EFFECTS OF CHEMICAL SURFACE MODIFICATIONS ON COMBRETUMDOLICHOPETALUM FIBER FOR SUSTAINABLE APPLICATIONS

Walter P. E[*]

Azeez T.O[**]

Onukwuli O. D[*]

AtuanyaC. U[***]

ABSTRACT

The need to minimize the environmental effect posed by *Combretum dolichopetalum*(*C. dolichopetalum*) fiber and its effective use in polymer applications necessitated this study. Effect of chemical surface treatments on*C. dolichopetalum* fiberfor sustainable applications was aimed to be investigated. Retting method was employed for extraction of *C. dolichopetalum* fiber and the proximate contents were determined using gravimetric analysis. The fibers were mercerized and acetylated separately by 0 – 15 % within time intervals of 10 – 50 minutes and 30 – 150 minutes respectively at room temperature. The tensile strength in accordance with ASTM standards, aspect ratio, water absorption and microstructural analysis were studied with aids of scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FTIR). The result shows that the fibers contain 59.22 % cellulose, 20.30 % hemicelluloses, 2.40 % lignin, 9.32 % pectin, 4.08 % moisture and 2.26 % wax. The tensile strength of the mercerized and acetylated fibers increased by 877.58 % and 601.73 % of the unmodified fibers at 6 and 12 % concentration for 30 minutes due to increased aspect ratio with reduced density and water adsorption. SEM and FTIR studies revealed changes in surface topography and surface chemistry of the treated fibers.

Keywords - *C. dolichopetalum* fiber, mercerization, acetylation, mechanical properties, physical properties

[*] Chemical Engineering Department, Nnamdi Azikiwe University, P. M. B. 5025, Awka, Anambra State Nigeria.

[**] Biomedical Technology Department, Federal University of Technology, P. M. B. 1526, Owerri, Imo State Nigeria.

[***] Metallurgical and Material Engineering Department, Nnamdi Azikiwe University, P. M. B. 5025, Awka, Anambra, State Nigeria.

Introduction

Environmental enlightenment, new rules and regulations with gradual depletion of petroleum resources worldwide are among factors that are drawing attention of researchers to the use of ecofriendly fibers for economic and technological sustainability in the World today (Sumaila et al, 2013; Zhong et al, 2010; SrinivasaandBharath, 2011).The numerous attractive properties of natural plant fibers such as renewability, cost effectiveness, availability, relative high specific strength and modulus, light weight, less abrasiveness, desirable fiber aspect ratio, minimal health hazards,and good thermal, electrical and acoustic insulating natureplaced them in high demand as possible replacement to synthetic fibers but increased the biomass on disposal to the environment and incompatibility with polymer matrix (Mohd et al, 2013; Azeez et al, 2013).

El-Shekial et al (2013) reported that the inherent polar and hydrophilic nature of plant fibers causes incompatibility with hydrophobic polymer matrix and poor orientation of the fibers in matrix altered the mechanical and thermal properties of the composites which truncates chances of forming good compositesRaju et al, 2012; Kim and Song, 1997; Mishra and Acharya, 2010). This hydrophilic behaviour which increases moisture absorption not only leads to swelling, creation off voids at the interface butreduces the extent of reinforcement with polymer matrix. These drawbacks may be attributed to molecular structure of cellulose contains alcoholic hydroxyl groups which forms intermolecular and intra-molecular hydrogen bonds with other cellulose macromolecule in the cell wall of the fibers (Osorio et al, 2012). Another factor that influences the high level of moisture absorption in fibers applications is their particular structure[6]. These limitations have been overcome by suitable physical, chemical or enzyme treatments which improves natural fiber hydrophilic nature, interfacial bonding between matrix and fiber, surface roughness and wettability with decreased moisture absorption (Tanobe et al, 2005). At optimal conditions, treatments improved the tensile properties and moisture absorption characteristics of fibers (Hossain et al,2013).Researchers reported that the mercerization and acetylation as chemical surface treatments significantly improved tensile properties and/or water absorption resistance (Punyamurthy et al, 2014; Noorunnisa et al, 2011; Hussain et al, 2011; Siva et al, 2013; Sampathakumar et al, 2012; Ejikeme et al, 2014).

*C. dolichopetalum*plantis commonly known as *sun birds wine*plant.Itis a specie of the genus *Combretum* which is part of a large family of herbs, shrubs and trees referred to as

Combretaceae. Gedson et al (2012) reported the usefulness of *Combretum*extracts extensivelyin traditional medicinebut disposal of the crystalline fiber to the environment, thereby, increased the biomass load. Thisresearch work is aimed to establish its possible characteristics, and effect of mercerization and acetylation on *C. dolichopetalum* fibers for sustainable polymer applications such as insulator, building and construction industries as panels for portioning and flooring or wall coverings, architectural landscaping to replace the hardwood currently used mainly for cost benefit, enhance performance and sustenance of applications.

