
EFFECT OF ALKALI TREATMENT ON THE PHYSICAL AND MECHANICAL PROPERTIES OF OKRO BAST FIBRE/EPOXY RESIN COMPOSITES

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ABSTRACTS

In recent years the development of polymer composites made from organic resources in the various sectors is increasing considerably due to the environmental issues and health hazard posed by synthetic fibres during disposal and manufacturing. Natural fibres are rapidly being used as the reinforcement material in polymer matrix composites due to their advantages like low cost, availability, low density, good mechanical properties, environment friendly, higher stiffness, and biodegradability characteristics. In present study, Okro bast fibres(OBF) were treated with 5% sodium Hydroxide and then used to fabricate composite using Epoxy Resin (ER) as the matrix material. OBF weight fractions of 3%,6%, 9%, 12% and 15% were used as the filler materials for all the composites. Composites Preparation and testing were conducted according to the ASTM standards. Tensile, hardness, impact, flexural strength and water absorption tests were conducted to find the mechanical and physical behaviour of the composites.

Keywords: *Epoxy Resin, Okro Bast Fibre, Alkali Treatment, Fibre Loading, Physical and Mechanical Properties.*

INTRODUCTION

The increasing demand concerning global warming and depleting petroleum reserve has led researchers and scientist to focus on the use of natural fibres as bio-fillers in thermosetting and thermoplastic polymers. Unlike the synthetic fibre, natural fibres are capable of imparting certain benefits to the composites such as low cost, high strength, low density, biodegradability and high degree of flexibility as ascertained by Sathish, *et al.* (2018). The natural fibres in their own quality have a positive impact to the environment which play a role in the emerging "green" economy based on energy efficiency. The use of natural fibre in composites adds

value to the material thereby reducing the rate of carbon emission in the environment.

However, natural fibres yield poor mechanical properties when used as fillers in thermoplastic or thermo set due to incompatibility between the fibre and the matrix. But with modification and advancement of polymer matrix composites, the mechanical properties tend to improve with addition of one or more fillers in the properties of composites. Natural fibre such as Okro, flax, ramie and cotton are used in place of synthetic fibres as reinforcement in polymer composites. Other fibres being used are hemp, kenaf, sisal, bamboo and jute.

Okro (*Abelmoschus esculentus* (L.) Moench), also known as *Hibiscus esculentus* L., is a member of the mallow (*Malvaceae*) family, which includes hibiscus and cotton among other species, it can be found as a tall-growing (2m tall and has leaves 10–20 cm long and broad, with lobes ranging from 5 to 7), warm-season annual or perennial (in India and Africa) that is well suited to a wide range of soil types (Shamsulalam and Arifuzzaman, 2007) It represents the only vegetable crop in the Malvaceae family, whose products have significant use in the food sector. In several parts of the world, it is known as Okro, Quingumbo, Lady's finger, and a variety of other names. The origin of Okro is disputable, but it seems to be native to the Abyssinian centre of origin of cultivated plants, an area that includes Ethiopia, Eritrea and the eastern part of the Anglo-Egyptian Sudan. It is currently grown throughout tropical Asia, Africa, the Caribbean and southern United States (Shamsulalam and Arifuzzaman, 2007). The chemical composition as well as the morphological microstructure of vegetable fibres is extremely complex due to the hierarchical organization and the different compounds present at various concentrations. The vegetable fibres are mainly composed of cellulose and non-cellulosic materials, such as hemicelluloses, lignin, pectin, waxes, and some water-soluble compounds. The lignin and pectin act as bonding agents (Muhanti *et al.*, 2005). Cellulose 67.5 % Hemicellulose 15.4 % Lignin, 7.1% pectic substances 3.4 %, waxes and fat 3.9 % water 2.7 % (Alam and Khan, 2007).

Okro bast fibre (OBF) obtained from the stem of the okro plant has extensively been used as a filler in the fabrication of biodegradable composites. Arrifuzaman *et al.*, (2014), in their study observed that as the filler content of OBF increases, tensile strength, young's modulus and

flexural strength of okro bast fibre/phenol formaldehyde resin (OBF/PFR) composites increases. Results suggested that the appropriate percentage of OBF in the composite is 29 wt%, but a larger amount of OBF may decrease the tensile strength and flexural strength of OBF/PFR composites. However, the presence of hydroxyl groups in OBF also increases water absorption and results in poor compatibility between the OBF and the hydrophobic phenol formaldehyde resin (PFR), but grafting hydrophobic character on the OBF increases its compatibility with phenol formaldehyde resin (PFR). Alkali treatment and bleached OBF are well distributed in PFR and gave higher mechanical properties of the prepared composites. The strength of natural fibre reinforced composites does not only depend on the matrix but also on a number of parameters such as, fibre orientation, fibre-matrix compatibility, volume weight fraction of fibres and the rate of aspect ratio. To this end, the objective of the present research work is to study the effect of okro bast fibre fillers on the mechanical and physical properties of epoxy composites.

