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ORIGINAL RESEARCH ARTICLE

EVALUATION OF THE MECHANICAL PROPERTIES OF SURFACE MODIFIED UKAM FIBRE REINFORCED POLYESTER COMPOSITES

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ABSTRACT

This work evaluated the properties of ukam fibre reinforced polyester composites. The ukam fibres were subjected to different surface treatments including alkaline, saline, permanganate and acetylation. Composites were produced at 5%, 10%, 15%, 20% and 25% fiber loadings and its mechanical properties determined. The results found that the most suitable mixture for excellent improvement in mechanical properties was obtained with the 25% fibre and 75% resin. Additionally, the results showed improvements in mechanical properties with surface treatments at all fibre loadings. Furthermore, it was observed that Fibre Surface treatment leads to compatibility which resulting in good load transfer between the modified fibre and matrix. Whereas acetylation treated composites exhibited the highest values of hardness at all levels of fibre loading, alkaline treated samples exhibited the highest tensile and compressive strengths at all fibre loadings. It is noteworthy that saline treated composites exhibited highest impact and flexural modulus of rupture. Interestingly, there was no significant improvement noticed with potassium permanganate treated reinforced fibre composites. Therefore, the results clearly showed that alkaline, saline, and acetylation fibre surface treatment methods could be used to produce ukam fibre composites with good mechanical properties for potential engineering applications.

1.0 Introduction

Natural fibres which are fibres obtained from plants, animals and mineral resources can be used as substitute for glass fibres in polymer composite materials because of the following advantages; low costs, low density, unlimited availability, biodegradability, renewability and recyclability (Okpanachi and Ogakwu, 2010) These characteristics makes natural fibres suitable for use as reinforcement for engineering polymer systems (Joffe et al., 2003) because natural fibre reinforced composites have comparable specific properties with glass fibre components and specific weight greater than synthetic fibre components (Westman et al., 2010). Similarly, a matrix reinforced with natural fibres provides an additional benefit because the properties of the plastic are markedly improved (Joffe et al., 2003). Conversely, the natural fibres possess some disadvantages which results to poor adhesion with the hydrophobic polymer matrix, propensity to coagulate during manufacturing and the tendency to soak up moisture which could affects its effectiveness significantly when used as a component in the reinforcement of polymers (Puglia et al., 2005).

Current research trends on fibre reinforcement methods have identified that surface treatments method which involves fibre reinforcement through adhesion and bonding between the fibre surfaces are potentially able to overcome the problem of incompatibility between fibre and matrix reinforced composites (Ghali et al., 2011). According to (Tserki et al., 2005) several modification processes on the fibre- matrix interfacial bonding have been proposed such as hydrophilizing agent coating, plasma modification, corona discharge and flame treatment (Molitor et al.,2001) and coupling agent (Pickering et al., 2016) These processes modify the membrane surfaces to increase the hydrophilicity, adsorption of molecules, ionic change and biocompatibility etc., without affecting their bulk properties(Zhang and Zhu, 2000). Also surface modification through efficient coupling of the fibre and matrix can be achieved by chemical treatment (Chen et al., 2011). In addition, chemical treatment other treatments such as alkaline, silane, acetylation and potassium permanganate can improve the adhesion of the fibre and matrix, reduces the hydrophilic nature of fibres and can be used in modifying the properties of natural fibres. The direct effect of the alkaline surface treatment is seen in the cellulosic fibril which leads to chemical reaction and the extraction of lignin and hemi cellulosic compounds (Jahn et al., 2002). As a result of silane treatment, siloxane bridge interlinking the fibre and the resin is created through the establishment of an interfacing region between the matrix and the fibre. This further prevents the fibre swelling into matrix through the formation of hydrocarbon chains (Sreekala et al., 2000). The Acetylation treatment ensures swelling of the fibre cell wall which prevents absorption of moisture resulting in improved dimensional stability and environmental degradation (Bledzki et al., 2008). While Potassium permanganate acts as an initiator for grafting and methylcrylate on fibres by Triathy et al. (1985) and Moharana et al. (1990).

