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Effect of chemical agents on morphology, tensile properties and water diffusion behaviour of *hibiscus sabdariffa* fibers

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Abstract

Effective utilization of *Hibiscus sabdariffa* fibers in composites applications as a reinforcing fibers in polymer matrix have been a major concern due to its poor mechanical and hydrophilic properties. It will be of benefits to environmental and technological advancement, if its properties are properly handled. In this study, the morphological, tensile and water absorption characteristics of *H. sabdariffa* fibers was aimed to be investigated. *H. sabdariffa* fibers was modified using sodium hydroxides, sodium lauryl sulphate and ethylene diamine tetraacetic acid. The morphology using scanning electron microscopy, tensile properties (strength, modulus, elongation and energy at break), water absorption and water diffusion behaviours were studied. Chemical modifications improved fiber surface and roughness, tensile strength and modulus, elongation and energy at break with reduced water absorption of *H. sabdariffa* fibers. The water diffusion behaviour is less - Fickian controlled by water penetration rate. Hence improved the hydrophobic nature of *H. sabdariffa* fibers.

Keywords: *Hibiscus sabdariffa* fibers, tensile properties, morphology, water diffusion behaviour

1. Introduction

H. Sabdariffa plant also known as roselle. It is a tropical plant found in parts of the world. Its extracted liquor from retting extraction is highly medicinal due to its antioxidant, antipyretic, anticancer, anti-inflammatory, hepatoprotective effects and its ability to improve visual acuity has been reported due to flavonoids, gossypetine, hibiscetine, sadderetine, delphinidin-3-monoglucoside, malvidin-3-mono-glucoside and their methyl-derivatives constituents reported (Abou-arab et al, 2011; Olusola, 2011; Fasoyiro et al, 2005) but disposal of its fiber becomes naissance in the environment due to increased biomass. Lignocellulosic fibers are natural polymers composed of crystalline cellulose microfibrils reinforced aromatic lignin and hemicelluloses matrix, as well as non-structural phytochemicals or extractives which influences their utilization (Agu et al, 2012). Natural fibers such as flax, hemp, jute, straw, wood fibers, rice husks, wheat, barley, oats, rye, cane (sugar and bamboo), grass reeds, kenaf, ramie, oil palm empty fruit bunch, sisal, coir, water hyacinth, pennywort, kapok, paper-mulberry, raffia, banana fiber, pineapple leaf fiber and papyrus have been used to reinforce polymers (Agu et al, 2012; Surata et al, 2014; Taj and Munawar, 2007). Fiber reinforced polymer composite have been employed in textile, construction, packaging, building and automotive industries due to lightweight, low cost, high specific strength, high modulus, reduced tool wear and safe manufacturing process (Benyahia et al, 2013; Bouatay et al, 2014; Brígida et al, 2010; Chandramohan and Bharanichandar, 2014). Despite the advantages, fibers possess poor wettability, incompatibility with some polymeric matrices and high moisture absorption which can be overcomes by chemical treatments (Azwin et al, 2009; Mohammed and Dauda, 2014; Singha and Rana, 2012). Hemicellulose is the cell wall polymer which responsible for the poor resistance to moisture or water absorption of natural fiber leading to swelling and presence of voids, hence poor mechanical properties and reduces dimensional stability of composites and microbial corrosion (Agu et al, 2012; Cristaldi et al, 2010). Many techniques such as electron beam irradiation (Han and Choi, 2010), mercerization (Chandramohan and Marimuthu, 2011; Herrera - Franco and Valadez - Gonzalez, 2005), use of coupling agents like silane (Azwin et al, 2009; Herrera - Franco and Valadez - Gonzalez, 2005; Liu and Dai, 2007) acetylation (Azeez et al, 2016), treatment with $NaHCO_3$ and $Cr_2(SO4)_3$ (Hossain et al. 2013; Mir et al, 2012), etherification (Kalia et al, 2009) have been employed to modify the fiber surfaces so as to improve both physical, chemical and mechanical properties but effectiveness differs in fibers with its composites. In this study, the effect of sodium hydroxide, sodium lauryl sulphate, and ethylene diamine tetraacetic acid on morphology, tensile properties, water absorption and water diffusion behaviour was aimed to be investigated.

