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Full Length Research Paper

Effects of particle size on the physico-mechanical properties of epoxy filled with dates palm pits *(Phoenix dactylifera)* particulate composites

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Two particles sizes of date palm pits particulate 150 µm and 300 µm, have been used to filled epoxy to produce composites DTP/EP 1)and DTP/EP 2, respectively. The effects of particle size on properties of the resultant composites were investigated using various techniques such as mechanical test, density, hardness and water absorption capacity. Composites prepared with 150 µm filler particles size gave higher mechanical strength in comparison with composites made from 300 µm filler particles size. Reducing the filler size of DTP/EP from 300 µm to 150 µm improved the tensile strength of the DTP/EP composites at 10 wt% filler loading by 10%. The flexural strength and impact strength were improved by 1.2% and 25%, respectively, upon particles size reduction of filler from 300 µm to 150 µm at 10 wt% filler loading. The result of water absorption based on the particle sizes showed that the composites with higher particle size absorbs more than the smaller particle size which can be attributed to the fact that the smaller particle sizes are more encapsulated by the matrix resulting in lower absorption values. Thus, the size of filler plays an important role in determining the mechanical properties of filled resin composites.

Keywords: Tensile strength, flexural strength, density, impact strength, hardness and matrix

INTRODUCTION

The use of polymer matrix composite has found wide application in our modern day world; this is as a result of the combination of properties which these materials possessed. Some of the properties of polymer matrix composites include specific strength, high modulus, good fracture and fatigue properties as well as corrosion resistance (Agunsoye and Edokpia, 2013). Polymer composites are now being used in both indoor and outdoor structural applications in housing, construction, automobile industry, aerospace etc.

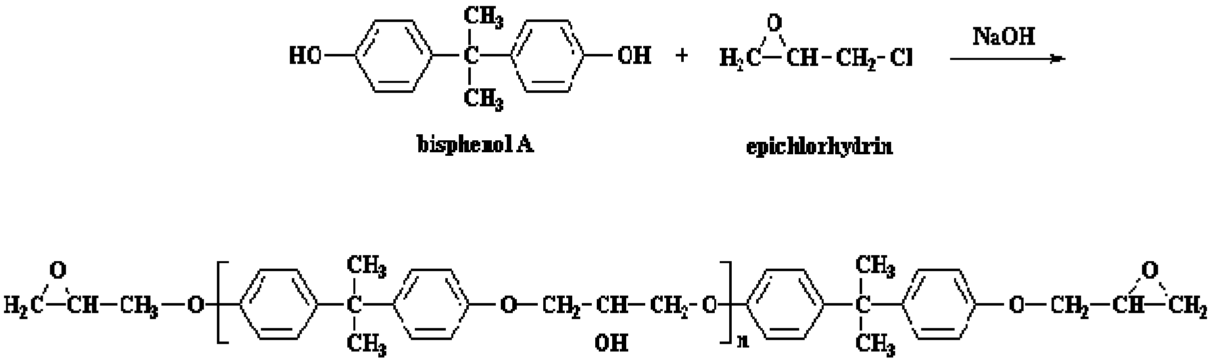
Natural fillers in the form of fibres or particulate have gained the attention of researchers in recent time as reinforcing materials in polymers, metals and ceramics. They are eco-friendly, low cost, low density materials; they are renewable in large amount when compared with synthetic fillers.

Epoxy Resins

Epoxies are thermosetting resin materials characterised by two or more oxirane rings or epoxy groups within their molecular structure. The commonest epoxy resin is the diglycidyl ether of bisphenol A (DGEBA), which is prepared by the reaction of epichlorohydrin (ECD) and bisphenol A (BPA). ECD is prepared from polypropylene (PP) by reacting chlorine with sodium hydroxide (Hodd, 1990) (Scheme 1).

Fillers

Fillers are defined as materials that are added to a polymer formulation to lower the compound cost or to improve properties. The filler properties such as particle



Scheme 1: Reaction for the synthesis of DGEBA-type epoxy resin.

Plate 1. Date palm pits.

size, size distribution and shape strongly influence the mechanical properties and play a central role in the properties of the formulation. The research aim at investigating the effect of particle size on the properties of epoxy filled with dates palm *(phoenix dactylifera)* pits particulate composites (Plate 1).