Although surface treatment of fibres could lead to improve dynamic mechanical analysis of the composites produced (Dash et al., 2000) the compatibility of a fibre reinforced polymer composite are greatly affected by interfacial characteristics existing between the reinforcing fibers and the polymer matrix (Suardana et al., 2011). It is however, important that the reinforcement and resin used should be compatible with each other r with good adhesion to ensure that stresses are transferred, and thus carried by the fibre. Similarly, it can be detrimental if adhesion is beyond the desired requirement because this can make the composite brittle as the excessive adhesion between the fibre and the resin does not allow the growth of cracks along the fibre in an impact situation.

According to Idrus et al. (2011) increase in fibre loading hardness can be achieved by increasing saw dust content used in the composites however the author further stated that e treated saw dust exhibit better hardness performance compared to raw saw dust at all fibre loading from 10 wt. % - 30 wt. % composition. Ramires and Froilini (2017) has shown that an increase in fibre content used in the composites lead to a significant increase in the impact strength and further research results on fibre properties have also shown that some improved mechanical properties can be achieve by increasing the fibre contents in the composites (Ugoamadi, 2011, Romanzini et al., 2013).

Ukam fibres (*Cochlospermum planchonii*) are cellulose based natural fibres that has excellent mechanical properties, low density, abundant and renewable. However, not much work has been done on the surface modification of ukam composites. The work of (Okpanachi and Ogakwu, 2010) seems to be the only work on surface modification of ukam fibre polyester composites. In their study however only the axial and compressive strength of the composites were determined using alkali solution for surface treatment. The hardness, impact, flexural properties and surface treatment methods including saline, potassium permanganate and acetylation were not considered. It is important that comprehensive treatment procedures and studies be carried out on the composites to allow a better understanding of the micromechanics of these processes on the overall mechanical properties. Therefore, the aim of this study is to evaluate the properties of ukam fibre reinforced composites under different surface treatment.

2. Materials and Methods

2.1 Materials

The main materials used in this study are: Ukam fibres, Polyester resin, polyvinyl alcohol (PVA) while Potassium permanganate ($KMnO_4$), Acetic acid, Silae solution, Sodium hydroxide, were used for chemical treatment of the ukam fibres. Also, Cobalt octane was used to act as accelerator, Methyl ethyl Ketone peroxide (MEKP) (1 % by volume) as catalyst. The modified fibres were used to produce the composites which were then evaluated for mechanical properties including hardness, tensile, compression, impact, and flexural strengths.

2.2 Fibre Surface Treatment

2.2.1 Alkali treatment

Sodium hydroxide solution was used in the alkaline treatment in accordance with Liu et al. (2009). The Ukam fibres were then soaked in NaOH of 5 % concentration at room temperature for 24 hours. To remove any alkali solution sticking to the fibre surface the fibers were washed severally with water which has been neutralized with dilute acetic acid and then washed again with water.

2.2.2 Silane treatment

Silane solution was dissolved in water and used for the silane treatment following Valadez et al. (2009). After the treatment process, Ukam fibres were immersed in silane dissolved in a water-ethanol mixture for 3 hours. The Potential of Hydrogen of the solution was 9.0. This contains 60 % ethanol and 40 % water mixed thoroughly which was allowed to stand for an hour.

2.2.3 Potassium permanganate ($KMnO_4$) treatment

0.5 % of potassium permanganate in acetone was used in soaking the alkali treated fibres for 30 minutes after it has been thoroughly washed with water. Distilled water was used in soaking the permanganate fibre for ten minutes in order to catalyse the reaction. Furthermore, the fibres were sun dried after it was decanted.

2.2.4 Acetylation

Ukam fibres were soaked in demineralized water for an hour, filtered and then placed in a flask, containing acetylating solution. Acetylating solution is made of 250 ml toluene, 125 ml acetic anhydride and a small amount of catalyst perchloric acid (60 %). The temperature for the acetylation process was 60 ^oC for the period of 1 to 3 hours. After modification, the fibre was washed thoroughly with distilled water until acid free. The treated fibres were sun dried before the manufacturing of the composites.

