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# TENSILE RESPONSES OF TREATED *CISSUS POPULNEA* FIBERS

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## ABSTRACT
***Improvement and effectiveness of polymers through reinforced materials coupled with environmental nuisance of the Cissus populnea fiber remains an area of concern. Tensile responses of chemically treated C. populnea fibers were investigated. Gravimetric analysis was used to determine the composition of C. populnea fibers. Sodium hydroxide (NaOH), acetic anhydride (AC) and ethylene diaminetetra-acetic acid (EDTA), respectively, were used for fiber treatment and optimized with variable parameters (concentration and time) using response surface methodology (RSM) with central composite design. Scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) were studied. At optimum treatment conditions, NaOH, AC and EDTA, respectively, increased the tensile strength of C. populnea fiber by 33.49, 274 and 194.52% as well as tensile modulus by 793.43, 20799.43 and 855%. Hence acetic anhydride treatment gave the best tensile properties of C. populnea fibers as corroborated by SEM with EDS. Thus, the effective use of C. populnea fiber in composite applications can be improved by chemical surface modifications.***

***Keywords:*** *Cissus populnea fiber; RSM; tensile properties; SEM; EDS.*

## 1. INTRODUCTION
*Cissus populnea* (*C. populnea*) is a tropical plant usually found in West Africa, particularly in Nigeria. It is called food gum plant. Its sap had been used as soup thickener, for treatment of venereal diseases and indigestion [1, 2], drug binder [3] and in ethno - medicine for treatment of male infertility [4]. About 51% of childless marriages result from fertility problems/sterility on the part of the male partners and demand the use of extract of the *C. populnea* plant [4]. This indicates high disposal of *C. populnea* fiber in Nigeria. Thus, disposal of *C. populnea* fiber causes environment nuisance through foul smell emissions and increased biochemical oxygen demand in the society. The composition, characteristics and usefulness of *C. populnea* fiber in composites applications are limited. Research in polymer composite applications is being directed towards the use of natural fibers as renewable resources for reinforcement. Lignocellulosic fibers also called ‘‘plant” or ‘‘natural” fibers such as bast, leaf or hard, seed, fruit, wood, cereal straw, and other grass fibers have been area of interest of researchers in polymer composites. Lignocellulosic fibers are materials rich in lignin, hemicellulose and cellulose that are used for various applications such as yarns and textiles, ropes, twines and nets, non-woven fabrics, tissues, paper and board products, packaging, building and construction materials, fiber boards, insulation, geotextiles, composites and automotive parts [5–8]. In composites, the applications of natural fibers depend on their composition, physical and mechanical properties [6]. The environmental benefits of natural fibers over synthetic fibers for industrial applications partly depends on the possibilities for replacement of the various fibers, energy requirement for the production process, product performance, functional life time and options for waste disposal. In Brazil, 93 % of natural fibers such as banana, jute, piassava, sponge-gourd, sugarcane, coco-nut, rice straw, sisal, ramie and coconut are replacing glass or other traditional reinforcement materials in composites [6, 9, 10]. The advantages of natural fibers include low density, high toughness, comparable specific strength properties, reduction in tool wear, ease of separation, reduced energy of fabrication, non – toxic, low cost, weight saving, reinforcing materials, improvement of stiffness and emerging saving of petroleum products [6, 11]. The

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undesirable properties of natural fibers may be attributed to relatively high hydrophilic behaviours which limits the application in industrial practice and become the most crucial issue in composites engineering. This affects the interfacial adhesion between the fiber and the polymer matrix, hence reducing the mechanical properties of the composites [12]. Surface modifications of natural fibers have been found to be very effective in improving the fiber-matrix interfacial adhesion. Surface modifications such as the use sodium hydroxide, silane, hydrogen peroxide, chromium sulphate, sodium hypochlorite, acetic anhydride with or without acetic acid, respectively, have been reported as promising techniques that improved the fiber wettability, modify microstructure, surface topography, surface chemical groups and tensile strength, through removal of lignins, hemicelluloses, pectins and other impurities [5, 6, 13, 14]. However, the use of catalysts in acetylation poses many problems as reported by Bledzki *, et al.*. [15]. Strong mineral acids or acid salts are known to cause hydrolysis of cellulose which damage the fiber structure. So, selection and optimization of catalyst is important for the acetylation of lignocellulosic fibers. The hydroxyl groups of lignin, hemicelluloses, and those of amorphous cellulose reacts with acetyl groups while the hydroxyl groups of crystalline region with close packing and strong interlock bonding are completely inaccessible [15]. Moreover, variation in fiber properties may be due to compositions, nature, extraction techniques and modification conditions [16, 17]. These changes have influence on the interaction between fiber and polymer (in case of composites), adsorbent and adsorbate (for use as adsorbent of metals and pigments), enzyme and support and/or functionalizing agent and support (for immobilization process) [6]. In this study, optimization of tensile properties of *C. populnea* fibers treated with sodium hydroxide (NaOH), acetic hydride (AC) and ethylene diaminetetra-acetic acid (EDTA) was investigated.

## 2. MATERIALS AND METHODS

White *C. populnea* plant was obtained from Gbana, Oriire Local Government Area of Oyo state, Nigeria. Sodium hydroxide, acetic anhydride and ethylene diamine tetra acetic acid are analytical grade chemicals obtained from Rovert scientific limited, Benin city in Edo state, Nigeria. Design of Experiment (DoE) software version 6.0.8 (2002 East Hennepin ave., Suite 480 Minneapolis, MN 55413, stat Ease, Inc.) was used to optimize the treatment of fiber.

### 2.1 Extraction and Composition of *C. populnea* Fibers

The stem of *C. populnea* plant was cut into 150 – 200 cm in length and beaten to soft the stems. 25 kg of *C. populnea* culms was immersed in 3L of deionized water for 18days, washed every 3days, then sun dried for seven days and later dried at a temperature of $60^0$C for 2 hours. The gravimetric analysis procedure described by Hänninen *, et al.*. [18] was used to determine the proximate composition of *C. populnea* fibers after milled into particles that could pass through 1mm sieve. The proximate composition of the fibers determined include moisture, dry matter, water soluble, ash, wax / fat, pectin, lignin, hemicelluloses and cellulose content.