Palm date fruits consist of three main parts: date flesh, date pit, and skin. That is, it is a drupe, an indehiscent fruit in which an outer fleshy part (exocarp, or skin; and mesocarp, or flesh) surrounds a shell (the pit, stone, or pyrene) of hardened endocarp (Marzieh *et al.,* 2010). It contains a single seed (pit) about 2–2.5 cm long and 6–8 mm thick. The pit is a major by-product of the date palm-processing industry. They contained 7.1–10.3 % moisture, 5.0–6.3 % protein; 9.9–13.5 % fat; 46–51 % acid detergent fibre; 65–69 % neutral detergent fibre; and 1.0–1.8 % ash. Date pit is mainly used as animal feed (Hamada *et al*., 2002). Yang *et al*., (2004) reported that the addition of filler to polymer system resulted in deterioration of tensile and impact resistance and this was attributed to the poor filler dispersion and poor filler-matrix interfacial bonding thus giving rise to the formation of large filler agglomerates in the polymer matrix, which influence the mechanical properties of the material. The interfacial adhesion between fillers and polymer has a determining influence in the mechanical properties of composites, however to improve mechanical properties through the use of fillers is by changing its particular size besides treating it with coupling agents. This may be attributed to the greater interaction and/or better dispersion of the finer particles in the PE matrix and also gave relatively higher absorption energy during the fracture process. The study is aim at investigating the effect of particle size on the physico-mechanical properties of epoxy filled with dates palm pits *(phoenix dactylifera)* particulate composites.

MATERIALS AND METHODS

Date palm fruits, aluminium foil, Epoxy Resin (commercially available epoxy resin (3554A) of density 1.17 g/cm^3) and polyamine amine (Hardener3554B) of density 1.03 g/cm^3 were procured from a local supplier in Ojota, Lagos, Nigeria. The date palm fruits were obtained from Gwagwalada market, F.C.T; Nigeria.

Experimental procedure

Filler Preparation

The date pits (DTP) were separated from their fruits manually, thereafter; they were washed and cleaned to remove contaminants. They were then dried and grounded with hammer mill to obtain filler powder. The fillers were screened to obtain different particle sizes of 150 μm and 300 μm. Thereafter, oven dried for 24 h at temperature of about 70°C to reduce the moisture content. Samples were thereafter stored in a sealed container prior to compounding.

Compounding

Five levels of filler loading (10 wt%, 20 wt%, 30 wt%, 40 wt%, and 50 wt%) were made from fillers particle sizes of 150 µm and 300 µm with the epoxy to give date pits/ epoxy composites DTP/EP 1 and DTP/EP 2 respectively. Neat resins without filler were equally prepared to serve as control.

Date epoxy composites

This was achieved by mixing the various ratios of the prepared fillers with the epoxy to form homogenous blends. The mixing was achieved via manual stirring method for 10 minutes; hardener was then added to the mixture. The volume ratio of resin to hardener was 2:1, and after thorough mixing with the filler, the mixture was poured onto the cavity of glass mould of dimensions 160 mm x 70 mm x 4.5 mm overlaid with aluminium foil to serve as releasing agent. The mixture was allowed to cure at room temperature for 24 h before removal from the mould (Plate 2 and b).

Characterization

Tensile test

The tensile test was carried out on a Universal testing machine (TIRA test 2810) with maximum load of 10 KN in accordance with ASTM D3039. Samples with dimension 160 mm, 20 mm, and 4.5 mm of length, breadth and height, respectively, were used for the test. A cross – head speed of 2 mm/min was used. The specimens were positioned in the grips of the testing machine and the grips were tightened evenly and firmly to prevent any slippage and as tensile test started, the specimen elongated, the resistance increased and is detected by the load cell. The load cell (F) was recorded until a fracture or rupture of the specimen occurred. Tensile strength and tensile modulus were expressed as:

Tensile strength (MPa) = $\frac{P}{bh}$, Where, P = Pulling force (N) b = Specimen Width (m)
h = Specimen thickness (m), Tensile modulus (MPa) = $\frac{\sigma}{\varepsilon}$
Where, $\boldsymbol{\sigma}$ = Stress (N/m^2)
$\boldsymbol{\varepsilon}$ = Strain

Five specimens for each composite were tested and statistical average for each set of results was recorded.

Flexural or 3-point Bending test (ASTM D790):

The 3-point bending test was carried out on a universal testing machine (TIRA test 2810) as shown below with maximum load of 1 KN in accordance with ASTM D790. A cross speed of 5 mm/min was used with sample dimension 160 mm, 20 mm by 4.5 mm i.e length, breadth by height respectively. The flexural strength of composites was found using the following equation.

Flexural strength = $\frac{3FL}{2bd^2}$ Where, F = Maximum load applied on test specimen (N),
L = Length of support span (mm), b = Width of specimen tested (mm),
d = Thickness of specimen tested (mm)

The flexural modulus can also be found using the following equation:
Flexural modulus = $\frac{ML^3}{4bd^3w}$ Where, M = Maximum load applied on test specimen (N)
L = Length of support span (mm), b = Width of Specimen, d = Thickness of specimen
w = Deflection at maximum force.

Impact strength test (ASTM D256 or ISO179)

The impact tests were performed according to ASTM D256 standard using impact testing machine (IMPat15). An unnotched charpy impact type test in which the specimens were held as a cantilever beam (usually horizontal position) thereafter broken by a blow delivered at a fixed distance from the edge of the specimen. Five specimens for each sample having sizes 160 mm, 20 mm and 4.5 mm thickness were prepared and tested. The specimen was clamped into the pendulum impact test fixture with the side edge facing the striking edge of the pendulum. The pendulum is released and allowed to strike through specimen.