2.3.2 Composites Production

The ukam fibre composites samples were produced using the hand lay-up method (Aly et al., 2010) in a wooden mould and the sample compositions are shown in Table 1

Table 1: Composition of Ukam Fibre and Resin

Sample	Ukam Fibre (wt%)	Resin (wt%)
1	5	95
2	10	90
3	15	85
4	20	80
5	25	75

2.4 *Determination of Mechanical Properties*

The process and procedure applied in determining the mechanical properties of the ukam fibre sample are presented as follows:

2.4.1 Hardness test

Hardness test was carried out in accordance with American Society for Testing and Materials D 785 - 90 using the Universal Hardness Tester Indentec, (model 8187.5 LKV). The dimension of the prepared test samples was 30 mm x 30 mm x 30 mm. From the test the hardness strength was determined.

2.4.2 Tensile test

Tensile strength test was conducted using Hounsfield Tensometer (model No.S/N 8889) according to ASTM D 638 - 03 (Croccolo et al., 2013). From the test elastic modulus, ultimate tensile strength and percentage elongation were determined.

2.4.3 Compression test

The test was conducted by following ASTM – C 109 - 88 using Universal (digital) testing machine. Sample specimen dimensions of 30 mm x 30 mm x30 mm were produced for the test. Compressive load was applied axially onto the specimen till it ruptured. The compressive strength was determined.

2.4.4 Impact test

Impact test on the composites was carried out in according to ASTM D 256 – 06a. The specimen dimensions were 60 mm x 12 mm x 5 mm. A pendulum swings are used to break the sample kept in cantilever. Impact strength of the composites is determined from the results.

2.4.5 Flexural test

Flexural test was conducted based on ASTM 790-07 using Universal (digital) testing machine. Sample dimensions of 100 mm x 10mm x 5 mm were produced for the test. The test sample was placed between rollers and force (using hydraulic handle) was applied until the sample ruptured. The MOR and MOE were determined. In each case three different samples were tested and the average value obtained.

3.0 Results and Discussion

3.1 Mechanical properties of ukam composites due to fibre loading and surface treatment

The experimental results for various samples under different fibre loading and surface treatment are given figure (1-8). In each of the figures, bar charts for different mechanical properties are plotted against different composition of fibre loading.

3.1.1 Hardness

Figure 1 presents the hardness properties of the composites sample due to fibre loading and surface treatment. In general, hardness was observed to increase with increase in fibre loading for all treatments.

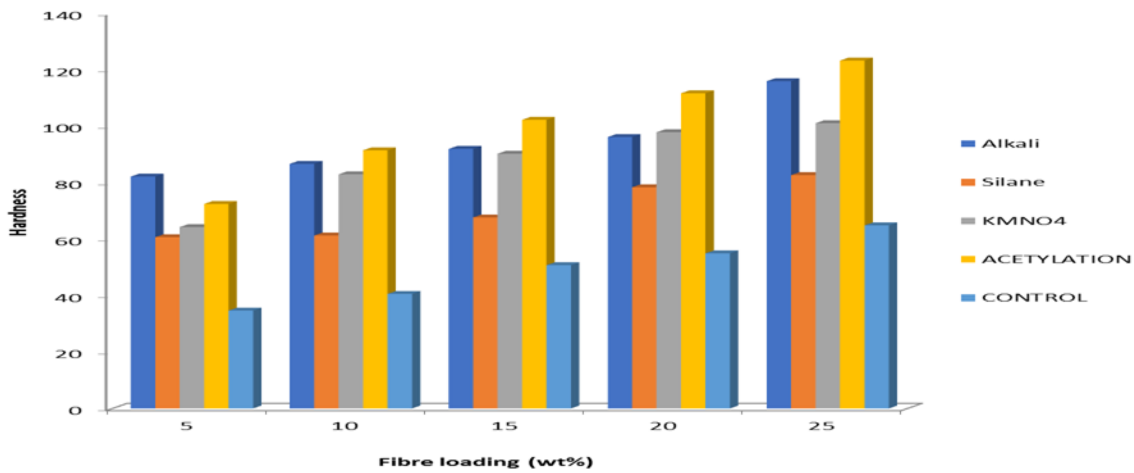

Figure 1: Hardness of composites due to surface treatment and fibre loading

Additionally, the work reported showed that the addition of natural fibres to a polymer matrix reduce flexibility of the resulting composite thereby increases the stiffness similar what was reported by Idrus et al. (2011) where hardness properties of a composite were enhanced as a result of the decrease in flexibility and increase in stiffness of the composites.

