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EFFECT OF CHEMICAL TREATMENT ON THE CONSTITUENTS AND TENSILE PROPERTIES OF OIL PALM LEAF FIBRE

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ABSTRACT

The effect of chemical treatment on the constituents and tensile properties of oil palm leaf fiber was investigated. Oil palm leaves were cut and buried underground close to the stream, and were regularly wetted with water to ensure proper fermentation for 20 days. The fermented leaves were washed and sun dried. The dried oil palm leaf fibers obtained were mechanically treated with chemicals, while some were left as control (untreated). The fibers were then characterized in terms of their percentage constituents, and their tensile properties determined with Instron universal testing machine. The tensile properties studied were ultimate tensile strength (UTS), elongation at yield and at break, Young's modulus of elasticity as well as resilience. The results show that chemical treatments enhanced the removal of lignin and hemicelluloses which are detrimental to the interfacial bond strength of composite produced from natural fibers, except the sample treated with alkaline peroxide. Tensile test results reveal that fiber sample treated sequentially with KOH, acetic acid, Nacl and Hcl produced the highest UTS, elongation at yield, elongation at break and resilience. This is attributed to the plasticizing effect of the chemical treatment. However, the untreated fiber sample exhibited the highest Young's modulus of elasticity. This observation was attributed to the highly crystalline nature of unmodified cellulose.

Keywords: Chemical treatment, Oil Palm Leaf, Constituents, Tensile Properties

INTRODUCTION

With the gradual depletion of petroleum resources in the last decade worldwide, fibers derived from plants have become fundamentally important for the production of biocomposites, which have the advantages of renewability, biodegradability, abundance, low cost, and reduced health hazard, and thus have received considerable attention with the possibility of replacing synthetic fibers based on petrochemical sources. Plant fibers are comprised of three major chemical components, namely cellulose, lignin, and hemicelluloses. The cellulose forms a skeletal frame that is surrounded and encrusted by the matrix substances (Hemicelluloses and lignin) (Reddy and Yang, 2005). Cellulose chains composed of glucose units are organized into the elementary fibril, which are arranged into micro fibrils with a diameter of 10 – 50nm in the presence of hemicelluloses and lignin (Paavilainen, 2001). The fiber/matrix interface plays an important role in the micro mechanical behaviors of composites.

Previous studies on the use of natural fibers as reinforcement in polymer composites have shown that lignin and hemicelluloses are detrimental to fiber/matrix interfacial adhesion leading to loss of strength in the composite (Matthew et al, 2005; Oksman et al, 2009; Huda et al, 2006; Lundquist et al, 2003). One difficulty that has hampered the use of natural fibers as reinforcement in composites is the lack of good adhesion with the polymeric matrix. In particular, the large moisture sorption of natural fibers adversely affects adhesion with the hydrophobic polymer matrix leading to poor interfacial adhesion and loss of strength in the composite. To prevent this phenomenon, fiber surface properties have been modified in order to promote adhesion (Khalil and Rozman, 2000). To reinforce thermoplastic composites, blue agave fibers were modified via esterification reaction (Tranc et al, 2007). The mechanical properties characterization of these fibers showed a change in the elastic modulus and an improvement in the impact resistance (Bessadok et al, 2008) in a recent study (Onyeagoro, 2010) on dispersion characteristics of yellow poplar wood fibers in cellulose acetate butyrate/yellow poplar wood fiber composites, it was found that the quality of fiber dispersion increased with fiber surface acetylation which improved fiber/matrix interfacial adhesion. Chlorine dioxide treatment had been used as a lignin modification to enhance the interfacial adhesion between fiber bundle and phenolic resin, while the cellulosic part was preserved (Hoareau et al, 2004; Trindade et al, 2005; Hoareau et al, 2006; Megiatto et al, 2007). On the other hand, the selective oxidation of lignin induced by chlorine dioxide also was found to cause a serious degradation of lignin units (Megiatto et al, 2007). This degradation is extremely important for the chlorine dioxide bleaching of pulp fiber in the paper-making industry, of which the main object is to remove as much lignin as practical (Broadon et al, 2005; Svenson et al, 2005, 2006). This study was carried out to investigate the effect of chemical treatment using various combinations of NaoH, H_2O_2, KOH, CH_3COOH, Nacl and Hcl on the chemical constituents and tensile properties of oil palm leaf fiber.

MATERIALS AND METHOD

Materials

The materials used in this work are oil palm leaves, distilled water, potassium hydroxide, hydrochloric acid, acetic acid and hydrogen peroxide.

Equipment

Instron universal tensile testing machine, weighing balance, shaker water bath, beakers, furnace, crucible.

Method

Preparation of oil palm fiber

The oil palm fiber used was obtained from the plant leaves. The leaves were cut and buried underground for 20 days so as to allow fermentation to take place. For the fermentation to take place normally, the leaves were buried close to Otamiri River that flows across Federal University of Technology, Owerri, Nigeria and were watered daily. The fermented leaves were washed and sun dried.

