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EFFECT OF SODIUM HYDROXIDE TREATMENT ON THE PHYSICAL AND MECHANICAL PROPERTIES OF EPOXY-BLACK DATE SEED PARTICULATE COMPOSITES

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ABSTRACT

The present investigation entails the development of epoxy/black date seed particulates (BDSP) composites fabricated by hand lay-up method. The BDSP with particle size 180µm was treated with sodium hydroxide (NaOH)solution and was loaded in the epoxy matrix from 0 to 20 wt% at 5 wt% interval to produce both treated (TDSP) and untreated (UDSP) composites. Physical (Density measurement) and mechanical (Tensile, impact, flexural and hardness) properties as well as Scanning electron microscopy (SEM) on tensile fractured surfaces were used to characterize the produced composites. The results obtained showed that the density of the composites (both TDSP and UDSP) and the control lied between 1.41 and 1.56 g/cm^3. Also, the hardness values increased with BDSP loading with those of TDSP (82.2 HV at 20wt% ASP) being higher than those of UDSP (44.4 HV at 20wt% ASP) and the tensile strength decreased progressively with increase in BDSP content. SEM images showed that TDSP produced composites contained rougher contours than that of UDSP. The produced composites exhibited properties that were within acceptable limits for useful engineering applications.

Keywords: Epoxy, Black date seed, Particulate, Hand lay-up, Sodium hydroxide

1.0 INTRODUCTION

Epoxy resins are finding substantial applications in the field of high-performance composites due to their outstanding mechanical properties, resistance to environmental degradation and chemicals, low shrinkage during curing and good thermal stability (Rana *et al.,* 2011). Use of epoxy-based composites in several high-end applications demands further improvement of matrix properties. Researchers have tried to modify epoxy resins either by adding rubber particles or by dispersing rigid inorganic fillers (Moloney and Kausch, 1985). Synthetic materials are the most commonly used fillers in composites processing due to their superior strength, high stiffness and lightweight. The increasing environmental consciousness and awareness of the need for sustainable development has raised interest in using natural materials as fillers in polymer composites (Karthikeyan*et al.,* 2014). The advantages of fillers derived from natural materials include low cost, low density, unlimited and sustainable availability, and low abrasive wear of processing machinery (Alves*et al.,* 2010).

Natural fillers reinforced composites have several drawbacks such as poor wettability, incompatibility with some polymeric matrices and high moisture absorption by the fibers. The main problem often encountered in its use is the filler-matrix adhesion due to the incompatibility between the hydrophilic natural fillers and the hydrophobic polymer matrices (Karthikeyan*et al.,* 2014). This shortcoming may be improved by chemical treatment of the filler. Alkali (NaOH) treatment is a common method to clean and modify the filler surface to lower surface tension and enhance interfacial adhesion between natural filler and a polymer matrix (Bledzki and Gassan 1999).

Black date, *Canarium schweinfurthii,* which is of interest in this present study, is an exotic tree introduced into Africa from Indonesia and are readily found in countries like Angola, Ethiopia, Ghana, Guinea Bissau, Liberia, Mali, Sudan, Zambia, Nigeria etc (Evans, 2004). In Nigeria, the trees are common in Ibadan, Ijebu, Benin, Calabar, Pankshin, Mangu, Barkin Ladi etc.

The fruit of the Black-date is called *Atili in Hausa* and *ube okpoko* in Igbo (Keay, 1964). The oil obtained from the seeds is classified as vegetable oil and the fatty acid compositionis oleic acid, Linoleic acid, Palmitic acid, Stearic acid, Linolenic acid and Myristic acids (Igbum and Eloka-Eboka, 2011).

On this account, the present study was conducted to investigate the effect of NaOH treatment on the properties of epoxy/black date seed particulates (BDSP) composites.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials used for this research include Epoxy resin, hardener, Sodium hydroxide (NaOH) solution, acetic acid, wooden mould, Vaseline, Black date seeds (Atile), paper foil and containers.

