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# Effect of Mercerization on the Properties of Acetylated Fibre Composites

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**Received:** March 20, 2022 **Accepted:** June 18, 2022

Abstract: Fibre composite manufacturers are in constant search for cost effective route of production. One potential way to achieve this is by employing a good fibre modification process that requires fewer chemicals. Thus, in this research an independent sample t-test was used to compare the properties of Acetylated fibre composites and fibre composites that were mercerized before acetylation, known as mercerized-acetylated fibre composites, in order to aid composite manufacturers in fibre-treatment decision making. Polyester based bamboo fibre composites were produced using acetylated and mercerized-acetylated fibres respectively. The produced composites were subjected to various physical and mechanical test. The results indicated that subjecting the fibres to mercerization before acetylation has no significant effect on hardness values, tensile strength and percentage elongation of the resulting composites. However, at 30 wt% and 40 wt% fibre contents, mercerized-acetylated fibre composites showed significantly higher Compressive strength, flexural strength, flexural modulus, and higher impact strength. Percentage moisture absorption of mercerized-acetylated composites was significantly lower than that of acetylated fibre composites at all levels of fibre content. This research will serve as a decision guide to composite manufacturers on the surface modification route to follow based on the economy, desired properties and applications of their product.
**Keywords:** Fibre treatment; Acetylation; Mercerization, Fibre Composite

## Introduction

Acetylation is carried out on natural fibres mainly to reduce their moisture absorption capacity and that of the resulting composites (Kai-Huang *et al*, 2014; Onyekwere *et al*, 2019). The main reaction that occurs during acetylation is the substitution of the hydroxyl groups in natural fibres with acetyl groups in order to render the fibre hydrophobic (Sjorstrom, 1981). On the other hand, one of the reasons for mercerization is to fibrillate the fibres in order to enhance the interfacial adhesion between fibre reinforcements and their matrix (Maya *et al*., 2008). Some researchers have undertaken acetylation of untreated natural fibres (natural fibres that have not been subjected to any form of modification prior to acetylation treatment) with improvement in moisture absorption properties (and some other properties) of the fibres and the resulting composites (Hassan *et al*, 2014; Yakubu *et al*, 2013; and Papadopoulos and Traboulay, 2002). Some researchers have also reported improvement in the mechanical properties of mercerized fibre composites over untreated fibre composites (Jayabal *et al*, 2012; Shehu *et al*, 2017; Wong, 2010; Somashekar and Shanthakumar, 2014; Prasad et al, 2014; Olorunnisola and Agrawal, 2013; Onyekwere and Igboanugo, 2019). A research was carried out by Anike *et al*, (2015) to compare the effect of mercerization and acetylation on some mechanical properties of raffia palm fibre polyester composite. They observed that composites of the acetylated fibres have improved tensile strength and micro hardness as compare to that of mercerized fibre composites while mercerized fibre composites have better tensile modulus and extension at break. Similarly, Hassan *et al* (2014) in their research on effect of mercerization and acetylation on mechanical properties of 'oil palm fruit bunch and rice husk' hybrid composite found that both acetylation and mercerization caused significant increase in tensile strength, impact strength and hardness values of the composite. In addition, acetylation led to significant reduction in percentage water absorption. However, the tensile modulus and compressive strength of the samples decreased due to acetylation. From the forgoing, it is observed that acetylation enhances some properties of natural fibre composites which mercerization does not improve, and vice versa. Since mercerization and acetylation individually improves some properties of natural fibre composites, it is likely that carrying out the two treatments consecutively could lead to positive synergetic effect on the properties of the resulting composites. However, there is no research, within the knowledge of the authors, on investigating the synergic effect of subjecting natural fibres to two consecutive treatments of mercerization and acetylation on the physical properties of their composites. Thus, this research tends to study the effect of acetylation of already mercerized fibre composites on its properties.

## Materials and Methods

### *Bamboo Fibre Extraction*

The Culms were split into strips of about 10 cm long. The strips were soaked in a solution containing; 8% v/v Sodium hypochlorite, 5% w/v Sodium hydroxide and 0.5% w/v Sodium chloride for 12 hours at room temperature. Thereafter, the bamboo strips were subjected to a pressure of 2 MPa in a hydraulic press to loosen the fibres. The fibres were extracted by manually scraping the pressed strips. The extracted fibres were rinsed with water and dried in an oven at 60°C until a steady weight was obtained (Onyekwere *et al*. 2019).

### *Surface Modification of Fibres*

The following forms of surface modification treatments were carried out on the natural fibres;

i. Mercerization
ii. Acetylation
iii. Mercerized-Acetylation

### *Route followed in the fibre treatment*

The route followed in the fibre treatment is depicted in fig. 1.

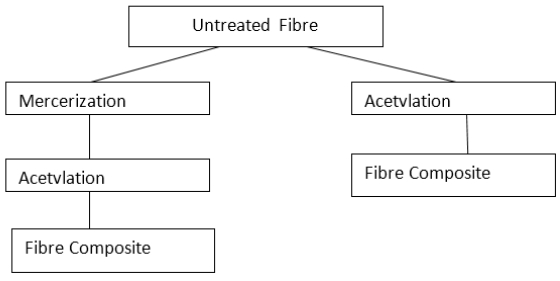


**Figure 1: Treatment route for mercerized-acetylated and acetylated fibre composites.**

***Mercerization treatment of fibre***

In this chemical treatment, alkali solutions wer prepared by diluting sodium hydroxide (NaOH) pallets in water, the amounts of NaOH was varied to achieve 4%, 6%, and 8% by weight of NaOH solutions. Bamboo fibres were immersed in the NaOH solutions each for different soaking time of 30, 60, and 90 minutes at room temperature. After the chemical treatment process, the fibres were washed under running tap water until all traces of excess alkali were completely removed. The fibres were oven dried at $60^0$C until a constant weight was achieved. The dried fibres were stored in plastic bags to avoid exposure to moisture.