Chemical treatment of fiber

Cellulose micro fibrils were prepared using different combinations of chemical and mechanical treatments. 100g oil palm fiber with fiber thickness of 0.2mm was divided into four equal portions of mass 25g each. Three portions were given three different treatments while the fourth portion (untreated) was left as control. The treatment was carried out to obtain samples A – NaOH, B – HCl and C-KOH as follows:

Sample a-NAOH

This sample was obtained by treating a portion of the fiber sample with 400ml of 0.5M NaOH inside shaker water bath at 40^0C for 9 hours. The insoluble residue was treated with 0.5M NaOH and 450ml of 3wt. % H_2O_2 solution at 45^0C for 8 hours, and then finally treated with 400ml of 2M NaOH solution at 50^0C for 3 hours.

Sample b – HCl

This sample was obtained by treating a portion of the fiber sample with 400ml of 0.5M NaOH inside shaker water bath at 40^0C for 9 hours. The insoluble residue was treated with 0.5M NaOH and 450ml of 3wt% H_2O_2 solution at 45^0C for 8 hours, and then finally treated with 400ml of 2M HCl at 80^0C for 3 hours in order to remove mineral traces.

Sample c – KOH

The sample was obtained by treating a portion of the fiber sample with 400ml of 5wt% KOH inside shaker water bath at 40^0C for 9 hours. The insoluble residue was de-lignified with 450ml of 1% NaCl solution at pH3, and adjusted with 10wt% acetic acid at 80^0C for 2 hours. Finally, the sample was soaked for 2 hours in 300ml of 1wt% HCl solution to remove mineral traces.

CONTROL SAMPLE

The untreated fiber sample was used as the control.

Determination of Chemical Constituents of Fiber

Determination of lignin content by gravimetric method

2.0g of the sample were weighed and placed inside a beaker. 72% H_2SO_4 was added and allowed to stand for 2 hours. 8% H_2SO_4 was later added and the solution refluxed for 3 hours. The residue was filtered with purpling cloth and washed severally with hot water. A crucible was weighed and the sample was scraped into it. The sample was oven dried at 110^0C for 1 hour and then cooled inside desiccators after which the weight was taken. The sample was ashed in a furnace at 500^0C for 3 hours. It was then cooled inside desiccators and finally weighed. The % lignin was calculated using equation 1:

$$\% \text{ Lignin} = \frac{W_2 - W_1 \times 100}{W_s} \qquad - \quad - \quad - \quad - \qquad (1)$$

Where,

W_1 = weight of ash sample + crucible

W_2 = weight of oven dried sample + crucible

W_s = initial weight of dried sample

Determination of cellulose content

1.5g of fiber sample was weighed into a beaker followed by addition of 20ml of 80% acetic acid, 1ml of concentrated nitric acid and 3 glass beads. The content was refluxed for 30 minutes. The sample was cooled and washed into 50ml centrifuge tube containing hot 95% ethanol, and then centrifuged at 15,000rpm for 5 minutes. Thereafter, the liquid was decanted and 95% ethanol was added, stirred and filtered by suction. The sample was washed three times with hot benzene, two times with 95% ethanol and once with ether. The sample was placed inside a weighed crucible and placed in the oven maintained at 110^0C for 1 hour. The crucible was then cooled in desiccators and weighed. For ash content determination, the crucible and its content was placed inside a furnace maintained at 500^0C for 3 hours after which it was cooled in desiccators and weighed. The % cellulose was calculated from equation 2.

$$\% \text{ cellulose} = \frac{W_2 - W_1 \times 100}{W_s} \qquad - \quad - \quad - \qquad (2)$$

Where,

W_1 = weight of crucible + sample after ashing

W_2 = weight of crucible + sample after drying

W_s = weight of sample

Determination of hemicelluloses

1g of fiber sample was weighed into two different conical flasks labeled A and B. Into flask A was added 5% KOH while 24% KOH was added into flask B. Both flasks were allowed to stand for 2 hours. Thereafter the mixtures were filtered with purpling cloth, washed with their corresponding KOH solution, and the filtrate was received into two different beakers. Ethanol was added to precipitate the hemicelluloses. The precipitated hemicelluloses were isolated by centrifuging for 15 minutes. The isolated hemicellulose was washed with ethanol and ether and, then transferred into two different crucibles. This was followed by drying in the oven for 1hr at 110^0C. Thereafter, they were transferred into desiccators and allowed to cool for 30 minutes and then weighed.

The fiber sample was also placed in a furnace maintained at 500^0C for 3 hours after which they were cooled inside desiccators and weighed. The weight of hemicelluloses precipitate A (W_A) was calculated while the weight of hemicelluloses precipitate B (W_B) was also calculated. The percentage hemicelluloses composition of the fiber samples were then calculated using equation 3.

$$\% \text{ Hemicelluloses (A)} = \frac{W_A}{W_2} \times 100 \quad - \quad - \quad (3)$$

Where,

W_A = dried weight of hemicelluloses precipitate A

W_2 = dried weight of fiber sample

$$\% \text{ Hemicelluloses (B)} = \frac{W_B}{W_2} \times 100$$

Where,

W_B = dried weight of hemicelluloses precipitate B

W_2 = dried weight of fiber sample

Determination of tensile properties of the fiber

Tensile properties of the fiber samples were determined on aInstron testing Machine following procedures from ASTM D638.