2.2 Preparation and treatment of black date seed particulate

The black date seeds (BDS) were washed with water and detergent to remove sand particles and other dirts from its surface and was handpicked to separate it from stones, sun dried for 3 days and then oven dried at 105 ^{o}C for 3 hours. The black date seeds were ground in a ball mill at Mineral Resources Engineering Department, Kaduna polytechnic, Kaduna, after they were sieved to 180µmsize.

The untreated particulates (UDSP) were treated with 2% w/w sodium hydroxide (NaOH) solution for one hour at room temperature after which it was washed with distilled water until all the NaOH was eliminated. A little quantity of acetic acid was added to help in the quick neutralization process. This was confirmed by using a pH meter which showed neutral state of 7.2. After washing, the particulates (TDSP) were dried in air for 3 days after which they were oven-dried at 105°C for 2hrs.

2.3 Preparation of epoxy/BDSP composites

The samples were manufactured with 0, 5, 10, 15 and 20 wt% of the BDSP for both treated and untreated composites. For each weight fraction of particulates, a calculated amount of epoxy resin and hardener (ratio of 2:1 by weight) were thoroughly mixed manually with a mild stirring to minimize air entrapment. For quick and easy removal of composite, Vaseline as a releasing agent was spread on the face of the mould which was placed on a flat surface; then, the mixture of the resin, hardener and the filler was poured into the mould, which was prepared to the various specimen tests specifications according to ASTM standards. The prepared composite samples were cleaned, trimmed of excess moulding materials and allowed to set at room temperature. The composite samples were then cured in an oven at 40°C for 2 hours then stored before further analysis.

2.4 Density measurement.

The densities of the samples were obtained using the Archimedes' principle first, by measuring the sample to get its mass (m), then the sample was immersed in a measuring cylinder half filled with water which would give the volume (v) of water displaced by the sample. The density of each sample was then calculated using the equation:

$$\text{Density} = \frac{mass}{volume}\ \text{g/cm}^3 \qquad (1)$$

2.5 Mechanical properties tests

2.5.1 Tensile test

Tensile test was conducted at room temperature using a Mosanto tensometer of capacity 2.5kN in line with ASTM D 638 method for both the treated and untreated samples as well as the control (unreinforced) sample. For each sample, three tensile test samples were tested and the mean values of the tensile strengths were calculated and recorded.

2.5.2 Hardness test

The test was conducted using a Fuel instruments and Engineers micro Vickers hardness testing machine with model number MV1-pv of capacity 500gF made by pvt, India. A diamond indenter was used to indent the surface of the test sample by the application of static load of 0.1kgf for fifteen seconds before it was removed. Three different points were indented for each sample and the average values were calculated and recorded.

2.5.3 Flexural test

Flexural test was carried out using the universal testing machine in line with ASTM D790. The samples had gauge length of 80mm and were each loaded at the mid span supported at both ends. The load applied at the mid span steadily increased until failure of the samples also, for each sample, three tests were conducted and the average flexural strengths were calculated and recorded.

2.5.4 Impact strength test

Impact strength test was carried out using a Hounsfield balanced charpy impact tester of capacity 15J. The samples were each placed on the machine and the pendulum was allowed to swing and hit them. The impact energy was obtained by reading the values directly from the machine. Three samples were tested for each composition and the average values were calculated and recorded.

2.5.5 Scanning Electron Microscopy (SEM) analysis.

The tensile fractured surfaces of the produced composites were studied by using JOEL-JSM 5600 series Scanning Electron Microscopes (SEM). The specimens were sputter coated with a thin layer of gold to avoid electrostatic charging during sample examination.