***Acetylation treatment of fibre***

Non-catalyzed room temperature acetylation method was employed in this study. 10grams of fibres, from each run in the experimental design, were soaked in a beaker containing 200mL acetic acid (The acetic acid was prepared for different concentrations of 5%, 10% and 15% and soaking time of 10, 30 and 50 minutes). The fibres were then transferred to a beaker containing 200mL acetic anhydride (the acetic anhydride was prepared for different concentrations of 5%, 10% and 15% for soaking time of 30, 60 and 90 minutes). The fibres were removed from acetic anhydride and washed with running water until acid free and dried in an oven at $80^0$C until a constant weight was obtained.

***Mercerized-acetylation treatment of fibre***

The already mercerized bamboo fibres were subjected to acetylation treatment resulting to a surface modification called 'mercerized-acetylated treatment'.

***Composite formulation***

Bamboo fibres were reinforced in polyester resin in order to produce bamboo fibre polyester composites. The bamboo fibre polyester composites were fabricated by conventional hand lay-up process followed by light compression moulding technique with five different fibre loading (10wt, 20wt, 30wt, 40wt and 50wt per hundred resins (PHR), which was coded in this study as P1, P2, P3, P4 and P5 respectively). Unsaturated polyester resin was mixed with 1wt % cobalt naphtenate accelerator and 1wt % MEKP catalyst. The fibres were placed in a mould and the resin mixture was poured evenly on the fibres and allowed to wet completely. A load of 50 kg was applied over the mould for 12 h during the curing process of the composites. Then the cast of composites were removed from the mould and post curing was done at $80^0$C for 4 hour. Silicon spray was used as a releasing agent for the easy removal of cured composite panels from the mould. Samples of proper dimensions, according to ASTM standards were cut out as test specimens from the sheet.

***Characterization of the composite***

The following tests were carried out on the formulated composite

***Tensile Test***

Tensile strength testing of all specimens was conducted as per ASTME 8 standard on composite samples of 15 mm × 200 mm × 3 mm. The gauge length between the two clamps was set at 100 mm. Three identical tests specimen for each section thickness per sample were tested at room temperature with a strain/ loading rate of 5 mm/min using a computerized Instron Testing Machine (Model 3369). The test piece which is of gauge length 100 mm was fixed at the edges of the upper and lower grip of the Instron testing machine tensile force applied until failure. Load displacement plots were obtained on an X – Y recorder and the testing machine displayed the ultimate tensile strength and yield strength.

***Compressive Test***

The compressive test was carried out in accordance with ASTM D 695-96. The specimens were cut to 25mm x 25mm x plate thickness and then ground with carbide sand paper in order to obtain smooth surface. The test was carried out in an Instron testing machine (Model 3369) equipped with a 50 kN load cell and a compression test fixture. Samples were placed on the machine and pressure was applied continuously at the rate of 2mm/min until the samples failed. Three replicas were tested.

***Impact Strength***

Impact testing for all the specimens was done based on ASTM/A29M-15. The tests were carried out using Izod Impact Testing method on Hounsfield Impact Testing Machine (Tensometer Ltd., Croydon, England) on samples having dimension 75mm x 15mm x 3mm. Specimen was notched at an angle of 45º from 28 mm end length of 75 mm. The specimen was subjected to impact blow and the amount of Impact energy absorbed by the specimen was read off on the calibrated scale attached to the machine as a measure of impact strength in Joules.

***Density***

The specimens were weighed in a weighing balance and their weights recorded. Their volumes were calculated from their dimensions. The density was calculated using equation 1.

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

***Hardness Test***

Brinell hardness test was conducted in accordance with ASTM E103. The samples were grinded using grinding machine and polished. After which the sample was fixed into tensiometer where it was subjected to compression load of 250kg for about 15 seconds after which the indented diameter was measured by eye scope. The Brinell hardness number was then calculated. The Brinell hardness (BHN) which is the pressure per unit surface area of the indentation in kg per square meter is calculated with equation 2.

$$\text{BHN} = \frac{w}{(\pi Ds/2)(Ds-\sqrt{Ds^2-d^2})} \qquad (2)$$

Where W is load on indenter, kg

Ds is diameter of steel ball, mm

d is average measured diameter of indentation, mm

***Flexural Test***

Flexural test were performed using 3-point bending method according to ASTM D790-03 procedure. During flexural test, rectangular specimens having dimensions of 100 mm x 20 mm x 3 mm was lied on support spans in Instron Testing Machine (Model 3369) and a load of 5 KN was applied to the centre of the specimen by the loading nose of the Instron machine producing a three point bending at a crosshead speed of 5 mm/min, at a room temperature. The test was stopped when the specimen broke. In each case three samples were tested and average value was reported.

***Moisture absorption***

The water absorption test was carried out according to ASTM D3171. One gram each of both modified and unmodified fibres was weighed and immersed in deionised water and placed in a water bath set at 300C. Samples were taken out after 1hour and 24hours respectively and weighed to determine the amount of water absorbed. The percentage water absorption was calculated using equation 3;

$$\mathbf{W_a} = \frac{w_f - w_o}{w_o} \text{ X } 100 \qquad (3)$$

Where Wa is the percentage water absorption, Wf is the Final weight, Wo is the initial weight.

**Results and Discussion**

The measured properties of acetylated and mercerized-acetylated fibre composites are shown in table 1.