### 2.2 Chemical Surface Treatments

*C. populnea* fibers were cut into 150 mm length, modified using NaOH with concentration range of 3 – 15 % for 10 – 50 minutes, AC with concentration range of 3 – 15 % for 30 – 150 minutes, and EDTA with concentration range of 1 – 5 % for 30 – 150 minutes, respectively, at room temperature. Fibers were then washed several times with deionized water until neutral pH of 7 was obtained and then, dried in an air oven at $60^0$C for 2 hours.

### 2.3 Tensile Properties of *C. populnea* Fibers

Tensile properties (tensile strength and modulus) were determined using Universal Testing Machine Instron 3369. A single fiber tensile test was conducted on three *Cissus populnea* fibers of length 150mm with gauge length of 100mm and average diameter of 0.11 $\pm$ 0.02mm. The average of three experiments conducted was used for this study. Then, optimization based on modified conditions was done using second order polynomial of response surface methodology (RSM) with central composite design (CCD) of Design - Expert software (version 6.0.8) and statistically analyzed using analysis of variance (ANOVA).

### 2.4 Scanning Electron Microscope Analysis

High resolution scanning electron microscope (SEM) of ASPEX 3020 model with energy dispersive X-ray spectroscopy (EDS) was used to study the morphology of surfaces of the *Cissus populnea* fibers with elemental composition at optimal treatment conditions. The surfaces of the fiber was examined directly by scanning electron microscope (SEM) ASPEX 3020 model at 20 KeV and 5.0 $x10^{-5}$ torr. Fiber sample was mounted on stubs with silver paste. To enhance the conductivity of the fiber, a thin film of platinum was vacuum -

evaporated before the photomicrographs or spectrum were taken.

## 3. RESULTS AND DISCUSSION

The extracted *C. populnea* fiber of 76.9 % plant bast was obtained with the proximate composition as presented in Table 1. It can be observed that *C. populnea* fibers rich in cellulose, hemicellulose, lignin content which contributed to the strength and stiffness of fibers and similar to the report of Kalia *, et al.* [19].

From Figure 1, the tensile strength of untreated *Cissus populnea* fibers (uCPF) of 21.16 MPa was obtained. NaOH treatment increased the tensile strength of *C. populnea* fibers to ultimate level based on concentration variation and treatment time. Treatment of uCPF with 3, 6, 9, 12, 15 % NaOH concentration, respectively, for 10, 50, 40, 50 and 30 minutes increased the tensile ultimate tensile strength of uCPF by 78.1, 114.8, 778.5, 314.0 and 746.8 %.

*Table 1: Proximate composition of C. populnea fibers*

| Composition | Percentage (w/w %) |
|---|---|
| Moisture | 3.94±0.23 |
| Dry matter | 96.06±0.20 |
| Ash | 1.59±0.14 |
| Wax | 2.94±0.31 |
| Water soluble | 2.33±0.27 |
| Pectins | 1.14±0.03 |
| Lignins | 11.52±0.27 |
| Hemicelluloses | 14.74±0.42 |
| Celluloses | 61.80±0.45 |

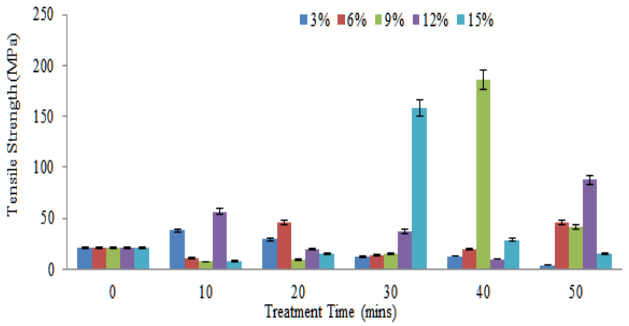



*Figure 1: Tensile strength of NaOH treated C. populnea fibers*

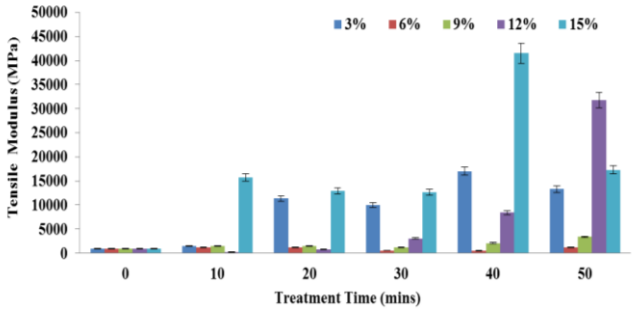



*Figure 2: Tensile modulus of NaOH treated C. populnea fibers*

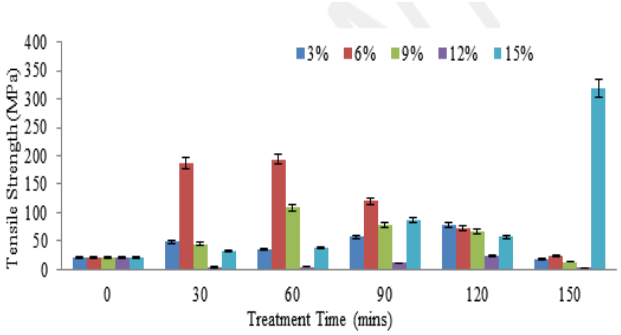



*Figure 3: Tensile strength of AC treated C. populnea fibers*

This shows that 9 % NaOH for 40 minutes gave the highest tensile strength of 185.9 MPa and found to be more effective on *C. populnea* fibers compared to double stages (0.5% $CrSO_4$ and $NaHCO_3$) treated coir fiber with 81.8 % improvement in tensile strength as reported by Hossain *, et al.*. [20] and Mir, *et al.*. [21], and 5% NaOH treated kenaf fibers with 57.4 % improvement in tensile strength as reported by Cao *, et al.*. [22].

The tensile modulus measured the stiffness of a material. From Figure 2, 6 % NaOH treatment was found to be ineffective on the tensile modulus of *C. populnea*. Treatment of fibers with 3, 9, 12 and 15 % NaOH, respectively, for 40, 50, 50 and 40 minutes increased the ultimate tensile modulus of uCPF by 1723.6, 263.3, 3311.1 and 4353.7 %. The increase in tensile modulus is more than that of kenaf fiber as reported by Cao *, et al.* [22]. The highest percentage increase in tensile modulus was obtained at treatment condition of 15% NaOH for 40 minutes. The increase in tensile properties may be attributed to surface removal of amorphous constituents (lignins, pectins, hemicellulose and other impurities) as reported by researchers [5, 13].