MATERIALS AND METHODS

Extraction of Fibre

The Okro stems were obtained from a small farm in Getso, Gwarzo local government area of Kano state. The fibres were extracted by water retting method. The okro bark was bundled in ribbon form and was immersed in water retting bath, little pressure was applied to the soaked bundle to ensure that the bark was fully submerged for a period of 10 days during which the cementing materials such as pectin, lignin, and hemicellulose must have loosened and softened. On 10th day, the retted ribbon was removed and washed with sufficient quantity of water until the pulp is completely detached from fibres; the fibres were shredded and combed to have finer fibres. Then the fibres were dried at room temperature for a period of fifteen days (15). After drying, the fibres were chopped into short lengths.

Alkaline Treatment of Okro Fibre

Sodium hydroxide solutions by weight concentration of 5%, was prepared using distilled water and sodium hydroxide. The Okro fibres were soaked in 5% sodium hydroxide solution at a temperature of 60°C for 2 hours with continuous stirring. After the stipulated period, the fibres were removed from the solution and rinsed with sufficient amount of water, then each batch of the treated fibres were neutralized with 1%

acetic acid and finally rinsed with distilled water. The fibres were allowed to dry at room temperature.

Fabrication of OBF/ER Composites

Short okro bast fibres were used for the composite's preparation using hand layup technique. This was achieved by mixing the various ratios of the prepared fibres (OBF) (3%,6%, 9%,12% and15) with the epoxy to form homogenous blends as in table 1. The mixing was achieved by manual stirring method for 10 -15 minutes, then the hardener was 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 200 mm x 100 mm x 4.00 mm overlaid with aluminium foil to serve as releasing agent. The mixture was allowed to cure at room temperature for 24hrs before removal from the mould. The composites were then cut based on characterizations in accordance with ASTM standard.

Table 1: Formulation table for OBF/ER composites

S/N	OBF fillers (wt%)	ER matrix (wt%)	Total (%)
1	0	100	100
2	3	97	100
3	6	94	100
5	9	91	100
6	12	88	100
7	15	85	100

OBF= okro bast fibre ER= epoxy resin

CHARACTERIZATION OF THE COMPOSITES

The characterization of the composites was performed inline with ASTM standards for testing of materials the composites were conditioned at room temperature and then subjected to various test.

MECHANICAL PROPERTIES

Tensile Properties

The tensile test was carried out using tensile properties tester (TM2101-T7 Multifunctional Electronic Fabric Strength Machine) according to ASTM D638 with maximum force of 10 KN. The samples dimensions were 100 x15 x 4 (mm) length, width and thickness respectively. A crosshead speed of 2 mm / min was used. The test specimens were held in the grips of the testing machine and tightened evenly and firmly to prevent any slippage as the test commenced. The resistance and elongation of the specimens were detected and recorded by load cell until a failure or rupture occurred. From the tensile test, tensile parameters (tensile strength (breaking point), elongation at break and tensile modulus) were determined and recorded.

FLEXURAL PROPERTIES

Izod Impact Test

The Impact test is usually carried out to determine the energy needed to initiate fracture and continue until the specimen is broken at a certain point in time. It is a test that determines the resistance of the material to impact from a moving pendulum. Izod test is used to identify the overall toughness of a material. The procedure involves specimens made with a notch which produces stress and concentration that increases the possibility of brittle failure. The notch in the specimen reduces or minimizes plastic deformation and direct fracture of the part behind the notch. The specimen is clamped into the fixture with the notched side facing the edge of the pendulum. The pendulum is allowed to hit the specimen. The Izod impact test was performed as per ASTM D256 with a standard specimen size of 64mm x 1.27 mm x 4 mm length, width and thickness. Impact strength was measured by dividing impact energy in joules by the thickness of the specimen. The greater number indicates the toughness of the material.