Also further analysis of the results reveals that surface modified composites have better hardness strength to those of untreated fibre composites at all fibre loadings. Hardness increased with treatment because treated fibre becomes more hydrophobic with increased interaction between fibre and matrix, which results in increases in strength, Stiffness, and interfacial adhesion. Generally, the pattern of the increased hardness based on surface treatment followed the order: untreated< saline < $KMNO_4$ < alkali < acetylation. Acetylation treated composites exhibited highest values of hardness at all levels of fibre loading. This scenario could be linked to better adhesion of the matrix to the fibre brought about by acetylation as reported by (Idrus et al., 2011). However, at 20 % fibre loading the pattern changed slightly to: *untreated < silane < alkali <* $KMNO_4$ *< acetylation.* The discrepancy in the pattern at 20 % fibre loading is caused by pores and voids (Pickering et al., 2016). It is observed that when the void fraction values become more pronounced, then the value of hardness decreases.

3.1.2 Tensile strength

Figure 2 indicates the change in the value of the tensile strength in relation to the respective change noticed in the fibre loading and surface treatment.

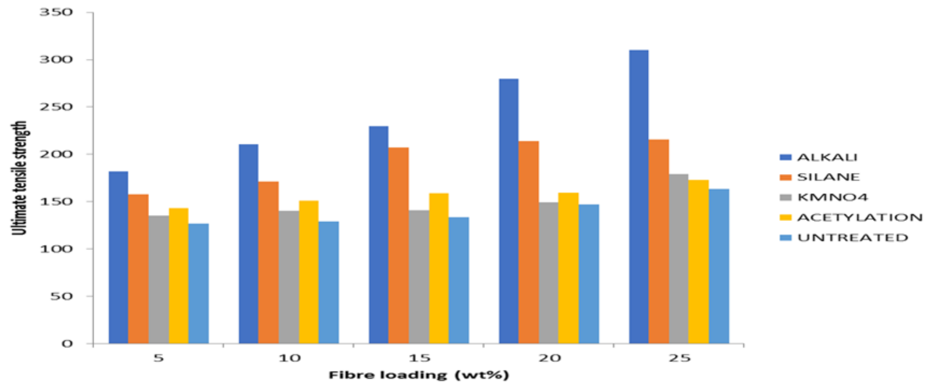

Figure 2: Tensile strength of composites as a result of fibre surface treatment and loading

The results showed increase in tensile strength with increase in fibre reinforced composites was consistently higher than the untreated composites. It is clear from this that treatment enhanced compatibility between the matrix and the fibres, allowing the required transfer of stresses between fibres and matrix with concurrent increase in values of tensile strength. At 5, 10, 15 and 20 % fibre loadings: untreated < $KMnO_4$ < acetylation < silane < alkali. The alkali treated samples exhibited the highest increase in tensile strength at all fibre loadings. This is because during alkaline treatment, hemicellulose and lignin are removed, the inter-fibrillar regions are likely to be soft, and that makes the fibrils to align themselves along the direction of tensile loading. Pickering et al. (2016) was of the opinion that physical and chemical treatments usually improve the wettability of fibre and thus improve the interfacial strength leading to improvement in mechanical properties as the cellulose structure is totally modified. However, at 25 % fibre loading the order became *untreated* < acetylation < $KMnO_4$ < silane < alkali. The discrepancy could be explained on the basis of chemical bonding which occurs when there are enough chemical groups between the fibre and matrix that can undergo reaction to form bonds between the fibre and matrix. This clearly shows that there was not enough chemical reaction for $KMNO_4$ treated fibres at 25 % fibre loading.

3.1.3 Modulus of Elasticity

The modulus of elasticity (MOE) increased with increase in Ukam fibre loadings for all the treatments (Figure 3).