Figure 3 shows the effect of acetic anhydride treatment on tensile strength of *C. populnea* fibers. It can be observed that acetic anhydride increased the tensile strength of the *C. populnea* fibers up to maximum levels at treatment conditions of 3, 6, 9, 12 and 15 %, respectively, for 120, 60, 60, 120 and 150 mins. This might be due to hydroxyl groups substituted by acetyl groups on *C. populnea* fibers forming strong covalent bond, thus reducing the hydrophilic nature of *C. populnea* fibers which makes it less susceptible to biological decay as reported by researchers [12, 23]. The highest percentage increase in tensile strength was found to be 1409.5% at treatment conditions of 15% AC for 150 mins.

From Figure 4, there seems to be no significant effect of the acetic anhydride on the tensile modulus of *C. populnea* fibers when treated with 6, 12 and 15 % concentration but influenced at concentration of 3 and

9 % of acetic anhydride, respectively. The highest percentage increase in tensile modulus for 3 and 9 %, respectively, at treatment time of 120 and 60 minutes was found to be 19144.5 and 28092.5% uCPF. The highest tensile modulus is 262807.8MPa which increased by 28092.5% uCPF.

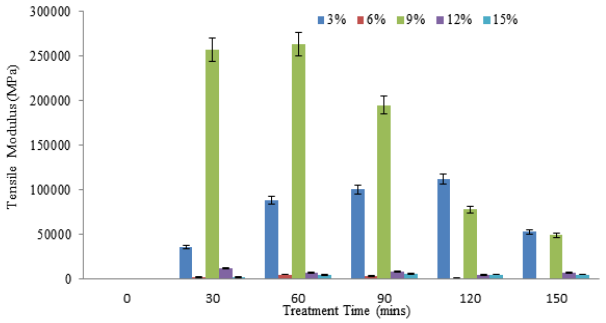



*Figure 4: Tensile modulus of AC treated C. populnea fibers*

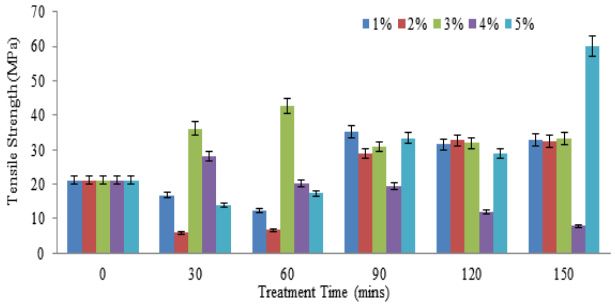



*Figure 5: Tensile strength of EDTA treated C. populnea fibers*

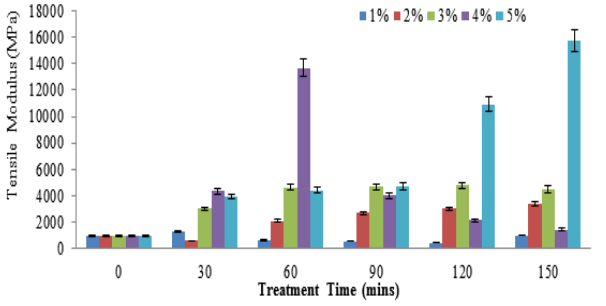



*Figure 6: Tensile Modulus of EDTA treated C. populnea fibers*

Figure 5 depicts that the tensile strength of 1, 2 and 5% EDTA treated *C. populnea* fibers, respectively, for 60 minutes, initially declined and, then rise to ultimate level of 35.29, 32.65 and 59.97MPa, with percentage increase in tensile strength by 66.8, 54.3 and 183.4% uCPF for 90, 120 and 150 mins. It can be deduced that 3 % EDTA for 30 mins and 4% EDTA for 60 mins, respectively, increased the tensile strength of uCPF by 32.1 and 101.5 %. The highest tensile strength of EDTA treated *C. populnea* fibers was obtained at 5% for 150 minutes.

Figure 6 shows the effect of EDTA treatment on tensile modulus of the fibers. It can be observed that the tensile modulus of EDTA treated *C. populnea* fibers varied with treatment conditions and it reaches maximum level at treatment conditions of 1, 2, 3, 4 and 5 %, respectively, for 30, 150, 120, 60 and 150 minutes, which increased by 138.0, 357.4, 511.1, 1469.0 and 1690.3 % uCPF. The ultimate tensile modulus was obtained at treatment concentration of EDTA of 5% for 150 minutes.

It can be deduced that the treatment conditions for *C. populnea* fibers to attain ultimate level of tensile strength and modulus varied. Though, the increase in tensile strength and modulus of treated *C. populnea* fibers compared with untreated fibers may be attributed to removal of amorphous constituents (waxes, lignins, pectins, hemicellulose and amorphous cellulose) and penetration depth of fibers by chemical treatment agents [24]. The need for optimization of fiber treatment is necessary due to variation in the fiber properties (tensile strength and modulus) with treatment conditions (concentration and time) at room temperature. The variation in fiber properties with treatment conditions may be attributed to kinetics of treatment, uneven distribution of hydroxyl groups, removal of amorphous constituents (lignin, pectins, hemicellulose and amorphous cellulose) and other uncontrollable factors. Optimizing the treatment conditions will ascertain the tensile properties of *C. populnea* fibers, avoiding wastage of materials and time.