HARDNESS TESTING (VICKERS HARDNESS)

The sample hardness was measured using (Vickers Hardness Tester MV1-PC, Mh-v CM. 07/2012-1329) with maximum capacity of 0.1 Kgf (150 HV) in accordance with ASTM D2240. The test was carried out at temperature (23 ± 2 °C). The specimen dimension was 30 x 10 x 4 (mm). The specimen was mounted on a specimen compartment and then the indentation point was focused thereafter five different points were

indented on each specimen and the hardness values were recorded. The average of the five readings were calculated and recorded.

PHYSICAL PROPERTIES

WATER ABSORPTION

Water absorption was carried out according to ASTM D570. The samples were conditioned in an oven at 45 °C for 72 hours. Then, placed inside desiccators for 24 hours and finally weighed (W_1) using metler weighing balance. The weighed samples were then immersed in a plastic container containing water for 24 hours. The samples were removed from the water, wiped with a clean cloth to eliminate moisture, then re-weighed (W_2) and the process continues for thirty (30) days.

RESULTS AND DISCUSSION

MECHANICAL PROPERTY

TENSILE STRENGTH

The result of tensile strength of untreated and treated Okro Bast Fibre/ER composites as shown in figure 1. The figure clearly shows the difference in tensile strength between the treated OBF and the untreated OBF/ER composites. The untreated OBF/ER composites were observed to have lower strength than the control sample and some of the treated OBF composites. However, the tensile strength of the untreated fibre decreases with increase in fibre content due to the presence of the cementing materials which lead to poor fibre matrix interfacial bond, it could also be due to formation of agglomerate which resulted to poor adhesion and non-uniform stress transfer within the interface of the formed composites. Sule et al., (2019), and Ali et al., (2016) reported similar findings.

Zin et al., 2018 also observed that the tensile strength of untreated pineapple leaf fibre is lower than the treated fibre. The alkali treatment resulted in increasing trend of the tensile strength properties of PALF/Epoxy composites, until the optimum point is reached where the tensile strength was observed to decline which could be due to excessive removal of waxy layers and lignin of the fibre in high alkaline concentration, resulting in weaker or damaged fibre. The result for treated okro bast fibre / epoxy resin composites shows that the sodium hydroxide treatment has played a vital role in improving the strength of the fibre by creating rough surface which led to better interaction between the fibre and the polymer matrix. According to Gassan and Bledzki

(1999) alkali treatment interfered with hydrogen bonds in the chemical structure of the fibres, thereby increasing the fibre surface roughness, which in turns promote crystallinity index of fibres, enhancing formation of hydrogen bonds between cellulose chains and chemical bonding between the fibres and the matrix of the composites. This is in line with the result obtained by Cao *et al* (2006) in their findings on mechanical properties of biodegradable composites reinforced with bagasse fibre before and after treatment. More so, tensile strength is affected by volume fractions, degree of adhesion between the filler and the matrix, level of dispersion of the filler and matrix and surface related defects. The increase in tensile strength of OBF/ER could also be due to better stirring during production process which led to better interaction, or good interfacial bond between the fibre and the matrix. The decrease in tensile strength of TOBF/ER with higher filler content resulted to weak adhesion between the filler and that of the matrix which accounts for the reason why the tensile strength 12% and 15% filler loading of treated okro bast fibre were lower than the control (neat) epoxycomposites. However, at 12% and 15% filler loading weight fraction a significant decrease in tensile strength was noticed which led to greater possibility of weak locations in the composites which caused poor interfacial bound between the filler and the matrix at the interface when tensile forces were applied.

Similarly, the control sample has the highest value of 48.82 MPa, the values obtained from 3%,6%,9% and 15% are 32.6,33,34.6,32.2 and 31.3 MPa. The values for treated OBF/Epoxy resin composites increase with increase in filler content to a certain level where it decreases with increase in filler content. Hazarika *et al.*,2015 reported that alkali treatment leads to an increased fibre surface roughness because of the removal of the non-cellulosic fibre components. He further stated that rough fibre surface improves the interlocking tendencies with matrix, thereby resulting in improvement of a stronger fibre matrix interfacial strength of a composite that will result to an improved mechanical property of the composite. Similar result was reported by Sugiman *et al.*,2019 in their research titled effects of alkali treatment of bamboo fibre under various conditions on the tensile and flexural properties of bamboo fibre/polystyrene-modified unsaturated polyester. Researchers such as Tezaraet *al.*, (2022) Ayyavoo, Kulendran, Anbarasu (2014) and Sudhakar, Dr. Naresh P, Sandip (2018) reported similar result.