3.0 RESULTS AND DISCUSSION

3.1 Density

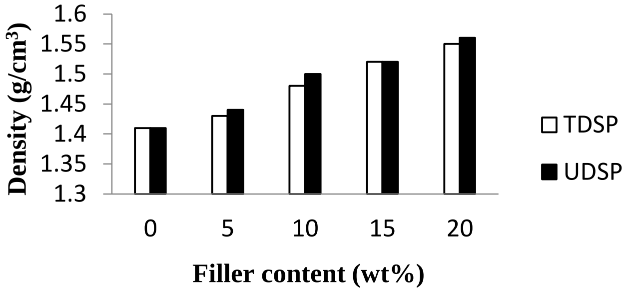

Figure 1: Density of epoxy/BDSP composites

It was observed that with increase in BDSP, density of the composites increased with those of the unreinforced slightly higher than the reinforced as shown in Figure 1. The density of the control sample was 1.41g/cm^3 while for the reinforced at 20 wt% BDSP were 1.55 g/cm^3 (TDSP) and 1.56 g /cm^3 (UDSP).The reduction of density values with surface treatment containing alkali could be attributed to the fact that alkali treatment removes surface impurities and makes the surface of filler rough (Yu *et al.*, 2010; Thao tran *et al.*, 2014). Fillers generally have low density which makes it possible to produce composite of low weights at low cost (Castellano *et al.*, 2004) and low weights are among the advantages of composites materials (Kim *et al.*, 2007).Essentially, the composites produced in this study were light weight which would be very useful in applications requiring low weight.

3.2 Tensile strength

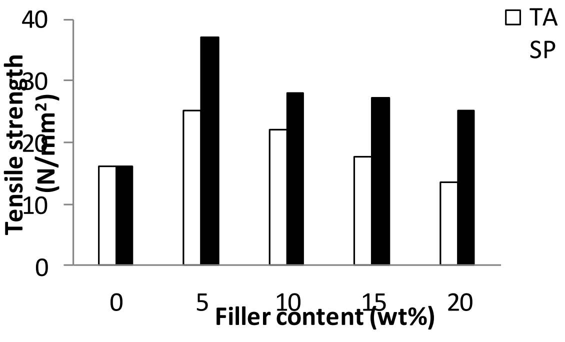

Figure 2: Tensile strength of epoxy/BDSP composites

Figure 2 shows the variation in the tensile strength of the composite materials. A general decrease in tensile strengths with filler loading was observed for the treated and untreated composites. However, the neat polymer (control) had lower tensile strength than the reinforced composites and the untreated composites exhibited higher tensile strengths than the treated samples. At 5 wt% BDSP, the tensile strength of the UDSP was 37.18 N/mm^2 while that of the TDSP was 25.32 N/mm^2 and these values decreased to 25.25 N/mm^2 (UDSP) and 13.62 N/mm^2 (TDSP).Decrease in tensile strengths of lingo cellulosic particle-filled composites is a general trend that have been variously reported by various investigators (Imoisili *et al.*, 2012, Baek *et al.*, 2013; Koutsomitopoulou *et al.*, 2014) although some other reports have shown increase in tensile strengths with filler loading (Onuegbu and Igwe, 2011; Ojha *et al.*, 2014). This trend could be attributed to the fact that fillers exhibit irregular shapes which may not be able to maximize the interfacial surface area and are rather poor in their ability to support the stress transfer from the matrix leading to weak bond between the flour and matrix which subsequently leads to reduction in tensile strength with filler loading (Ewulonu and Igwe, 2011; Chun *et al.*,2012 and Viet *et al.*, 2012).It was reported that the tensile strength of particle-filled composites is more sensitive to the matrix properties (Razavi-Nouri *et al.*, 2006) but with increase in the filler content, the matrix content decreases which could probably lead to strength reduction (Ibrahim *et al.*, 2012).