Hardness (Shore D) ASTM D2240

The specimen was first placed on a hard flat surface. The indicator of the instrument is then pressed into the specimen making sure that it is parallel to the surface. The hardness is read within fifteen second.

Density (ASTM D792)

The test was carried out in accordance with ASTM D792. The weight of each specimen having been dried in an oven at 105 °C to a constant weight was taken. Thereafter, the weights of the specimen when immersed

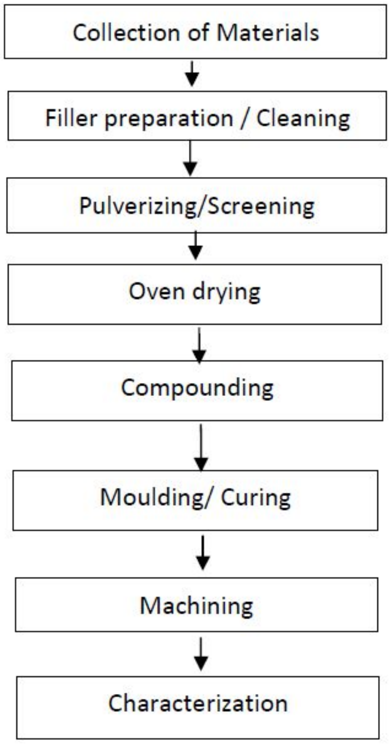


Figure 1. Process steps for the research.

a) b)

Plate 2 a. Composite block and, b. Machine samples.

in water were equally recorded. The density (ρ) is then determined from the relationship below.

$$\rho_c = \frac{W_c}{W_w} * \rho_w$$

Where

W_c = weight of composite in air
W_w = weight of composite when immersed in water
ρ_w = density of water at room temperature (1000 kg/m^3) or 1 g/cm^3
ρ_c = density of the composites specimen.

Water absorption test (ASTM D570)

The specimens for the test were machined into 40 mm, 20 mm and 4.5 mm of length, breadth and thickness respectively. Three specimens for each composite were tested and the average values were taken for each composite. The specimens were dried in an oven for 4 h at 70°C and then placed in desiccators to cool for 2 h immediately; upon cooling the specimens were weighed. The materials were then immersed in water at room temperature of 25°C for 24 h. Specimens are removed, patted dry with a lint free cloth, and weighed immediately in an analytical weighing balance to the nearest milligram.

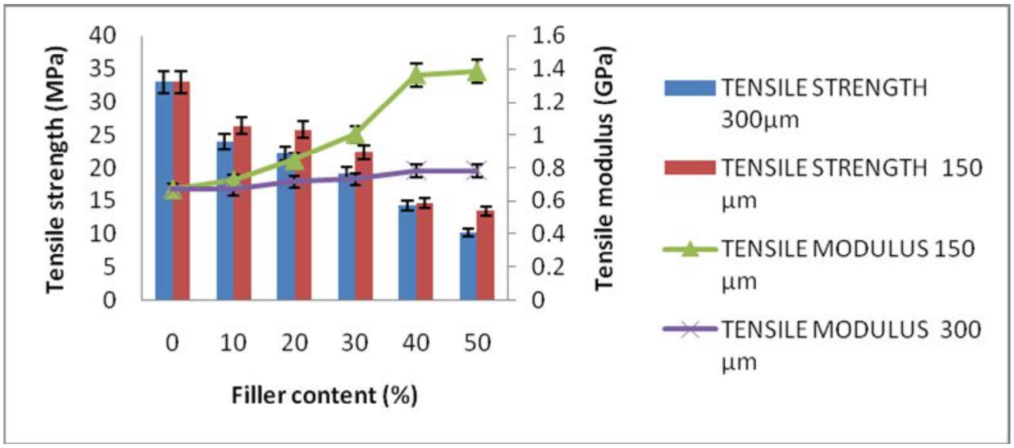


Figure 2. Effect of filler loading and particle sizes on the tensile strength and modulus of date pits/ epoxy composites (DTP/EP 1 and 2).

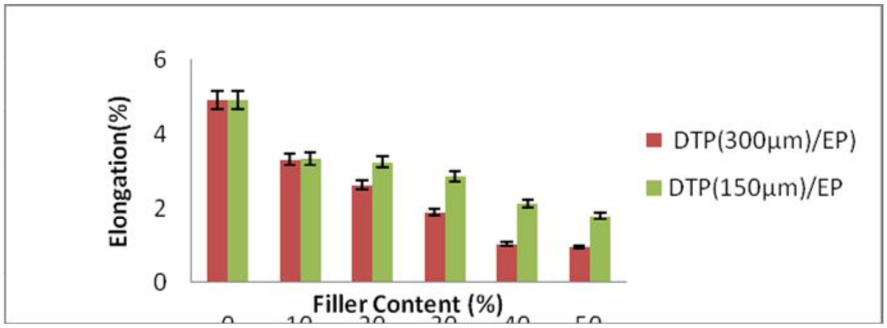


Figure 3. Effect of filler loading on the percentage elongation at break date pit/ epoxy of (DTP/EP 1 and 2) composites.