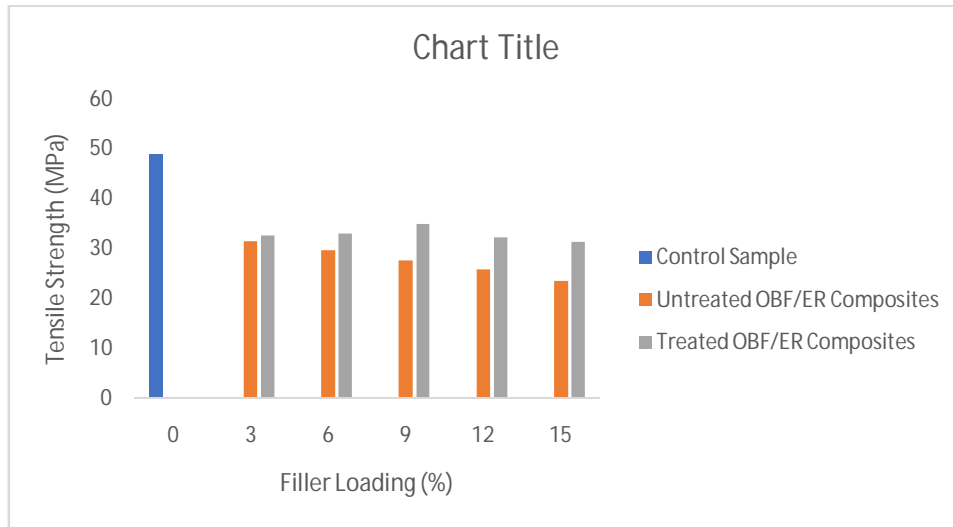


Figure 1: Tensile Strength of Untreated and treated Okro Bast Fibre/Epoxy Resin Composites

Tensile Modulus

Figure 2 illustrates the results of impact strength of OBF/ER composites, it can be seen that the modulus increased with increase in filler loading up to about 9% filler loading for treated OBF/Epoxy composites. The maximum values of 6.35 GPa was obtained for treated OBF/Epoxy composites which is higher than the control sample and the untreated OBF/ER composites. The incorporation of the fibre (OBF) into the matrix attributed to the stiffening effect and rigidity to the composites. Nitya (2016), observed that composite structure with high modulus and high tensile strength yield more rigid structure. Fibre treatment impacts better adhesion between the fibre and the matrix which increased the stress involved to break the samples and consequently increased modulus. Composites of OBF/Epoxy modulus is higher than that of the control sample with the values of 5.90, 5.9, 6.35, 5.33 and 5.15 GPa for composites filled with 3%, 6%, 9%, 12%, 15%, 18%, and 21% respectively. This increase in modulus property could be attributed to a proper adhesion, compatibility and interaction between constituent OBF and the matrix. Bello et al., (2021).

However, a decrease in modulus was observed for the untreated OBF/ER composites which showed that the modulus decreases with decrease in filler content and attain its maximum value at 6% filler loading with a value of 4.5 GPa. At 12% and 15% filler loading a remarkable decrease in

modulus was noticed this may be due to uneven fibre distribution in the matrix that led to poor interfacial adhesion and increase in the possibility of stress centers leading to premature failure of the composites. Ahmad et al.,2015 also reported that variation in the modulus observed in the various researches could be attributed to the variation in the weight fractions of the fibres, matrix and the nature of the fibres used.

According to Sair et al.,2017 in their findings observed that alkali treatment improves both tensile and modulus of hemp fibres. They also stated that the observed increase in mechanical properties of hemp fibre is due to the partial removal of hemicellulose and lignin which causes the increase in the crystallinity index of the fibres and therefore the fibre become more rigid. However, the removal of a certain amount of lignin and hemicellulose allows relaxation of the microfibrils and their reorganization along the principal axis of the fibre which gives rise to a more rigid structure. The presence of hemicellulose, lignin and other impurities on the untreated okro bast fibre resulted to weak adhesion which in turn resulted to poor bonding between the filler and the matrix.