3.2.1 Young's modulus

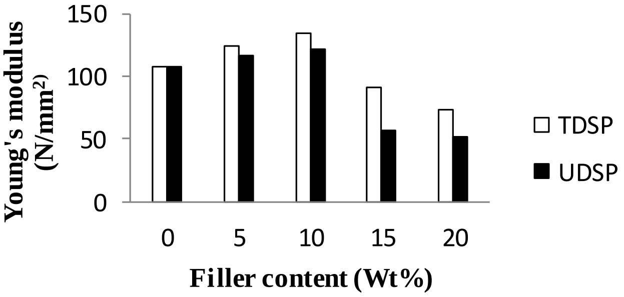

Figure 3: Young's modulus of epoxy/BDSP composites

The Young's modulus of the composite increased progressively from 0 wt% to 10wt% BDSP loading and then decreased afterwards. Also, composites with treated particulates exhibited higher Young's moduli as compared to those with untreated filler as shown in Figure 3.The results obtained are in agreement with various other findings (Imoisili *et al.*, 2012; Fakhrul *et al.*, 2013) that also documented increase in Young's modulus with filler loading. Increase in Young's modulus with filler loading is commonly attributed to the inherent rigidity of fillers which exhibit higher stiffness than the polymer matrices (Salmah *et al.*, 2012; Eze *et al.*, 2013). It was also observed that NaOH treated fillers gave the composites better Young's moduli as compared with those of the untreated. This could be due to improved adhesion of the filler with the matrix. However, further filler addition led to a progressive decrease in the Young's moduli which might be connected to the rise in closed pores content with increasing filler addition creating more sites for crack initiation and subsequently lowering the load bearing limit of the composites. Some investigators such as (Chun *et al.*, 2012; Salmah *et al.*, 2012) reported improvement of Young's moduli with surface treatments over the untreated composites, others such as (Tserki *et al.*, 2006; Rosa *et al.*, 2009) reported otherwise.

3.3 Hardness

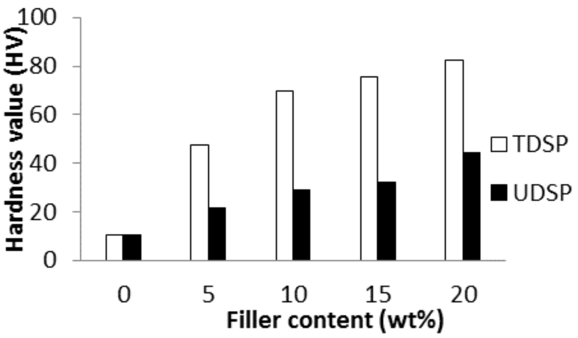

Figure4: Hardness values of epoxy/BDSP composites

Figure 4 presented the results of the hardness test on the epoxy/BDSP composites. The results showed that hardness values increase substantially with BDSP loading with those of TDSP being higher than those of UDSP. The control sample had a hardness of 10.5HV while 20wt% BDSP had hardness values of 82.2 HV (TDSP) and 44.4 HV (UDSP).The increase in hardness values with filler loading could be attributed to formation of a more compact structure on the surface of the composite leading to generation of greater resistance to penetration and consequently, higher hardness values are obtained for the composites with filler loading (Părpăriţă *et al*., 2014).

3.4 Flexural strength

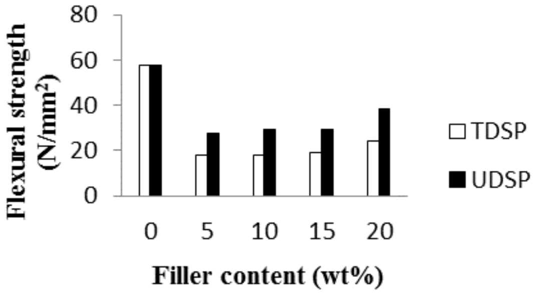

Figure 5: Flexural strength of epoxy/BDSP composites

Figure 5depicts the effect of filler addition on the flexural strength of the produced composites. Accordingly, the flexural strength increased progressively with the filler content for the composites but were all lower than that of the control sample. The flexural strength for the UDSP composites were higher than those of the TDSP composites similar to the tensile strength trend. Koutsomipolou *et al*., (2014) reported that flexural strength decreased with filler loading. Suardana *et al*., (2011) reported that silane treatment did not affect the flexural strength of the composites as observed in their study. Thamae *et al*., (2009) reported that probably due to the compressive component of flexural strength which is not interface dependent, they observed an improvement in flexural strength of their studied composites with flour loading. However, at higher flour content, there was a decrease probably due to agglomeration of the particles.