Materials and Methods

Fiber extraction

C. dolichopetalum fiber was extracted from the plant by retting extraction process using deionized water in accordance with method described by Sumaila et al[1] in which bast plant were immersed in deionized water and washed every 3 days for 21 days, then sun dried for 7 days and oven dried at temperature of 60^0C for 2hours.

Proximate composition of *C. dolichopetalum* fiber

The gravimetric analysis procedure described by Thygesen et al (2007)was used to determine the proximate composition of *C. dolichopetalum*bast plant and its fiber after milled into particles that could pass through 1 mm sieve. The proximate composition of the fiber quantify include moisture content, dry matter, water soluble, ash, wax / fat, pectin, lignin, hemicelluloses and cellulose.

Mercerization andAcetic anhydride modification

The strand of *C. dolichopetalum* fiber were cut into 150 mm length and treated with 0 – 15 % NaOH as well as acetic anhydride at room temperature for 10 – 50 minutes and 0 – 150 minutes respectively. The fibers were then washed severally with deionized water until neutral pH was obtained. The fibers were finally dried in an air oven at 60^0C for 2 hours.

Tensile strength

Tensile properties were evaluated using a Universal Testing Machine Instron 3369 in accordance with ASTM Test Method D638-03. Strand of fibers were cut to 150mm and the tensile test was conducted on the fiber gauge length of 100mm with average diameter of 0.48± 0.03 mm (range for both untreated and treated of *C. dolichopetalum* fiber.

Fiber aspect ratio

The length of 20 randomly sampled *C. dolichopetalum* fiber were measured and recorded. The fibers' diameters were measured at different locations along their length using micrometer screw gauge and the average diameter of 10 randomly sampled *C. dolichopetalum* fiber were measured at 5 positions as described by Brindha et al (2005). The mean aspect ratio was calculated using equation (1) as given by Herrera – Franco and Valadez – Gonzalez (2005).

$$Aspect\ ratio = \frac{L_f}{D_f} \quad (1)$$

Where $L_f$is the fiber length and $D_f$is the fiber diameter.

Fiber density

The method described by Brigida et al (2010) was used to measuredensity of the fibers by pyknometry. Fiber samples were selected and bound into a bundle and its mass measured on a digital weighing balance with resolution 0.001 g. The volume of this fixed mass of *C. dolichopetalum* fiber. The density was calculated using equation (2):

$$\rho_f = \frac{M}{V} \quad (2)$$

Where $\rho f$is density of fiber measured in grams per cubic centimeters, M is the fiber quantity immersed in deionized water in grams and V is the volume water displaced by the fiber.

Water absorption

The test was carried out as per ASTM D – 570. Prior to testing, the *C. dolichopetalum* fibers were dried in an oven at 70^0C for 24 hours. The fibers were then soaked in deionized water for 24 hours at room temperature. The fibers were removed, rid of surface water and immediately weighed and the moisture absorption determined by weight difference. The process was continued until equilibrium was attained. Moisture absorption was determined by percentage mass gain and calculated as given by Sinha and Rout (2007);

$$Water\ absorption\ (\%) = \frac{M_t - M_0}{M_0} x100\% \quad (3)$$

Where M_t is the mass of the sample after conditioning in grams (wet weight), M_o is the mass of the sample before conditioning in grams (dry weight).

Scanning electron microscope (SEM)

The SEM micrographs of untreated *C. dolichopetalum* fibers and treated fibers were taken. To avoid electron - charging effects, fiber samples were spotter coated with gold.

Fourier transform infrared (FTIR)

Infrared spectra of raw and chemically modified *C. dolichopetalum* fiber were measured. In order to obtain good resolution spectra, the fibers were crushed into small particles in liquid nitrogen.

RESULTS AND DISCUSSION

Fiber chemical composition

Theresult of chemical composition*C. dolichopetalum bast plant and* fiber were presented in Table 1 with high cellulose content of *C. dolichopetalum* fiber which indicated fiber possesses significant strength and it may be used to reinforce polymers. Cellulose, hemicelluloses, pectin and lignin contain polar groups which responsible for hygroscopic nature and moisture content of *C. dolichopetalum* fibers, and hydrogen bonds build up between the rich carbonyl groups of pectin and polar solvents facilitates moisture absorption with reduced mechanical properties of the fibers (Celino et al, 2014). More so, the significant amount of hemicelluloses and lignin in *C. dolichopetalum* fiber is an indication that the *C. dolichopetalum*fiber may be biodegradable, thermal degradable and responds to chemical treatments (Saira and Khan, 2007).