RESULTS AND DISCUSSION

Effect of Chemical Treatment of Fibers on Fiber Constituents

The effect of chemical treatment of fibers on fiber constituents is shown in Figure 1. The results show that chemical treatment, with the exception of alkaline peroxide (sample A) contributed to the removal of lignin and hemicelluloses contents. The resistance to extraction with alkaline peroxide (sample A) of the sugars that are the main constituents of hemicelluloses is attributed to the very strong associated between xyloglucan and cellulose. Xyloglucan probably binds not only to the surface of cellulose micro fibrils, but can also be entrapped within the micro fibrils (Habibi et al, 2008).

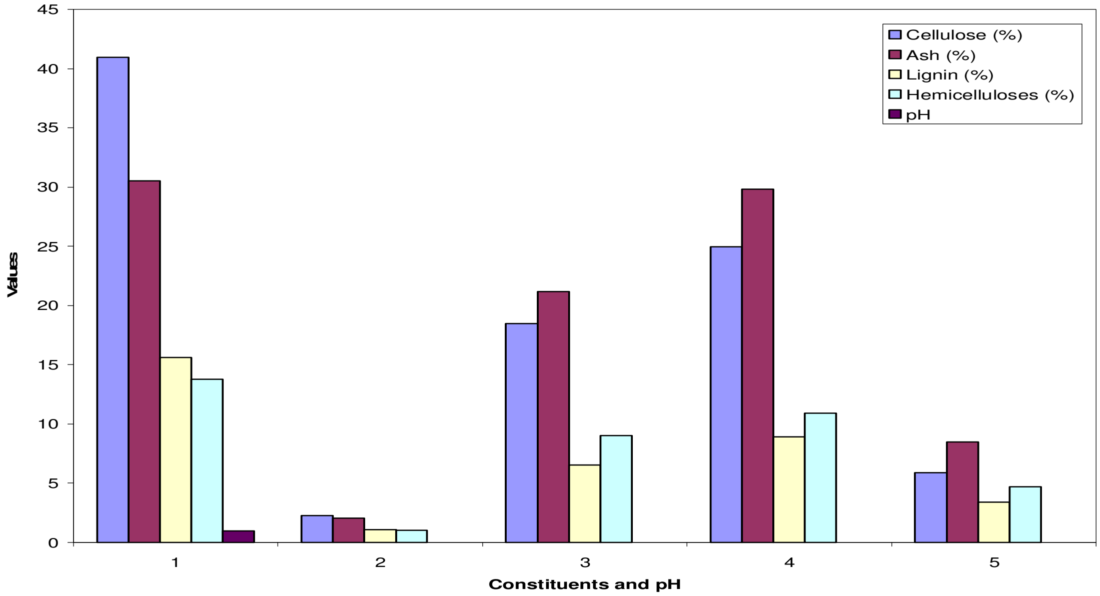

Figure 1.Plot of chemical constituents of oil palm leaf fiber and pHvalues for both treated and untreated (control) fibers

Xylans, xyloglucans and glucomannans are all able to bond onto cellulose fibrils in a manner similar to the interchange bonding of cellulose itself. The results show that sample A gave higher percentages of lignin and hemicelluloses than the control sample, while sample B and sample C had lower values. However, sample B exhibited the least values for both constituents. This observation is consistent with the report on the study of abrasive and tensile property performance of treated sugarcane fiber-reinforced polymer composites (EL-Tayeb, 2008). Figure 1 also reveals the effect of chemical treatment on cellulose and ash contents as well as the pH values of the fibers. The results show a progressive decrease in cellulose and ash contents from control sample down to sample C. However, the pH value of the fiber did not follow the same pattern of response to chemical treatment. The highest pH value of 8.5 was exhibited by sample A that was treated with alkaline peroxide, while sample B that was treated with HCl in addition to alkaline peroxide gave the lowest pH value of 3.4.

Response of Tensile Properties of Fibers to Chemical Treatments

The results of the tensile properties obtained for the treated and untreated (control) fiber samples are shown in figures 2 – 9.

Response of Tensile Stress and Ultimate Tensile Strength (UTS) Of Fibers to Chemical Treatment

Figures 2 – 6 show the response of tensile stress and UTS of fibers to chemical treatment. The results show that fiber sample coded C-KOH that was treated with a combination of KOH, CH_3COOH, NaCl and HCl exhibited the highest UTS value of 0.96N/mm^2. The UTS decreased in the order C-KOH (0.96N/mm^2) > A-NaOH (0.73N/mm^2) > Control (0.5N/mm^2) > B-HCl (0.3N/mm^2).

