Properties of White Roselle (Hibiscus sabdariffa) Fibers

T O Azeez1* and D O Onukwuli2

*1Department of Biomedical Technology, School of Health Technology, Federal University of Technology, P M B 1526, Owerri, Nigeria
2Department of Chemical Engineering, Faculty of Engineering, Nnamdi Azikiwe University, P. M. B. 5025, Awka, Anambra State, Nigeria

Received 04 May 2017; revised 18 December 2017; accepted 15 June 2018

The chemical composition, physical (aspect ratio, density and water absorption) and tensile properties are determinant factors of fibers quality, effectiveness and dictate its usefulness in composite applications. Fiber physical and tensile properties of white Hibiscus sabdariffa (H. sabdariffa) fibers modified with sodium hydroxide (NaOH) and sodium lauryl sulphate (SLS) was aimed to be investigated for effective and quality use in polymer composite applications. The chemical compositions of white H. sabdariffa bast and fibers were analyzed using gravimetric method after retting extraction. Tensile properties, aspect ratio, density, water absorption behaviour using Peleg’s model and Power law expression, scanning electron microscope (SEM) with X-Ray energy dispersive microscope (EDS) analysis were determined and studied. NaOH and SLS treatments, respectively, improved the tensile strength and modulus of H. sabdariffa fibers by 282.31 and 182.07 %, and 49.38 and 2448.28 % with increased aspect ratio at reduced density and water absorption. SEM with EDS results corroborate the improvement in tensile properties. The water absorption of H. sabdariffa fibers exhibit less Fickian behaviour. Properties of white H. sabdariffa fibers modified with NaOH and SLS can be used in polymer composite applications.

Keywords: H Sabdariffa Fibers, Tensile Properties, SEM, EDS, Physical Properties

Introduction

The promising nature of eco friendly materials, natural fibers in composites applications have been reported1-3 as reinforcement of plastics in automotive, packaging, building materials, textiles and rehabilitation medicine (implants and tissue generations) applications4-6. Quality and properties of fibers depend on maturity, processing methods adopted for the extraction, seed rate, constituents, thickness, the fibrillar structure and the lamellae matrix2,5,7. Low strength, modulus and high water absorption are limitation of natural fibers as reinforcement compared with synthetic fibers8 and may be improved through physical, biological and chemical means9-14. The ultimate conditions of fiber modifications varied with properties which may be attributed to type, extraction processes15 and source16-20. The tensile and physical properties of white Hibiscus sabdariffa fibers with modifications using NaOH and SLS for potential applications was aimed to be investigated.

Materials and methods

Materials

White H. sabdariffa plant was obtained from Iyana Ogojo, Ogbomoso North Local Government Area of Oyo state, Nigeria. NaOH and SLS are analytical grade chemicals obtained from Rovert scientific limited, Benin city in Edo state, Nigeria.

Fiber extraction

The water retting method described by Phong et al21 was employed. The bast of roselle plant was removed from the stem and cut into 50-70 cm in length. 20 kg of roselle culms was immersed in 3 liters of deionized water for 18 days, washed every 3 days, and dried at a temperature of 60°C for 2 hours. The dried fibers are designated as untreated H. sabdariffa fibers (uHSF).

Proximate composition of H. sabdariffa fibers

The gravimetric analysis procedure described by Thygesen et al7 was used to determine the proximate composition of H. sabdariffa fibers after milled into particles that could pass through 1 mm sieve. The proximate composition of the fibers determined contains moisture content, dry matter, water soluble, ash, wax / fat, pectin, lignin, hemicelluloses and cellulose.
Chemical surface modifications

The strands of *H. sabdariffa* fibers were cut into 150 mm length modified using NaOH and SLS with concentration range of 3-15 % room temperature for 10-50 minutes. The fibers were then washed severally with deionized water until neutral pH of 7 was obtained. The fibers were finally dried in an air oven at 60°C for 2 hours.