Table 1: Chemical constituents of *C.dolichopetalum* plant and fiber

Sample	Moisture content (%)	Dry matter (%)	Ash content (%)	Wax content (%)	Water soluble (%)	Pectins (%)	Lignins (%)	Hemicelluloses (%)	Celluloses (%)
C_b	3.65	93.65	1.29	6.42	2.31	9.6	22.69	54.78	2.92
C_f	4.08	95.92	2.26	4.7	2.4	9.32	20.3	59.22	1.8

Subscript *b* and *f* represent bast and fiber respectively.

Mechanical properties analysis of the fiber

The tensile properties of unmodified and modified*C. dolichopetalum* fiber at variable concentration and time of alkali and acetic anhydride modification of the fibers were illustrated in Figures 1(a) and 1(b) respectively. Tensile strength of unmodified *C. dolichopetalum* fiber represented at zero treatment conditions of fibers.It can be deduced from Figure 1(a)that the tensile strength of 6 and 9 % NaOH treated *C. dolichopetalum* fiberdecreases for the 10 minutes

which may be attributed to adaptation to new operating conditions and it is in agreement with the report of Khalil et al (2007)and later increasesto ultimate tensile strength of 511.54 and 153.99 % of unmodified fiber for50 and 40 minutes respectivelyand 3% NaOH treated *C. dolichopetalum* fibergave ultimate tensile strength of 229.69 % of the unmodified *C. dolichopetalum* fiber for 10minutes which indicated that fiber may not experiences the lag phase. 12 and 15 % NaOH treatment resulted to anincrease in tensile strength up to ultimate level of601.73and 546.93% for 30 and 10minutes respectively.In the case of acetic anhydride treatment of *C. dolichopetalum* fiber as illustratedin Figure 1(b), the tensile strength increase to ultimate level of 527.06, 877.58, 202.21, 355.70 and 630.61% of unmodified fiber for 3, 6, 9, 12 and 15 % of acetic anhydride at treatment time of 30, 30, 150, 150 and 60 minutes respectively.

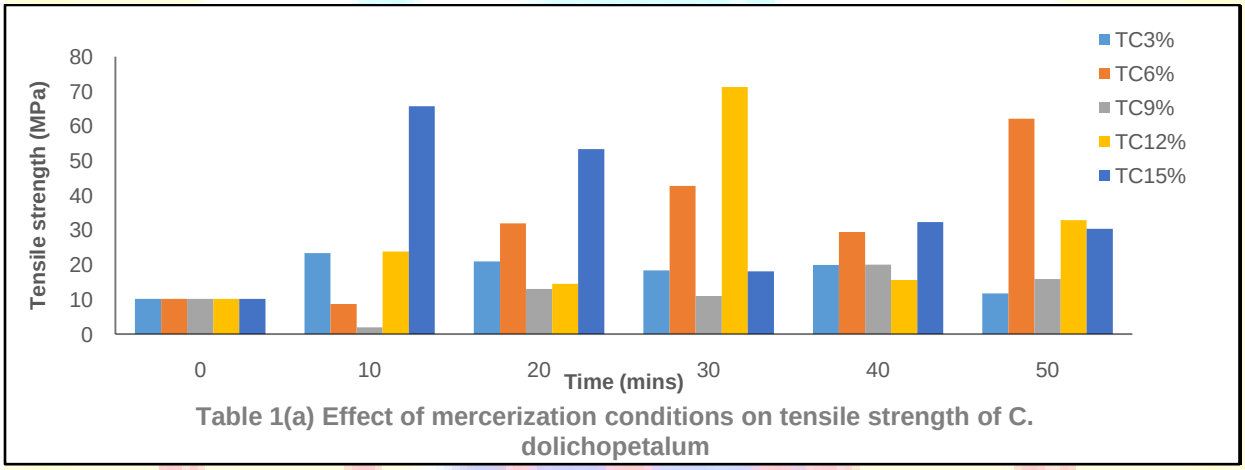


Table 1(a) Effect of mercerization conditions on tensile strength of C. dolichopetalum