**Table 1: Properties of acetylated and mercerized-acetylated bamboo fibre polyester composites**

| Treatment/Fibre Content | Physical properties | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Compressive Strength | Impact strength | Tensile strength | Tensile modulus | Elongation | Hardness | Percentage moisture absorption | Density | Flexural strength | Flexural modulus |
| **A1** | 69.54 | 110.89 | 49.27 | 1762.63 | 9.36 | 31.68 | 2.061 | 1.1 | 9.70 | 163.66 |
| **A2** | 68.01 | 171.22 | 57.92 | 1786.27 | 7.79 | 34.99 | 2.092 | 0.87 | 29.97 | 497.52 |
| **A3** | 10.89 | 173.17 | 55.16 | 1802.09 | 9.10 | 34.71 | 2.624 | 0.80 | 20.19 | 434.58 |
| **A4** | 11.92 | 135.98 | 56.82 | 1898.69 | 7.72 | 39.75 | 2.972 | 0.75 | 11.89 | 668.29 |
| **A5** | 17.64 | 132.07 | 58.27 | 1992.42 | 7.79 | 49.88 | 3.106 | 0.71 | 37.95 | 772.19 |
| **MA1** | 59.42 | 126.34 | 60.41 | 1707.16 | 8.41 | 31.95 | 1.261 | 1.04 | 17.24 | 308.23 |
| **MA2** | 48.13 | 135.15 | 69.91 | 2304.61 | 8.01 | 34.33 | 1.392 | 0.84 | 28.31 | 671.68 |
| **MA3** | 23.12 | 145.65 | 60.07 | 2112.52 | 7.39 | 35.67 | 1.924 | 0.78 | 33.04 | 1050.54 |
| **MA4** | 22.55 | 154.68 | 69.38 | 2064.30 | 8.09 | 40.34 | 2.001 | 0.73 | 18.99 | 1024.43 |
| **MA5** | 24.36 | 192.01 | 72.96 | 2420.11 | 6.90 | 55.42 | 2.176 | 0.70 | 37.24 | 1096.35 |

Note: A = Acetylated; MA = Mercerized-Acetylated. 1,2,3,4 and 5 represents 10 percent, 20 percent, 30 percent, 40 percent and 50 percent fibres in matrix respectively.

Independent sample t-test was used to compare the means of Acetylated fibre composites and mercerized-acetylated fibre composites for each response being measured at each level of fibre content.

***Effect of mercerization on the compressive strength of acetylated fibre composites***

Figure 2 shows a comparison of the compressive strength at various loads of acetylated and mercerized-acetylated fibre composites. The result shows higher values for acetylated fibre composites at lower fibre contents of P1 and P2. As the fibre content increased from P3 to P5, the values of compressive strength of mercerized-acetylated fibre composites became higher. Acetylation introduces plasticisation to cellulose fibres. Natural fibres that have been rendered near hydrophobic by acetylation forms better bond with hydrophobic polymer matrix. However, as the fibre content increase, there is need to enhance adhesion between fibres. Mercerization enhances interfacial adhesion among fibres and between fibres and their matrix. At higher fibre content, the frayed mercerized fibres forms enhanced interlock within the fibres and between the fibres and the matrix thereby improving the compressive strength of the mercerized-acetylated fibre composites over the acetylated fibre composites.

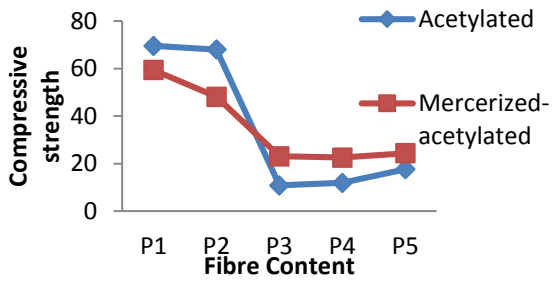


**Figure 2: Comparison of Compressive strengths of Acetylated and Mercerized-acetylated fibre composites**

Table 2 contains the result of independent t-test carried out to compare the statistical significant of the differences in the compressive strength of acetylated fibre composites and mercerized- acetylated fibre composites. At low fibre content of P1 and P2, there is no significant difference in the compressive strength of acetylated and mercerized-acetylated fibre composites. For fibre content P3, the independent sample t-test indicated that compressive strengths were significantly higher in mercerized-acetylated fibre composites (M = 23.12; SD = 5.97) than for acetylated fibre composites (M = 10.89; SD = 5.46), t (8) = 3.38, p = .010, d = 2.14. Similar observation was made in fibre content P4 where the compressive strength of mercerized-acetylated fibre composites (M = 22.55; SD = 6.30) were found to be significantly higher than that of acetylated fibre composites (M = 11.92; SD = 5.89), t (8) = 2.76, p = .025, d = 1.74. At fibre content P5, the compressive strength of mercerized-acetylated fibre composites (M = 24.37; SD = 3.7) were marginally significantly higher than that of acetylated fibre composites (M = 17.65; SD = 5.71), t (8) = 2.21, p = .058, d = 1.40. Taken together, at low fibre contents there is no significant difference in compressive strength of mercerized-acetylated and acetylated fibre composites. However, at higher fibre contents the compressive strengths of mercerized-acetylated fibre composites were significantly higher than that of acetylated fibre composites. This shows that the synergetic effect of acetylation and mercerization lead to enhanced bonding and improved compressive strength.

**Table 2: t-test for equality of means of compressive strength between acetylated and mercerized-acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | 1.443 | 8 | .187 | 10.1197 | 7.0151 | -6.05712 | 26.2966 |
| P2 | 2.198 | 8 | .059 | 19.87118 | 9.03914 | -.97311 | 40.71547 |
| P3 | -3.381 | 8 | .010 | -12.2317 | 3.61731 | -20.5732 | -3.89011 |
| P4 | -2.755 | 8 | .025 | -10.6283 | 3.85832 | -19.5257 | -1.73109 |
| P5 | -2.208 | 8 | .058 | -6.72313 | 3.04519 | -13.7454 | .29909 |

***Effect of mercerization on the hardness values of acetylated fibre composites***

Figure 3 shows a comparison of the Hardness value at various fibre loads of acetylated and mercerized-acetylated fibre composites. Figure 3 indicates no observable difference in the hardness values at fibre contents of P1 to P4. As the fibre content increased to P5 the hardness values of mercerized-acetylated fibre composites became higher than that of acetylated fibre composite.

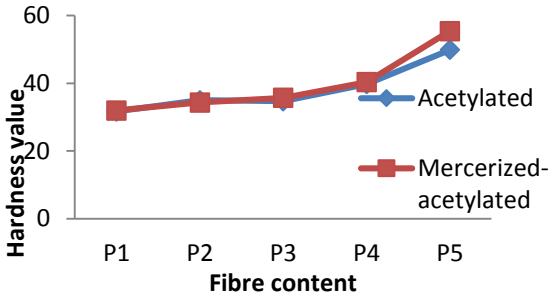


Figure 3: Comparison of Hardness values of Acetylated and Mercerized-acetylated fibre composites

Table 3 contains the result of independent t-test carried out to test for statistical significant of the mean differences in the hardness values of acetylated fibre composites and mercerized- acetylated fibre composites. For all levels of fibre contents, the independent sample t-test indicates that there is no significant difference between the hardness values of mercerized- acetylated and acetylated fibre composites.