Tensile properties of *H. sabdariffa* fibers

Tensile properties (tensile strength and modulus) were obtained using Universal Testing Machine Instron 3369. The tensile test was conducted on five strand of fibers of length 150 mm with gauge length of 100mm and average diameter of 0.002 ± 0.0003 mm. Then, optimized based on modified conditions using response surface methodology (RSM) with central composite design (CCD) of Design - Expert software (version 6.0.8). The concentration of chemical (NaOH and SLS) and treatment time were considered as factors of responses (tensile strength and modulus) as presented in Table 1.

Fiber aspect ratio

The length of 20 randomly sampled *H. sabdariffa* fibers were measured and recorded. The fibers’ diameters were measured at different locations along their length using micrometer screw gauge. The mean aspect ratio was calculated using equation (1) as given by Azeez *et al*[

\[
\text{Aspect ratio} = \frac{L_f}{D_f} \quad \ldots (1)
\]

Where \(L_f\) is the fiber length and \(D_f\) is the fiber diameter.

Fiber density

The method described by Brígida *et al*[

\[
\rho_f = \frac{M}{V} \quad \ldots (2)
\]

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 in accordance with ASTM D-570. Prior to testing, the *H. sabdariffa* fibers were dried in an oven at 60°C for 24 hours and weighed. 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 using equation (3) as given by Thiruchitrambalam *et al*[

\[
\text{Water absorption (\%)} = \frac{M_t - M_0}{M_0} \times 100\% \quad \ldots (3)
\]

Where \(M_t\) is the mass of the sample after conditioning in grams (wet weight), \(M_0\) is the mass of the sample before conditioning in grams (dry weight).

| Table 1 — Experimental CCD matrix of NaOH and SLS treated *H. sabdariffa* fibers with response |
|---|---|---|---|---|---|
| Run | C | t | \(T_{sa}\) (MPa) | \(T_{ma}\) (MPa) | \(T_{sa}\) (MPa) | \(T_{ma}\) (MPa) |
| 1 | 9 | 30 | 68.32611 | 1400.914 | 92.57809 | 3502.955 |
| 2 | 17.4853 | 30 | 53.05472 | 4237.798 | 74.40254 | 10753.21 |
| 3 | 3 | 50 | 179.2424 | 3945.683 | 27.32869 | 1688.951 |
| 4 | 3 | 10 | 88.06377 | 17253.51* | 87.44217 | 1315.7 |
| 5 | 9 | 1.7157 | 111.7469 | 7097.408 | 187.8348* | 1762.396 |
| 6 | 15 | 10 | 114.7032 | 3200.71 | 77.85574 | 2490.045 |
| 7 | 0.5147 | 30 | 43.76256 | 7793.986 | 52.00147 | 73222.33* |
| 8 | 9 | 30 | 68.32611 | 1400.914 | 92.57809 | 3502.955 |
| 9 | 9 | 30 | 68.32611 | 1400.914 | 92.57809 | 3502.955 |
| 10 | 15 | 50 | 58.75847 | 6374.277 | 159.178 | 6211.797 |
| 11 | 9 | 30 | 68.32611 | 1400.914 | 92.57809 | 3502.955 |
| 12 | 9 | 30 | 68.32611 | 1400.914 | 92.57809 | 3502.955 |
| 13 | 9 | 58.2843 | 182.8458* | 6197.661 | 121.4714 | 5411.239 |

C, t, \(T_{sa}\) and \(T_{ma}\) represent treatment concentration, time, actual tensile strength and modulus respectively. Superscript (*) indicates maximum tensile strength and modulus.
Water absorption mechanism of *H. sabdariffa* fibers

Peleg’s and Power law models were employed to study the kinetics of water sorption of *H. sabdariffa* fibers using Equation (4) and (5), respectively, as given by Afolabi *et al.*\(^{21}\) and Razavi-Nouri *et al.*\(^{23}\)

\[ M_t - M_0 = t /k_1 - k_2 t \] \hspace{1cm} (4)

Where \( M_t \) is percentage water absorption content at any time \( t \), \( M_0 \) is the initial percentage of water content, \( k_1 \) and \( k_2 \) are Peleg’s rate constant which relates to initial sorption rate and Peleg’s maximum moisture capacity constant, respectively.

\[ M_t /M_m = k t^n \] \hspace{1cm} (5)

where \( M_t \) is the water content at specific time \( t \), and \( M_m \) is the equilibrium water content (EMC) or water absorption at saturation point. \( k \) and \( n \) are constants. The magnitude of \( n \) indicated whether the water diffusion through fiber is governed by Fickian diffusion model or Non-Fickian diffusion model. The magnitude of \( k \) and \( n \) were evaluated as intercept and slope, respectively, of \( M_t /M_m \) versus \( t \) in the log - log plot of water absorption with time.