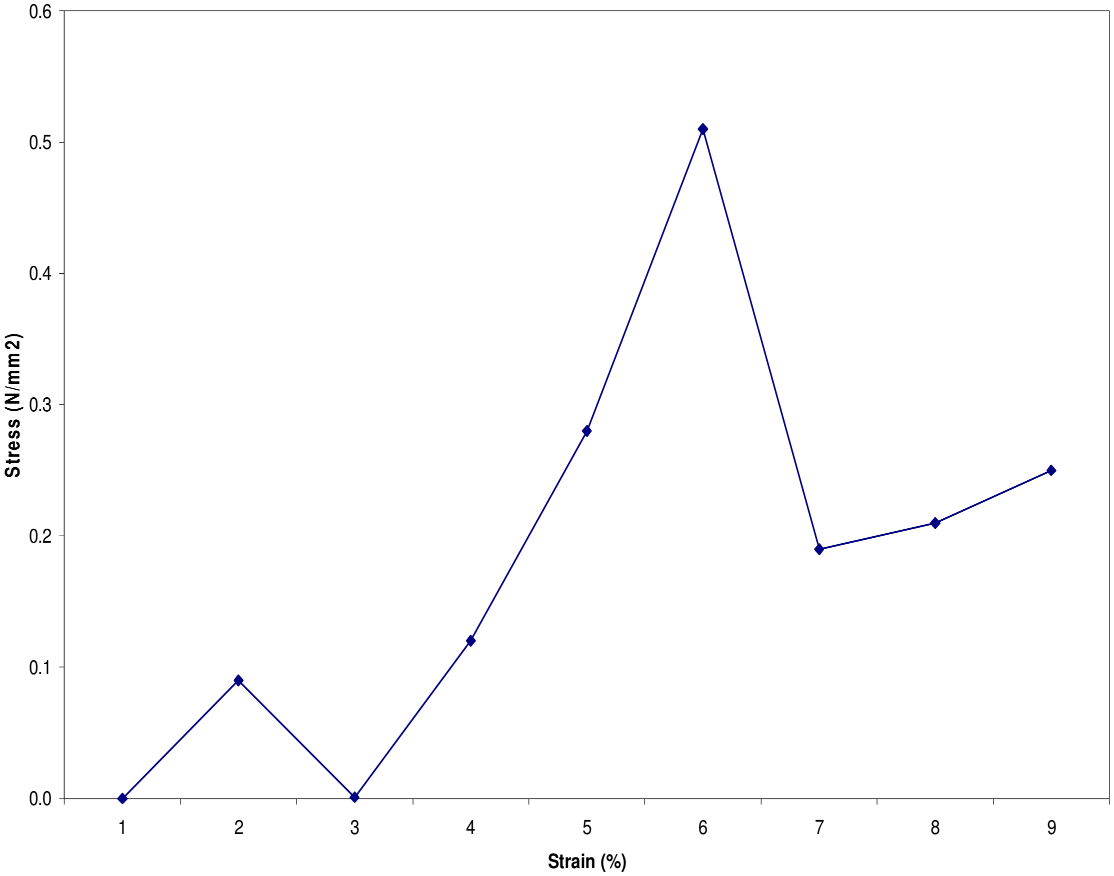

Figure 2. Plot of stress versus strain for the control (untreated) sample

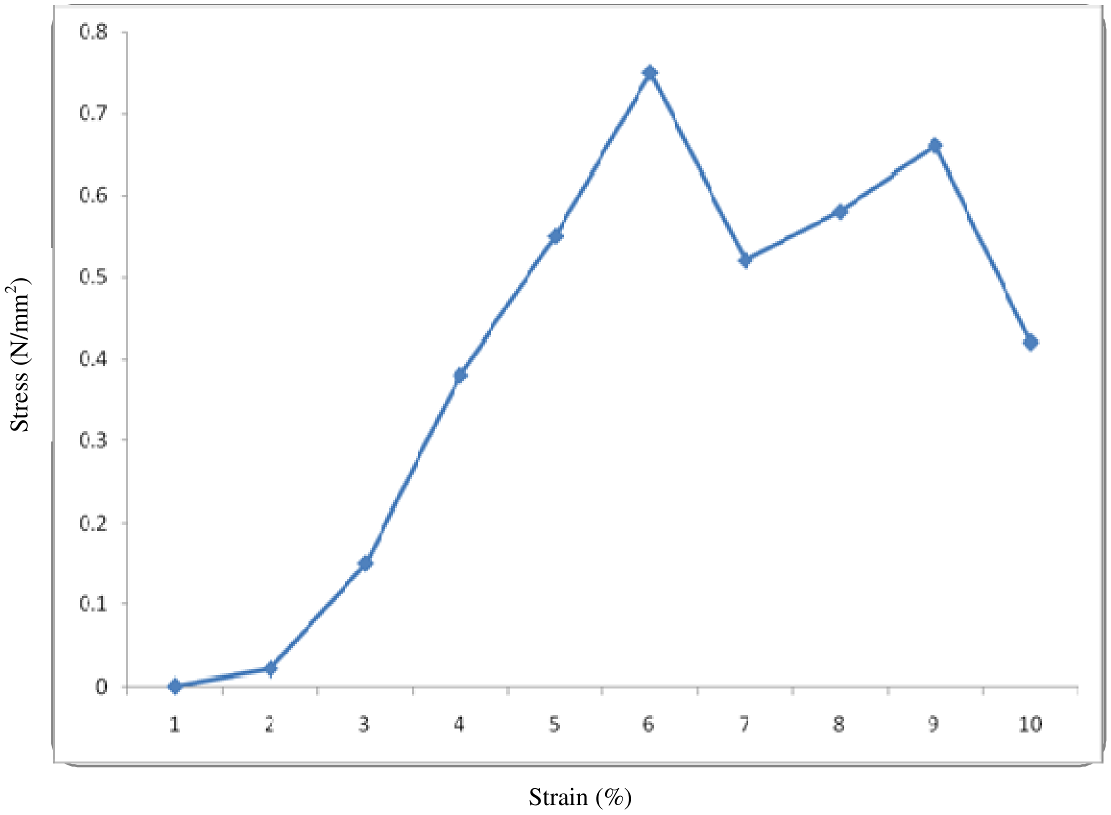

Figure 3.Plot of stress versus strain for treated sample A-NaoH