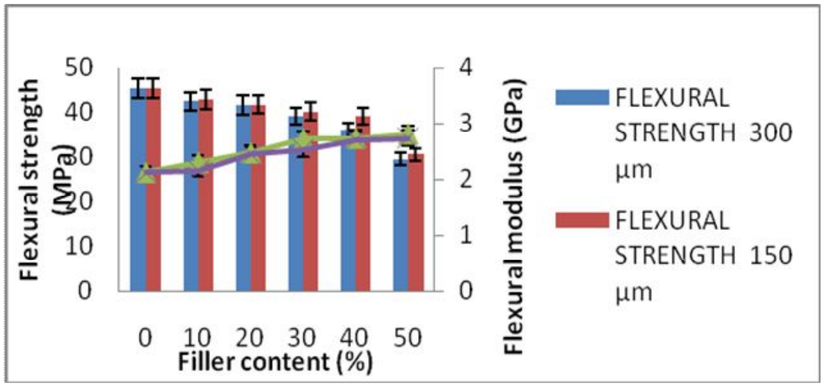


Figure 4. Effect of particle size and filler loading on the flexural strength and modulus of date pits/ epoxy (DTP/EP 1 and 2) composites.

The hydration capacity of the composites was then monitored for period of 32 days.

RESULTS

The results of the mechanical properties of epoxy filled with date pits particulate composites are shown in (Figures 2 to 6).

Water absorption

The effect of time on hydration capacity of date pits particulate composite are illustrated in (Figures 8 and 9), while the effect of filler ratio on rate of absorption of the fabricated composites are as depicted in (Figures 10 to 11).

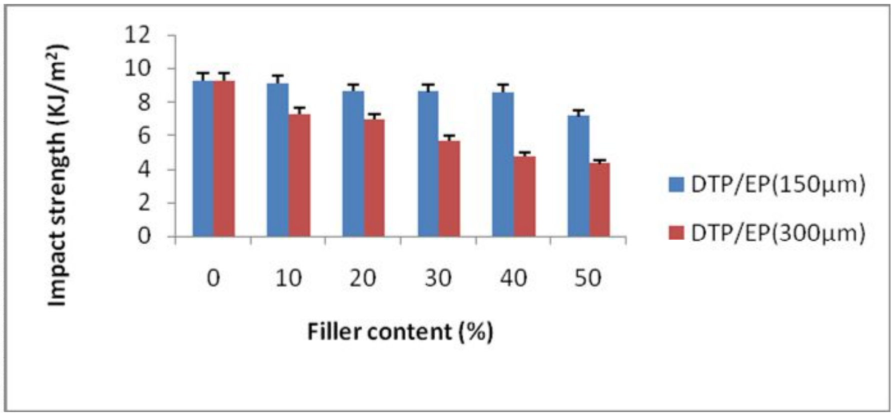


Figure 5. Effect of filler loading on the impact strength of date pits/epoxy composites.

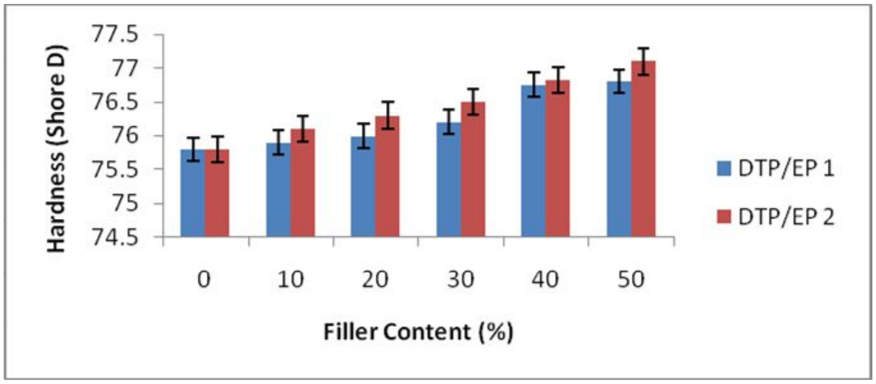


Figure 6. Effect of filler loading and particle size on the hardness of date pits/ epoxy (DTP/ EP) composites.