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 fiber (treated and untreated) surfaces at optimal conditions. The fiber surfaces was examined directly by scanning electron microscope (SEM) ASPEX 3020 model with EDS at 20 KeV and 5.0 \( \times \) 10\(^{-5} \) torr which 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.

Results and Discussion

The extracted *H.sabdariffa* fibers obtained was 79.6 % of bast plant. The average proximate composition of cellulose (60.93±0.32), lignin (12.70±0.18), hemicellulose (13.32±0.65), moisture (3.88±0.05), wax (1.82±0.29), water soluble (3.77±0.35) and pectins (2.04±0.34) were obtained from *H. sabdariffa* fibers. The tensile strength and modulus of 44.14402 and 2361.7429 MPa, respectively, obtained for untreated *H. sabdariffa* fibers (uHSF). The experimental CCD matrix of NaOH and SLS modified *H. sabdariffa* fibers with tensile strength and modulus as response are presented in Table 2. It can be observed that mercerized *H. sabdariffa* fibers at 9% NaOH for 58.2843 mins gave the maximum tensile strength of 182.8458 MPa and maximum tensile modulus of 17253.51MPa was obtained at 3% for 10 mins and. This implies that NaOH increased the tensile strength and stiffness of uHSF by 314.2 and 630.54%, respectively. It can also be observed that the ultimate tensile strength of 187.8348 MPa was obtained at 9 % SLS for 1.7157 mins and that of ultimate tensile strength of NaOH modified *H. sabdariffa* fiber

<table>
<thead>
<tr>
<th>Model term</th>
<th>DF</th>
<th>model coefficient</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F Value</th>
<th>Prob &gt; F</th>
<th>R(^2)= 0.9074, Adj R(^2)=0.8413, Adeq Precision=11.2148</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>5</td>
<td>55.558</td>
<td>22230</td>
<td>4445.9</td>
<td>13.7188</td>
<td>0.0017</td>
<td>Tensile strength of NaOH modified <em>H. sabdariffa</em> fiber</td>
</tr>
<tr>
<td>c</td>
<td>1</td>
<td>10.951</td>
<td>814.1</td>
<td>814.13</td>
<td>2.5122</td>
<td>0.157</td>
<td>28559.3 216135669.2 43227133.84 15.957344 0.0017</td>
</tr>
<tr>
<td>t</td>
<td>1</td>
<td>-2.7783</td>
<td>2305</td>
<td>2304.6</td>
<td>7.1114</td>
<td>0.032</td>
<td>-2701.01 34667037.29 34667037.29 12.793757 0.009</td>
</tr>
<tr>
<td>c(^2)</td>
<td>1</td>
<td>-0.1909</td>
<td>328.7</td>
<td>328.7</td>
<td>1.0143</td>
<td>0.347</td>
<td>73.5541 36189887.49 36189887.49 13.359537 0.0081</td>
</tr>
<tr>
<td>t(^2)</td>
<td>1</td>
<td>0.10642</td>
<td>12607</td>
<td>38.9000</td>
<td>0.0004</td>
<td>0.0047</td>
<td>7.40942 61105554.03 61105554.03 0.0021</td>
</tr>
<tr>
<td>ct</td>
<td>1</td>
<td>-0.3065</td>
<td>5411</td>
<td>5411.3</td>
<td>16.6978</td>
<td>0.0047</td>
<td>34.3362 67909098.83 67909098.83 25.068719 0.0016</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Model term</th>
<th>DF</th>
<th>model coefficient</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F Value</th>
<th>Prob &gt; F</th>
<th>R(^2)= 0.8277, Adj R(^2)=0.7617, Adeq Precision=11.406</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>5</td>
<td>170.08</td>
<td>17104</td>
<td>3420.8</td>
<td>7.686</td>
<td>0.0064</td>
<td>Tensile strength of SLS modified <em>H. sabdariffa</em> fiber</td>
</tr>
<tr>
<td>c</td>
<td>1</td>
<td>4.33348</td>
<td>2962</td>
<td>2962.3</td>
<td>6.6558</td>
<td>0.0326</td>
<td>110240 3819534784 763906956.8 8.1532824 0.0078</td>
</tr>
<tr>
<td>t</td>
<td>1</td>
<td>-6.975</td>
<td>659.6</td>
<td>659.63</td>
<td>1.4821</td>
<td>0.2581</td>
<td>-15655.2 1189807986 1189807986 12.698898 0.0092</td>
</tr>
<tr>
<td>c(^2)</td>
<td>1</td>
<td>-0.5537</td>
<td>2934</td>
<td>2933.8</td>
<td>6.5919</td>
<td>0.0333</td>
<td>-1242.11 24920876.14 24920876.14 0.2659839 0.6219</td>
</tr>
<tr>
<td>t(^2)</td>
<td>1</td>
<td>0.06448</td>
<td>4913</td>
<td>4913</td>
<td>11.0387</td>
<td>0.0105</td>
<td>-3.32798 57405842.47 57405842.47 0.4594</td>
</tr>
<tr>
<td>ct</td>
<td>1</td>
<td>0.29466</td>
<td>5001</td>
<td>5001</td>
<td>11.2365</td>
<td>0.0101</td>
<td>126.713 554904990.9 554904990.9 5.9225761 0.0452</td>
</tr>
</tbody>
</table>