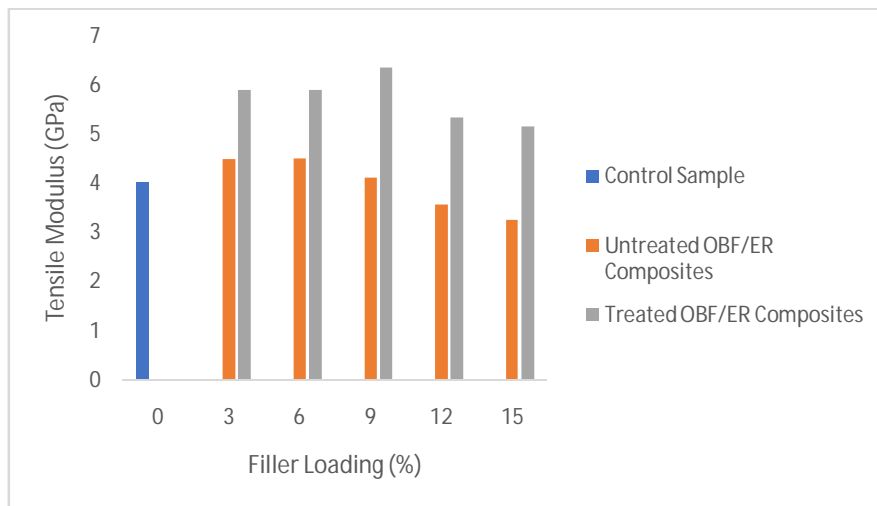


Figure 2: Tensile Modulus of Untreated Okro Bast Fibre/Epoxy Resin Composites

FLEXURAL STRENGTH

Figure 3 gives the Flexural Strength values of treated and untreated OBF/ER composites.

From the results, it can be seen that sodium hydroxide treated okro fibre composites of epoxy have superior flexural property when compared to untreated fibre composites. This shows that the fibre treatment has improve the adhesive character in combination with the matrix which promotes good wet ability and enhance bonding of the fibre in the polymer matrix. Arifuzzaman et al., (2014) observed that NaOH treatment greatly improve the flexural strength as well as the tensile strength of the okro bast fibre reinforced phenol formaldehyde composites. After the NaOH treatment fibre surface became rougher which gave better interlocking between the filler and the matrix which led to better flexural property than the untreated okro fibre reinforce phenol formaldehyde resin composites. The result obtained for treated OBF/epoxy composites shows that the flexural strength of the composites increased with increase in fibre loading and then decrease with higher amount of fibre volume fraction having its highest value at 9% fibre content with a value of 78.9 MPa, the increase in strength could be attributed to improved adhesion that assist in providing adequate stress transfer between the matrix and the fibre. The improvement in flexural strength could be due to surface modification which creates rough surface topography there by resulting to effective stress transfer from the fibre to the matrix.

Similar behavior was reported by Sugiman et al., (2019) they observed that flexural strength and modulus increases with treatment of bamboo fibre/polystyrene-modified unsaturated polyester composites. Nadendla (2015) also reported that flexural strength, flexural modulus and impact strength increase with increase in filler loading of okro fibre reinforced with unsaturated polyester resin composites. Silas and timothy (2020) reported that alkali treatment increases crystallinity index of fibres, enhancing formation of hydrogen bonds between cellulose chains and hence chemical bonding between the fibres in composites which may be responsible for increase in flexural strength.

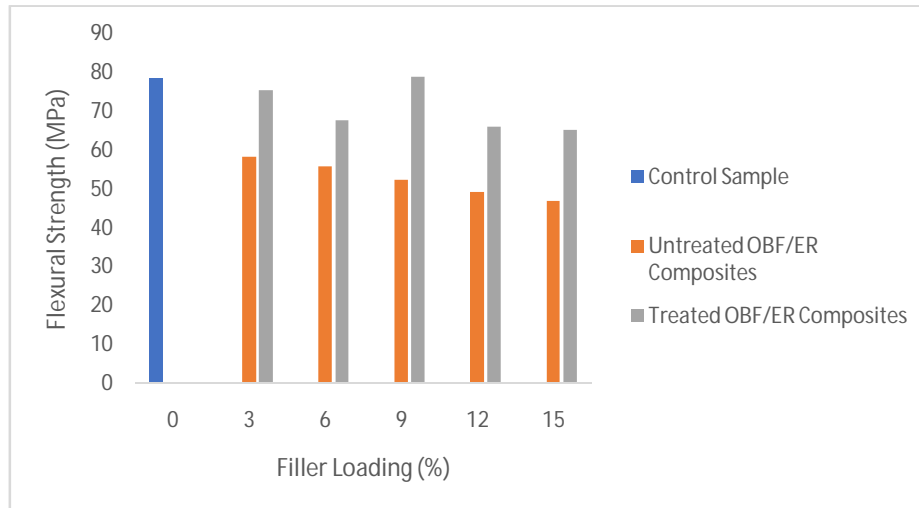


Figure 3: Flexural Strength of untreated and treated Okro Bast Fibre/Epoxy Resin Composites

