



MODIFIED FOOD GUM (*Cissus populnea*) FIBERS: MICROSTRUCTURAL BEHAVIOUR, PHYSICO - MECHANICAL PROPERTIES AND KINETICS OF WATER ABSORPTION

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ABSTRACT

The aim of this study is to investigate the effect of chemical modifications on the properties of *Cissus populnea* fibers. The method involve chemical treatments of *C. populnea* fibers using sodium hydroxide (NaOH), sodium lauryl sulphate (SLS) and ethylene diamine tetraacetic acid (EDTA). Microstructural behaviour (scanning electron microscope 'SEM' and fourier transform infrared 'FTIR' analysis), mechanical properties (tensile strength, modulus, elongation and energy at break), the physical properties (aspect ratio, density and water absorption) were determined and compared with untreated *Cissus populnea* fibers. SEM topography and FTIR spectra, respectively, revealed the change in morphology and structure after treatments which resulted to increase in tensile properties of *Cissus populnea* fibers. The treatments increased the aspect ratio with reduced density and water absorption capacity of *Cissus populnea* fibers. SLS and EDTA treatments, respectively, increased the tensile strength of *Cissus populnea* fibers by 298.46 and 250.86 %. Hence, SLS is superior as surface modification for improving tensile strength while EDTA treatment superior for improving tensile modulus and hydrophobic nature of *Cissus populnea* fibers.

Keywords: *Cissus populnea* fibers, SEM, FTIR, Peleg's model, water diffusion.

1. INTRODUCTION

Cissus populnea plant is a plant climber found in the western part of Nigeria called food gum plant. Its gum is used as soup thickener, venereal diseases, indigestion [1] and drug binder [2]. Its fiber may be obtained using retting extraction. The liquor are used traditionally in ethno - medicine for treating male infertility [3] and disposal of the fiber causes naissance in the environment due to increased biomass. Natural fibers are green products abundantly available for enhancement of plastics in composites applications. Natural fibers are also known as plants fibers. It is probably one of the most ubiquitous and abundant biopolymers on the planet and employed as a renewable raw material in a wide range of applications [4, 5]. Its proximate composition include cellulose, hemicellulose, lignin, pectins, wax, water soluble and ash contents as reported by researcher [6]. They are materials 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 due to their richness in lignin, hemicellulose and cellulose [4, 5, 7]. In composites, the applications depends on their composition, physical and mechanical properties of fibers [7]. The extent of natural fibers as environmental benefits over synthetic fibers in industrial applications partly depends on the possibilities for substitution of the various fibers in the processing, the energy requirement of the production process, the product performance and the functional life time, including options for waste disposal. Natural fibers such as banana, jute, sponge-gourd, sugarcane, coco-nut, rice straw, sisal and coconut are produced as a replacement for glass or other

traditional reinforcement materials in composites. The advantages include low density, high toughness, comparable specific strength properties, reduction in tool wear, ease of separation, decrease in energy for fabrication, non - toxic, low cost, weight saving, reinforcing materials, improvement of stiffness and emerging saving of petroleum products, thereby, relieving the nation economy from melting down [7, 8]. The limitation of natural fibers for industrials applications may be attributed to its undesirable properties of relatively high hydrophilic character resulted to poor interfacial attraction with the matrices and poor quality of the composites [9, 10]. The development of composites with better mechanical properties and environmental performance can be achieved through improve in hydrophobicity of the natural fibers and interfacial adhesion between the matrix and natural fibers, which may increase the compatibility and dispersability of fiber in the matrix [11]. Chemical treatments are considered in modifying the fiber surface properties which can enhance the bond strength between fiber and matrix due to differential hydroxyl group and also can reduce water absorption of the natural fiber [4]. Yet, the driving force of water absorption through the fibers have not been verified, though it might be differed from fiber to fiber. In this study, the effect of chemical treatments on microstructural (morphology and FTIR analysis) behaviour, physical properties (aspect ratio, density and water absorption), kinetics of water absorption and water diffusion behaviour of modified *C. populnea* fiber using sodium hydroxide (NaOH), sodium lauryl sulphate (SLS) and ethylene diamine acetic acid (EDTA) at optimum treatment conditions was aimed to be investigated.



2. MATERIALS AND METHODS

2.1 Materials

White *C. populnea* fibers with composition of moisture content (3.94 %), water soluble (2.33 %), ash (1.59 %), wax (2.94 %), pectin (1.14 %), lignin (11.52 %), hemicelluloses (14.74 %) and cellulose (61.8 %) was used and modified using analytical grade chemicals of sodium hydroxide, sodium lauryl and ethylene diamine tetraacetic acid obtained from Rovert scientific limited, Benin city in Edo state, Nigeria. The chosen chemicals were based on low cost, availability and effectiveness.