\( c \) is the concentration used for the treatment and \( t \) is the time treatment time (mins)
modulus of 73222.33 MPa was obtained at treatment concentration of 0.5147 % for 30 mins. This implies that the ultimate tensile strength and modulus of uHSF, respectively, increased by 325.51 and 3000.35%. The increase in tensile properties may be attributed to removal of amorphous constituents (lignin, hemicellulose and pectin) in the secondary layers of fiber surface, thereby, clean the fiber surfaces. Moreover, the ultimate tensile strength and modulus of modified *H. sabdariffa* fibers varied with ultimate treatment conditions. The observed inconsistencies in fiber property variation with treatment conditions are unusual, considering several uncontrollable factors such as extraction techniques, climatic conditions, maturity, variety, soil characteristics, decortification, and disintegration that may affect the properties of natural fibers. The need to optimize the treatment conditions with response properties is necessary for effective use of *H. sabdariffa* fibers in composite applications. The tensile strength response models for *H. sabdariffa* fibers modified with NaOH and SLS are represented by equations (6) and (7), respectively while that of tensile modulus response models are represented by equations (8) and (9), respectively.

\[
T_{sp} = 55.558 + 10.951c - 2.7783t - 0.19094c^2 + 0.10642t - 0.30651ct \\
T_{mp} = 170.08 + 4.3335c - 6.9750t - 0.55367c^2 + 0.064483t^2 + 0.29466ct
\]

(6)  
(7)

\[
T_{sp} = 28559.33 - 2701.0c - 824.88t - 73.554c^2 + 7.4094t^2 - 34.336ct \\
T_{mp} = 110239.8 - 1565.521c - 1242.108t + 496.369c^2 - 3.328t^2 + 126.713ct
\]

(8)  
(9)