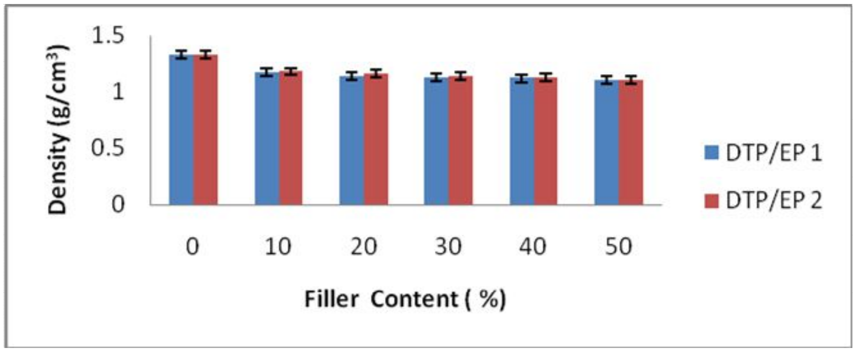


Figure 7. Effect of filler loading and particle size on the density of date pits/epoxy (DTP/EP) composites.

Rate of water absorption

DISCUSSION

The effects of filler loading on the mechanical properties, water absorption, and rate of water absorption at a specific period, hardness, and density of the composites are discussed below.

Tensile tests

The results of the effect of filler loading on the tensile

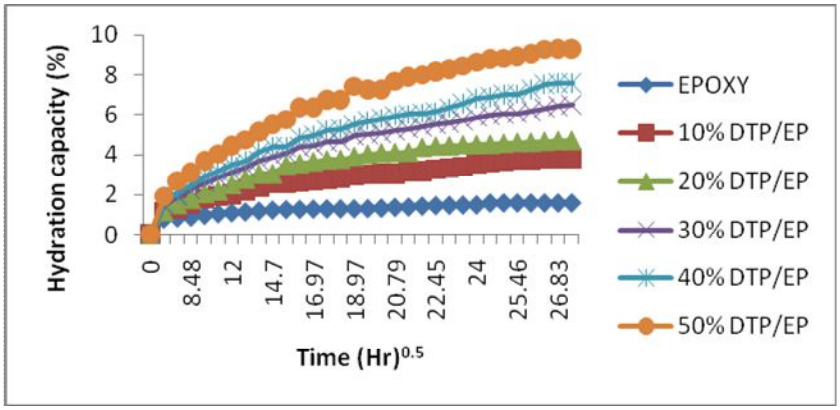


Figure 8. Water absorption curves of date pits/ epoxy (DTP /EP 1) composites.

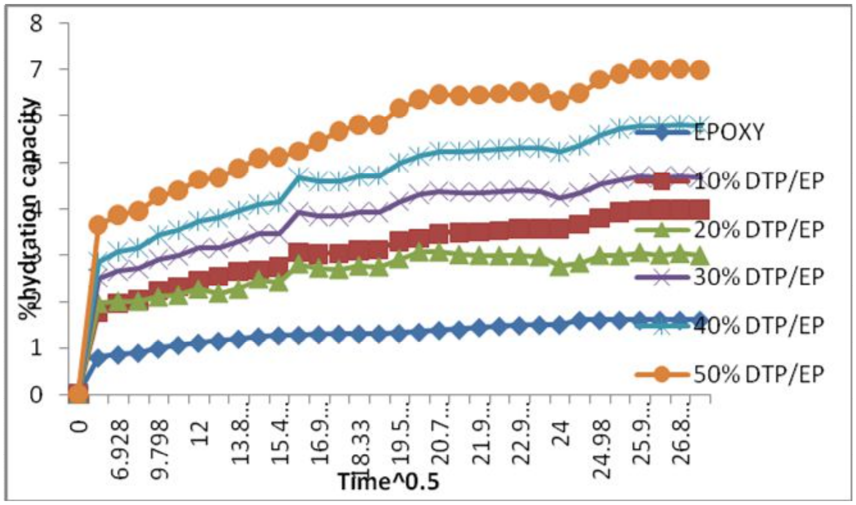


Figure 9. Water absorption curves of date pits/ epoxy (DTP/EP 2) composites.

strength and modulus of epoxy filled with date pits particulate size 150 µm composites (DTP/EP 1) and date pits particulate size 300 µm composites (DTP/EP 2) as shown in Figures 2. The figure shows the relationship between tensile strength, the modulus and the filler ratio. It can be seen that an increase in the filler loading produces a corresponding decrease in the tensile strength of the composite while modulus of the date pits/ epoxy composite increases as the filler ratio increases. The decrease of tensile strength can be attributed to the physical properties of the filler and interaction of this filler with the epoxy matrix (Moczo and Pukanszky, 2008). The tensile strength of date pits/ epoxy was at maximum and minimum value at 10 wt% and 50 wt% filler loading with 26.34 MPa and 13.44 MPa respectively while that of the unfilled resin was found to be 33 MPa.