2. Materials and Methods

2.1 Materials

Hibiscus sabdariffa fibers with proximate composition of moisture content (3.88 %), water soluble (3.77 %), ash (1.54 %), wax (1.82 %), pectin (2.04 %), lignin (12.7 %), hemicelluloses (13.32 %) and cellulose (60.93%) was used. The dried fibers are designated as untreated *H. sabdariffa* fibers (uHSF). Sodium hydroxide and sodium lauryl sulphate (analytical grade chemicals), respectively, used for fiber modification at optimum conditions of 3.0% for 50 mins and 3.0 % for 10 mins. The chemical used were obtained from Rovert scientific limited, Benin city in Edo state, Nigeria.

2.2 Scanning electron microscope (SEM) analysis

SEM microscopy analysis was conducted on modified *H. sabdariffa* fibers using High resolution scanning electron microscope (SEM) of ASPEX 3020 model to study the morphology of surfaces of *H. sabdariffa* fibers at optimal 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. The fiber sample was mounted on stubs with silver paste. A thin film of platinum is vacuum-evaporated before the photomicrographs or spectrum were taken in order to enhance the conductivity of the fibers.

2.3 Tensile testing

Tensile test was conducted using Universal Testing Machine Instron 3369 in accordance with ASTM Test Method D 638-03. Strand of fibers were cut to 130 mm 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 *H. sabdariffa* fiber.

2.4 Water absorption

H. sabdariffa fibers were dried in an oven at 60^0C for 2 hours before water absorption analysis. 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. Water absorption determined as percentage by mass gain using equation (3) as given by (Singha and Rana, 2012). The process was continued until equilibrium was attained.

$$Water\ absorption\ (\%) = \frac{M_t - M_0}{M_0} x100\% \quad (1)$$

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

2.5 Water diffusion mechanisms

The diffusion phenomenon was studied through water absorption method as described by (Herrera - Franco and Valadez - Gonzalez, 2005). The water diffusion mechanism through interphase of the fibers was study using Becker's model and power law expression respectively as given by equations (2) and (3);

$$M_t - M_o = \alpha_s t^{1/2} + M_p \quad (2)$$

Where M_t is water content at any time t, α_s is the water sorption rate, M_o is the initial moisture content, M_p is the initial water gain due to fast capillary action.

$$\frac{M_t}{M_m} = kt^n \quad (3)$$

Where M_t and M_m represents the water content at any time t and water absorption at saturation point, and k and n are constants which were determined as intercept and slope, respectively, of M_t/M_m versus t in the log - log plot of water absorption with time. The magnitude of n indicated whether the composite is governed by Fickian diffusion model or Non – Fickian diffusion model. The water diffusion coefficient (D_{wf}) through the fibers for short time exposure was evaluated using the equation (4);

$$D_{wf} = \pi \left[\frac{h}{4M_m}\right]^2 [S]^2 \quad (4)$$

Where $S = \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}$, M_m, h, M_1 and M_2 are the maximum percentage of water content, fiber thickness, percentage of water content at specific time $t_1$and t_2 selected in the linear portion of the plot of water sorption (M_t) versus $\sqrt{t}$. S was evaluated as gradient plot of M_t against$\sqrt{t}$ based on equation (4).

3. Results and Discussion

3.1 Morphology of the *H. sabdariffa* fibers

Figure 1 reveals the morphology of untreated and treated *H. sabdariffa* fiber. It can be observed that *H. sabdariffa* fiber are in bundle of individual cells that held together by lignin with presence of impurities on surface as shown in Figure 1(a). Figure 1(b) shows the roughness and serrated surfaces with shrinkage in fiber surface. It can be observed in Figure 1(c) that the fiber surfaces are clean and separated with irregular surfaces. This may be due to removal of lignin, hemicellulose, wax and other impurities at the fiber surfaces when SLS treatment was used. In Figure 1(d), disorganized fibril and weaken of the gel structure with separated fiber surface was observed which might be due to removal of wax, lignin, hemicellulose of *H. sabdariffa* fibers when treated with EDTA. This is an indication that EDTA serves as chelating agent and improved fiber separation, thereby, improved interfacial adhesion.