**Table 3: t-test for equality of means of hardness values between acetylated and mercerized- acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | -.147 | 6 | .888 | -.27 | -1.839 | -4.77 | 4.23 |
| P2 | .506 | 6 | .631 | .66 | 1.304 | -2.53 | 3.85 |
| P3 | -.484 | 6 | .645 | -.962 | 1.987 | -5.83 | 3.901 |
| P4 | -.190 | 6 | .855 | -.59 | 3.099 | -8.173 | 6.993 |
| P5 | -2.337 | 6 | .058 | -5.545 | 2.373 | -11.350 | .2606 |

***Effect of mercerization on the flexural strength of acetylated fibre composites***

Comparison of the Flexural strength at various loads of acetylated and mercerized-acetylated fibre composites is shown in figure 4. The result shows higher values of flexural strength for mercerized-acetylated fibre composites at fibre contents of P1, P3 and P4. There is no observable difference between the mean flexural strengths of mercerized-acetylated and acetylated fibre composites at P2 and P5.

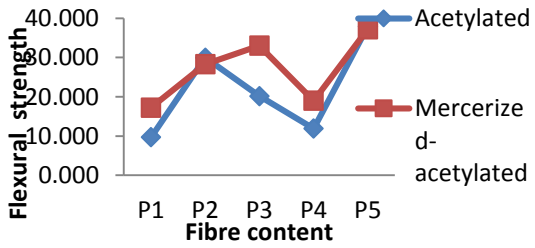


**Figure 4: Comparison of Flexural strengths of Acetylated and Mercerized-acetylated fibre composites**

Table 4 contains the result of independent t-test carried to compare the statistical significant of the differences in the flexural strength of acetylated fibre composites and mercerized- acetylated fibre composites. As can be seen from table 4, there is no significant difference between the flexural strength of mercerized-acetylated and acetylated fibre composites at fibre contents of P1, P2 and P5. For fibre content P3, the independent sample t-test indicated that flexural strengths were significantly higher in mercerized-acetylated fibre composites (M = 33.04; SD = 5.2) than for acetylated fibre composites (M = 20.19; SD = 3.29), t (8) = 3.38, p = .010, d = 2.96. Similar observation was made in fibre content P4 where the flexural strength of mercerized-acetylated fibre composites (M = 18.99; SD = 4.66) were found to be significantly higher than that of acetylated fibre composites (M = 11.89; SD = 2.22), t (8) = 2.76, p = .025, d = 1.94. This shows that as the fibre content increased from P2 to higher levels, the synergic effect of good interlocking of frayed fibres that result from mercerization and near hydrophobic fibres that results from acetylation lead to improvement in the flexural strength.

**Table 4: t-test for equality of means of flexural strength between acetylated and mercerized- acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | -2.934 | 6 | .26 | -7.540 | 2.569 | -13.83 | -1.252 |
| P2 | .315 | 6 | .763 | 1.658 | 5.265 | -11.23 | 14.54 |
| P3 | -4.180 | 6 | .006 | -12.852 | 3.074 | -20.374 | -5.329 |
| P4 | -2.750 | 6 | .033 | -7.095 | 2.579 | -13.408 | -.782 |
| P5 | .138 | 6 | .895 | .710 | 5.152 | -11.898 | 13.318 |

***Effect of mercerization on the flexural modulus of acetylated fibre composites***

Figure 5 shows a comparison of the flexural modulus at various loads of acetylated and mercerized-acetylated fibre composites. The result shows higher values of flexural modulus at all fibre content for mercerized-acetylated fibre composites.

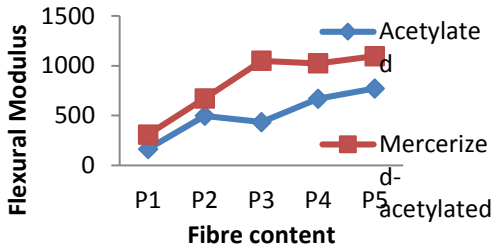


**Figure 5: Comparison of Flexural modulus of Acetylated and Mercerized-acetylated fibre composites**

Table 5 contains the result of independent t-test carried to compare the statistical significant of the mean differences in the flexural modulus of acetylated fibre composites and mercerized-acetylated fibre composites. Table 5 shows that there is no significant difference in the flexural modulus of mercerized-acetylated and acetylated fibre composites at fibre contents of P1, P2 and P5. For fibre content P3, the independent sample t-test indicated that flexural modulus were significantly higher in mercerized-acetylated fibre composites (M = 1050.54; SD = 184.21) than for acetylated fibre composites (M = 434.58; SD = 112.79), t (8) = 3.38, p = .010, d = 4.03. Similar observation was made in fibre content P4 where the flexural modulus of mercerized-acetylated fibre composites (M = 668.29; SD = 170.73) were found to be significantly higher than that of acetylated fibre composites (M = 1024.43; SD = 236.79), t (8) = 2.76, p = .025, d = 1.73. Generally, the flexural modulus showed similar trend with the flexural strength.

**Table 5: t-test for Equality of Means of Flexural modulus between acetylated and mercerized-acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | -1.793 | 6 | .123 | -144.564 | 80.606 | -341.801 | 52.671 |
| P2 | -1.428 | 6 | .203 | -174.166 | 121.98 | -472.64 | 124.312 |
| P3 | -5.703 | 6 | .001 | -615.96 | 107.998 | -880.223 | -351.7 |
| P4 | -2.440 | 6 | .050 | -356.136 | 145.958 | -713.283 | 1.0116 |
| P5 | -1.510 | 6 | .182 | -309.565 | 204.989 | -811.157 | 192.025 |

***Effect of mercerization on the tensile strength of acetylated fibre composites***

Figure 6 shows a comparison of the tensile strength at various loads of acetylated and mercerized-acetylated fibre composites. The result shows higher values of tensile strength at all levels of fibre contents for mercerized-acetylated fibre composites over the acetylated fibre composites.