Also, the figure compares the effects of filler loading and particle sizes on the tensile strength and modulus of DTP/EP composites. It can be seen that the lower particle size gave more strength than the higher particle sizes. It is expected that the smaller particle size will have more surface area of interaction with the resin thereby improving the stress transfer process that will ultimately increased the tensile strength. Extender filler could perform as functional filler when particles size is reduced (Nura, 2008). Reduction of the filler particle size from 300 µm to 150 µm increases the strength of the composites by 10% and 32% at 10 wt% and 50 wt% filler loading respectively. Higher values of strength were generally observed for filler with particles size 150 µm. Sreekanth *et al.* (2009) in their study of effect of particle size and concentration of flyash on properties of polyester thermoplastic elastomer composites also confirmed that smaller particles size gave higher strength.

On the other hand, the modulus increased as the filler loading increases. However, it can be seen from the

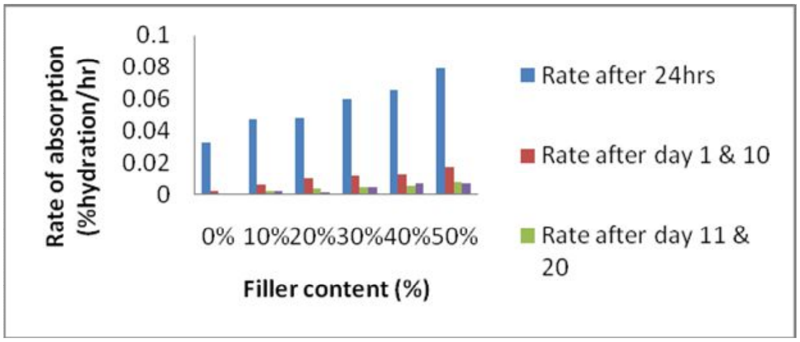


Figure 10. Effect of filler loading on rate of absorption of water by date pits/epoxy (DTP/EP 1) composites.

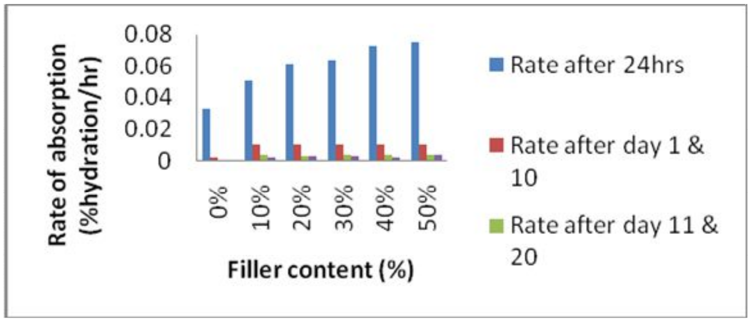


Figure 11. Effect of filler loading on rate of absorption of water of date pits/epoxy (DTP/EP 2) composites.

(Figure 2), that the modulus of the lower filler size improved greatly more than that of higher filler size. Modulus of date pits/ epoxy (DTP/EP 1) composites at 40 wt% was 1.36 GPa while that of date pits/ epoxy (DTP/EP 2) composite at the same filler weight percent was 0.78 GPa. Since, modulus is an indication of stiffness, improvement in the modulus as the particle size reduces might be as result of more interaction between the filler and the matrix at lower particle size. It can also be due to lack of proper dispersion at higher filler size resulting into lower strength and modulus.

Elongation at break

Figure 3 shows the percentage elongation at break of date pit/ epoxy (DTP/EP 1) composites. The results show that as the filler loading increases, the elongation at break of the composites decreases, signifying reduction in the ductile nature of the composite. The addition of filler caused the matrix to lose its elastic properties, in other words, the material is more brittle (Lassaad *et al*., 2011). Incorporation of DTP into EP matrix drastically reduced the elongation at break value. It was reported that reduction of elongation at break of mica/epoxy composites was due to reduction of volume of the matrix (Suradi *et al*., 2011), since the elastic properties were only obtained from the matrix (Firoozian *et al*., 2010). The rigidity of composite was also due to the restriction of the matrix mobility and deformability by the addition of filler content. The lower values of elongation at break shown by composites have been confirmed by their low impacts strength values. Generally, a high elastic material is an indication of the high value of elongation at break. It can be seen from Figure 3 that (DTP/EP 1) composite produces the higher elongation at break than (DTP/EP 2). Date pits/ epoxy (DTP/EP 1) and date pit/ epoxy (DTP/EP 2) gave 3.33% and 3.31%, respectively. The minimum values were obtained at 50 wt% filler ratio with following values of 1.81% and 0.95% respectively, for (DTP/EP 1) and (DTP/EP 2), respectively. The elongation at break of the unfilled epoxy reduces by 32 % and 63 % upon addition of 10 wt% and 50 wt% filler loading of date pits particulate, respectively.