3.2 Tensile properties of *H. sabdariffa* fibers

Table 1 reveals the tensile properties of the *H. Sabdariffa* fibers based on chemical treatment conditions. It can be deduced that NaOH, SLS and EDTA treatment, respectively, increased the tensile strength by 292.38, 102.73 and 302.83 % of untreated *H. Sabdariffa* fiber. This indicated that all chemical treatment used really improved load carrying capacity of the fibers. The tensile modulus is a measure of the stiffness of the fibers. It can also be observed that NaOH, SLS and EDTA treatment, respectively, improved the tensile modulus of *H. Sabdariffa* fiber by 58.3, 36.86 and 81.93 % of untreated *H. Sabdariffa* fibers. EDTA gave the highest effect based on strength. This is similar to the report of (Troedec et al, 2007). However, it can be deduced that the NaOH treated *H. Sabdariffa* fibers gave highest elongation with about 380.47% increase in elongation of untreated *H. Sabdariffa* fibers, followed by EDTA (224.84 % increase) and SLS gave least elongation. This is to say that all treatments improved ductility of *H. Sabdariffa* fiber. It can be observed that the treated *H. Sabdariffa* fibers with NaOH, SLS and EDTA, respectively, required 979. 7, 303.15 and 988.65 % energy of untreated *H. Sabdariffa* fibers for failure to occur. This reveals that about 75 % of tensile properties of *H. Sabdariffa* fibers gave EDTA treatment an edge as a better modifying agent, though all treatment improved tensile properties of *H. Sabdariffa* fibers. This may be attributed to removal of impurities from the *H. Sabdariffa* fiber surfaces, thereby, causes a reduction in diameter fiber. This can help for better interlocking between matrix and fiber as reported by (Cao et al, 2007; Thiruchitrambalam et al, 2009; Troëdec et al, 2007).

3.3 Water absorption of *H. Sabdariffa* fibers

Figure 1 reveals the water absorption which is determined as the weight gained of the *H. Sabdariffa* fibers based on time of exposure to water as environmental conditions. It can be observed that the maximum water absorption or saturation condition for untreated, NaOH, SLS and EDTA treated *H. Sabdariffa* fibers, respectively, attained at exposure time of 25, 30, 40 and 50 minutes. This shows that all chemical treatments increased the exposure time of *H. Sabdariffa* fibers to attained water absorption saturation point with reduced water absorption. This means that chemical treatments employed reduced the hydrophilic nature of *H. Sabdariffa* fibers.

3.3.1 Water absorption behaviour of *H. Sabdariffa* fiber fibers using Becker's model

Figure 3 shows the simulated water absorption parameters for untreated and treated *H. Sabdariffa* fibers using Becker's model (Equation 1) with its fitness. The coefficient of determination (R^2) indicates the fitness of Becker's model. It can be observed that chemical treatments improved the fitness of Becker's model of water absorption of *H. Sabdariffa* fibers as presented in Table 2. The water absorption rate constant (α_s) for untreated and treated *H. Sabdariffa* fiber does not follow a definite profile. It can be deduced that EDTA and SLS increased the magnitude of α_s by 1.67 and 2.14 times of untreated *H. Sabdariffa* fibers, respectively, while NaOH reduced by 0.039 times of untreated *H. Sabdariffa* fibers. Initial water absorption (M_P) due to fast capillary action were found to be lower for NaOH, EDTA and SLS treated *H. Sabdariffa* fibers, respectively, compared with untreated. This also reveals the decrease in water absorption capacity and improvement on hydrophobic nature of *H. Sabdariffa* fibers based on treatment as presented in Table 2.