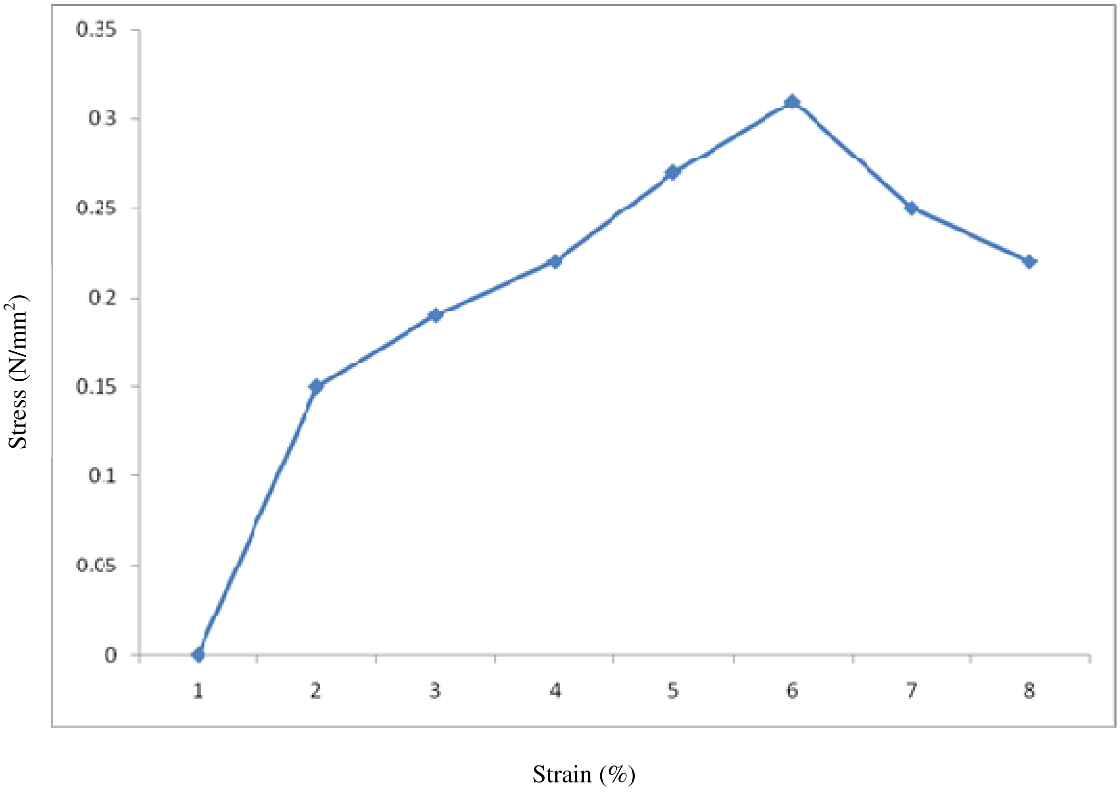

Figure 4.Plot of stress versus strain for treated sample B-HCl

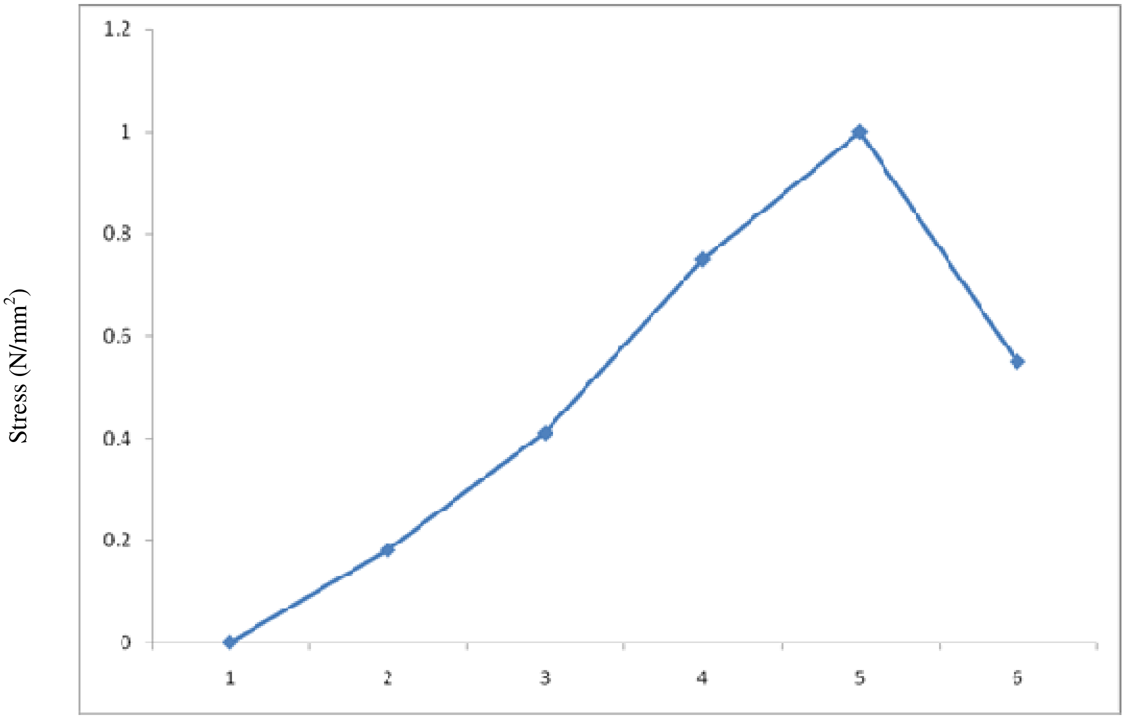

Figure 5.Plot of stress versus strain for treated sample C – KOH

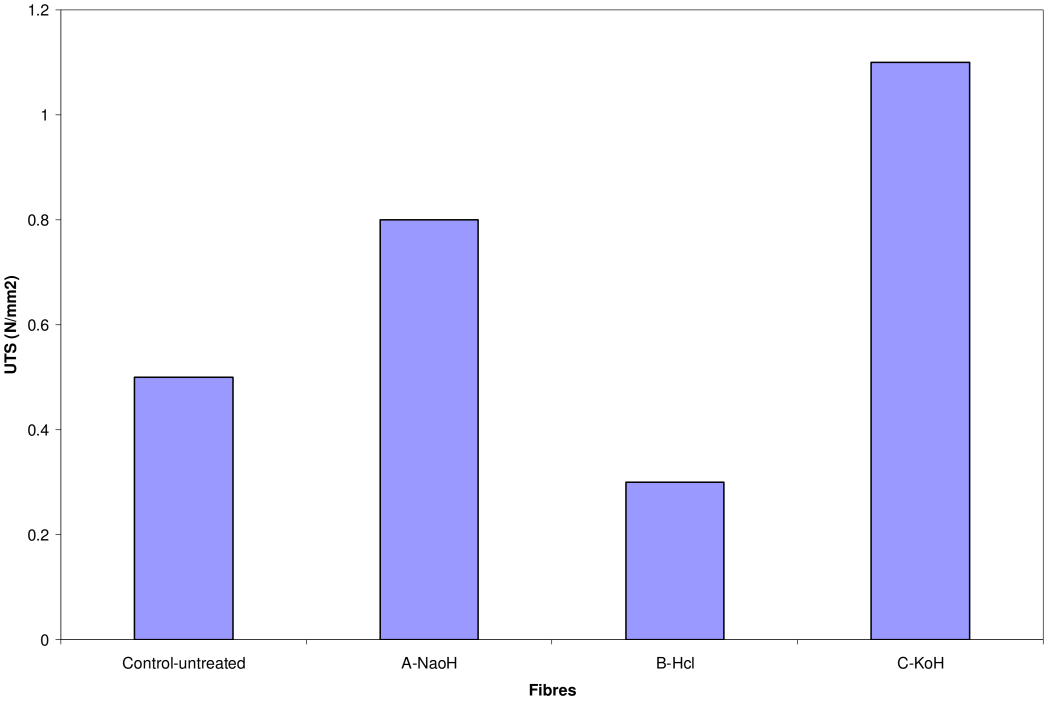