The ANOVA results of tensile strength response model are presented in Table 2. The tensile strength response model for *C. populnea* fibers treated with NaOH, AC and EDTA are represented by equations (1), (2) and (3), respectively. The fitness of experimental results of NaOH, AC and EDTA treated *C. populnea* fibers, respectively, can be explained by the value of coefficient of determination ($R^2$). The value $R^2$ for NaOH, AC and EDTA treated *C. populnea* fibers, respectively, was found to be 0.994, 0.8382 and 0.9099 explain 99.4, 83.82 and 90.99 % of the observed variability in tensile strength with concentration and treatment time. The residue measured the tensile strength error of fibers. The residue of 0.6, 16.18 and 9.01%, respectively, obtained for NaOH, AC and EDTA treatment. This may be due to uncontrollable factors such as nature, maturity and thickness of fibers, and penetration rate of chemical used. Moreover, adequacy precision greater than 4 justified $R^2$. This implies that NaOH, AC and EDTA, respectively, adequate for modification of *C. populnea* fibers and modified fibers can be used in composite applications since adequacy precision > 4.

$$T_{sf} = 88.070129 - 13.33872c - 2.918828t + 0.472196c^2 + 0.0.201245t^2 + 0.2876018ct \quad (1)$$

$$T_{sf} = 11.115651 + 2.839207c + 1.201999t - 0.192764c^2 - 0.006488t^2 + 0.003402ct \quad (2)$$

$$T_{sf} = 22.329979 - 16.3122c + 0.3703311t + 1.8468837c^2 - 0.00199t^2 + 0.0875185c \quad (3)$$

Where $T_{sf}$, $c$ and $t$ represent tensile strength, concentration and time, respectively.

*Table 2: ANOVA for response surface model of tensile strength of treated C. populnea fibers*

| Source | Model cefficient | Sum of Squares | DF | Mean Square | F Value | Prob > F | $R^2$ | Adj $R^2$ | Adeq Precision |
|---|---|---|---|---|---|---|---|---|---|
| *C. populnea* fibers *with NaOH* | | | | | | | | | |
| Constant | 88.070129 | 13611.25 | 5 | 2722.25 | 233.0735 | < 0.0001 | 0.994 | 0.9898 | 49.3116 |
| c | -13.33872 | 4134.37 | 1 | 4134.37 | 353.9765 | < 0.0001 | | | |
| t | -2.918828 | 2461.55 | 1 | 2461.55 | 210.753 | < 0.0001 | | | |
| $c^2$ | 0.472196 | 2010.21 | 1 | 2010.21 | 172.1102 | < 0.0001 | | | |
| $t^2$ | 0.0201245 | 450.78 | 1 | 450.78 | 38.5948 | 0.0004 | | | |
| ct | 0.2876018 | 4764.37 | 1 | 4764.37 | 407.9157 | < 0.0001 | | | |
| *C. populnea* fibers *with AC* | | | | | | | | | |
| Constant | 11.115651 | 3981.3894 | 5 | 796.27788 | 6.215217 | 0.0229 | 0.8382 | 0.7033 | 6.5233 |
| c | 2.8392073 | 5.8793982 | 1 | 5.8793982 | 0.0458907 | 0.8375 | | | |
| t | 1.2019988 | 228.8247 | 1 | 228.8247 | 1.7860538 | 0.2299 | | | |
| $c^2$ | -0.192764 | 67.987919 | 1 | 67.987919 | 0.5306686 | 0.4938 | | | |
| $t^2$ | -0.006488 | 3675.0984 | 1 | 3675.0984 | 28.68538 | 0.0017 | | | |
| ct | 0.0034016 | 3.5990052 | 1 | 3.5990052 | 0.0280914 | 0.8724 | | | |
| *C. populnea* fibers *with EDTA* | | | | | | | | | |
| Constant | 22.329979 | 2140.1215 | 5 | 428.02431 | 12.119686 | 0.0043 | 0.9099 | 0.8348 | 12.8442 |
| c | -16.3122 | 224.00191 | 1 | 224.00191 | 6.342707 | 0.0454 | | | |
| t | 0.3703311 | 938.74209 | 1 | 938.74209 | 26.580871 | 0.0021 | | | |
| $c^2$ | 1.8468837 | 320.16203 | 1 | 320.16203 | 9.0655205 | 0.0237 | | | |
| $t^2$ | -0.00199 | 216.02923 | 1 | 216.02923 | 6.1169571 | 0.0482 | | | |
| ct | 0.0875185 | 441.18629 | 1 | 441.18629 | 12.492373 | 0.0123 | | | |

The significance of the response model term can be determined by the $p < 0.05$. It can be observed that all the model terms with constant parameters are significant for the NaOH and EDTA treated *C. populnea* fibers since $p < 0.05$. This shows the contribution of concentration, treatment time, quadratic of concentration and time as well as interaction of concentration and time for NaOH and EDTA treatments, respectively, on the tensile strength of modified *C. populnea* fibers. In the case of *C. populnea* fibers treated with acetic anhydride, quadratic of treatment time ($t^2$) contributes significantly to the tensile strength of fibers. This is also illustrated in Figure 7. The above observation shows a limitation in the report of researchers on improvement of tensile strength of some natural fibers as functions of concentration alone without varying the time, as their observed tensile strength may not be optimal since time is a significant term [25, 26]. Global maximum can be observed from the surface plots (Figure 7) for NaOH and AC treated *C. populnea* fibers. The statistical data of tensile modulus response using ANOVA are presented in Table 3.

The quadratic response surface model for tensile modulus of NaOH, AC and EDTA treated *C. populnea* fibers, respectively, are represented by equations (4), (5) and (6). The $R^2$ value of 0.9081, 0.9985 and 0.9044, respectively, for tensile modulus of NaOH, AC and EDTA treated *C. populnea* fibers were favourably correlated which explain 90.8, 99.9 and 90.4% of the observed variability in tensile modulus based on treatment conditions. Moreover, with adequate precision of 11.1086, 55.9988 and 9.014 (> 4), the quadratic model for tensile modulus of *C. populnea* fibers treated with NaOH, AC and EDTA, respectively, may be adequately used in composite design applications. The value of 8.19, 0.15 and 9.56%, respectively, measured the residue which cannot be explained due to uncontrollable factors that were not put into consideration for this study.

The model terms for tensile modulus of *C. populnea* fibers modified with NaOH ($t^2$ and ct), AC (c, t, $c^2$, $t^2$ and ct) and EDTA (c, $c^2$ and $t^2$) were significant since $p < 0.05$.

