Effect of Alkali Treatment on the Mechanical Properties of Raffia Palm Fibres/Oil Bean Pod Shell Reinforced Epoxy Hybrid Composite

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This study investigates the effects of alkali (sodium hydroxide “NaOH”) treatment of raffia palm fibres (RPF) on some mechanical properties of raffia palm fibres/oil bean pod shell particulates/epoxy hybrid composite. The raffia palm fibres were subjected to a 5, 10, 15 and 20% alkali (NaOH) solution treatment for 60, 90, 120, and 150 minutes respectively. The matured oil bean pod shells were dried, grounded and sieved with a 300 µm stainless steel sieve. The composite matrix was prepared by mixing epoxy resin and hardener in a ratio of 7:3 (wt%). The composite hybrid samples were prepared by partial reinforcement of the matrix with 30% combination of raffia palm fibres (RPF) and OBPS particulates in the ratio of 1:1 (wt%); and their tensile and flexural properties of the samples were tested in according to ASTM standards. Results obtained from the tests showed an improvement in the tensile and flexural properties of the treated samples, when compared with results obtained from the untreated (control) samples. The tensile strength increases from 10.672 to 26.437 MPa, Young’s modulus increase from 2.514 to 5.831 GPa, Transverse rupture strength increase from 23.897 to 43.873 MPa, while the flexural modulus increase from 0.784 to 3.386 GPa as the alkali concentrations increased from 5% to 20%. On the average, the best results were obtained at 15% NaOH concentration after 60 and 90 minutes treatment. In contrast, high NaOH concentration (20% NaOH) for longer treatment periods makes the samples become weaker and brittle. Results obtained from this study showed that optimizing the treatment conditions of the raffia palm fibres will certainly enhance their mechanical properties, thereby eliminating time and material wastage.

Keywords: Hybrid composite, raffia palm fibre, particulates, alkali treatment, mechanical properties.

INTRODUCTION

The African oil bean (Pentaclethra macrophylla Benth) is a tropical tree belonging to the family Leguminosae (Mimosoideae) family. The tree bears fruits which are encased in a pod; each pod contains about 8 seeds, which splits explosively at peak maturity dispersing the enclosed fruits about 30 m from the tree (Oghenerukevwe and Uguru, 2018). Raffia palms (Raphia) are a genus of about twenty species of palms native to tropical Africa. Raffia palm fibre is obtained from the membrane found on the underside of the raffia palm frond. When this membrane is peeled off from the palm frond, a long thin fibre is produced. Raffia fibres have many uses, especially in the textiles industry; also, they are used in ropes, roof coverings and shoe production (Tucker et al., 2010; Uguru and Umurhrhu, 2018).

Composite materials are composed of two or more constituents combined in a specific ratio, such that the product is distinct but identifiable. The constituents should be able to improve some strength properties of the composite; therefore, the product often compensates for the weaknesses of each constituent (Verma et al., 2015; Umurhrhu and Uguru, 2019). Some composite types include random-fiber or short-fiber reinforcement, continuous-fiber or long-fiber reinforcement, particulates reinforcement, flake reinforcement, and fillers reinforcement (Kennedy, 2018). There are documented

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results of the high performance of fibres and fillers reinforced epoxy matrix composites. However, these composites still have some drawbacks concerning their matrix-dominated properties that limit their applicability range. The development of newer composite materials addressing these issues is thus of great significance for several engineering applications, broadening the potential structural applications of composites (Baptista et al., 2016). Carbon fibre reinforced epoxy matrix composites are currently used in orthopedics, mainly because of their exceptional strength-to-weight characteristics and high biocompatibility (Scholz et al. 2011). Boob (2014) investigated the suitability of wood sawdust as a constituent material in sandcrete production by partial replacement of the fine aggregates with sawdust. His results showed that an increase in the percentage of sawdust in the sandcrete blocks led to a corresponding reduction in their densities and compressive strength; hence, sawdust can be used for the production of lightweight sandcrete blocks.