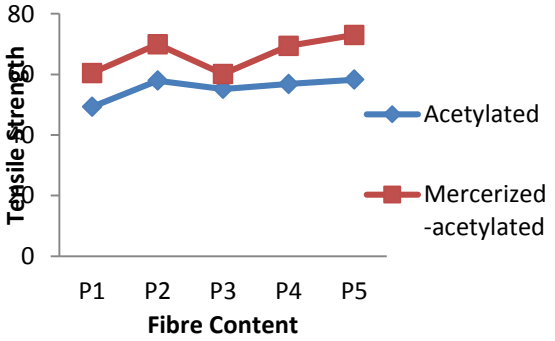


**Figure 6: Comparison of Tensile strengths of Acetylated and Mercerized-acetylated fibre composites**

Table 6 contains the result of independent t-test carried out to test for statistical significant of the mean differences in the tensile strength of acetylated fibre composites and mercerized-acetylated fibre composites. For all levels of fibre contents, the independent sample t-test indicated that there was no significant difference between the tensile strength of mercerized- acetylated and acetylated fibre composites.

**Table 6: t-test for equality of means of tensile strength between acetylated and mercerized- acetylated fibre composites**

| **Fibre Content** | **t-test for Equality of Means** | | | | | | |
|---|---|---|---|---|---|---|---|
| | **t** | **df** | **Sig.(2-tailed)** | **Mean Difference ("Acetylated" - "Mercerized Acetylated")** | **Std. Error Difference** | **95% Confidence Interval of the Difference** | |
| | | | | | | **Lower** | **Upper** |
| P1 | -.917 | 8 | .386 | -11.1318 | 12.143 | -39.13 | 16.869 |
| P2 | -.903 | 8 | .393 | -11.991 | 13.274 | -42.6 | 18.618 |
| P3 | -.373 | 8 | .719 | -4.9128 | 13.163 | -35.207 | 25.441 |
| P4 | -.924 | 8 | .383 | -12.559 | 13.599 | -43.917 | 18.799 |
| P5 | -1.16 | 8 | .278 | -14.694 | 12.636 | -43.834 | 14.44 |

***Effect of mercerization on the tensile modulus of acetylated fibre composites***

Figure 7 shows a comparison of the tensile modulus at various fibre loads of acetylated and mercerized-acetylated fibre composites. The result shows higher values of tensile modulus at all levels of fibre contents for mercerized-acetylated fibre composites over the acetylated fibre composites except for P1, where there is no observable difference in tensile modulus.

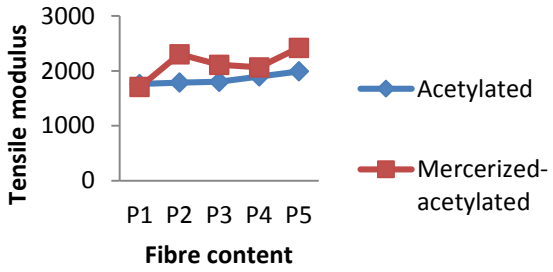


Figure 7: Comparison of Tensile modulus of Acetylated and Mercerized-acetylated fibre composites

The result of independent t-test carried out to test for the statistical significant of the mean differences in the tensile modulus of acetylated fibre composites and mercerized-acetylated fibre composites (Table 7) shows that, for P1, P3, P4 and P5 levels of fibre contents, there was no significant difference between the tensile modulus of mercerized-acetylated and acetylated fibre composites. However, at P2 fibre content, the mean tensile modulus of mercerized-acetylated fibre composites (M = 2304.61; SD = 436.85) were significantly higher than for acetylated fibre composites (M = 1786.28; SD = 191.57), t (8) = 3.38, p = .010, d = 1.54.

**Table 7: t-test for equality of means of tensile modulus between acetylated and mercerized- acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | .355 | 8 | .732 | 55.471 | 156.399 | -305.185 | 416.128 |
| P2 | -2.430 | 8 | .041 | -518.332 | 213.323 | -1010.25 | -26.4069 |
| P3 | -2.041 | 8 | .076 | -310.433 | 152.130 | -661.246 | 40.380 |
| P4 | -.966 | 8 | .362 | -165.607 | 171.467 | -561.011 | 229.797 |
| P5 | -1.755 | 8 | .117 | -427.690 | 243.743 | -989.763 | 134.383 |

***Effect of mercerization on the percentage elongation of acetylated fibre composites***

Comparison of the percentage elongation at various fibre loads of acetylated and mercerized- acetylated fibre composites is shown in figure 8. The result shows higher values for acetylated fibre composites at fibre contents of P1, P3 and P5. The difference between the mean percentage elongation of mercerized-acetylated and acetylated fibre composites at P2 and P5 seems very marginal from figure 8.

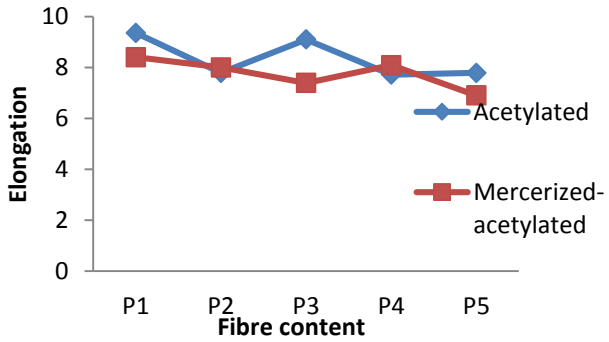


**Figure 8: Comparison of percentage elongation of Acetylated and Mercerized-acetylated fibre composites**

Table 8 contains the result of independent t-test carried out to test for the statistical significant of the mean differences in the percentage elongation of acetylated fibre composites and mercerized-acetylated fibre composites. For all levels of fibre contents, the independent sample t-test indicated that there was no significant difference between the percentage elongation of mercerized-acetylated and acetylated fibre composites.