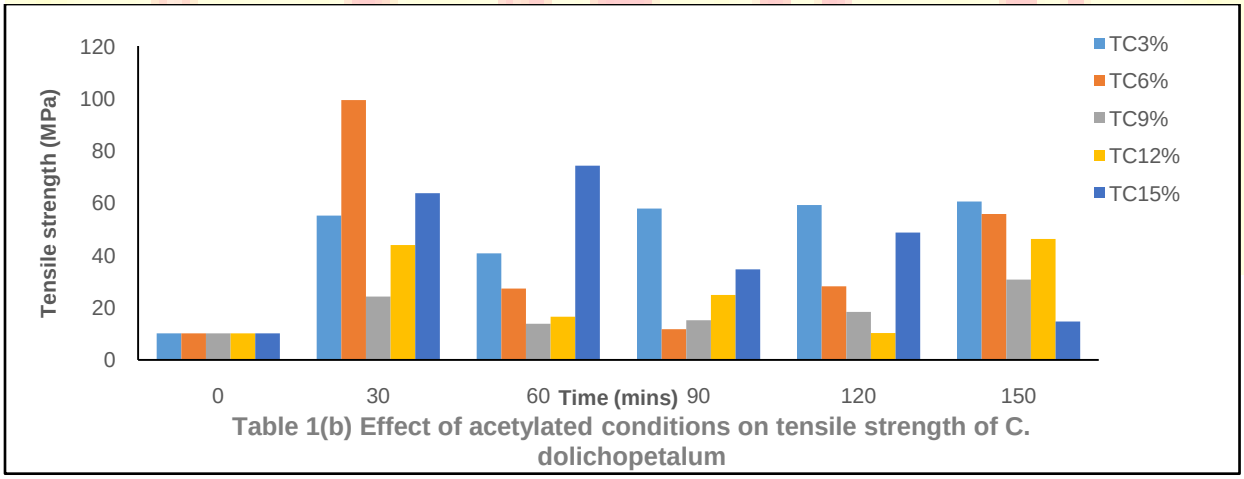


Table 1(b) Effect of acetylated conditions on tensile strength of C. dolichopetalum

However, the ultimate tensile strength of mercerized and acetylated fiber was obtained at treatment conditions of 12 % NaOH and 6 % acetic anhydride for 30 minutes while that of

tensile modulus at treatment condition of 9 and 6 % for 90 and 10 minutes as illustrated in Figure 2(a) and 2(b) respectively. The decrease in tensile strength of fiber may be attributed to structural