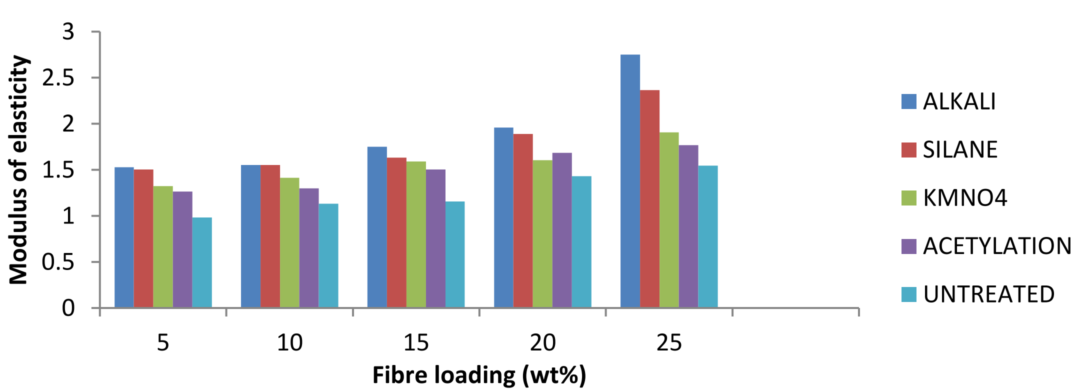

Figure 3: Modulus of elasticity of tensile strength composite as a result of fibre surface treatment and loading

The increased in MOE of the composites with increase in Ukam fibres loadings is attributed to the inherent high stiffness strength of the fibres. The sodium hydroxide treated composites gave the highest value of MOE (2.7521 MPa) at 25 % fibre loading for same reason as for tensile strength. Surface treatment could improve the stiffness of fibers and accelerate the reorganization of fibrils along the direction of tension force after removal of binding materials by treatment (Jacob et al., 2004). The MOE values at 5, 10, 15, 20 and 25 % ukam fibre loadings for the treatments varied from 1.0218 – 1.5276 MPa, 1.0364 – 1.5531 MPa, 1.1582 – 1.7504 MPa, 1.1801 – 1.8012 MPa and 1.2016 – 1.8342 MPa respectively. Also the percentage elongation increased with increase in fibre loading and treatments for similar reasons. Figures 3 and 4 respectively present the MOE and the percentage elongation of fibre surface treatment at various loadings.

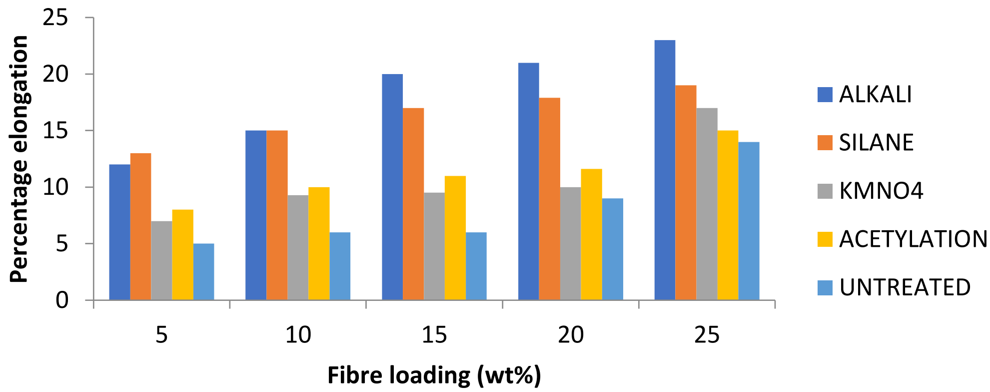

Figure 4: Percentage elongation of composites as a result of fibre surface treatment and loading

3.1.4 Impact Strength

The influence of surface treatment and fibre loading on impact properties is shown in Figure 5. It can be noted that for all applied treatments, there was a proportional increase in the Impact strength of the composite with respect to increase in the fibre loadings. The results clearly showed that impact energy of reinforced composites was enhanced by four treatments which is in agreement with a study by Ramires and Frallini (2017).

