added.

2.2 Sample Preparation

The OP fillers was dried at 60°C for 12h before being stored in a desiccator to cool and to avoid humidity until it was utilized in preparation. OP filler of (1, 3, 5, and 10 wt%) was added to the epoxy resin using a high speed continuous stirrer, using Eq.(1): 

\[
\text{wt}\% = \frac{w_m}{(w_m+w_p)} \times 100\% 
\]  

where \(w_m\) and \(w_p\) are the weight of the mixture and the weight for the polymer, respectively.

The epoxy and hardener stoichiometric ratio (2:1) was maintained. The mixture was physically mixed at room temperature for at least 20min. The liquid was then put into a stainless-steel mold and allowed to cure at room temperature (30°C) for 24 hours. To simplify the extraction of the composite samples, a silicon spray release agent was utilized in the mold. The time taken to complete the sample preparation was an hour to completely mix the materials and make them cohesive. It also took another 24 hours to be ready for the testing.

Epoxy/OP/ZnO nanocomposites were prepared (ZnO) nanoparticles, with average size of 10-30nm, were added to epoxy/OP composite at different mass ratios (1, 2, 3, 5wt%) at the percent of 5% from (epoxy/OP) which was employed as an alcohol dispersion. The mixture was then mixed at a shear rate and mixing time of 2000rpm and 20min, respectively, the samples were poured into molds and prepared for mechanical testing, as shown in Fig.1.

2.3 Fourier Transforms Infrared Spectroscopy (FTIR)

The Fourier transform infrared spectroscopy (FTIR), at a range of 4000-400cm\(^{-1}\) spectra were analyzed, in order to analyze the chemical bonding of the modified nanoparticles and assure their effectiveness.

2.4 Mechanical testing

2.4.1 Tensile strength

Tensile strength, elongation, and modulus were measured at break of the epoxy/OP and epoxy/OP/ZnO composites. Universal Instron testing equipment was used to measure the nano composites. The composite samples were cut in standard sizes according to ASTM D- 638 [12]. A typical head displacement of 5 mm/min was
used, and the averaged tensile strength, modulus, and elongation at the point of breakage of the sample were recorded.

![Figure 1: Samples of tensile test.](image)

The sample (of known dimensions, including length and cross-sectional area) was inserted between "knobs" that hold the material in situ. Load was imparted on the sample that has been seized at one end and clamped at the other. The changes in the length of the sample were measured as the load was increased.

### 2.4.2 Impact test

This method is for calculating the amount of energy that causes the sample surface to fail under certain conditions, the free-fall effect of a stock method is to determine the minimum height from which the collider falls, at an energy rate of (2J), which causes mechanical damage to the sample surface from a height of one meter. The dimensions of the sample should have a thickness of (5 mm), length (55 mm), and width (10 mm) according to ISO-179 [13]. It is a standard high strain-rate test that assesses how much energy material absorbs during fracture. This absorbed energy is a measure of a material's notch toughness and may be used to analyze the temperature-dependent ductile-brittle transition.

### 2.4.3 Hardness test

A durometer (Shore D) was used to test the hardness of the samples. A hardness test is normally carried out by pressing a precisely dimensioned and loaded item (indenter) into the material surface being tested. The hardness of a material is determined by measuring the depth of indenter penetration or the size of the imprint made by an indenter. A tensile test was carried out on all types of samples made which were flat samples. The procedure was carried out at an accidental speed, the thickness of the sample should not be less than 5 mm.
2.4.4 Wear test

Wear testing were performed in accordance with ASTM G-99 [14] utilizing a wear and friction monitor test setup of the pin-on-disc kind (provided by DUCOM). The counter body is a reinforced grind steel disc of (269HB) toughness with a track of 6cm radius and a speed of 357rpm. The specimen, the composite, is maintained still, and the disc rotates while a normal weight is provided through a lever system. Under standard loads of 500N, a series of tests were performed with three sliding velocities of (6cm/sec). A precise electronic device is used to detect material loss from the composite surface and the wear rate can be estimated by the following relation:

\[
WR = \frac{\Delta w}{S_D}
\]  

(2)

where: WR: Wear rate (g/cm), \(\Delta W\): Weight difference (g) and \(S_D\) is the sliding distance which is equal to:

\[
S_D = 2\pi R t
\]  

(3)

R: is the radius of the rotating center, t: is the test time (sec), and n: is the number of cycles per time (375 rpm).

3. Results and Discussion

Fig.2 depicts the band assignment for the pure resins and indicates that the C-O distortion band is located at 929 cm\(^{-1}\). At 3059 cm\(^{-1}\), C-H stretching of the terminal oxidizing group is seen. The band at 3390 cm\(^{-1}\) is attributed to hydroxyl group O-H stretching. There are other bands at 1029-1107 cm\(^{-1}\) that correlate to the ether linkage. The peaks at 1504–1643 cm\(^{-1}\) correspond to the primary amine (NH), peaks corresponding to 1454 cm\(^{-1}\) and 1238 cm\(^{-1}\) shows C-C and C-O stretching, 1029–1107 cm\(^{-1}\) correspond to C-N group and 821 cm\(^{-1}\) is related to the aromatic ring of the epoxy resin. The fundamental C-H stretching is centered at 3730 cm\(^{-1}\) in epoxy.