Impact Strength

From the result, it was observed that a positive treatment effect was observed with the treated okro bast fibre composites compared to the untreated okro bast fibre/Epoxy resin composites. The untreated OBF/ER composites showed a decreasing trend that is the impact strength decrease with increase in fibre loading. The observed decrease could be due to poor compatibility that exists between the fibre and the matrix, in addition to voids and agglomeration of the fibres that occur with higher percentage of fibres in the polymer matrix. However, with alkali treatment impact strength of OBF/ER increased with increase in fibre loading. This increase may be attributed to the increase in the fibre surface roughness, resulting in better mechanical interlocking.

Suradiet al., (2010) reported that impact strength of modified oil palm fibre composites presented better and promising result that unmodified fibre composites as stated in their study on Oil Palm Bio-Fibre Reinforced Thermoplastic Composites-Effects of Matrix Modification on Mechanical and Thermal Properties. The impact strength of TOBF/ER composites shows that at 9% fibre loading, an optimal impact strength was observed with a value of (0.36 J/m²). But as the fibre loading increases, the impact strength starts increasing at the initial stage but further decrease with increase in fibre loading. The impact strength was reduced to 0.27J/m², 0.26J/m² for OBF/ER at 12% and 15% with higher fibre loading,

resulting in lower impact strength compared to other composites with 3%,6% fibre loadings.

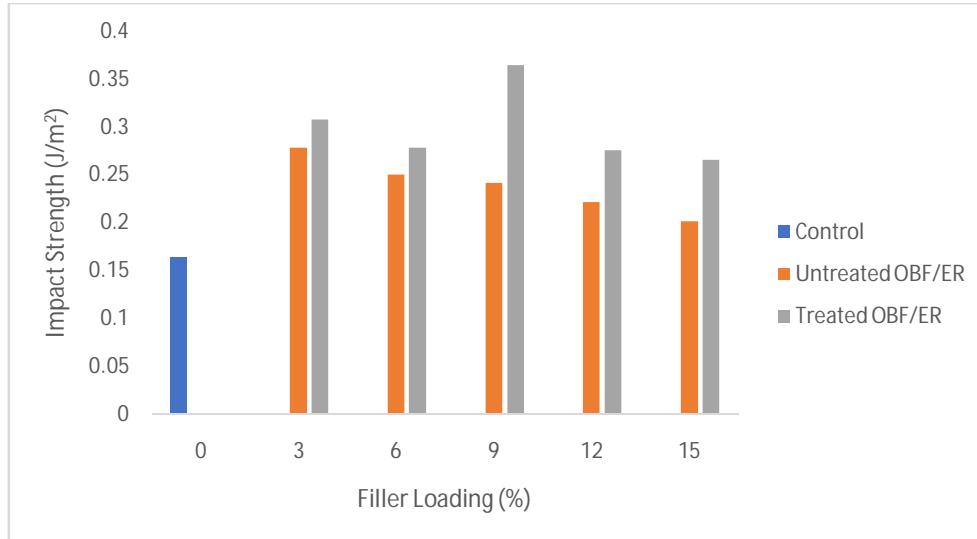


Figure 4: Impact Strength of Untreated and treated Okro Bast Fibre /Epoxy Resin Composites