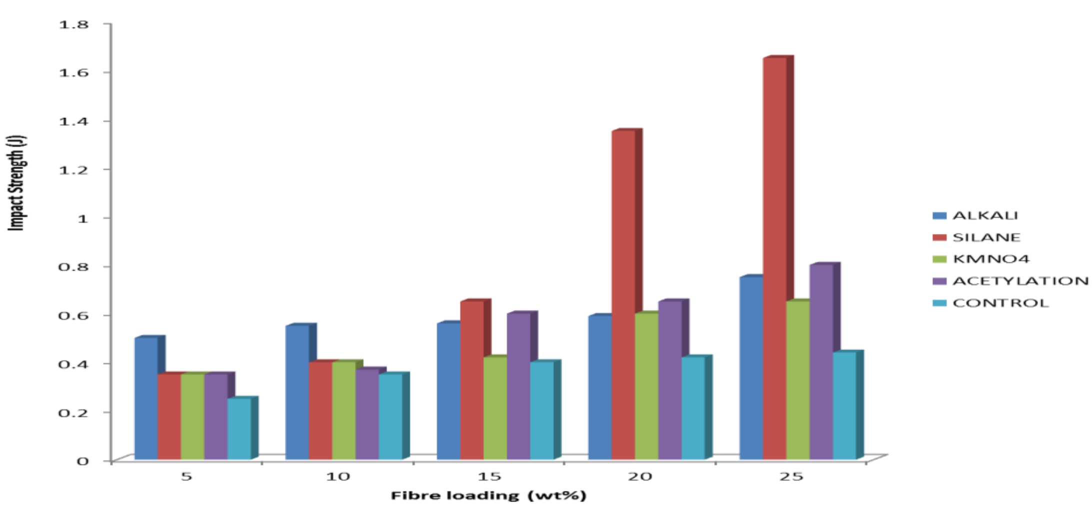

Figure 5: Impact strength as a result of fibre surface treatment and loading of composites

In addition, the results show that silane treated composites impact strength was the highest compared to other treatments, especially at higher fibre loading ratios. At lower fibre loading ratios, however, the differences were not significant. The results show that the average impact energy of the composites increased by 28.09%, 20.07%, 14.06%, 15.44%, and 22.34% for composites with 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%, and 25 wt.%, respectively, over the values obtained for the untreated composites using the same weight fractions. The higher values of impact energy for the treated composites over the untreated composites can be attributed to the improved mechanical interlocking due to rougher surfaces of the treated sisal fibres and improved interfacial bonding. The silane treated composite at 25 % fibre loading had the highest impact strength of 1.65 J. This is for the fact that silane used for treatment of fibre have different functional groups at either ends such that interaction at one end can occur with hydrophilic groups whilst the other end can interact with hydrophobic groups in the matrix to form a bridge between them (Pickering et al., 2016).

3.1.5 Compressive Strength

Figure 6 presents the compressive strength of the composites due to fibre loading and surface treatment. The result shows an increase in compressive strength with increases in fibre content and treatment. This is because an increase in fibre content increases the micro-packing of fibres in the cured matrix, which causes mechanical compaction of the fibrous ingredients with resultant high compression strength. Interestingly, Alkali treated composite showed the highest compressive strength values at all fibre loadings, with highest value of 143.03 MPa at 25 % fibre loading. In general, the order of increase in compressive strength with fibre treatments was*,* alkali > silane > $KMnO_4$ > acetylation > untreated. The lowest values of compressive strength exhibited by untreated ukam fibre in the matrix of polyester are that untreated fibres are incompatible with the matrix due to its hydrophilic nature (Jacob et al., 2004). The compressive strength values for other treatments at 25 % fibre loading were 94.14, 68.03, and 66.09 MPa for silane, acetylation, and $KMNO_4$ when compared with the value of 45.62 for the untreated fibre.