Figure 6. Plot of UTS versus fiber for both treated and untreated (control) samples

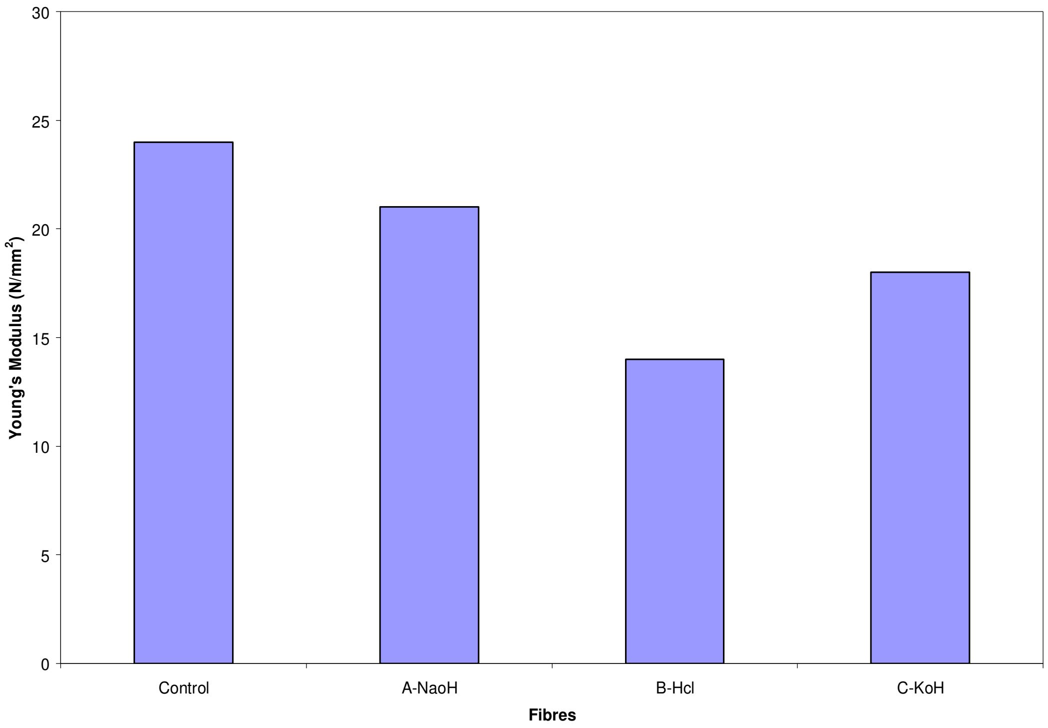

Figure 7.Plot of Young's Modulus versus Fibers for both treated anduntreated samples