Hardness Test

Hardness is the measure of wear resistance of any materials to surface indentation, which also serve as a function of stress required to produce a specific type of deformation and values obtain are used to evaluate or estimate the mechanical strength of each composite. Figure 4 illustrate the hardness values of treated and untreated OBF/ER composites. It can be seen that the hardness value of all the treated and untreated composites of OBF/ER increases with increase in fibre loading, the highest value indicates greater resistance of the composites to indentation. However, an increase in hardness value of treated OBF/ER composites was noticed with increase in fibre loading content with the observed values of 63 to 84.5Hv all values observed are greater than that of the control/Neat sample and the untreated OBF composites. The increase in hardness value could be attributed to the increase in stiffness, uniform fibre dispersion, minimisation of voids and stronger interfacial bonding between the matrix and the fibre as a result of alkaline treatment (Rajesh Kumar 2020).The decrease in hardness value could be attributed to poor/weak interfacial bonding between the OBF/ER composites. On introduction of OBF into the epoxy matrix air may be trapped inside

which leads to voids formation in the interface under loading and non-uniform stress transfer due to the fibre agglomeration in the matrix. (Sanjay et al., 2009). The values for untreated OBF/ER are lower than that of treated OBF/ER composites. This shows that the surface modification has increased the fibre stiffness and enhanced the interfacial bonding between the fibre and the polymer matrix. Similar result was reported by Sudhakar, Naresh and Sandip (2018) in their findings on study of mechanical properties of bamboo fibres before and after alkali treatment.

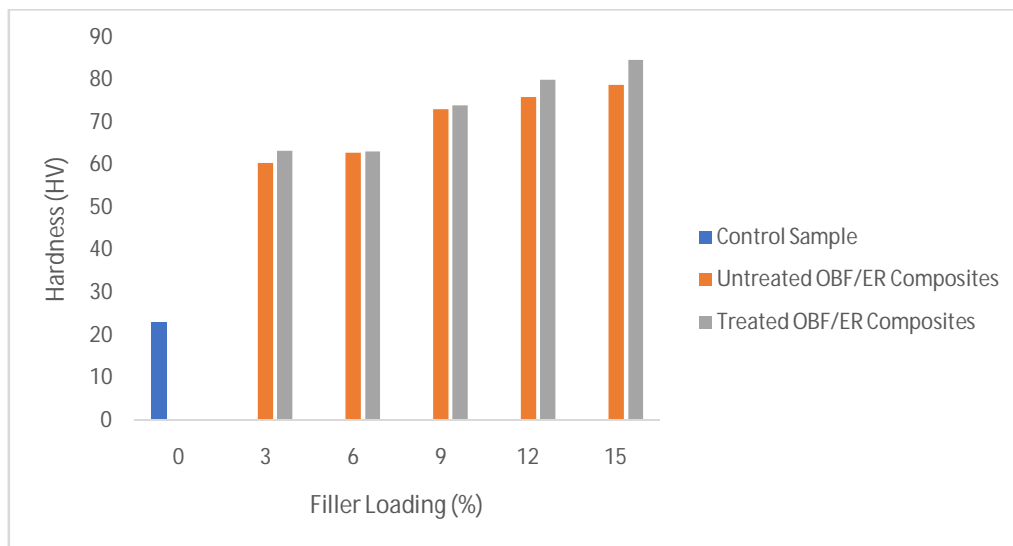


Figure 5: Hardness Value of Untreated and Treated Okro Bast Fibre/Epoxy Resin Composites

PHYSICAL PROPERTIES

Water Absorption

In order to find out the quantity of water absorbed by the composites water absorption test was carried out to find out the quantity of water absorbed for thirty (30) days. Water absorption depends on certain parameters such as matrix, fibre content/filler loading, method of fabrication and environment/ weather condition. Water absorption of treated and untreated OBF/ER composites as shown in figure 6 and 7.

The absorption of water by the fibre may be due to the presence of hydroxyl groups which absorb water through the formation of hydrogen bonding. It could also be due to the facts that natural fibre is said to be

hydrophilic while polymers are hydrophobic in nature. Both treated and untreated composites show increment in water uptake at the beginning 48 hours and later reached a saturation point where no water is absorbed after 22-30 days with the maximum water absorption at 15% filler loading for both untreated and alkali treated OBF/ER composites. The amount of water uptake was typical of Fickian diffusion, which explains that rapid water absorption takes place at the beginning of contact of matter with water and then subsequently, a saturation point is reached. However, OBF is a natural fibre that is hydrophilic and contains cellulose of hydroxyl groups which leads to water absorption. The low percentage of water absorption is attributed to the hydrophobic nature of epoxy resin and the effect of alkali treatment on the OBF. The alkali treated fibre reinforced epoxy composite shows lower water absorption rate this is because alkali treated fibre is less hydrophilic as number of hydrophilic hydroxyl groups were reduce by NaOH which leads to lower water absorption from substrate of composite. From the results in figure 6 and 7 (untreated and treated) it can be observed that 6% fibre loading absorbed more water than 9 and 12% fibre loadings. This behaviour could be attributed to the cracks or voids paving ways for water to diffuse into the core of the composites. Researchers such as Madhusudhan *et al.*, (2018) and Arifuzzaman *et al.*, (2014) reported similar findings.