There has been a growing awareness in the utilization of biomaterials as reinforcements in epoxy composites in both the automobile and construction industries. According to Ishak et al. (2013) natural fibres and fillers, reinforced composites are suitably applicable in the aerospace, construction, leisure, containerizing, and automotive industries. Many synthetic fibres create several ecological and health hazard problems for the environment and the workers employed in those composites manufacturing companies. As a result, there is a growing interest in the utilization of biomaterials in the production of composites with added advantage due to their availability, light weight, low cost, biodegradability, etc. (Poostforush et al., 2013; Oghenerukevwe and Uguru, 2018). The engineering properties of the composite depend on the volume of the fillers or fibres, the direction of fibres, moisture content of the fillers or fibres, particle size of the fillers, distribution and the interaction among the constituent materials, etc. Umurhurhu and Uguru (2019) reported that filler volume statistically affects the tensile strength of mahogany particulates reinforced epoxy resin composite, as the tensile strength of the samples increased with an increase in the fillers loading. The mechanical properties of short random oil palm fibre reinforced epoxy (OPF/epoxy) composites were studied by Yusoff et al. (2010). They reported that the tensile and flexural properties of the composites decreased with increase in the fibre loading. According to Pramono et al., (2015) the bidirectional bamboo fibre reinforced composite has higher punch shear strength and lower tensile strength than that of the unidirectional fiber composite. Raju and Kumarappa, (2012) studied the mechanical behaviour of epoxy resin composite boards prepared using groundnut shell particulates and reported that the particulate size affects the tensile strength, flexural strength and impact strength of the composites boards. Furthermore, Henry (2013) studied some mechanical properties of raffia palm interspersed fibre (RPIF) filled high density polyethylene (HDPE) at different filler loadings (0 to 60 wt%); and reported that the tensile strength of the composite decreased (27 MPa to 11 MPa) as the raffia palm interspersed fibre increased from 0 to 60%.

Despite the attractive nature of biomaterials reinforced composites, they suffer from a lowered modulus, lowered strength, and a relatively poor moisture resistance compared to synthetic fibre reinforced composites and other conventional materials (Thwea and Liao, 2003). These drawbacks can be overcome through chemical treatment (modification) of the fibres and fillers. Alkali treatment is one of the chemical treatments administered on natural fibres and fillers to enhance their mechanical properties. Sodium hydroxide is one of the most common alkali employed to improve fibre-matrix interfacial bonding of natural fibres; as it increases the surface roughness and removes some lignin and oils covering the surfaces of the fibre’s cell wall (Uguru and Umurhuru, 2018). Uguru and Umurhuru (2018) in their study reported that tensile strength of alkali treated raffia palm fibres was higher (107.07 MPa) when compared with the results obtained from the untreated fibres (67.73 MPa). In addition, Mir et al. (2012) observed an increment in the tensile strength (from 50.4 to 84.8 MPa) of coir fibres after alkali treatment. From the literature search, there is a dearth of information about the effect of treatment duration on the mechanical properties of hybrid composites. Therefore, the objectives of this study were to: (i) treat raffia palm fibres with NaOH at four concentration levels (5%, 10%, 15% and 20%) and four treatment periods (60, 90, 120 and 150 minutes); (ii) evaluate the effect of fibres treatment on some tensile and flexural properties of raffia palm fibre/oil bean pod shell particulates/epoxy hybrid composites.

**MATERIALS AND METHODS**

**Materials**

**Epoxy resin and hardener:** The epoxy resin (LY556) with the corresponding hardener (HY951) 951 were purchased from a chemical shop at Onitsha, Anambra State, Nigeria. Epoxy resin (LY556) has good impregnation properties and exhibits excellent mechanical, dynamic, and thermal properties. It also exhibits extremely high resistance to alkali and good solvent resistance (Raju and Kumarappa, 2012).

**Oil bean pod shells (OBPS) particulates:** The oil bean pod shells used for this study were collected from the premises of Delta State Polytechnic, Ozoro, Nigeria. They were sundried for two weeks before they were ground into particulates using a Burr mill. The particulates were sieved with a 300 µm stainless steel sieve.

**Raffia palm fibres:** The raffia palm fronds, from which the fibres used for this study were prepared, were obtained from the forest of Delta State, Nigeria. The fibre is the thin
membrane located on the underside of the raffia palm frond. This fibre (membrane) was separated from the raffia palm frond manually and air-dried in the laboratory for five days (Figure 1), before the alkali treatment. The dried but untreated fibres were designated as control fibres.

Figure 1: Untreated raffia fibres

METHODS

Alkali treatment (modification) of the raffia palm fibres: To reduce the effect of moisture absorption on natural fibres and improve their mechanical properties, as well as provide better adhesion with the matrix, the fibres were treated with NaOH solution. The raffia palm fibres were treated with sodium hydroxide (NaOH) aqueous solution, at five concentration (5, 10, 15 and 20 wt%) levels for four treatment duration (60, 90, 120 and 150 minutes) at ambient temperature. After each treatment, the fibres were neutralized with distilled water containing 1% H₂SO₄ acid for 10 min, and then rinsed severally with fresh tap water. The treated fibres were then air-dried at ambient temperature for 24 hours in the laboratory; then oven dried at 60°C for another 6 hours, before they were used to prepare the composite sample. The fibres were cut into 5 mm size before they were used to prepare the composite samples.