There are no absorption peaks within the annotation of Fig.2. We talked about the range of bond distortion and the bonding of epoxy resin with molecule materials.

![Figure 2: FTIR of pure epoxy resin.](image-url)
The stretching vibration of C-O-C groups at 1257.59 cm\(^{-1}\) is clearly visible in the FTIR spectra of epoxy and epoxy/OP (Fig. 3). In addition to the other distinctive band of the resin rings at the wavenumber between 879.54 – 810.10 cm\(^{-1}\). The terminal methyl groups (\(\text{CH}_3\)) of the triglyceride chains exhibited a significant C-H stretching band at 2920.23 cm\(^{-1}\), whereas methylene molecules (\(\text{CH}_2\)) in the saturated fatty chain showed a stretching band at 2862.38 cm\(^{-1}\) and a C-H in-plane distortion band at 1381.03 cm\(^{-1}\).

![Figure 3: FTIR of epoxy/OP composites.](image)

After combining ZnO nanoparticles with epoxy/OP, minor vibrations at 1573 cm\(^{-1}\) corresponding to aromatic moieties were noticed (Fig.4), as well as a strong band at 1600 cm\(^{-1}\) due to the stretching vibration of the aromatic ring and CO-H groups. The existence of N-H bonds is responsible for the peak at 3473 cm\(^{-1}\), the group at 3047 cm\(^{-1}\) for C–H and 925 cm\(^{-1}\) for C–O, NH\(_2\) vibration absorption at 3336 – 3200 cm\(^{-1}\), C–OH at 3400–3600 cm\(^{-1}\) and –CN group vibration at 1103 cm\(^{-1}\)[8].

![Figure 4: FTIR of epoxy/OP/ZnO composites.](image)
3.1 Tensile Strength, elongation, and Young Modulus

Tensile properties are shown in Fig. 5. The influence of varied OP filler loads on the strength properties of epoxy composites decreased from 22.78 to 19.3 (MPa) due to the inefficient stress transmission from the epoxy matrix during loading circumstances caused by a hydrophilic factor or poor adhesive capacity between the oil palm fiber and the epoxy matrix. When adding the fillings (1, 2, 3 and 5 wt %) of ZnO, the tensile strength fundamentally increased because nanofiller particles are harder and stiffer, nanocomposites created by adding nanofiller were significantly stiffer and stronger than epoxy and epoxy/OP composites (as shown in Fig. 6) [10].

![Figure 5: Samples after tensile test.](image)

**Figure 5: Samples after tensile test.**

![Tensile Strength MPa](chart)

**Figure 6: The values of tensile strength for epoxy/OP (with different wt% of OP) and for epoxy/OP/ZnO (with different wt% of ZnO nanoparticles) composites.**

<table>
<thead>
<tr>
<th></th>
<th>Tensile Strength MPa</th>
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<tbody>
<tr>
<td>pure</td>
<td>22.78</td>
</tr>
<tr>
<td>OP 1%</td>
<td>21.56</td>
</tr>
<tr>
<td>OP 3%</td>
<td>20.36</td>
</tr>
<tr>
<td>OP 5%</td>
<td>20.2</td>
</tr>
<tr>
<td>OP 10%</td>
<td>19.03</td>
</tr>
<tr>
<td>OP/ZnO 1%</td>
<td>21.15</td>
</tr>
<tr>
<td>OP/ZnO 2%</td>
<td>22.2</td>
</tr>
<tr>
<td>OP/ZnO 3%</td>
<td>27.08</td>
</tr>
<tr>
<td>OP/ZnO 5%</td>
<td>29.24</td>
</tr>
</tbody>
</table>

The observed continually decreasing elongation at break of composites (Fig. 7) with increasing OP fiber loading is attributable to natural fibers’ low elongation at break, which restricts polymer molecules from moving past one another. Furthermore, when the OP fiber load was raised, the stiffness and fracture toughness of the hand sheet was nearly as high, leading in a decrease in ductility.
Fig. 7: The values of elongation for epoxy/OP (with different wt% of OP) and for epoxy/OP/ZnO (with different wt% of ZnO nanoparticles) composites.

Fig. 8 shows that Young modulus increased for all the samples of epoxy/OP and epoxy/OP/ZnO composites. This indicates the improvement of stiffness due to the addition of the rigid epoxy matrix which has a fiber and a nanofiller. Tensile strength has increased due to the use of the nanoparticles. The highest value of Young modulus (4.3 MPa) at 5% nanofiller was attributed to the improved homogenous dispersion and significant improvements in the interface between the nanofiller and the epoxy/OP matrix included inside the nanocomposites. The incorporation of a greater number of distinct nanofiller particles into the polymer matrix increases the reinforcement volume fraction and concentration leading to a rise in inter-particle interaction within the epoxy matrix. This agrees with the work of Ghazilan et al. [10].