Table 2 shows the ANOVA for the response surface quadratic models of tensile strength and modulus of modified *H. sabdariffa* fiber using NaOH and SLS, respectively. The results of the experimental data for modified *H. sabdariffa* fibers with NaOH and SLS treatment, respectively, revealed that concentration and treatment time contribute 90.74 and 82.77% of the observed variability in tensile strength based on the magnitude of coefficient of determination (R²). The variability in tensile strength of modified *H. sabdariffa* fibers may be attributed to plant variety, maturity, harvesting techniques, fiber diameter and many other factors that are not put into consideration for modelling. The value of R² is judged by the adequacy precision > 4 for a model to be considered adequate. Thus, with adequacy precision of 11.2148 and 9.5218 (> 4), for the quadratic model of tensile strength of *H. sabdariffa* fibers modified with NaOH and SLS, respectively, the model is adequate for all treatment. The significance of model terms were judged based on the p < 0.05. The model terms obtained for tensile strength of *H. sabdariffa* fibers: modified with NaOH (t, r² and ct) and SLS (c, c², r² and ct) were significant since p < 0.05. It can be observed that quadratic relationship exists between tensile strength and treatment time as well as interaction (ct) for SLS treatment, while quadratic relationship exists between the tensile strength and concentration of NaOH. More so, the tensile modulus of NaOH and SLS modified *H. sabdariffa* fibers, respectively, with R of 0.9193 and 0.8535 revealed that the concentration and treatment time contribute 91.93 and 85.35% of the observed variability in tensile modulus during the treatment process and model were significant since p<0.05. With adequacy precision of 11.8406 and 10.9026 (>4), the quadratic models for tensile modulus of NaOH and SLS modified *H. sabdariffa* fibers, respectively, were adequate. The model terms of tensile modulus of uHSF modified with NaOH (c, t, c², t² and ct) and SLS (c, c² and ct) were significant since p<0.05. The results show contributions of varying NaOH and SLS concentration and time (interaction) on observed improvement in tensile modulus of *H. sabdariffa* fibers. It can be observed that quadratic relationship exist between the tensile modulus, concentration and treatment time of NaOH. Whereas, only a quadratic relationship between concentration and tensile modulus when modified with SLS. The significant interaction between concentration and time on tensile modulus indicated that the variables do not function independently. The above observation revealed a limitation on improvement of tensile properties 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 for treatments. It can be observed from Table 3 that the tensile strength and modulus errors between the actual value and predicted value for SLS and NaOH modified *H. sabdariffa* fibers, respectively, were considerably small. At optimum treatment conditions of *H. sabdariffa* fibers using NaOH and SLS, respectively, the tensile strength of uHSF improved by 282.31 and 182.07% and that of tensile modulus of uHSF improved by 49.38 and 2448.28%. This revealed that NaOH modification of
H. sabdariffa fibers is superior over SLS modification for load carrying capacity applications while SLS modified is superior for stiffness applications. It can be deduced that modification of H. sabdariffa fibers at optimum conditions using NaOH and SLS were effective compared with kenaf fiber using NaOH and coir fibers using a mixture of CrSO₄ and NaOH as reported by Cao et al.¹⁴ and Mir et al.²⁰, respectively. The physical properties of NaOH and SLS modified H. sabdariffa fibers as well as that of unmodified H. sabdariffa fibers are also presented in Table 3. The aspect ratio for NaOH and SLS modified H. sabdariffa fibers, respectively, was observed to be higher than unmodified H. sabdariffa fibers by 23.53 and 16.67% due to reduction in diameter of fibers. The increased aspect ratio shows the effectiveness of surface modifications on amorphous constituents (lignin, hemi cellulose and other impurities) removal as reported by researchers.¹¹,¹²,¹⁷. It can also be deduced that NaOH and SLS, respectively, reduced the density of uHSF by 28.79 and 61.09%. The reduction in density may be due to substitution of hydrogen atoms from hydroxyl groups by sodium atoms as reported by Nikoghosyan et al.²⁵. This indicated that the reduction in density of H. sabdariffa fibers may not only make the fiber to be lighter but rather make its composites application models to be lighter. More so, water sorption of uHSF reduced by 9.09 and 14.77% when treated with NaOH and SLS, respectively. This shows that NaOH and SLS improved the hydrophobic nature of H. sabdariffa fibers. This may be due to reduction in amorphous constituents and similar to the report of researchers.²⁶-³⁰. Figure 1(a) shows that Peleg’s model accurately described the water sorption kinetics of unmodified and modified H. sabdariffa fibers due to high value of $R^2$. NaOH and SLS, respectively, increased the Peleg’s rate constant ($k_1$) of uHSF by 0.18 and 4.97 times. It can also be observed that NaOH modification increased the water holding capacity of uHSF but otherwise when