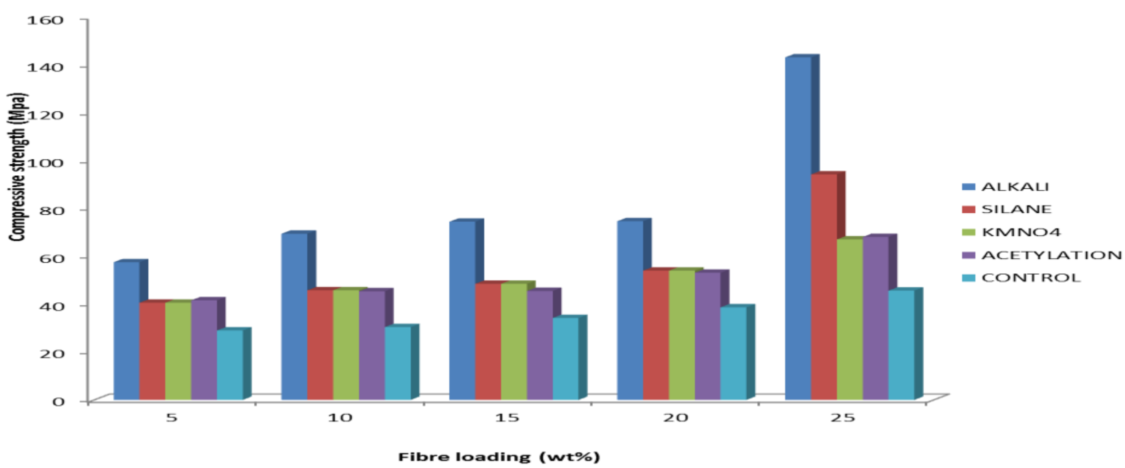

Figure 6: Compressive strength of composites as a result of fibre surface treatment and loading

3.1.6 Flexural strength

Furthermore, the Modulus of elasticity of composites as a result of fibre surface treatment and loading due to flexural strength is shown in Figure 7. It was revealed that Flexural strength increased with increasing fibre loading and treatments for all the composites investigated which is in agreement with the work of Romanzini et al. (2013). Silane treated composites at 5 – 15 % fibre loadings has the highest values of MOR (16-24 MPa). The order of increase in MOR with treatments were; at 5 and 10 % fibre loading, silane > $KMnO_4$ > alkali > acetylation > untreated, while at 15, 20 and 25 % fibre loadings respectively, were, silane > alkali > $KMnO_4$ > acetylation > untreated, $KMnO_4$ > silane > alkali > acetylation > untreated and acetylation > silane > alkali >acetylation > $KMnO_4$ > untreated.

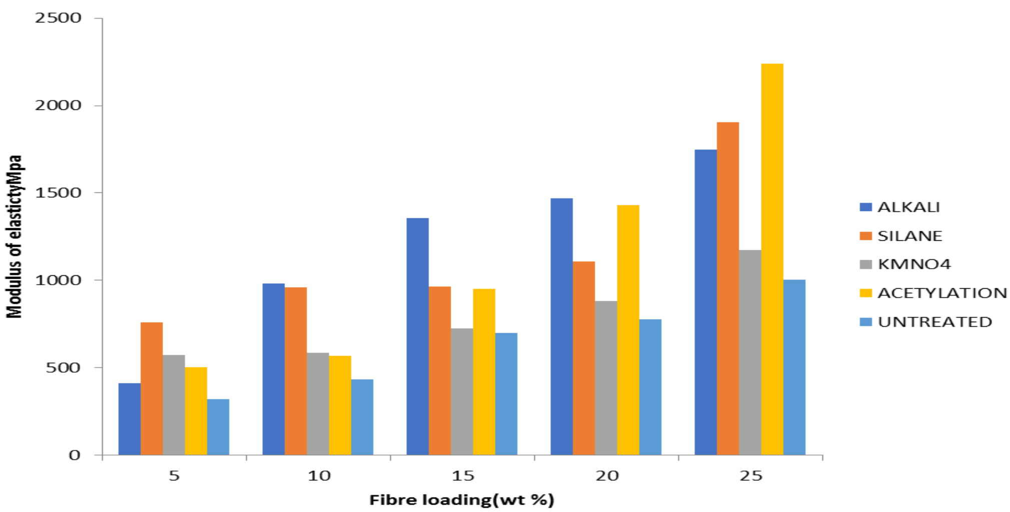

Figure 7: Modulus of elasticity of composites as a result of fibre surface treatment and loading due to flexural strength

From the results obtained Acetylation treatment gave the highest value of MOR at 25 % fibre loading while the MOR values of the composites at 5 % were 7, 16.06, 16.05, 7.99 and 15.94 MPa respectively, for untreated, silane, potassium permanganate, acetylation and alkali treated composites. The values of MOR increased to 19.23, 26.77, 27.37, 24.1, 31.78 MPa at 25 wt% Ukam fibres addition for untreated fibres, alkali, silane, potassium permanganate, acetylation treatment respectively. Acetylation treated Ukam fibre composite gave the highest modulus of rupture at 25 % fiber loading, compared with other treated the composites sample.