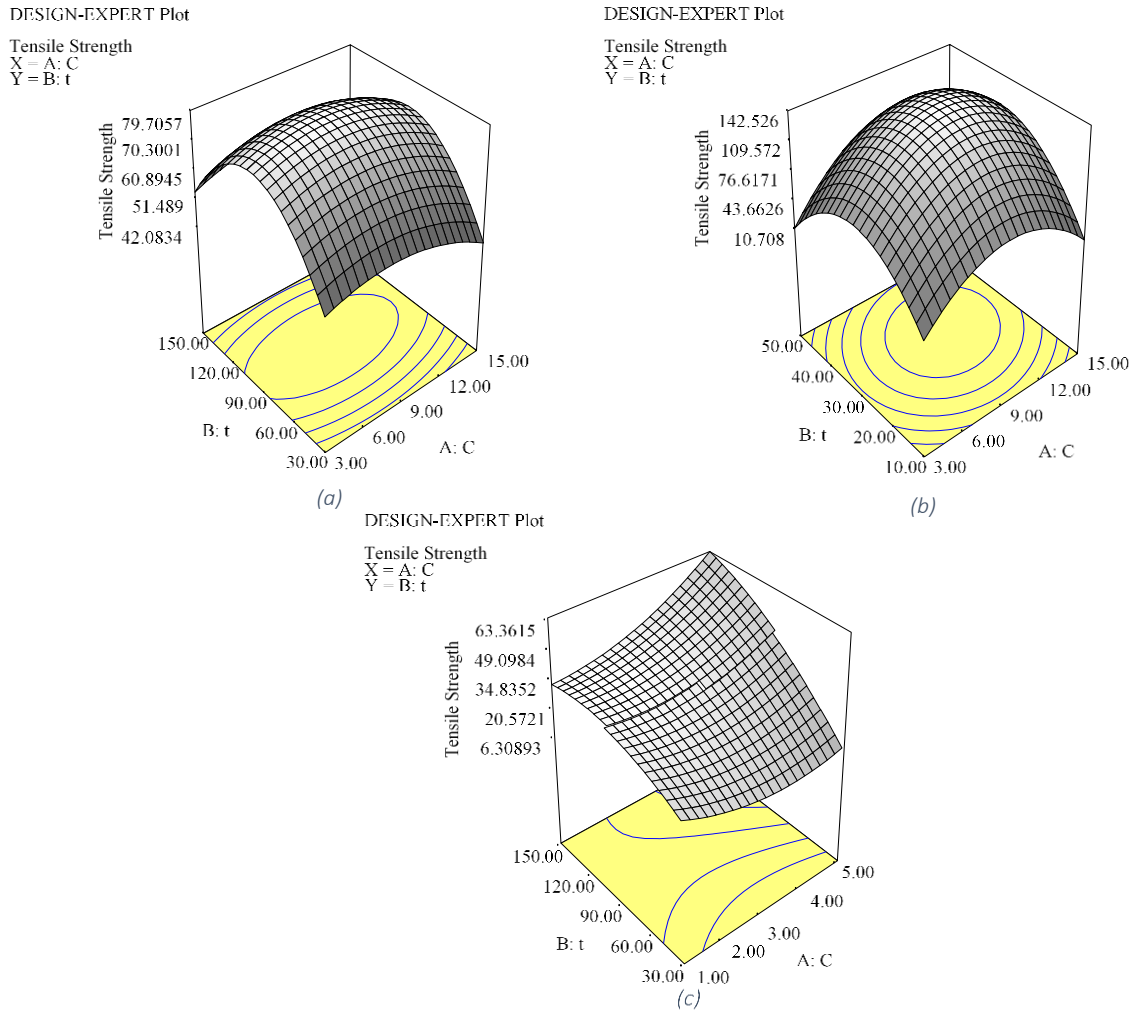



*Figure 7: 3-D response surface of tensile strength of treated C. populnea fibers with (a) NaOH (b) AC (c) EDTA*

$$T_{mf} = 327.08099 + 1209.7766c - 240.23855t + 27.29295c^2 + 1076815t^2 - 54.49786ct \quad (4)$$

$$T_{mf} = -183480.47 + 51442.66c + 3494.3753t - 2146.2912c^2 - 16.7391t^2 - 110.8072ct \quad (5)$$

$$T_{mf} = -2339.7757 - 2625.8593c + 43.8281t - 360.50427c^2 - 0.217551t^2 + 0.765237ct \quad (6)$$

Where $T_{mf}$, $c$ and $t$ represent tensile modulus, concentration and time, respectively.

*Table 3: ANOVA for response surface quadratic model of tensile modulus of treated C. populnea fibers*

| Source | Model coefficient | Sum of Squares | DF | Mean Square | F Value | Prob > F | $R^2$ | Adj $R^2$ | Adeq Precision |
|---|---|---|---|---|---|---|---|---|---|
| | | | | *C. populnea* fibers *with NaOH* | | | | | |
| Constant | 327.08099 | 260447156.6 | 5 | 52089431.31 | 11.855787 | 0.0046 | 0.9081 | 0.8315 | 11.1086 |
| C | 1209.7766 | 1258860.684 | 1 | 1258860.684 | 0.2865223 | 0.6117 | | | |
| t | -240.23835 | 473982.1111 | 1 | 473982.1111 | 0.1078804 | 0.7537 | | | |
| $c^2$ | 27.292945 | 9525649.639 | 1 | 9525649.639 | 2.1680804 | 0.1913 | | | |
| $t^2$ | 10.768148 | 78115714.41 | 1 | 78115714.41 | 17.779485 | 0.0056 | | | |
| ct | -54.497858 | 171072949.7 | 1 | 171072949.7 | 38.936967 | 0.0008 | | | |
| | | | | *C. populnea* fibers *with AC* | | | | | |
| Constant | -183480.47 | 72367670958 | 5 | 14473534192 | 676.11064 | <0.0001 | 0.9985 | 0.9970 | 55.9988 |
| c | 51442.662 | 6343184455 | 1 | 6343184455 | 296.31288 | <0.0001 | | | |
| t | 3494.3753 | 10247570158 | 1 | 10247570158 | 478.70072 | <0.0001 | | | |
| $c^2$ | -2146.2912 | 29076528936 | 1 | 29076528936 | 1358.2689 | <0.0001 | | | |
| $t^2$ | -16.739129 | 24499326481 | 1 | 24499326481 | 1144.4513 | <0.0001 | | | |
| ct | -110.80723 | 2201060927 | 1 | 2201060927 | 102.81944 | 0.0002 | | | |
| | | | | *C. populnea* fibers *with EDTA* | | | | | |