Fig. 8: The values of Young modulus & for epoxy/OP (with different wt% of OP) and for epoxy/OP/ZnO (with different wt% of ZnO nanoparticles) composites.

### 3.2 Impact test

Impact strength provides a measure of the material capacity to withstand impact loading, which is essential because components are regularly subjected to such pressure during service. Figs. 9 and 10 show that the impact strength increased for epoxy/OP composites which indicate that depending on the type and the volume of the fiber is more likely to be related to the degree of internal damage during impact loading. This composite fiber had the greatest cellulose concentration in its chemical...
makeup, which contributed to its increased mechanical strength and less internal damage during stress, than the homogeneous epoxy specimen, irrespective of the type of fiber because that epoxy itself is brittle in nature, which reduces the capacity to absorb impact force.

The value of impact strength rose with the addition of ZnO nanoparticles, owing to the reinforcements of ZnO having a favorable effect on bearing impact force and increasing the required impact energy for fracturing the specimen. The impact strength characteristics of epoxy/OP/ZnO composites were found to be somewhat greater than those of pure epoxy due to the sitter nature of the palm filler and the ZnO.

![Figure 9: The samples of composites after the impact test.](image)

![Figure 10: The values of impact strength for epoxy/OP (with different wt% of OP) and for epoxy/OP/ ZnO (with different wt% of ZnO nanoparticles) composites.](image)

### 3.3 Hardness test

Fig.11 shows the values of hardness of epoxy/OP composites for the different wt% of composite. Adding ZnO to epoxy /OP composites resulted in the increase of the hardness values with increasing ZnO and improving when using particles ZnO as reinforced epoxy/OP. The reason for such significant behavior might be related to increased strength in particulate composite materials by increasing fracture surface area, which arises from an uneven crack route caused by particles serving as an obstruction in front of crack propagation, and the requirement for more energy for crack growth [11].
3.4 Wear test

Wear rate results for epoxy/OP composites are presented in Fig. 12 which show that wear rate decreased slowly when a 5N load was applied for all the wt% of oil palm. This is because reinforcing epoxy with oil palm fiber can improve the weight lost performance of the polymer composite because, in most applications, epoxy has already a high friction coefficient and when compared to epoxy-containing composites, it has a low wear resistance. To reduce the friction coefficient, as well as the wear rate, fillers have been added.

The wear rate for epoxy/OP/ZnO nanocomposites as compared with that of epoxy /OP composites has improved because the distribution of the natural fiber (oil palm in the polymer matrix) and nano ZnO helped to reduce the friction between the counter face and the specimen surface (i.e., the oil palm worked as a self-lubricant). Finally, the composites were improved because it was reinforced with oil palm fiber, and so, in turn, the composite could withstand heavier loads and also because that nanometer ZnO is hypothesized to offer toughness and wear reduction by restraining fractures, improving ductility, compartmentalizing damage and improving ductility.
4. Conclusions

The results showed that the tensile strength was low for the pure epoxy samples but increased for epoxy/ palm oil/ ZnO samples. Young modulus increased with decreasing elongation with ZnO. The impact of pure epoxy and epoxy/OP and epoxy/OP/ ZnO nanocomposites increased between (6.94 to 10.8 kJ/m²). Hardness increased between (80.8 to 84.55) for all the composites under study, the inclusion of small percentages of nanoparticles to epoxy/OP composites has significantly boosted their strength.

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Conflict of interest

Authors declare that they have no conflict of interest.

References


Tensile Mechanical Property of Oil Palm Empty Fruit Bunch Fiber Reinforced Epoxy Composites

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Abstract

The research focuses on using epoxy resin and oil palm waste, which is shaped through casting at different percentages (1, 3, 5, and 10% by weight) using a high-speed mechanical mixer, and the effect of zinc oxide nanoparticles (ZnO) on the behavior of composites containing different percentages (1, 2, 3, 5, and 10% by weight) of ZnO nanoparticles. The results show a strong interaction between zinc oxide and oil palm fibers and epoxy resin, and the mechanical properties of the composites, the tensile strength decreased from (22.78 MPa to 19.03 MPa) and increased to (29.224 MPa) for composites containing zinc oxide, while the elongation decreased from (9.6%) to (6.8%) with an increase of the original elongation of (84.55) to (80.8) at (8.94 kJ/m²). The Tribological characterization of the composites containing zinc oxide was evaluated. It was found that the addition of zinc oxide nanoparticles increased the tribological properties of the composites compared to the original composites. Therefore, the results indicate that the composites containing zinc oxide nanoparticles are promising materials for the production of new composites with high mechanical and tribological properties.