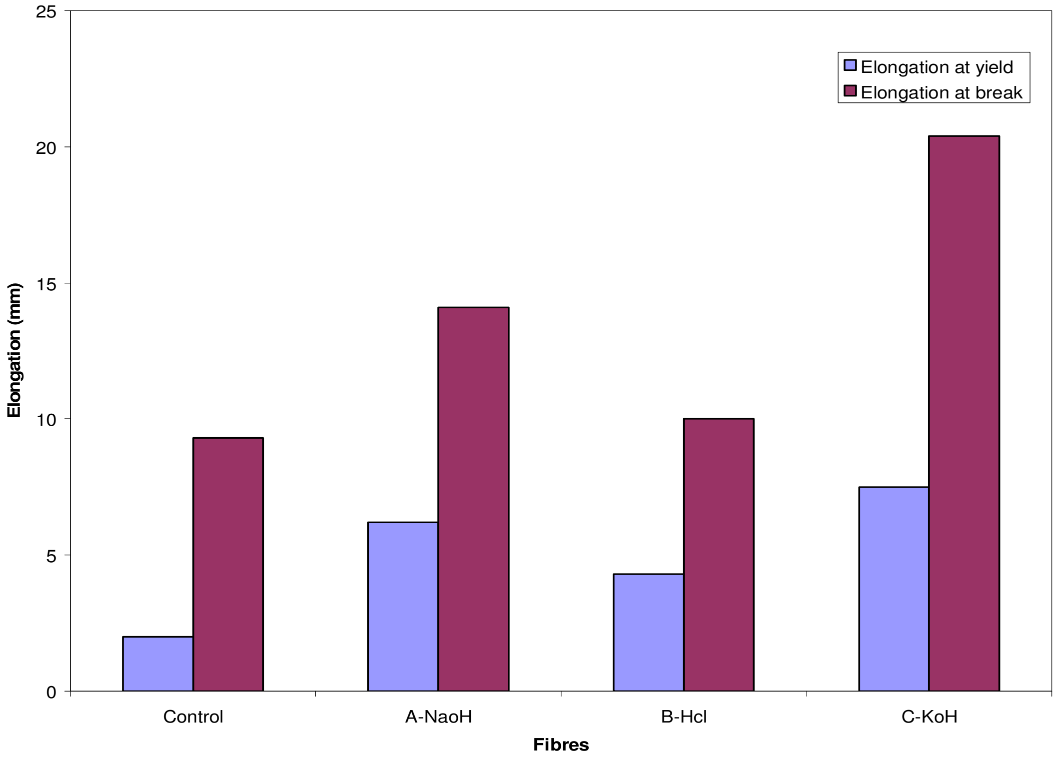

Figure 8. Plot of Elongation against Fibers prepared with both treated anduntreated fibers at 20wt% fiber content