3.5 Impact energy

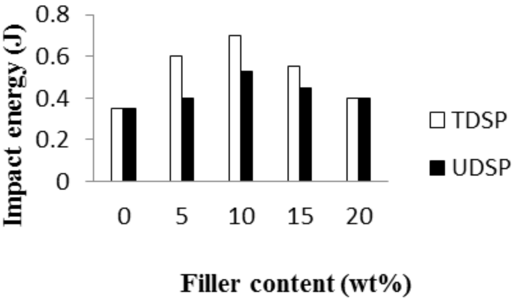

Figure 6: Impact energy of epoxy/BDSP composites

Figure 6illustrates the effect of filler content on impact energy of the composites. The impact energy increases considerably with filler addition up to 10 wt% BDSP for both the TDSP and UDSP composites and then decreased steadily. Khanjanzadeh *et al*., (2012) reported that typically, a polymer matrix with high loading of fillers has less ability to absorb impact energy. Fillers disturb matrix continuity and individual filler is a site of stress concentration, which can act as a micro-crack initiator. The NaOH treatment made the filler surface rougher thereby increasing the mechanical bonding between particulates and matrix and thus improved the impact energy better than UDSP. Similar result was reported by Sutharson*et al.,* (2012).

3.6 SEM Analysis

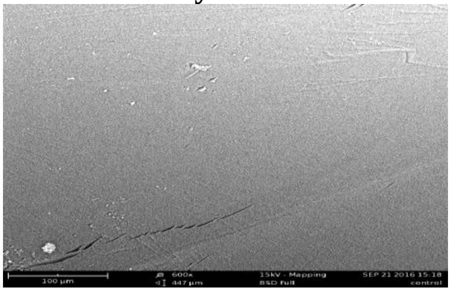

Plate I: Tensile fractured surface of the epoxy material

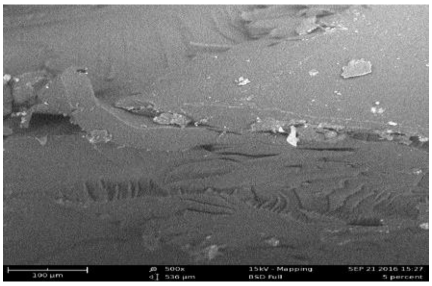

Plate II: Tensile fractured surface of the epoxy material.

Epoxy matrix Epoxy/5 wt% UDSP composite

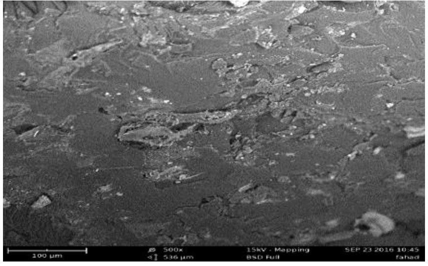

Plate III: Tensile fractured surface of Epoxy/5 wt% TDSP composite

The tensile fractured surface of the epoxy material (Plate I) is characteristic of brittle fracture observed in thermosetting materials. It was observed in the SEM image that several opened-up cracks generated by tear fractures during the deformation of the epoxy material could be attributed to such behaviours. Similarly, filler debonding produces a field of contours on the tensile fractured surfaces of the epoxy/BDSP composites. These contours are smoother in the epoxy/UDSP composites when compared to the epoxy composites reinforced with TDSP (Plates II and III). This explains the impact energy exhibited by epoxy/TDSP composites (Figure 6).This is similar to the observation of Rana*et al.*, (2011).

4.0 CONCLUSIONS

The effect of NaOH treatment and BDSP loading on the mechanical and physical properties of the epoxy/BDSP composites were studied. At the end of the study, the following conclusions are drawn:

1. The density of the composites increased with filler loading with those of the untreated being higher than those of the treated.
2. There was progressive decrease in tensile strength with filler loading. The values were higher for the untreated samples. However, the control sample had lower tensile strength as compared to the reinforced.
3. The hardness values of the composites increased with filler loading.
4. The impact strength increased with filler loading progressively from 0 wt% BDSP (control sample) to 10wt% BDSP addition then subsequently decreased afterwards.
5. The SEM analysis showed brittle fractured surfaces.
6. The composites produced are light weight and could be used in applications requiring light weights such as packaging.

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