Flexural test

The results of the effects of filler loading on the flexural strength and modulus of the epoxy filled date pits particulate sizes 150 µm and 300 µm composites are as shown in Figure 4. It can be seen that an increase in the filler loading produces a corresponding decrease in the strength of the composite while modulus of the date pits/ epoxy composite increases as the filler ratio increases. The flexural properties of composites depend critically on the microstructure of the composite and the interfacial bonding between the reinforcement and the matrix (Subita and Pardeep, 2013). From the results of DTP/EP 1, it was observed that the flexural strength of the unfilled resins was reduced by 5.2% on addition of 10% filler loading. Further increase in the filler loading to 20 % resulted to a decreased in the value by 7.9%. Changing the filler loading from 40% to 50% causes the strength to decrease by 22% whereas the change from 30 % to 40 % filler loading decreased the flexural strength by 2.6 %. The later decreased in flexural strength at 50% filler loading is due to agglomerate formation at higher concentration of the particulate (Sarojini, 2013). Similar results have been reported by other researchers that the flexural strength decreased after 40 wt% of filler loading (Raju *et al*., 2012). The maximum flexural strength of 42.92 MPa was observed at 10% filler loading while the minimum value of 30.5 MPa was observed at 50% filler loading.

On the other hand, the study shows that the flexural modulus increases as the filler ratio increased which was in agreement with several previous reports on the effects of lignocellulosic filler content on the modulus of polymeric composite. It has been observed by many researchers that the modulus increases with increase in filler loading (Raju *et al.,* (2012). This behaviour may possibly lead to the conclusion that the flexural modulus might also depend on the filler content rather than particle–matrix interface. Furthermore, increase in flexural modulus for higher filler loading is due to the higher stiffness of the reinforcing particles than the matrix material. Thus, increase in modulus as a result of the increased in the filler ratio imparts stiffness to the composite. According to Adams *et al.* (1969) the relative stiffness of a material is indicated by its modulus. Figure 4 clearly showed that the lower particle size gave more strength than the higher particle sizes. Sreekanth *et al.* (2009) in their study of effect of particle size and concentration of flyash on properties of polyester thermoplastic elastomer composites also confirmed smaller particles size gave higher strength. It is expected that the smaller particle size will have more surface area of interaction with the resin thereby improving the stress transfer process that will ultimately increased the flexural strength. Reduction of the filler particle size from 300 µm to 150 µm increases the strength of 10% filler weight composite from 42.41 MPa to 42.92 MPa respectively, while the strength of 50% filler weight ratio was improved from 29.5 MPa to 30.5 MPa. Higher values of strength were generally observed for filler with particles size 150 µm. On the other hand, the modulus increased as the filler loading increases. However, it can be seen from the Figure 4 that the modulus of the higher filler size slightly improved more than that of lower filler size. Modulus of date pits/ epoxy (DTP/EP 1) composites at 50 % was 2.73 GPa while that of date pits/ epoxy (DTP/EP 2) composite at the same filler weight percent was 2.81 GPa while the modulus for the unfilled resin was 2.12 GPa.

Impact strength

Figure 5 showed the result of the effect filler loading on the impact strength of 150 µm and 300 µm date pits/epoxy composites. It can be seen from result that as the filler loading is increased, the impact strength decreases. Addition of 10% filler loading into epoxy resin decreases the impact strength 150 µm particle sizes by only 1.3% while that of 300 µm reduces by 21 %. The impact strength of the unfilled resin reduces from 9.249 KJm^{-2} to 9.128 KJm^{-2} at 10 wt% filler ratio of 150 µm particle size filler as in Figure 5. The material under study shows lower values of impact strength for 300 µm particle size filler in which the minimum impact strength values of 4.02 KJm^{-2} (56% reduction) was recorded at 50 % filler loading. The decrease in impact strength of the composites is due to decrease in elasticity of the composite thereby decreasing the deformability of the matrix. As the concentration of the filler increases, there is poor interfacial adhesion between the polymer matrix and the particles resulting into occurrence of micro-cracks at the point of impact which decreases the impact strength. The decrease in impact strength might also be due to the inability of the reinforcement's filler to block the crack propagation resulting in reduction of the impact strength (Sabu *et al.,* 2012).

Hardness (Shore D)

Hardness implies a resistance to indentation, permanent or plastic deformation of material. In a composite material, filler weight fraction significantly affects the hardness value of the composite material. Figure 6 shows the result of the effect filler loading on the hardness of 150 µm and 300 µm date pits/epoxy composites. It can be seen from result that as the filler loading is increased, hardness also increased. Addition of 10 % filler content into epoxy resin increases the hardness by insignificant increase of 0.13 %. The hardness of the unfilled epoxy increased from 75.8 to 75.9 on shore D scale at 10 % filler weight ratio as shown in Figure 6. The values show that increased in filler loading increases the hardness. Hardness test is a simple one and gives good information on the microstructure relationships of polymer composites (d'Almeida and Manfredini, 2001). For date pits/ epoxy (DTP/EP) composite, the unfilled resin hardness increases slightly upon addition 10% filler loading. The hardness continues to increase as filler loading increases up to maximum of 76.8 and 77.1 for 150 µm and 300 µm particle sizes respectively. This might be attributed to the nature of the filler since the properties of composite depends on nature and type of the filler. Naturally, date pit is a hard stony material that can be termed as composite containing cellulose as reinforcement in a lignin matrix.