<table>
<thead>
<tr>
<th>Sample</th>
<th>B</th>
<th>B$_{NaOH}$</th>
<th>B$_{SLS}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>C (%)</td>
<td>0</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>t (mins)</td>
<td>0</td>
<td>50</td>
<td>10</td>
</tr>
<tr>
<td>pH</td>
<td>7±0.1</td>
<td>12.6±0.2</td>
<td>7.6±0.2</td>
</tr>
<tr>
<td>T$_{sa}$ (MPa)</td>
<td>43.5326</td>
<td>166.43</td>
<td>122.79</td>
</tr>
<tr>
<td>T$_{sp}$ (MPa)</td>
<td>44.144</td>
<td>167.864</td>
<td>123.64</td>
</tr>
<tr>
<td>T$_{se}$ (%)</td>
<td>1.4</td>
<td>0.86</td>
<td>0.69</td>
</tr>
<tr>
<td>T$_{mn}$ (MPa)</td>
<td>2294.6</td>
<td>3472.6</td>
<td>58473</td>
</tr>
<tr>
<td>T$_{mp}$ (MPa)</td>
<td>2361.743</td>
<td>3548.12</td>
<td>58789</td>
</tr>
<tr>
<td>T$_{me}$ (%)</td>
<td>2.84</td>
<td>2.13</td>
<td>0.54</td>
</tr>
<tr>
<td>d$_f$ (mm)</td>
<td>0.021</td>
<td>0.017</td>
<td>0.018</td>
</tr>
<tr>
<td>L$_f$ (mm)</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>A$_r$</td>
<td>4761.9</td>
<td>5882.35</td>
<td>5556</td>
</tr>
<tr>
<td>$\rho_f$ (g/cm$^3$)</td>
<td>0.514</td>
<td>0.366</td>
<td>0.2</td>
</tr>
<tr>
<td>W$_i$ (%)</td>
<td>366.67</td>
<td>333.33</td>
<td>312.5</td>
</tr>
<tr>
<td>k$_1$</td>
<td>0.5945</td>
<td>0.7008</td>
<td>3.549</td>
</tr>
<tr>
<td>k$_2$</td>
<td>0.0025</td>
<td>0.0027</td>
<td>0.002</td>
</tr>
<tr>
<td>n</td>
<td>0.0946</td>
<td>0.1001</td>
<td>0.4125</td>
</tr>
<tr>
<td>k</td>
<td>0.4785</td>
<td>0.4578</td>
<td>0.0367</td>
</tr>
</tbody>
</table>

B, C, t, T$_{sa}$, T$_{sp}$, T$_{mn}$, T$_{se}$ and T$_{me}$ represent roselle fibers, treatment concentration, time, actual tensile strength, predicted tensile strength, actual tensile modulus, predicted tensile modulus, error in tensile strength and modulus, respectively. d$_f$, L$_f$, A$_r$, $\rho_f$, W$_i$, k$_1$, k$_2$, n and k represent fiber diameter, length, aspect ratio, density, water absorption, peleg’s water rate constant, initial water absorption, water absorption index and water absorption rate constant.

Fig. 1 — Kinetics of water absorption behaviour (a) Peleg’s model (b) Fick’s model
modification using SLS. The increase in water absorption may be due to affinity of –OH group on cellulosic backbone of water. Kinetics parameters of water absorption of *H. sabdariffa* fibers using Fickian’s model were determined from Figure 1(b). It can be deduced that unmodified and modified *H. sabdariffa* fibers using NaOH and SLS, respectively, exhibit less - Fickian behaviour since n < 0.5 (water penetration rate is much more below fiber relaxation rate). This is in agreement with the report of Gierszewska-Drużyńska and Ostrowska-Czubenko 28. Moreover, the experimental results were fit due to high value of $R^2$ with the generated model. It can be deduced that NaOH and SLS, respectively, reduced the sorption rate constant ($k$) with increase in sorption index ($n$). This might be due to fiber source and processing, shrinkage in fiber, reduction in porosity or void creation, and reduction in hemicellulose and lignin content which makes the *H. sabdariffa* fibers to become more hydrophobic in nature 28. Optimizing the fiber modifications for effective use in composite applications will not misleading on mechanical and physical properties of *H. sabdariffa* fibers, rather enhanced the fiber properties, avoidance of wastage and time consumption. This is in agreement with the report of Vardhini et al 31 on optimization of alkali treatment of banana fibers on lignin removal.