Composite samples preparation: The composite matrix was prepared by mixing epoxy resin and hardener in a ratio of 7:3 (wt%). While the hand-lay-up technique, which was followed static compressive loading was employed to prepare the composite samples. The composite samples were prepared by partial reinforcement of the matrix with 30% combination of raffia palm fibres (RPF) and OBPS particulates in the ratio of 1:1 (by weight). To ensure the fast release of the composite sample from the mould, a thin film of wax was applied to the mould before the casting. During the composite samples preparation, measured quantities of the RPF (uniform length of 5 mm), OBPS particulate and resin were poured in a plastic container and stirred thoroughly for 20 minutes to obtain a homogeneous mixture. Then the hardener was poured into the mixture and stirred thoroughly for another 10 minutes before the mixture was poured into the already prepared mould. The casted composite was compressed uniformly using a roller to further expel any trapped air within the mixture, kept under a 25 kg load for 24 hours to expel any entrapped air within the composite before demoulding. The composite samples produced were later cured in the laboratory at ambient temperature for 21 days. Composite samples used for this study were produced with five different NaOH concentrations (5%, 10, 15% and 20%), and their corresponding treatment duration (60, 90, 120 and 150 minutes).

Mechanical testing

Tensile Test: The tensile test of the composite samples was carried out by using the Universal Testing Machine (UTM) (Testometric model). The test was done following the ASTM D638 standard procedure. During the testing process, the machine gripped each end of the sample, slowly pulling it until fracture occurred (Figure 2). The dimension of each sample was 150 mm × 30 mm × 3 mm. At the end of each test, the tensile strength and Young’s modulus of the sample were calculated automatically by the machine and results displayed on the screen. Five samples were tested, and the average values of tensile strength and Young modulus were calculated. The tests were carried out at room temperature (27±3°C).

Flexural Test: Three-point bend tests were performed by using the Universal Testing Machine (UTM) (Testometric model), in accordance with ASTM D790M-81 recommended standard. The dimension of the sample was 100 mm × 30 mm × 5 mm. At the end of each test, the transverse rupture strength and flexural modulus of the sample were calculated automatically by the machine and results displayed on the screen. Five samples were tested and the average value was recorded. The tests were carried out at room temperature.
RESULTS AND DISCUSSIONS

Tensile properties

**Tensile strength:** The effects of alkali concentration and treatment duration on the tensile strength of the raffia palm fibres/oil beam pod shell particulate epoxy composite are presented in Figure 3. The results presented in Figure 3 showed that the NaOH treatment of the fibre significantly improved the tensile strength of the composite produced. The tensile strength increased from 10.672 MPa (for the untreated fibres composite) to 26.056 MPa (for the fibres treated with 15% NaOH for 120 minutes). As shown in Figure 3, prolong treatment of the fibres at higher NaOH concentration had a negative effect on the tensile strength of composite samples produced. The best tensile strength results were obtained between the treatment condition of 10% and 15% NaOH concentration, while further increment in the NaOH concentration (20%) led to an average decrease in the tensile strength of the composite samples. This could be attributed to the destruction of the fibre cells wall by the stronger alkali solution, and the inability of the fibre cells wall to withstand the alkali solution for longer period of time. Our result is in a similar trend with the previous studies by Mir et al. (2012) and Azeez et al. (2016). Azeez et al., (2016) reported improvement in the tensile behaviour of Combretum dolichopetalum fibres after NaOH and acetic anhydride modification. Besides, Salim and Sorya, (2015) reported that the tensile strength results obtained from alkali-treated Spartium junceum fibre composites were higher when compare with the results obtained from untreated Spartium junceum fibre composites. Girisha (2012) studied the tensile strength of as sisal-coconut spathe, sisal-ridge gourd, and coconut spathe-ridge gourd hybrid composites, and observed an increase in the tensile strength of the composites as the fibres volume varied from 5% to 30 wt%. Tensile strength is the ability of a material to withstand a pulling force; it is one of the most important properties of materials used in structural applications.

![Figure 3: Tensile strength of the epoxy hybrid composite after alkali treatment](image)

**Young’s Modulus:** The results of the Young’s modulus of the composite samples are presented in Figure 4. With reference to Figure 4, Alkali treatment of the raffia palm fibres significantly improved the Young’s modulus of the composite samples. The highest Young modulus (6.352 GPa) was obtained at treatment condition of 15% NaOH for 90 minutes. As shown in Figure 4, the Young’s modulus of the composite samples declined between treatment conditions of 15% and 20% NaOH concentration; and was higher for the 150 minutes treatment period, when compare with the results obtained for the 60 minutes treatment period. From the results of the study presented in Figure 4, it can be observed that at treatment duration of 150 minutes, the Young’s modulus started declining from 10% NaOH concentration, portraying the unsuitability of the fibres to long treatment periods. Mir et al. (2012) observed that the Young’s modulus of chemically modified coir fibre was higher (3.83 GPa) when compared with the results of raw (unmodified) coir fibre (3.69 GPa). According to Boopathi et al., (2012) alkali treatment caused the cellulosic content of the fibres to swell, removing all the impurities from their surfaces during the process. This will lead to a better fibre wetting characteristics and fibre-matrix adhesiveness in composite production; making composites produced from them (the fibres) to have improved mechanical properties (Boopathi et al., 2012). Cai et al. (2015) reported that the Young’s modulus of the abaca fibres treated with 5% NaOH solution for 30 minutes was increased by 41%. The increase in tensile properties may be attributed to surface removal of amorphous constituents (lignins, pectins, hemicellulose, and other impurities) as reported by researchers (Bouatay et al., 2014; Salim and Sorya, 2015).