3.3.2 Water absorption and diffusion behaviour of *H. Sabdariffa* fibers using Fickian's model

It can be deduced that untreated and treated *H. Sabdariffa* fibers with NaOH, SLS and EDTA, respectively, are Less Fickian behaviour since $n < 0.5$ (rate of water penetration is much below the fiber chain relaxation rate) as presented in Table 3. This is in agreement with the report of Gierszewska-Drużyńska and Ostrowska-Czubenko (2012). However, the experimental results obtained for *H. Sabdariffa* fibers based on untreated, treated with NaOH, SLS and EDTA treatments, respectively, were fit due to high value of R^2 0.8234, 0.8443, 0.9749 and 0.949 explains 82.34, 84.43, 97.49 and 94.9 % of the observed variability for water absorption as shown in Figure 4 and 17.66, 15.51, 2.51 and 5.1 % indicates the residue which cannot be explain. It can also be observed that NaOH, SLS and EDTA treatments reduced the water absorption rate constant (k) with sorption index (n) of *H. Sabdariffa* fibers (Table 3). Moreover, NaOH treatment reduced the water diffusion coefficient of *H. Sabdariffa* fibers, otherwise for the case of SLS and EDTA treatments. The reduction in water diffusion coefficient of NaOH treated *H. Sabdariffa* fibers might be due to shrinkage in fiber, decrease in porosity or void creation, and reduction in hemicellulose and lignin content which makes the *H. Sabdariffa* fibers to become more hydrophobic in nature. However, the increase in water diffusion coefficient of SLS and EDTA treated *H. Sabdariffa* fibers may be due to large void created in the fibers.

4. Conclusion

Based on the results obtained, the following conclusion can be drawn:

The morphology reveals the cleaning of fiber surfaces through removal of removal of impurities by NaOH, SLS and EDTA, thereby, improved the hydrophobic nature and surface properties of *H. Sabdariffa* fibers.

Sodium hydroxide, sodium lauryl sulphate and ethylene diamine tetraacetic acid treatments, respectively, improved the tensile properties and ductility of *Sabdariffa* fiber with superiority for the case of ethylene diamine tetraacetic acid treatment.

All treatments used in this work improved the fitness of the Becker's model and power law expression with increased water absorption rate of *H. Sabdariffa* fibers treated with SLS and EDTA while NaOH treatment reduced the water absorption rate of *H. Sabdariffa* fibers. Initial water holding capacity of SLS and EDTA treated *H. Sabdariffa* fibers reduced and vice-versa for the case NaOH.

Both untreated and treated *H. Sabdariffa* fibers water absorption were Less Fickian behaviour since the rate of water diffusion is below the relaxation rate of fibers.