| Source | Model coefficient | Sum of Squares | DF | Mean Square | F Value | Prob > F | $R^2$ | Adj $R^2$ | Adeq Precision |
|---|---|---|---|---|---|---|---|---|---|
| Constant | -2339.7757 | 26891787.34 | 5 | 5378357.467 | 11.349254 | 0.0051 | 0.9044 | 0.8247 | 9.014 |
| c | 2625.8593 | 8639488.513 | 1 | 8639488.513 | 18.230798 | 0.0053 | | | |
| t | 43.828139 | 2114137.623 | 1 | 2114137.623 | 4.461192 | 0.0791 | | | |
| $c^2$ | -360.50427 | 11932305.03 | 1 | 11932305.03 | 25.179204 | 0.0024 | | | |
| $t^2$ | -0.2175507 | 4185618.263 | 1 | 4185618.263 | 8.8323704 | 0.0249 | | | |
| ct | 0.765237 | 20237.90767 | 1 | 20237.90767 | 0.0427054 | 0.8431 | | | |

The above observation indicated that there exist a quadratic relationship between tensile modulus of modified *C. populnea* fibers using NaOH, AC and EDTA, respectively, with the concentration and treatment time as illustrated in Figure 8. The global maximum tensile modulus of fiber was found for AC at optimum treatment conditions. Table 4 shows the experimental, optimal and error values for tensile strength of treated *C. populnea* fibers using NaOH, AC and EDTA, respectively. The experimental value for tensile strength of NaOH, AC and EDTA treated *C. populnea* fibers, respectively, increased by 33.49, 274 and 194.52% uCPF while stiffness increased by 793.43, 20799.43 and 441.2 % uCPF. This indicated that acetic anhydride proves to be superior for modification of *C. populnea* fibers in composite applications that required high strength and stiffness, while EDTA is a better modifying agent of *C. populnea* fibers compared to NaOH treatment for effective use of *C. populnea* fibers in composite applications. This is similar to the report of Azeez , *et al.*. [27] on modification of Combretum dolichopetalum fibers using NaOH and acetic anhydride. This work is also in agreement with Troedec , *et al.*. [28] report on modified hemp fibers for reinforcement of lime matrix using NaOH, $Ca(OH)_2$, EDTA and polyethylene imine. However, the closeness of tensile properties from experimental and response model indicated that the RSM approach using CCD shows the extent of accuracy of DoE for optimizing the tensile properties of *C. populnea* fibers with process conditions and will not misleading to determine the strength and modulus since tensile errors were comparably small. This shows that AC (9.01 % for 90.07mins) and EDTA (5.00 % for 146.13 mins) are more effective on tensile strength of *C. populnea* fibers compared to double stages (0.5% $CrSO_4$ and $NaHCO_3$) treated coir fiber with 81.75 % improvement [20, 21].

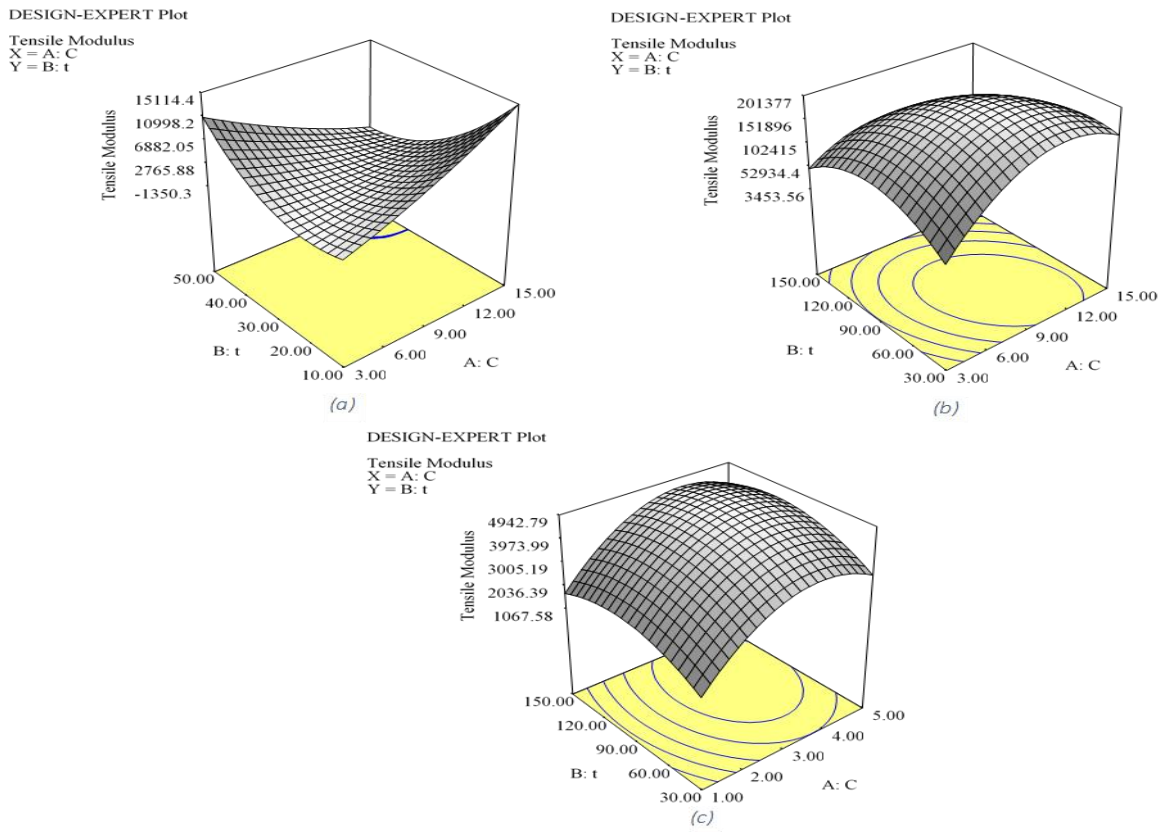



*Figure 8: 3-D response surface of tensile modulus of treated C. populnea fibers with (a) NaOH (b) AC (c) EDTA*