![Figure 4: Young’s modulus of the epoxy hybrid composite after alkali treatment](image)

**Flexural properties**

**Transverse rupture strength:** The transverse rupture strength of composite samples prepared with varying concentrations of NaOH and treatment duration are...
presented in Figure 5. As shown in Figure 5, the composite samples produced with treated fibres showed a general improvement in their transverse rupture strength, when compared with untreated fibres. The transverse rupture strength improvement of the composite samples became more predominant with increasing NaOH concentration and treatment duration. The composite samples prepared with fibres treated with 20% NaOH showed maximum improvements at 20 minutes treatment duration. The transverse rupture strength of the composite produced with fibres treated with 20 wt% NaOH after 60 minutes was 43.87 MPa in contrast to 23.89 MPa for the composite produced with untreated fibres. With reference to the results presented in Figure 5, 15% NaOH showed the maximum prospect in fibres treatment; the fibres were able to maintain good flexural properties, at longer treatment duration (90 – 120 minutes). It is advisable to use lower NaOH concentration (5 and 10%), at long treatment duration (150 minutes). Similar observations for alkali-treated jute reinforced composites were reported by Ray et al. (2001). According to Ray et al., the maximum transverse rupture strength was obtained at 4 hours treated fibres composites at 35 wt% reinforcements; after which, the transverse rupture strength started to decline with an increase in the treatment period.

**Figure 5:** Transverse rupture strength of the epoxy hybrid composite after alkali treatment

**Flexural modulus:** The results of the flexural modulus of the composite prepared from fibres by varying their treatment duration (60 to 150 minutes) and alkali concentration (5% to 20% NaOH solution) are presented in Figure 6. From the results, the composites prepared with fibres treated with 20% NaOH for 60 minutes showed the maximum improvement (3.386 GPa) for all treatment duration. The improvement in the flexural modulus of the composite was more predominant at 15% NaOH concentration with the treatment duration ranging from 60 minutes to 120 minutes. The flexural modulus of the composite prepared with fibres treated with 5% NaOH for 60 minutes had the poorest improvement (1.342 GPa) when compared with the results obtained from all the composites prepared with treated fibres. As seen in the result (Figure 6), as the treatment duration increased from 60 to 150 minutes, for 5% and 10% NaOH concentration, there was a gradual improvement in the flexural modulus of composite samples. But at 15% and 20% NaOH concentration, an increase in the treatment duration resulted in a decline in the flexural modulus of the composite samples. Ray et al. (2001) reported the same trend for the flexural modulus of treated jute fibres composites. According to Ray et al. (2001), as the treatment duration of fibres increase at higher alkali concentrations; the fibres became more rigid but brittle. Therefore, when composite produced from them are subjected to stress, they suffered breakage easily due to their brittleness, thus lowering their strength. The increase in tensile properties may be attributed to surface removal of amorphous constituents (lignins, pectins, hemicellulose and other impurities) as reported by researchers (Bouatay et al., 2014; Salim and Sorya, 2015). Optimizing the treatment conditions of the raffia palm fibres will certainly enhance their mechanical properties, thereby eliminating time and material wastage.

**Figure 6:** Flexural modulus of the epoxy hybrid composite after alkali treatment

**CONCLUSION**

This study was conducted to evaluate the effect of alkali treatment on some mechanical properties (tensile strength, Young’s modulus, flexural modulus, and transverse rupture strength) of raffia palm fibres/oil bean pod shell particulates epoxy hybrid composite. The raffia palm fibres were treated with 5, 10 15 and 20% NaOH solution for a duration of 60, 90, 120 and 150 minutes. Results obtained from the study shown significant improvement in the mechanical properties of the hybrid composite samples produced with treated fibres. The tensile strength increased from 10.672 to 26.437 MPa, Young’s modulus increased from 2.514 to 5.831 GPa, Transverse rupture strength increased from 23.897 to 43.873 MPa, while the flexural modulus increased from 0.784 to 3.386 GPa as the alkali concentrations increase from 5% to 20%. On average, the best results were
obtained at 15% NaOH concentration after 60 and 90 minutes treatment. The results obtained from this study showed that optimizing the treatment conditions of the raffia palm fibres will certainly establish their mechanical properties, thereby eliminating time and material wastage.

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