Density

The basic reason for a composite is to have material that combined good mechanical strength with light weight. Therefore, determination of the density of the prepared composites is important. The result of the effect of filler loading and particle size on the density of date pits/epoxy (DTP/EP) composite depicted in Figure 7 reveals that an increase in filler loading of particulate of date pits in the resin decreases the density of the composites. This decrease in density can be related to the fact that the fillers are lighter. That is, the density of the filler is less than that of the unfilled resin which necessitates the decrease in the composite density as the filler loading increases. Consequently, the density of the composite decreased with regards to the epoxy composite as both fillers are less dense to the epoxy resin. As observed from the Figure 7, the density of DTP/EP 1 and DTP/EP 2 shows similar values indicating that the effect of particle size on density might not be relevant since the two particle sizes are from the same filler having the same density value.

Water absorption

The percentage hydration of (DTP/EP 1) at room temperature of about 25°C has the following values after 24 hours of absorption as shown in (Figure 8) 1.13%, 1.16%, 1.44%, 1.58%, and 1.91% at 10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt% filler loading respectively while the unfilled epoxy resin gives 0.789%. The test shows that the absorption continues to increase daily and after 768 h (32 days), the following values were obtained: 3.82%, 4.71%, 6.48 %, 7.56 %, and 9.82 % for the corresponding 10 wt%, 20 wt%, 30 wt%, 40 wt%, and 50 wt% filler content. The daily absorption is primarily due to the hydrophilic nature of the lignocellulosic filler. The unfilled epoxy reached maximum absorption value of 1.61% after 648 h (27days). However, the rate of absorption for all the composites was at maximum after the first 24 h of absorption. The result in (Figure 9) shows that date pits/ epoxy (DTP/EP 2) has the following percentage hydration: 1.79%, 1.97%, 2.05%, 2.86 %, and 3.65% at 10 wt%, 20 wt%, 30 wt%, 40 wt% and 50 wt%% filler weight ratio respectively. The result of absorption based on the particle sizes showed that the composite with higher particle size absorbs more than that of smaller particle sizes. It may be logical to say that the smaller particle sizes are more trapped or enclosed by the matrixes resulting in lower absorption values. This may be attributed to the fact that at lower particle size, the hydrophilic fillers were more encapsulated by the matrix than the higher particle size. Generally, from the results, water absorption increases as the filler loading increases.

Rate of water absorption

From the result shown in Figure 10, the rate of absorption of DTP/EP composite gives the following values after 24 h 0.047%/h, 0.048%/h, 0.060%/h, 0.066%/h and 0.080%/h at 10 wt%, 20 wt%, 30 wt%, 40 wt%, and 50 wt% filler loading respectively. Thus, indicating the maximum rate of absorption for the filler loading. The unfilled epoxy showed the least rate of absorption of 0.033%/h at the first 24 h and 0.002%/h within the first ten days. The rate of absorption of the composite reduces drastically within the first 10 days to the following values 0.007%/h, 0.107%/h, 0.012%/hr, 0.013%/h, and 0.018 %/h for 10wt %, 20 wt%, 30 wt%, 40 w% and wt % filler loading, respectively, while day 21 to 32 gave the minimum rate of 0.001%/h, 0.003%/h, 0.003%/h, 0.004 %/h, 0.007%/h, and 0.007%/h at 0 wt%, 10 wt%, 20 wt%, 30 wt%, 40 wt%, and 50 wt% filler loading, respectively. The rate of absorption for all the composites was at maximum after the first 24 h of absorption.

Conclusion

The addition of 10 wt% and 50 wt% ratio of date pits filler into epoxy resin reduces the tensile strength by 20% and 59% respectively, while the flexural strength was reduced by 5.2% and 32.7%. Composites prepared with 150 µm filler particles size gave higher mechanical strength in comparison with composites made from 300 µm filler particles size.

Reducing the filler size of DTP/EP from 300 µm to 150 µm improved the tensile strength of the DTP/EP composites at 10 wt% filler loading by 10%, while the flexural strength was improved by 1.2%. The impact strength of DTP/EP composites was improved by 25% and respectively, upon particles size reduction of filler from 300 µm to 150 µm at 10 wt% filler loading. Water absorption of the composites can be considered to be moderately low based on the result, in comparison to other lignocellulosic materials given in literatures. The DTP/EP composite gave 1.1% and 1.9% for 10 wt% and 50 wt% filler loading at the first 24 hours respectively, while 3.86% and 9.8% were obtained for 10 wt% and 50 wt% filler loading respectively after 768 h (32 days) of immersion.

Authors' declaration

We declared that this study is an original research by our research team and we agree to publish it in the journal.

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