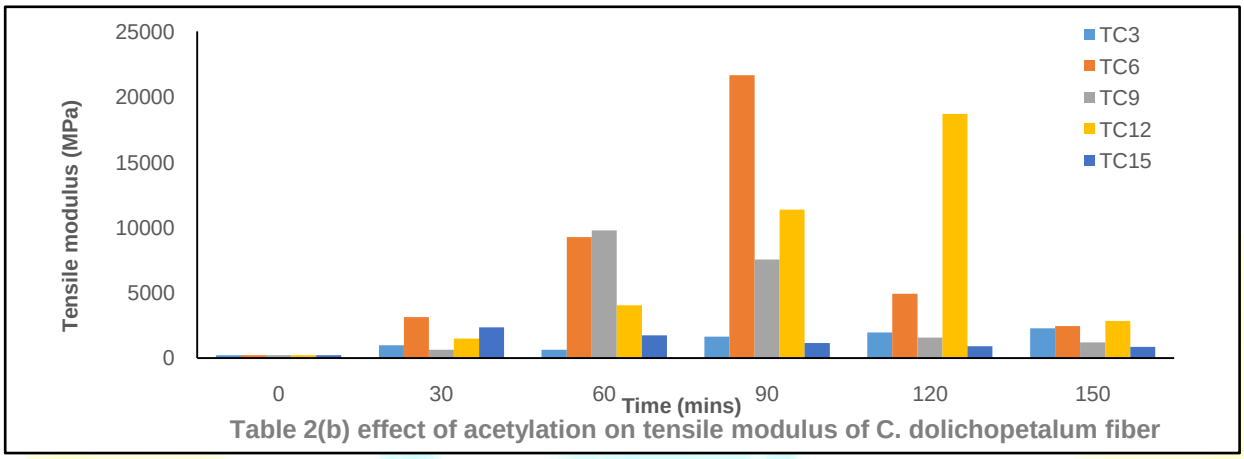


Table 2(b) effect of acetylation on tensile modulus of C. dolichopetalum fiber

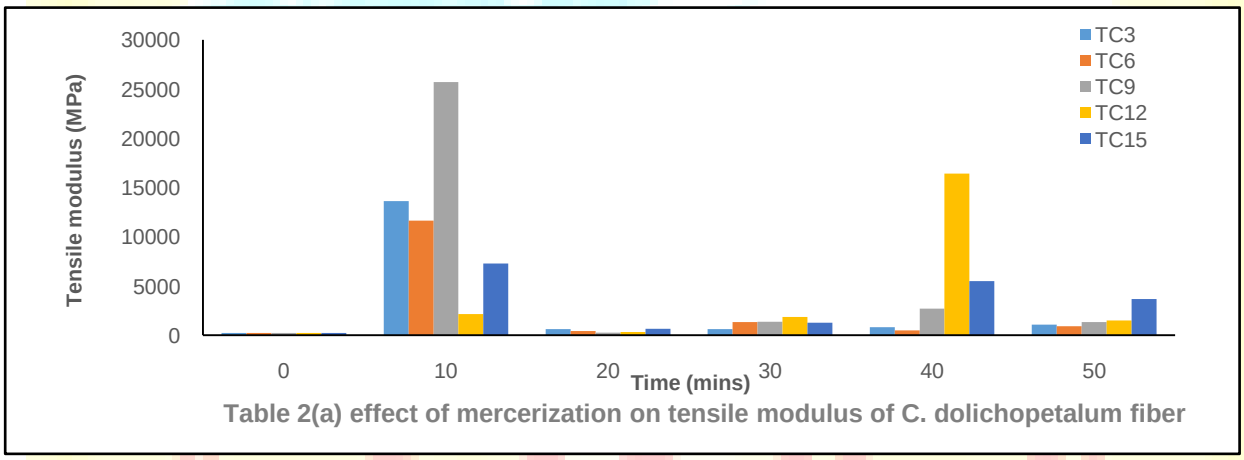


Table 2(a) effect of mercerization on tensile modulus of C. dolichopetalum fiber

changes of the fiber surface and cell wall after treatment by cellulose delignification and degradation, thereby, disrupting bonding and causes increase in surface roughness (Khalil et al, 2007; Rong et al, 2001).

Fiber aspect ratio and density

The mean aspect ratio and density of modified and unmodified*C. dolichopetalum* fiber at ultimate treatment conditions were presented in the Table 2. It was observed that there is decrease in diameter of modified fiber with NaOH and acetic hydridedue to removal of surface constituents, thereby, resulted in shrinkage of cell wall thickness, thus increased the aspect ratio. This is in similar to the report of Osorio et al (2012).It was observed that increase in aspect ratio resulted in remarkable improvement in fibertensile properties as result of NaOH and acetic anhydride treated *C. dolichopetalum* fiber is in agreement with report ofAshori and Nourbakhsh

(2010).It was obtained that mercerization and acetylation of *C. dolichopetalum*fiber reduced the density by 2.58 and 1.72 % of untreated respectively which may be attributed to removal of lignins, hemicelluloses and pectins content.

Table 2: Aspect ratio of *C. dolichopetalum* fibers

Sample	Average length (mm)	Diameter (mm)	Aspect ratio	Density (g/cm^{-3})
Raw fiber	100	$0.0505^{a}\pm 0.025^{b}$	1980.2	0.233
Mercerized fiber	100	$0.0465^{a}\pm 0.014^{b}$	2150.5	0.227
Acetylated fiber	100	$0.0470^{a}\pm 0.053^{b}$	2127.7	0.229

Superscript a and b represent mean and standard deviation of the number respectively

Water absorption

Figure 3 shows the water uptake of unmodified and modified *C.dolichopetalum* fiber at room temperature for period of 24 hours. The water absorption of unmodified, mercerized and acetylated fibersincreases with time until it reaches a maximum water absorption.The graph also shows that the percentage water absorption of the raw fiber is 9.16 % compared to 6.79 % and 6.55 % for mercerized and acetylated fiber respectively which indicated that water absorption of mercerized and acetylated fiber decreased by 9.93 and 13.75 % of unmodified fiber. The acetic anhydride treatment of *C. dolichopetalum* fiber is more stable compared with NaOH treatment due to hydrophilicity of the cellulose fiber. Water molecules penetrate the microcrack within the fiber induced fiber swelling and eventually resulted to failure. This may be attributed to the ability of the polar molecular sites of the untreated fibers to form hydrogen bonds with the water molecules. The removal of amorphous constituents (pectin, lignin, hemicelluloses and impurities) from the fiber surface by mercerization removes the polar group and prevents the formation of hydrogen bonds with water molecules which reduced the water absorption while the coupling of the hydroxyl groups of the non-cellulose constituents with acetyl groups fromacetylationreduced the ingress of water molecule into the fiber which makes it to be more hydrophobic in nature.This is similar to the report of researchers (SavitaandPrecti, 2012; Shah et al, 2012; Zafeiropoulos et al, 2002; Ramadevi and Sampathkumar, 2012; Arifuzzaman et al, 2009; Rao et al, 2007).