**Morphology of fibers**

Figure 2 shows the morphology of *H. sabdariffa* fibers using scanning electron microscope. It can be observed that the uHSF are in bundle of individual cells that held together by lignin with weak intermolecular bonds as seen in Figure 2(a). It can be observed that NaOH modified *H. sabdariffa* fibers shows roughness and separated with reduction in fiber diameter as observed in Figure 2(b), while that of SLS modified *H. sabdariffa* fibers are clean and rough with reduction in diameter (see Figure 2(b)) when compared with Figure 2(a). This may be attributed to removal of lignin, hemicellulose and wax or fatty substances which hold the fiber unit cells firmly. This is in agreement with report of Thiruchitrambalam et al 1 and Nikoghosyan et al 24. The presence of elemental atoms like carbon, oxygen and bromine atoms with compositions for unmodified *H. sabdariffa* fibers can be observed in spectra of EDS as shown in Figure 3. The presence of sodium...
atoms and counts of elemental compositions was observed and indicated an increase in mechanical properties of fibers due to modification by NaOH and SLS, respectively. H. sabdariffa fibers modified with NaOH gave the highest count of elemental compositions of carbon from crystalline cellulose, sodium, and bromine atoms compared with that of SLS modified H. sabdariffa fibers. The oxygen content shows the presence of micropores. The low oxygen content and presence of sodium atoms replaced the hydrogen atoms of H. sabdariffa fibers modified with SLS indicated high tensile properties compared with unmodified fibers.

**Conclusion**

Based on the result obtained on physical, tensile and microstructural behaviour of white Hibiscus sabdariffa fibers modified with NaOH and SLS. The followings conclusion can be drawn: NaOH and SLS modifications, respectively, improved the tensile properties of H. sabdariffa fibers and possess a quadratic and interaction relationship of concentration and time. At optimal modified conditions, NaOH improved the tensile strength and modulus of uHSF, respectively, by 282.31 and 49.38%, while that of SLS improved by 182.07 and 2448.28% which corroborated with SEM and EDS results. Hence, NaOH and SLS modification, respectively, found to be superior for high strength and stiffness applications. At optimal conditions of H. sabdariffa fibers, NaOH and SLS, respectively, reduced the density and water absorption, thereby, reduced the heaviness and improved the hydrophobic nature of H. sabdariffa fibers with improved aspect ratio. NaOH and SLS, respectively, increased the water sorption rate of H. sabdariffa fibers and the water diffusion behaviour was found to be less-Fickian behaviour since n < 0.5. With these modified conditions, H. sabdariffa fibers may be better in composites applications compared with kenaf and coir fibers.

**Acknowledgment**

We appreciate the support of Federal University of Technology, Owerri and Nnamdi Azikiwe University, Awka, Nigeria as well as Engineering Material Development Institute, Akure, Nigeria for assistance in mechanical analysis.

**List of symbols and abbreviations**

- \( C \) = Concentration of the chemical used (%)
- \( t \) = time (mins)
- \( T \) = Tensile property (MPa)
- \( L \) = length
- \( A_s \) = aspect ratio
- \( d \) = diameter
- \( \rho \) = density
- \( M \) = percentage of water absorption (%)
- \( uHSF \) = B = unmodified H. sabdariffa fibers
- \( mHSF \) = mercerized H. sabdariffa fibers
- NaOH = Sodium hydroxide
- SLS = Sodium lauryl sulphate
- SEM = Scanning electron microscope
- EDS = Energy X-ray diffractive spectroscope
- FTIR = Fourier transform infrared
- RSM = response surface methodology
- CCD = central composite design
- NaOH = sodium hydroxide
- SLS = sodium lauryl sulphate

**References**