**Table 8: t-test for equality of means of percentage elongation between acetylated and mercerized-acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | .693 | 6 | .514 | .956 | 1.38 | -2.42 | 4.332 |
| P2 | -.642 | 6 | .545 | -.2111 | .3289 | -1.0159 | .594 |
| P3 | .935 | 6 | .411 | 1.7097 | 1.829 | -3.7156 | 7.135 |
| P4 | -.722 | 6 | .497 | -.37689 | .52187 | -1.6538 | .900 |
| P5 | .829 | 6 | .462 | .8858 | 1.0683 | -2.315 | 4.0867 |

***Effect of mercerization on the impact strength of acetylated fibre composites***

Figure 9 shows a comparison of the impact strength at various loads of acetylated and mercerized-acetylated fibre composites. For acetylated fibre composites, the impact strength increased from P1 up to P3 and declined. Similar observation was made by Obasi *et al* (2018) on acetylated Piassava fibre reinforced polystyrene composite. They attributed the decline in impact strength to the inability of the matrix to wet the fibres as the fibre quantity increased. However, a steady increase in impact strength with increase in fibre content for mercerized- acetylated fibre composites was observed. High impact strength at high fibre contents for mercerized-acetylated fibre composites could be attributed to improved wetting of the alkaline-frayed mercerized fibres which enhance bonding and stress transfer. Figure 9 shows higher impact values for mercerized-acetylated fibre composites at fibre contents of P1 and P4 and P5. While Acetylated fibre composites indicated higher impact strength values at fibre contents of P2 to P3.

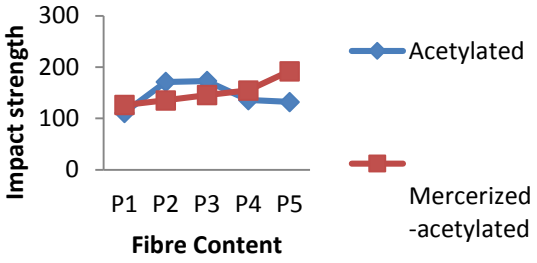


**Figure 9: Comparison of Impact strengths of Acetylated and Mercerized-acetylated fibre composites**

Table 9 contains the result of independent t-test carried to test the statistical significant of the differences in the mean impact strength of acetylated fibre composites and mercerized-acetylated fibre composites. At fibre content P1, the independent sample t-test indicated that impact strengths were significantly higher in mercerized-acetylated fibre composites (M = 126.35; SD = 14.47) than for acetylated fibre composites (M = 110.87; SD = 3.90), t(10) = 2.53, p = .047, d = 1.46. Similar observation was made in fibre content P5 where the impact strength of mercerized-acetylated fibre composites (M = 192.01; SD = 13.03) were found to be significantly higher than that of acetylated fibre composites (M = 132.07; SD = 13.64), t (10) = 7.78, p < .001, d = 4.49. At fibre content P4, no significant difference was observed between the mean impact strength of mercerized-acetylated fibre composites and acetylated fibre composites. At fibre content P2, the independent sample t-test indicated that the impact strengths were significantly higher in acetylated fibre composites (M = 171.22; SD = 16.7) than for mercerized-acetylated fibre composites (M = 135.16; SD = 6.1), t (10) = 4.97, p = .002, d = 2.87. Similar observation was also made in fibre content P3 where the impact strength of acetylated fibre composites (M = 173.18; SD = 16.37) were found to be significantly higher than that of mercerized-acetylated fibre composites (M = 145.66; SD = 7.99), t (10) = 3.7, p = .004, d = 2.14. Generally, at lower fibre contents of P2 and P3 the impact strength of acetylated fibre composites was significantly higher. However, at high fibre contents of P5 the impact strength of mercerized-acetylated fibre composites became higher.

**Table 9: t-test for equality of means of impact strength between acetylated and mercerized- acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | -2.53 | 10 | .047 | -15.45 | 6.12 | -30.60 | -.30 |
| P2 | 4.97 | 10 | .002 | 36.07 | 7.26 | 18.52 | 53.62 |
| P3 | 3.70 | 10 | .004 | 27.52 | 7.44 | 10.95 | 44.08 |
| P4 | -1.87 | 10 | .090 | -18.707 | 9.98 | -40.95 | 3.54 |
| P5 | -7.78 | 10 | .000 | -59.94 | 7.70 | -77.10 | -42.77 |

***Effect of mercerization on the percentage moisture absorption of acetylated fibre composites***

Figure 10 shows the mean comparison of the percentage moisture absorption at various fibre loads of acetylated and mercerized-acetylated fibre composites. The result shows lower values of percentage moisture absorption at all levels of fibre contents for mercerized- acetylated fibre composites over the acetylated fibre composites. Fibre modification through the alkalisation process can decreases the hydrogen bonding capacity of cellulose and eliminates open hydroxyl groups that tend to bond with water molecules. It also dissolves lignin and hemicelluloses (which is the most hydrophilic part of natural fibre), thus reducing the moisture absorption capacity of fibre (Dittenber & GangaRao 2012; Wong *et al*., 2010).

Alkali treatment also enhances the interfacial adhesion between the matrix and bamboo fibres thereby reducing voids and pores within the composite. With the reduction in voids within the specimens, less moisture was accommodated by the composite, which led to less gain in weight. Thus, subjecting the bamboo fibres to both mercerization and acetylation before composite formulation showed better moisture absorption resistance than acetylated fibre composites.