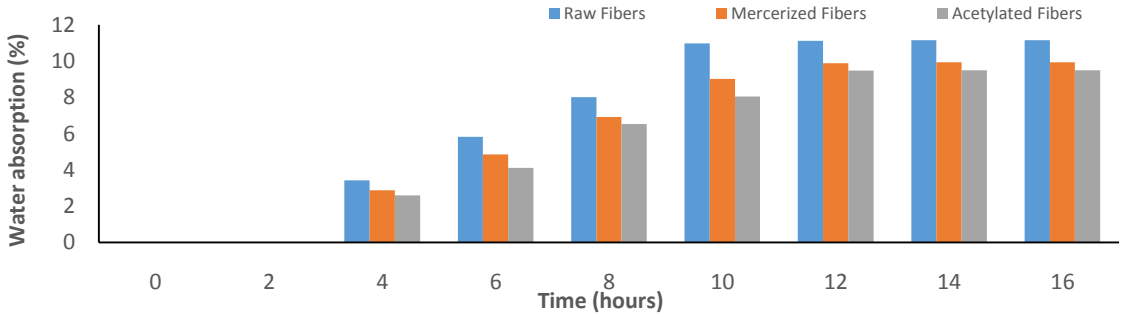


Figure 3: Effect of chemical surface modificationof C. dolichopetalum fiber on water absorption at room temperature

Surface topography studies of fiber by SEM

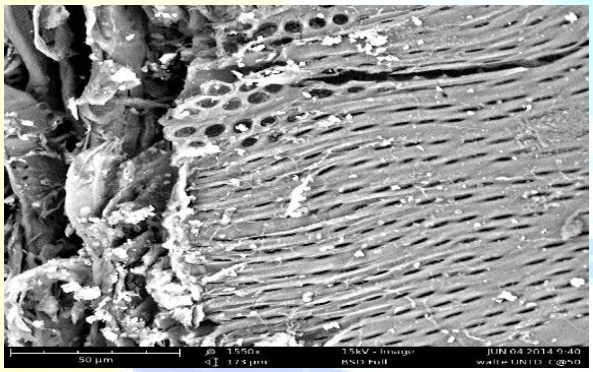


Figure 4 shows SEM micrograph of the unmodified and modified *C. dolichopetalum* fibers.

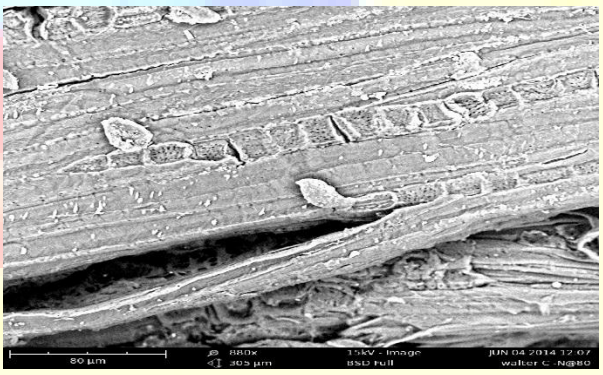


Figure 4(a) shows an irregular surface having variable roughness with fibrillar structure as revealed from the fiber topography with a better mechanical interlocking of the matrix in composites development. Figure 4(b) shows that the rough surface of the mercerized fiber is produced by the dissolution and leaching out of lignin and hemicelluloses components of the fiber, with clean pores and thinning of the fiber. However, in the micrograph of acetylated fiber as shown in the Figure 4(c), the fiber surface is irregular, thinning and rough compared with

mercerized fiber but clean than that of untreated one which increases fiber adhesion as reported by Ishidi et al (2011).

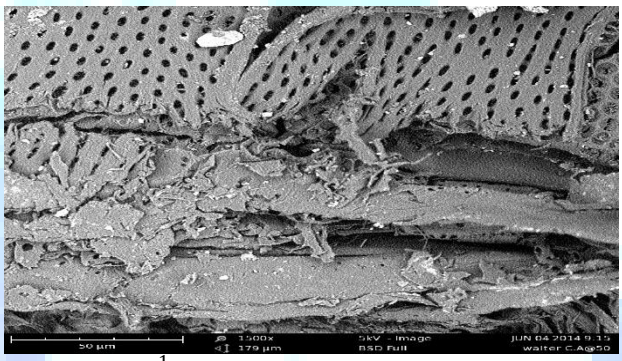
(a) (b)

(c)

Figure 4: SEM micrographs of *C. dolichopetalum* fiber at a magnification of x1500 (a) unmodified (b) mercerized (c) acetylated