Figure 8 presents the effect of surface treatment on MOE obtained from flexural strength for both untreated and treated composites. The work reported here shows that the modulus of elasticity of the composite was observed to increase with increasing fibre loading and treatments, and order of increase was thus, silane > $KMnO_4$ > acetylation > alkali > untreated at 5 % fibre loading, alkali > silane >$KMnO_4$ > acetylation > untreated at 10 % fibre loading. At 15, 20 and 25 % fibre loading the orders were respectively, alkali > silane > acetylation > $KMnO_4$ > untreated and acetylation > silane > alkali > $KMnO_4$ > untreated. The MOE values of the composites at 5 % fiber loading were 519.23, 760 MPa, 671, 603 and 613 MPa for untreated, acetylation, potassium permanganate and silane treatment. These values increased to 1005.58, 1749.5, 1903.22, 1171.79 and 2140.71 MPa at 25 % Ukam fibres addition for untreated fibres, alkali, silane, potassium permanganate, acetylation treatment respectively. Acetylation treated Ukam fibre loading at 25 % composite had the highest value of modulus of elasticity (2140.38 MPa).

Based on the results the highest increase recorded in both the MOE and MOR of the composite with increasing fibre loading could be attributed to higher stiffness of the Ukam fibres. This shows that addition of Ukam fibres has positive effect on flexural strength and flexural modulus of both treated and untreated composite with highest performance recorded at 25 wt%. Composition of fibre loading. Again, the highest flexural strength recorded can also be attributed to the enhanced interfacial adhesion between polyester resin and ukam fibres and the efficient stress transfer achieved through surface treatment. The results obtained in this study are consistent with other studies (Kim et al., 2001, Romanzini et al., 2013).

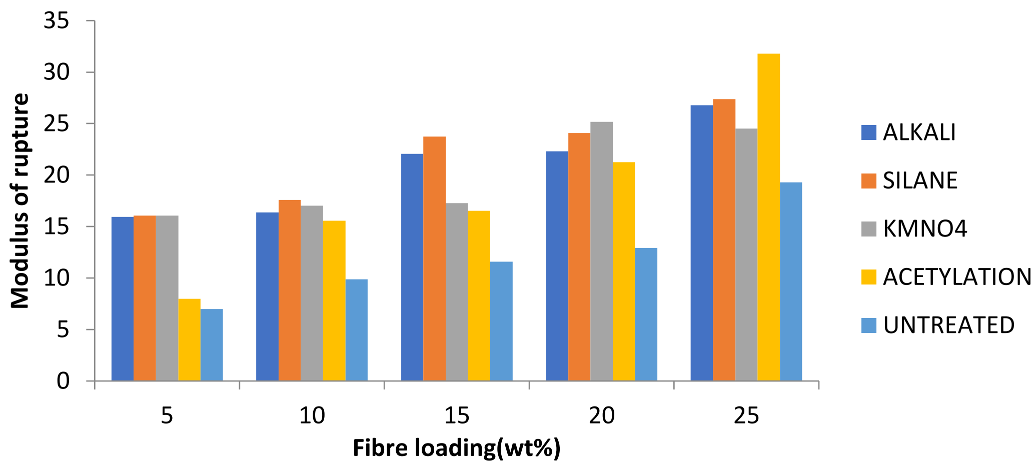

Figure 8: Modulus of rupture of composites as a result of fibre surface treatment and loading

4. Conclusion

The mechanical properties (hardness, tensile, impact and flexural strengths) of treated ukam fibre reinforced composites have been evaluated and the results presented to show that surface treatment processes improved the mechanical properties of treated fibre reinforced composites. The hardness and tensile strength exhibited in the Acetylation and Alkali treated composites respectively are higher than those in the other treated composites under all levels of fibre loading. Also the modulus of elasticity (MOE) and percentage elongation are found to increase with increase in fibre loading for all treatments.

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