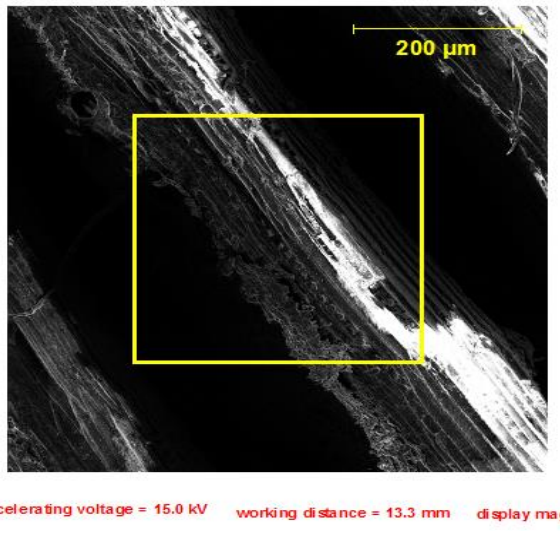
*Table 4: Experimental and predicted tensile properties of fibers at optimal treatment conditions*

| *Fiber sample* | *c (%)* | *t (mins)* | *pH* | *$d_f$ (mm)* | *$T_{sa}$ (MPa)* | *$T_{sp}$ (MPa)* | *$T_{se}$ (%)* | *$T_{ma}$ (MPa)* | *$T_{mp}$ (MPa)* | *$T_{me}$ (%)* |
|---|---|---|---|---|---|---|---|---|---|---|
| D | 0.00 | 0.00 | 7±0.1 | 0.110 | 21.1592 | 21.1592 | - | 932.1931 | 932.1931 | - |
| $D_{NaOH}$ | 15.00 | 18.88 | 12.6±0.2 | 0.104 | 28.2446 | 27.7474 | 1.79 | 8328.45 | 8483.75 | 1.87 |
| $D_{AC}$ | 9.01 | 90.07 | 2.3±0.2 | 0.088 | 79.1349 | 79.4376 | 0.38 | 194823 | 194809 | 0.00 |
| $D_{EDTA}$ | 5.00 | 146.13 | 6.7±0.2 | 0.100 | 62.3172 | 62.5153 | 0.32 | 4113.04 | 4095.68 | 0.44 |

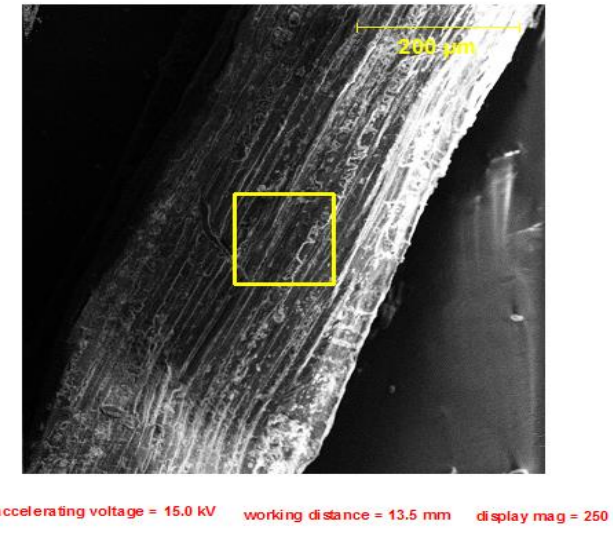
D, C, t, pH, $d_f$, $T_{sa}$, $T_{sp}$, $T_{se}$, $T_{ma}$, $T_{mp}$ and $T_{me}$ represent *C. populnea* fibers, concentration of the chemical used, treatment time, pH of the chemical solution, fiber diameter, actual tensile strength, predicted tensile strength, tensile strength error, actual tensile modulus, predicted tensile modulus, tensile modulus error.

Figure 9(a) – (d), respectively, shows the surface morphology of untreated, NaOH, AC and EDTA modified *C. populnea* fibers using high resolution scanning electron microscope (SEM) of ASPEX 3020 model. Figure 9(a) shows that there is deposition of lignin, hemicellulose and wax on primary layer of uCPF. It can be observed that NaOH modified *C. populnea* fibers revealed the roughness and smooth surfaces as shown in Figure 9(b). This indicates the removal of lignin, hemicellulose and wax substances on fiber surfaces and resulted to reduction in fibers' diameter as presented in Table 4 using micrometer screw gauge and increased the reactive sites, which may improve the adhesion of the *C. populnea* fibers to polymer. This is similar to the report of researchers [29, 30]. From Figure 9(c), surface shrinkage of acetic anhydride modified *C. populnea* fibers was observed, thereby, caused decrease in diameter. Figure 9(d) shows disorganized fibril and weakening of the gel structure which revealed the partial removal of wax and lignin from *C. populnea* fibers when treated with EDTA. This is an indication that EDTA serves as chelating agent and improved fiber separation, thereby, improved interfacial adhesion of fibers. The surface of the treated fibers appears free of any residual particles and smoother. Hence, this revealed the effectiveness of NaOH, AC and EDTA treatments in removing impurities surface of fibers. This is in agreement with the report of [24]. Treated *C. populnea* fiber may be alternative to glass and other alternative fibres in composite [31, 32].