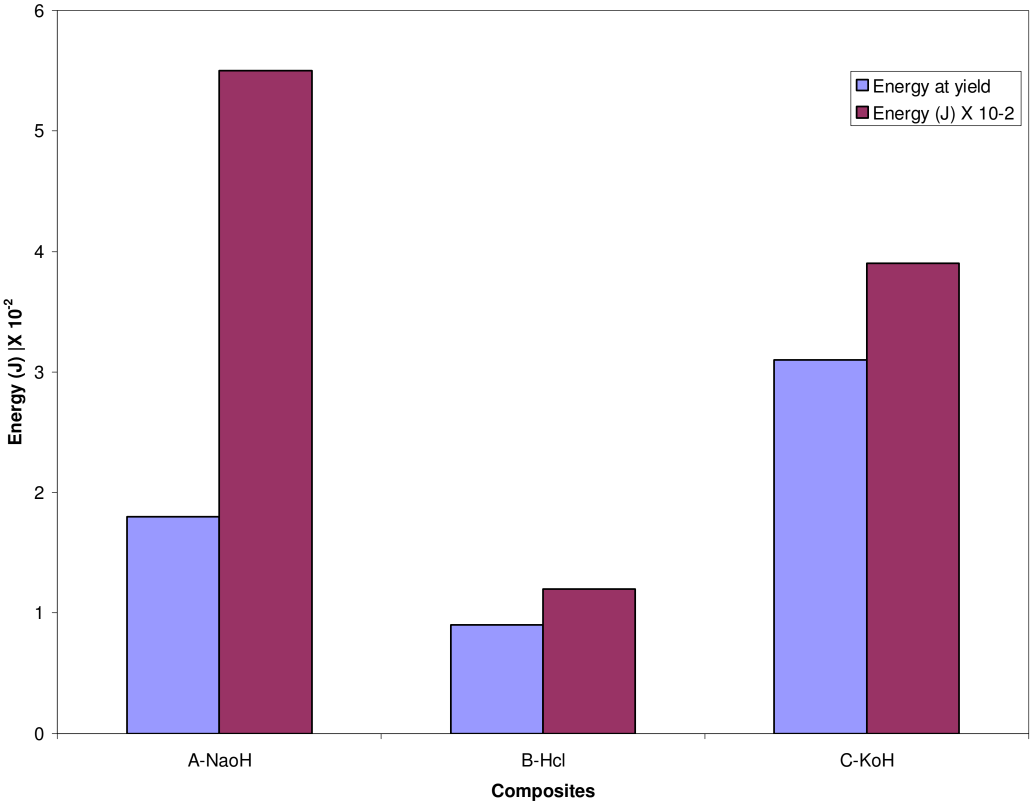

Figure 9. Plot of energy versus fibers for both treated and untreated samples

Response Of Young's Modulus Of Fibers To Chemical Treatment

The response of Young's modulus of elasticity of fibers to chemical treatment is shown in Figure 7. The untreated (control) sample produced the highest modulus of 23.9N/mm^2. The Young's Modulus of elasticity decreased in the order: control (23.9N/mm^2) > A – NaOH (23.1N/mm^2) > C – KOH (17.9N/mm^2) > B – HCl (13.5N/mm^2). This observed was attributed to the highest crystalline nature of unmodified cellulose and also due to reduction in molecular weight of fibers by chemical treatment via oxidative degradation. This reduction in molecular weight became more pronounced in the fibers treated with NaOH, H_2O_2 and finally washed with HCl (sample B – HCl) which resulted to the lowest value (13.5N/mm^2) of Young's modulus of elasticity produced by this fiber sample. Sun et al, 2004 have shown in a previous study on isolation and characterization of cellulose from sugarcane bagasse that acid and alkali treatment of sugarcane fibers causes oxidative degradation and reduction in molecular weight of the fibers.

Response of Elongation at Yield and At Break of Fibers to Chemical Treatment

The response of elongation at yield and at break of fibers to chemical treatment is shown in Figure 8. The results show that fiber sample C-KOH produced the highest elongation at yield of 7.5mm. Elongation at yield decreased in the order: C-KOH (7.5mm) > A – NaOH (6.2mm) > B – HCl (4.3mm) > Control (2mm). Yield point is defined as the point at which materials experience a major deformation at the micro-structural level. It is a point where additional strain occurs without any increase in stress load on the material (Egor and Toader, 1991). In the unmodified form, cellulose is a highly crystalline polymer and as expected, it produced the least elongation at break (2mm). The fiber samples C-KOH and A-NaOH produced higher values of elongation at break due to the plasticizing effect of KOH and NaOH which reduced the glass transition temperature (Tg) of cellulose. The lower value of elongation at break (4.3mm) produced by fiber sample B – HCl may be due to the degrading effect of HCl on cellulose. Figure 8 also reveals that the untreated (control) sample exhibited the lowest value (9.3mm) of elongation at break. Elongation at break decreased in the order: (C-KOH (20.4mm) > A-NaOH (14.1mm) > B – HCl (10mm) > control (9.3mm). The breaking load is the load that brings about material failure (Bolton, 1988). The results therefore show that fiber sample C-KOH can withstand the load generating the stress that will cause the fibers to fail for longer period than the other fiber samples. Similarly, chemical treatment produced an increase in elongation at break with fiber sample C-KOH showing the highest value.

Response of Absorbed Energy of Fibers to Chemical Treatment

Figure 9 shows the response of the absorbed energy of fibers to chemical treatment before yield and at break (failure). The energy parameter is a measure of the resilience of a material (Dotan et al, 1966). Figure 9 shows that fiber sample C-KOH absorbed the highest energy at yield of 0.03J. The absorbed energy decreased in the order C-KOH (0.03J) > A-NaOH (0.019J) > B–HCl (0.006J) > Control. The highest value of absorbed energy exhibited by fiber sample C–KOH (which suggests very high resilience) lends credence to the highest value of energy at yield shown by this fiber. However, it was observed that fiber sample A–NaOH exhibited the highest value of energy at break of 0.054J, followed by sample C–KOH with a value of 0.039J, while sample B–HCl produced the least value of 0.008J.

CONCLUSION

The following conclusions can be drawn from this study:

The chemicals applied in this work are combinations of NaOH, H_2O_2, KOH, CH_3COOH, NaCl and HCl as follows:

a. Combination I: NaOH and H_2O_2 with NaOH wash
b. Combination II: NaOH and H_2O_2 with HCl wash
c. Combination III: KOH, CH_3COOH and NaCl with HCl wash

Chemical treatment was found to improve the tensile properties of oil palm fiber by removing the deleterious constituents such as lignin, hemicelluloses and ashes which affect the interfacial bonding strength between the fibers and polymer matrix. Fiber samples treated with combination III exhibited the highest ultimate tensile strength (UTS), elongation at yield and at break as well as resilience followed by the fiber samples treated with combination I. However, the untreated fiber samples produced the highest Young’s modulus of elasticity before fracture.

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