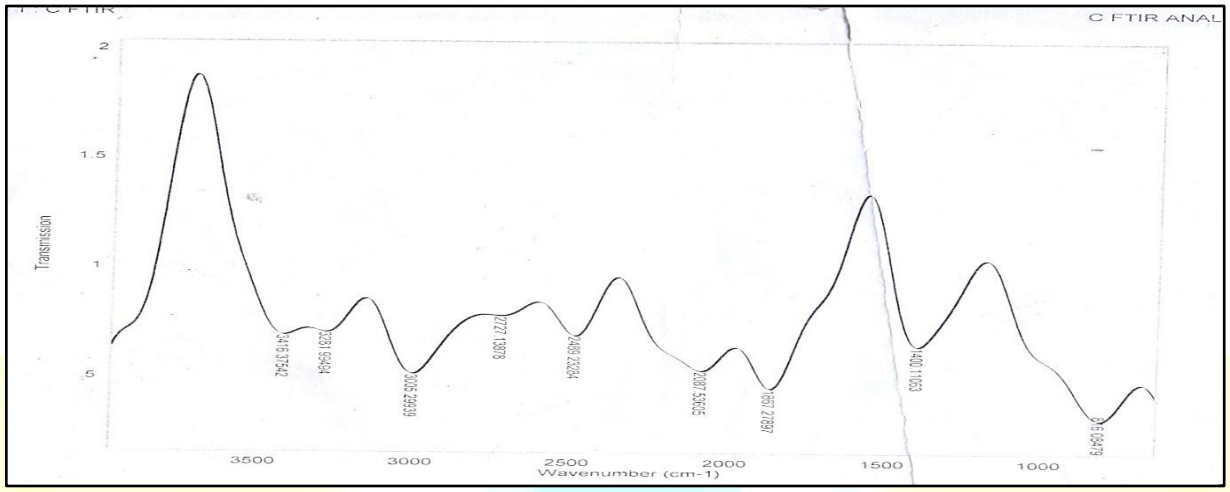
FTIR analysis of fiber

Figure 5 shows the FTIR spectrum of *C. dolichopetalum* fiber. It was obtained in Figure 5(a) that the absorption peak at 816.08479 cm^{-1} corresponds tounsaturated -CH stretching of branch



alkenes. The band at 1400.11063 cm^{-1} is attributed to carbon – carbon stretching of the aromatic ring in the lignin components, while a strong peak at 1867.27897 cm^{-1} is observed due to carbonyl stretching present in lignin and other cellulose components. The absorption peaks at 2087.53605 cm^{-1} and 2489.23284 cm^{-1} are linked to –C=C and –C-O-C bonds in the cellulose chain respectively. The band peak at 2727.13878 cm^{-1} is attributed to H-C=O:C-H functional group of aldehydes stretching while 3005.29939 cm^{-1} is associated with –CH stretching of aromatics in the non-cellulose constituents. The peaks at 3281.99494 cm^{-1} and 3416.37542 cm^{-1} arises from –C=C stretching and the characteristics –OH stretching vibration. Figure 5(b) illustrates the spectrum of mercerized *C. dolichopetalum* fiber. The spectrum shows a reduction in unsaturated –CH stretching intensity of alkane branches and the shifting of the peak from 816.0848 cm^{-1} to 924.708 cm-1 indicates participation of some unsaturated –CH branch of alkenes in the reaction. The band peak at 1386.69725 cm^{-1} is associated with –CH deformation while the sharp peak at 1664.22277 cm^{-1} is attributed to the –C=C stretching of alkenes. The cleansing effect of mercerization is the probable reason for the reduction of absorption peak of carbonyl groups present in lignin and other cellulose components and the shifting of the peak