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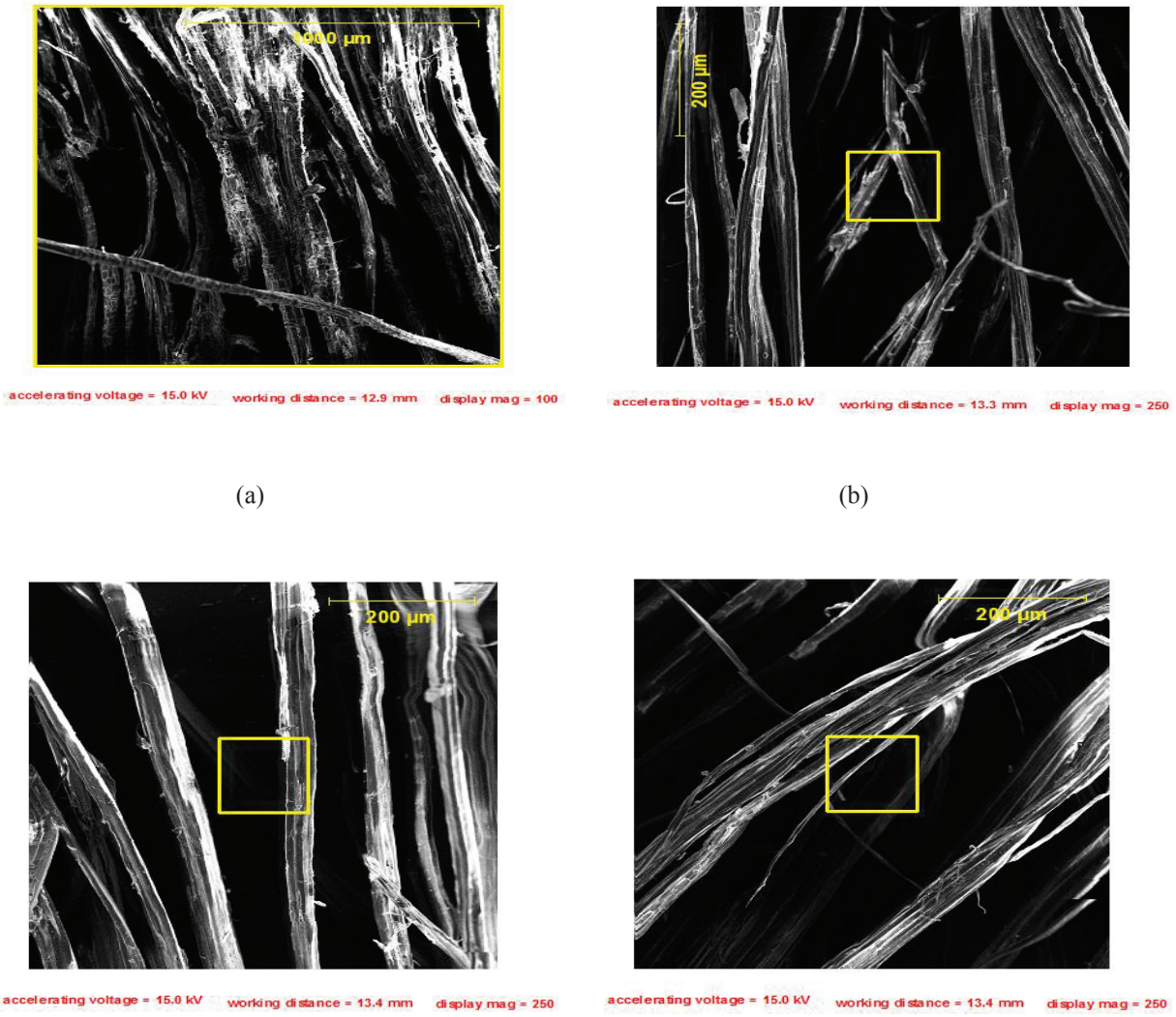

Figure 1: SEM of *H. Sabdariffa* fibers: (a) untreated (b) modified with NaOH (c) modified with SLS (d) modified with EDTA