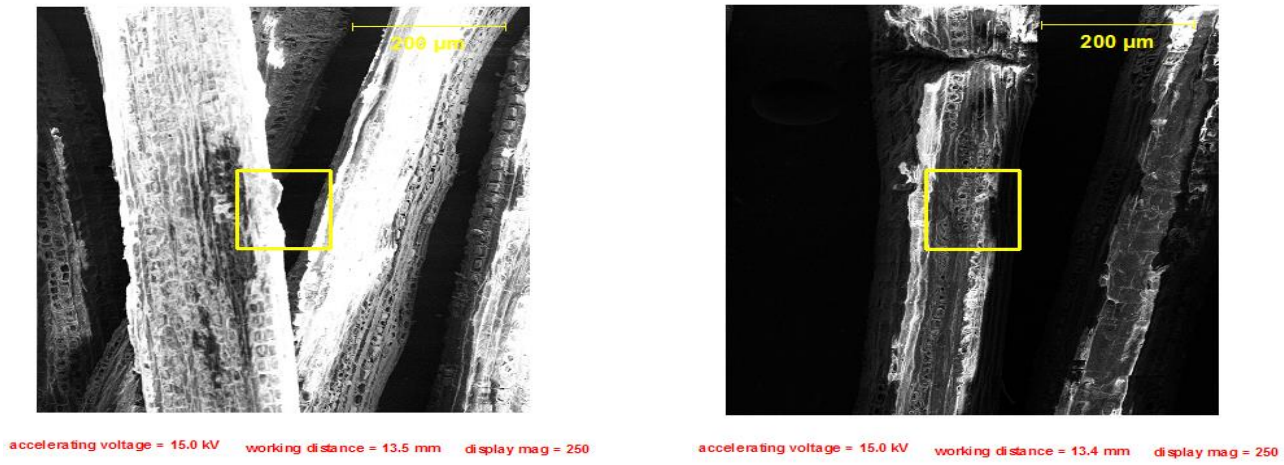
The EDS spectra of untreated and treated *C. populnea* fibers are presented in Figure 10. The presence of elemental atoms like carbon, oxygen and bromine atoms with elemental compositions can be observed in Figure 10(a). The presence of sodium atoms based on acquisition time was observed and indicated an increase in tensile properties of the fibers as seen in Figure 10 (b) – (d). It can also be observed that *C. populnea* fibers treated with NaOH gave the least counts of elemental compositions, especially, carbon from crystalline cellulose as shown in Figure 10(b) compared to Figure 10(a), (c) and (d). More so, the highest counts of elemental composition for carbon of crystalline cellulose was observed when *C. populnea* fiber treated was with acetic anhydride as shown in Figure 10(c), followed by *C. populnea* fiber treated with EDTA as shown in Figure 10(d).




*(a)*

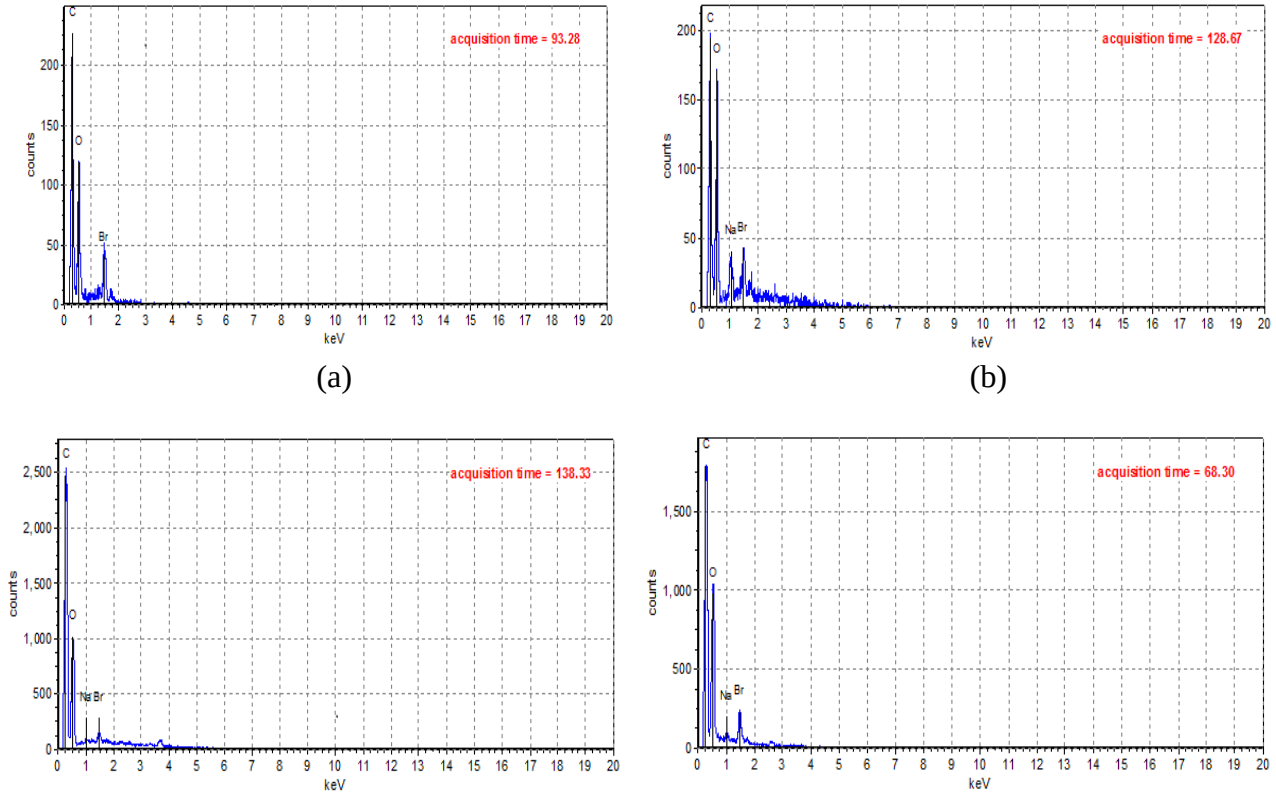



*(b)*




(c) (d)

*Figure 9: SEM of C. populnea fibers: (a) untreated (b) treated with NaOH (c) treated with AC (d) treated with EDTA*




(a) (b)

(c) (d)

*Figure 10: EDS of C. populnea fibers with (a) untreated (b) treated with NaOH (c) treated with AC (d) treated with EDTA*

## 4. CONCLUSIONS

Based on the results obtained, the ultimate tensile strength and modulus varied with concentration and treatment time of the fibers. NaOH, AC and EDTA treatment, respectively, increased the tensile strength and modulus of *C. populnea* fibers but reduced the thickness or diameter of uCPF at optimum treatment conditions. Acetic anhydride proved to be superior agent for improvement of tensile properties of *C. populnea* fibers compared with NaOH and EDTA treatments. Thus, the improvement in tensile properties of *C. populnea* fibers by NaOH, AC and EDTA, respectively, corroborated by SEM and EDS results. The residual values obtained may be minimized and improved the quality of the fibers for composite

applications if the thickness and penetration rate of chemical treatment can be put into consideration.

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