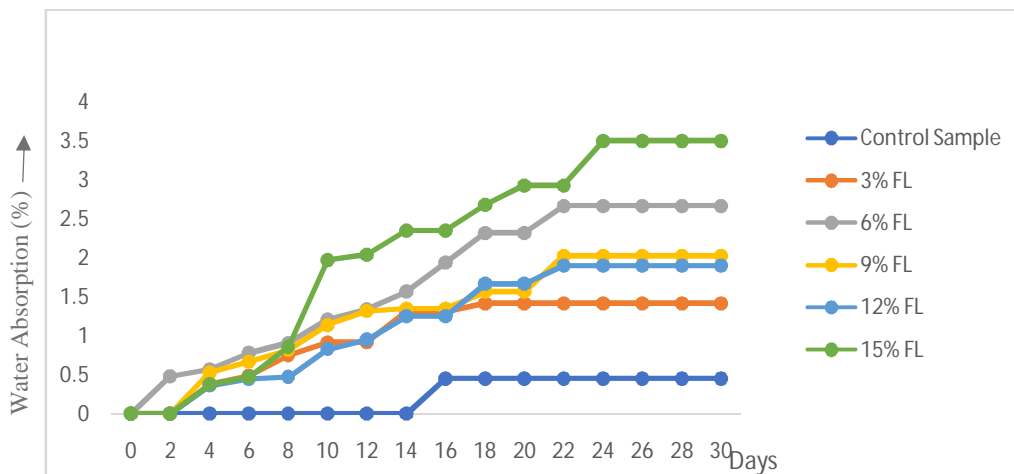


Figure 6: Water Absorption of Untreated Okro Bast Fibre/Epoxy Resin Composites

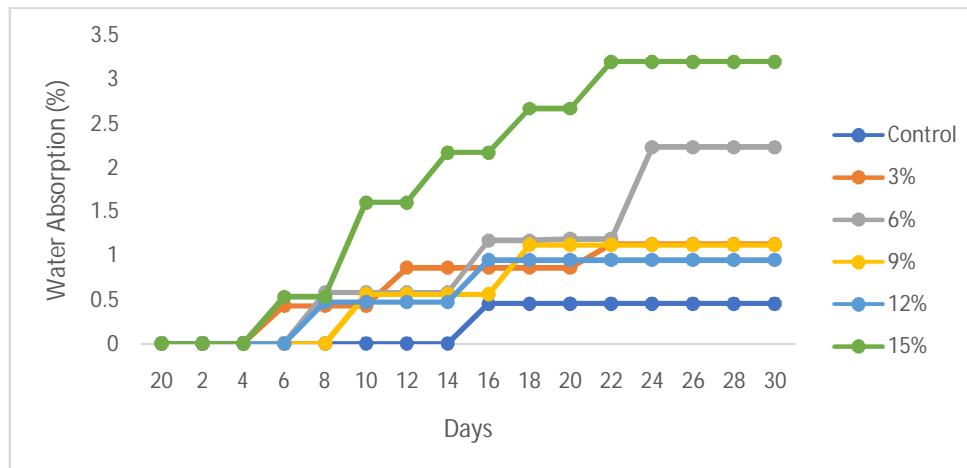


Figure 7: Water Absorption of Treated Okro Bast Fibre/Epoxy Resin Composites

CONCLUSION

Composites of OBF with ER were produced using the hand layup technique. The alkali treated OBF/ER composites were produced and analysed. The result shows that the addition of OBF into Epoxy resin enhances the mechanical properties of the formed composites. The hardness value of both treated and untreated composites increased with addition of fibre loading, the untreated OBF/ER composites absorb more water than the treated OBF/ER composites. The tensile strength, flexural strength and impact strength of the treated OBF/ER composites have their highest value at 9% fibre loading. OBF/ER composites could be used in producing materials that can find application in areas where much strength is not required. Applications like ceiling board, partition board/walls, can be the areas where OBF/ER composites can find useful applications. Similarly, with the recent findings value has been added to okro bast fibre by transforming waste OBF to wealth.

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