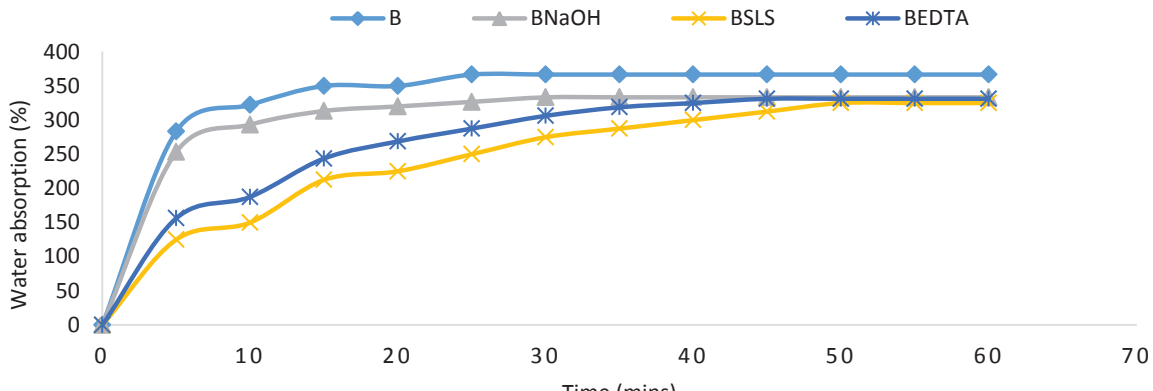

Figure 2: Water absorption of *H. Sabdariffa* fibers with exposure time

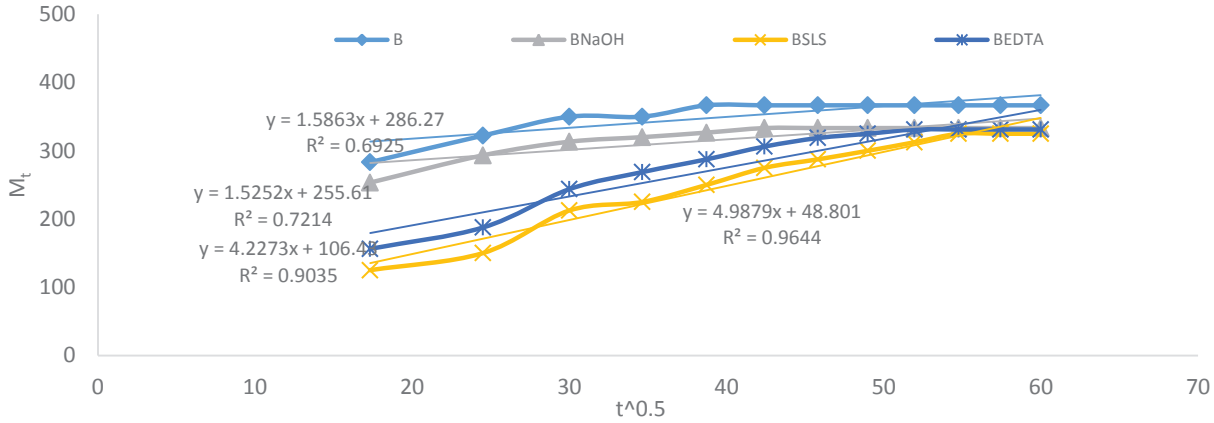

Figure 3: Simulation of water sorption of *H. Sabdariffa* fiber using Becker's model

Table 1: Tensile properties of H. Sabdariffa fiber based on treatment conditions

Fiber sample	*C (%)*	*t (mins)*	*pH*	T_{sa} *(MPa)*	T_m *(MPa)*	*Elongation (%)*	*Energy at break (J)*
B	0	0	7±0.1	43.1326	2302.8	2.01536	0.00793
B_{NaOH}	3	50	12.6±0.2	169.2432	3645.6828	9.68312	0.07769
B_{SLS}	3	10	7.6±0.2	87.4422	3151.7	3.46656	0.02404
B_{EDTA}	3.845	81	6.6±0.2	173.75	4189.5	6.54673	0.0784

B is H. Sabdariffa fiber *respectively, subscript NaOH, SLS and EDTA are chemical treatments of modified fiber*

Table 2: Fitness of *H. sabdariffa* fiber using Becker's model Parameters based on treatments

Sample	Treatment conditions		α_s ($min^{-1/2}$)	M_P (%)	R^2
	C (%)	t (mins)			
B	0	0	1.5863	286.27	0.6925
B_{NaOH}	3	50	1.5252	255.61	0.7214
B_{SLS}	3	10	4.9878	48.801	0.9644
B_{EDTA}	3.945	80.42	4.2273	106.43	0.9035

B is H. Sabdariffa fiber *respectively, subscript NaOH, SLS and EDTA are chemical treatments of modified fiber*

Table 3: Diffusion behaviour of roselle, food gum and kapok fibers using Fickian model

Fiber sample	*C (%)*	*t (mins)*	*pH*	d_f *(μm)*	*n*	*k*	D_{wf} *(mm^2/s)*	R^2
B	0	0	7±0.1	0.021	0.0946	0.4785	1.6207E-09	0.8234
B_{NaOH}	3	50	12.6±0.2	0.017	0.1001	0.4578	1.1880E-09	0.8443
B_{SLS}	3	10	7.6±0.2	0.018	0.4125	0.0367	1.6207E-08	0.9749
B_{EDTA}	3.945	80.42	6.6±0.2	0.016	0.3209	0.0788	8.1862E-09	0.949

B is H. Sabdariffa fiber *respectively, subscript NaOH, SLS and EDTA are chemical treatments of modified fiber*