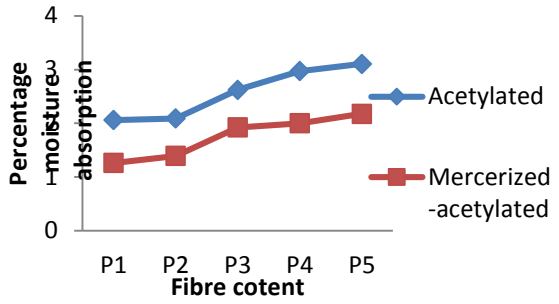


**Figure 10: Comparison of Percentage moisture absorption of Acetylated and Mercerized- acetylated fibre composites**

Table 10 contains the result of independent t-test carried to test the statistical significant of the differences in the percentage moisture absorption of acetylated fibre composites and mercerized-acetylated fibre composites. For fibre content P1, the independent sample t-test indicated that percentage moisture absorption were significantly lower in mercerized-acetylated fibre composites (M = 1.26; SD = 0.039) than for acetylated fibre composites (M = 2.06; SD = 0.039), t (8) = 32.61, p < 0.001, d = 20.61. Similar observation was made in fibre content P2 where the percentage moisture absorption of mercerized-acetylated fibre composites (M = 1.39; SD = 2.09) were found to be significantly lower than that of acetylated fibre composites (M = 2.09; SD = 0.057), t(8) = 19.56, p < .001, d = 12.37. At fibre content P3, the percentage moisture absorption of mercerized-acetylated fibre composites (M = 1.92; SD = 0.023) were significantly lower than that of acetylated fibre composites (M = 2.62; SD = 0.023), t (8) = 48.13, p < .001, d = 30.43. At fibre content P4, the observed difference between the percentage moisture absorption of mercerized-acetylated fibre composites (M = 2.00; SD = 0.0014) were found to be significantly lower than that of acetylated fibre composites (M = 2.97; SD = 0.001), t (8) = 1431.07, p < .001, d = 905.17. The difference between the percentage moisture absorption of mercerized-acetylated fibre composites (M = 2.18; SD = 0.015) were also found to be significantly lower than that of acetylated fibre composites (M = 3.11; SD = 0.014), t (8) = 99.55, p < .001, d = 62.97, at fibre content P5. Summarily, at all levels of fibre contents, the mean percentage moisture absorption of mercerized-acetylated fibre composites were significantly lower than that of acetylated fibre composites.

**Table 10: t-test for equality of means of percentage moisture absorption between acetylated and mercerized-acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | 32.608 | 8 | .000 | .800 | .02453 | .74342 | .85658 |
| P2 | 19.557 | 8 | .000 | .700 | .03579 | .61746 | .78254 |
| P3 | 48.131 | 8 | .000 | .700 | .1454 | .66646 | .73354 |
| P4 | 143.072 | 8 | .000 | .971 | .00068 | .96904 | .97216 |
| P5 | 99.547 | 8 | .000 | .930 | .00934 | .90846 | .95154 |

***Effect of mercerization on the density of acetylated fibre composites***

Figure 11 shows the mean comparison of the density at various fibre loads of acetylated and mercerized-acetylated fibre composites. The result shows marginally, higher values at all levels of fibre contents for acetylated fibre composites over the mercerized-acetylated fibre composites. Acetylation is a substitution reaction which increases the weight of natural fibres (Yakubu et al, 2013; Kai-Huang et al, 2014). On the other hand, mercerization produces more cleaning effect removing natural and artificial impurities from the fibres. Thus, mercerized-acetylated has lower density than acetylated fibre composites.

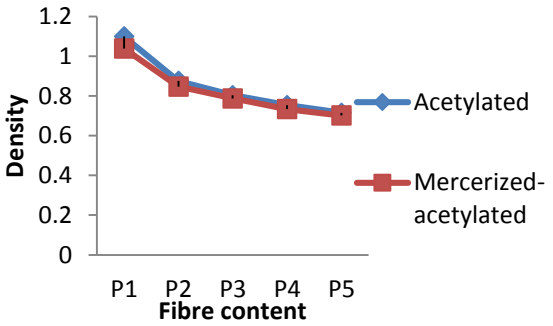


**Figure 11: Comparison of Density of Acetylated and Mercerized-acetylated fibre composites**

Table 11 contains the result of independent t-test carried out to test the statistical significant of the differences in the mean density of acetylated fibre composites and mercerized-acetylated fibre composites.

**Table 11: t-test for equality of means of density between acetylated and mercerized- acetylated fibre composites**

| Fibre Content | t-test for Equality of Means | | | | | | |
|---|---|---|---|---|---|---|---|
| | t | df | Sig.(2-tailed) | Mean Difference ("Acetylated" - "Mercerized Acetylated") | Std. Error Difference | 95% Confidence Interval of the Difference | |
| | | | | | | Lower | Upper |
| P1 | 2.739 | 8 | .026 | .06 | .0219 | .00948 | .11052 |
| P2 | 3.960 | 8 | .004 | .028 | .00707 | .01169 | .04431 |
| P3 | 1.600 | 8 | .148 | .016 | .0100 | -.00706 | -.03906 |
| P4 | 1.622 | 8 | .143 | .02 | .01233 | -.00843 | -.04843 |
| P5 | 1.807 | 8 | .108 | .014 | .00775 | -.00386 | -.03186 |

For fibre content P1, the independent sample t-test indicated that density were significantly lower in mercerized-acetylated fibre composites (M = 1.04; SD = 0.042) than for acetylated fibre composites (M = 1.1; SD = 0.024), t(8) = 2.739, p =

.026, d = 1.73. Similar observation was made for fibre content P2 where the density of mercerized-acetylated fibre composites (M = 0.85; SD = 0.0083) were found to be significantly lower than that of acetylated fibre composites (M = 0.88; SD = 0.013), t (8) = 3.96, p < .001, d = 2.50. At higher level of fibre content, the difference in density was found not to be statistically significant.

**Summary**

Independent sample t-test was used to compare the means of Acetylated fibre composites and mercerized-acetylated fibre composites for each response being measured at each level of fibre content. The results indicated that subjecting the fibres to mercerization before acetylation has no significant effect on hardness values, tensile strength and percentage elongation of the resulting composites. However, at higher fibre contents of P3 and P4, mercerized-acetylated fibrecomposites showed significantly higher Compressive strength, flexural strength, flexural modulus, and higher impact strength at fibre content P5. Percentage moisture absorption of mercerized-acetylated composites was significantly lower than that of acetylated fibre composites at all levels of fibre content. It was also observed that the density of mercerized-acetylated fibre composites were significantly lower than that of acetylated fibre composites at lower fibre content levels of P1 and P2. However, no significant difference was found in density between mercerized-acetylated and acetylated fibre composites at higher levels of fibre contents. Table 12 summarised the results of the study.