from 1867.27897 cm^{-1} to 1884.45757 cm^{-1}.Figure 5(c) shows the spectrum of acetylated *C. dolichopetalum* fiber. The fibers shows a reduction in –CH stretching intensity of alkane branch and the shifting of the peak from 816.08479 cm^{-1} to 936.543161 cm^{-1} which indicated presence of more of unsaturated –CH alkane branches in acetylation reaction. The strong band peak at 1397.1415 cm^{-1} shows a slight reduction of –C-C stretching of aromatic in the lignin components. A slight reduction of the absorption peak of carbonyl group present in lignin and other cellulose component, and the shifting of the peak from 1867.27897 cm^{-1} to 1892.28693 cm^{-1} indicate that some of the carbonyl group present in lignin participate in the chemical reaction. The disappearance of the band peak at 2087.53605 cm^{-1}. Figure 5 shows the complete destruction of the carbon-carbon triple bond (-C≡C-) in cellulose chain by the acetic anhydride treatment. The peak at 2540.34583 cm^{-1} shows a slightly higher absorption of –C-O-C bond in the cellulose chain due to acetylation reaction. The bands at 2900.65684 cm^{-1} and 3020.4448 cm^{-1} are due to –CH deformations of alkanes and alkenes respectively. The increase in intensity and shifting of peak from 3416.37542 cm^{-1} to 3616.91068 cm^{-1} is associated with increased absorption or removal of –OH functional groups due to the coupling of acetyl group to the molecule.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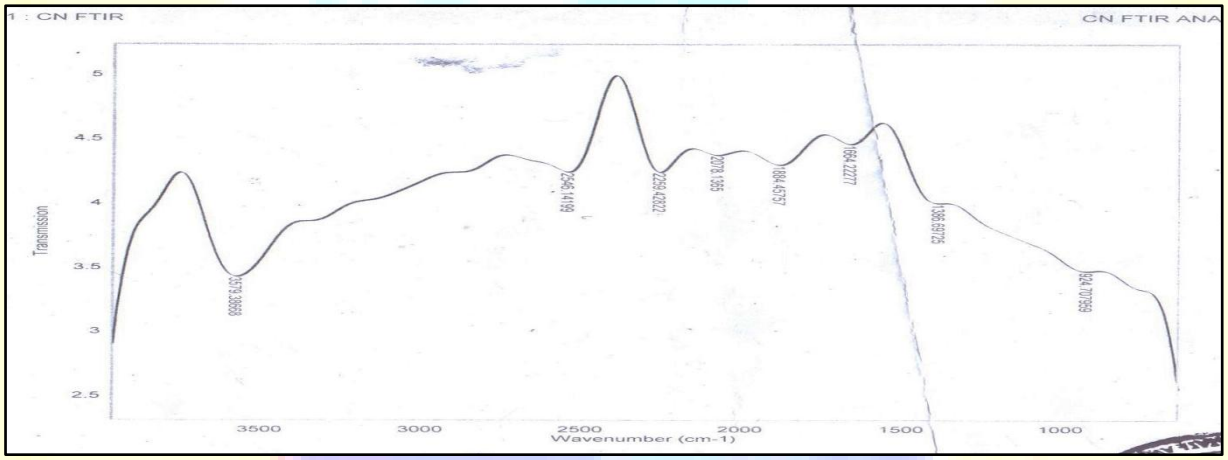
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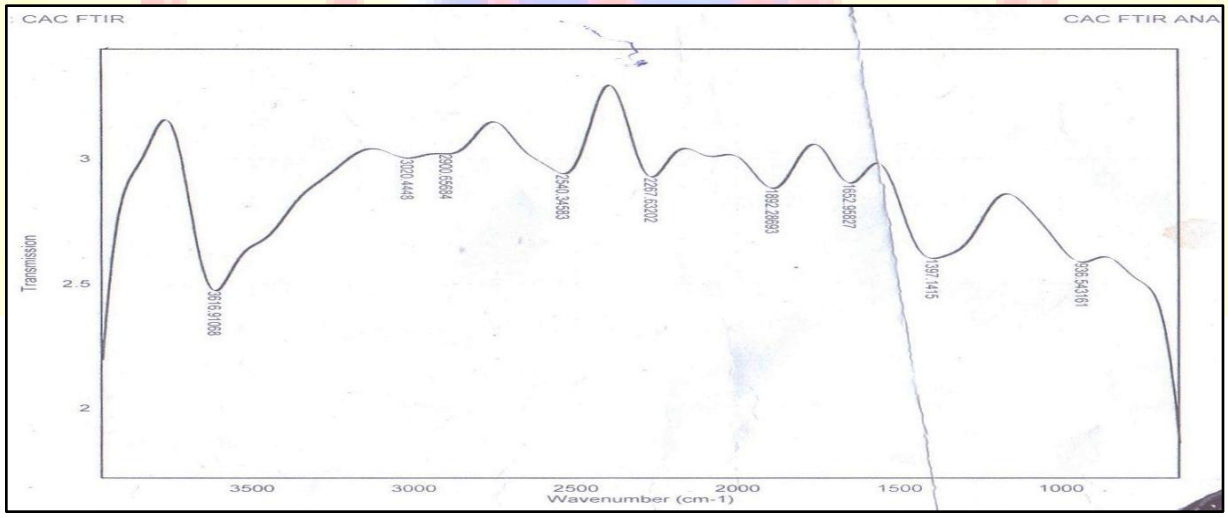
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(b)

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Figure 5: FTIR spectrum of (a) unmodified (b) mercerized (c) acetylated *C. dolichopetalum* fibers

CONCLUSION

Proximate analysis of *C. dolichopetalum*fiber revealed that the fiber is rich in crystalline cellulose and may be used for the reinforcement in polymer matrix applications. The ultimate tensile strength of *C. dolichopetalum* fiber increased with mercerization and acetylation processes, with increased aspect ratio, and reduced density and water sorptionattributed to shrinkage of the fiber diameter and leaching of amorphous cellulose constituents of the fiber surface as revealed by microstructural analysis using SEM and FTIR for changes in the surface topography and surface chemistry of the treated fibers compared to the unmodified fiber. The effects of chemical treatments enhanced the ultimate tensile strength and physico – chemical properties of *C. dolichopetalum* fiber. This indicated that the use *C. dolichopetalum* fiber will not only reduced the environmental dangerbut enhanced by mercerization and acetylation in composite applications.

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