**Table 12: Effect of mercerization on the physical properties of acetylated composite using independent sample t-test**

| Treatment/Fibre Content | Physical properties | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Compressive Strength | Impact strength | Tensile strength | Tensile modulus | Elongation | Hardness | Percentage moisture absorption | Density | Flexural strength | Flexural modulus |
| **P1** | 0 | + | 0 | 0 | 0 | 0 | + | + | 0 | 0 |
| **P2** | 0 | - | 0 | + | 0 | 0 | + | + | 0 | 0 |
| **P3** | + | - | 0 | 0 | 0 | 0 | + | 0 | + | + |
| **P4** | + | 0 | 0 | 0 | 0 | 0 | + | 0 | + | + |
| **P5** | 0 | + | 0 | 0 | 0 | 0 | + | 0 | 0 | 0 |

0 = no difference observed, + = Positive significance difference, - = Negative significant difference

***Conclusion***

In this study, tests were carried out to determine if there are differences in the properties of composites produced using acetylated fibres (fibres that were acetylated directly from crude fibres) and composites made from mercerized-acetylated fibres (fibres that were subjected to mercerization before acetylation). The obtained results show that mercerizing of fibres before acetylation has significant effect on some properties of acetylated fibre composites. Therefore, this research should serve as a decision guide to composite manufacturers on the surface modification route to follow based on the desired properties and the particular application of their composite product.

***References***


Anike, D. C., Onuegbu, T. U., Ugochukwu-Aniefuna, A. A., And Ezuh, C. S. (2015). Comparison of Acetylation and Alkali Treatments on the Physical and Morphological Properties of Raffia Palm Fibre Reinforced Composite. Science Journal of ChemistryScience Journal of Chemistry, 3 (4), 72-77.

Dittenber, D. B. and Ganga, R., (2012). Critical Review of Recent Publications on Use of Natural Composites in Infrastructure, Composites Part A: Applied Science and Manufacturing, 43 (8), 1419-29.

Hassan, M.A., Onyekwere, O. S. and Ibrahim, M. (2014). Stripped Oil Palm Fruit Bunch Fibre-Rice Husk Hybrid Composite For Automobile Applications. European International Journal of Applied Science and Technology, 1 (4), 85-94.

Hassan, M. A., Onyekwere, O. S., Yami, A., and Raji, A. (2014). Effects of Chemical Modification on Physical And Mechanical Properties of Rice Husk - Stripped Oil Palm Fruit Bunch Fibre Polypropylene Hybrid Composite. IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE), 11 (4), 1-5.

Jayabal, S., Sathiyamurthy, S., Loganathan, K. T. and Kalyanasundaram, S. (2012). Effect of soaking time and Concentration of NaOH Solution on Mechanical Properties of Coir– Polyester Composites. Bulletin of Material Science, 35 (4), 567-574. Kai-Huang, M. Z., Guangzhi Z, X. J. and Dan, H. (2014). Acetylation Modification of Rice Straw Fibre and Its Thermal Properties. Cellulose Chemistry and Technology, 48 (3-4), 199- 207.

Maya, J., Rajesh, J. and Anandjiwala, D. (2008). Recent Development in Chemical Modification and Characterisation of Natural Fibre-Reinforced Composite. Polymer Composites, 29, 187-207.

Obasi, H. C., Nwanonenyi, S. C., Chiemenem, L. I., and Nwosu-Obieogu, K. (2018). Effects of Fibre Acetylation and Fibre Content on the Properties of Piassava Fibre Reinforced Polystyrene. Futo Journal Series, 4 (1), 475-491.

Olorunnisola, A. O., and Agrawal, S. P. (2013). Effects of Sodium Hydroxide Concentration and Fibre Content on Cement-Bonded Composites from Eucalyptus Veneer Waste. Pro Ligno, 9 (4), 504 - 509.

Onyekwere, O. S., Igboanugo, A. C. and Adeleke, T. B. (2019). Optimisation of Acetylation Parameters for Reduced Moisture Absorption of Bamboo Fibre Using Taguchi Experimental Design and Genetic Algorithm Optimisation Tools. Nigerian Journal of Technology, 38 (1), 104-111

Onyekwere, O. S. and Igboanugo, A. C. (2019). Optimal Parameter Setting for Mercerization of Bamboo Fibres. Journal of Science and Technology Research, 1(1), 12-22

Papadopoulos, A. N., and Traboulay, E. (2002). Dimensional Stability of OSB made from Acetylated Fir Strands. Holz-Werkstoff, 60, 84-87.

Prasad, L. S., Kumar, S. M., and Rajesh, G. (2014). Effect of Fibre Loading and Successive Alkali Treatments on Tensile Properties of Short Jute Fibre Reinforced Polypropylene Composites. International Journal of Engineering Science Invention, 3 (3), 30 - 34.

Shehu, U., Isa, M. T., Aderemi, B. O. and Bello, T. K. (2017). Effects of NaOH Modification on the Mechanical Properties of Baobab Pod Fibre Reinforced LDPE Composites. Nigerian Journal of Technology, 36 (1), 87 - 95.

Sjorstrom, E. (1981). Wood Chemistry: Fundamentals and Applications. London: Academic Press.

Somashekar, S. and Shanthakumar, G. C. (2014). Effect of Alkali Treatment on Mechanical Properties of Sisal -Reinforced Epoxy Polymer Matrix Composite. International Journal of Mechanical Engineering and Robotic Research, 3 (4), 441 - 450.

Wong, K. J., Yousif, B. F. and Low, K. O. (2010). The Effects of Alkali Treatment on the Interfacial Adhesion of Bamboo Fibres. Journals of Material Design and Application, 224, 139 - 148.

Yakubu, A., Gabriel. A. O., Cheku, M. and Paul, A. M. (2013). Acetylation of Wood Flour from Four Wood Species Grown in Nigeria Using Vinegar and Acetic Anhydride. International Journal of Carbohydrate Chemistry, 20, 1-6