2.2 Chemical surface modifications

C. populnea fibers were cut into 120 mm length modified using NaOH, SLS and EDTA, respectively, at optimal level of 15 % for 20.94 mins, 5.84 % for 20.22 mins and 5.0 % for 150 mins at room temperature. The unmodified and modified *C. populnea* fibers were washed severally with deionized water until neutral pH of 7 was obtained. The fibers was finally dried in an air oven at 60°C for 2 hours.

2.3 Surface morphology

SEM microscopy analysis was conducted on modified *C. populnea* fibers using high resolution scanning electron microscope (SEM) of ASPEX 3020 model to study the morphology of surfaces of *C. populnea* 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 was taken in order to enhance the conductivity of the fibers.

2.4 Fourier transform infrared (FTIR) Analysis

4.0g of unmodified and modified *C. populnea* fibers sample was crushed into pellets. The spectrum was run by applying fiber sample on KBr cell covering the range of frequencies from 600 – 4000 cm⁻¹ with scanning period of 20 seconds. The crushed powder sample (0.1g) was mixed with dry KBr (0.4g) and transferred to sample compartment of the Buck Scientific M500 Infrared Spectrophotometer. The spectrophotometer was set at 100% transmittance with pure KBr pellet and the transmittance reading was obtained and stored.

2.5 Tensile strength analysis on *C. populnea* fibers

Tensile test was conducted on a single fibers using Universal Testing Machine Instron 3369. A single fiber tensile test was conducted on three *C. populnea* fibers of length 120mm with gauge length of 100mm and average diameter of 0.11 ± 0.02mm. The tensile strength, extension and modulus were determined and study.

2.6 Fiber aspect ratio

The length of 5 randomly sampled *C. populnea* fibers were measured and recorded. The fibers' diameters were measured at different locations along their length using micrometer screw gauge and the average diameter of 5 randomly sampled *C. populnea* fibers were measured at 5 positions. The mean aspect ratio was calculated using equation (1) as given by Herrera - Franco and Valadez - Gonzalez [12]

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

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

2.7 Fiber density

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. populnea* fiber. The density was calculated using equation (2):

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

Where ρ_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.

2.8 Water absorption

The test was carried out in accordance with ASTM D - 570. Prior to testing, the *C. populnea* fibers were dried in an oven at 60°C 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. The process was continued until equilibrium was attained. The water absorption was determined by percentage mass gain using equation (3) as given by [13, 14];

$$\text{Water absorption (\%)} = \frac{M_t - M_o}{M_o} \times 100\% \quad (3)$$

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).

2.9 Kinetics of water absorption and diffusion behaviours

The diffusion phenomenon was studied through water absorption method The kinetics of water absorption was evaluated using Peleg's model and Power law represented by equations (4) and (5) respectively as reported by Afolabi et al [15] and Osman et al [16];

$$M_t - M_o = \frac{t}{k_1 + k_2 t} \quad (4)$$

$$\frac{M_t}{M_m} = kt^n \quad (5)$$



Where M_t , M_0 and M_m are the water content at specific time t , initial water absorption and the equilibrium water content (EWC), respectively. k_1 and k_2 are Peleg rate constant which relates to initial absorption rate and Peleg capacity constant relate to maximum attainable water content, respectively. k and n are constants obtained as an 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 water absorption through the fiber 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 (6) as given by Osman et al [16]:

$$D_{wf} = \pi \left[\frac{h}{4M_m} \right]^2 [S]^2 \quad (6)$$

Where $S = \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}$, M_m is the maximum percentage of water content, h is the fiber thickness, M_1 and M_2 are percentage of water content at respective 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 (6).

3. RESULTS AND DISCUSSIONS

3.1 Surface morphology

Morphology of *C. populnea* fibers using high resolution scanning electron microscope (SEM) of ASPEX 3020 model are presented in Figure-1(a) - (b). In Figure-1(a), it can be observed that there is presence of lignin, hemicellulose and wax deposition on the surface of untreated *C. populnea* fibers. Figure-1(b) shows the roughness and serrated surfaces which indicated the removal of lignin, hemicellulose and wax substances from fiber surface. This leads to reduction in fibers' diameter and increased the reactive sites, hence improved the adhesion of the *C. populnea* fibers. This is similar to the report of researchers [17, 18]. Figure-1(c) depicts clean and smooth fiber surfaces, thereby, reduced the diameter of fibers. Disorganized fibril with weakened of the gel structure of cellulose fibers was observed in Figure-1(d). This may be due to removal of wax and lignin from *C. populnea* fibers when modified with EDTA. This is an indication that EDTA serves as chelating agent.

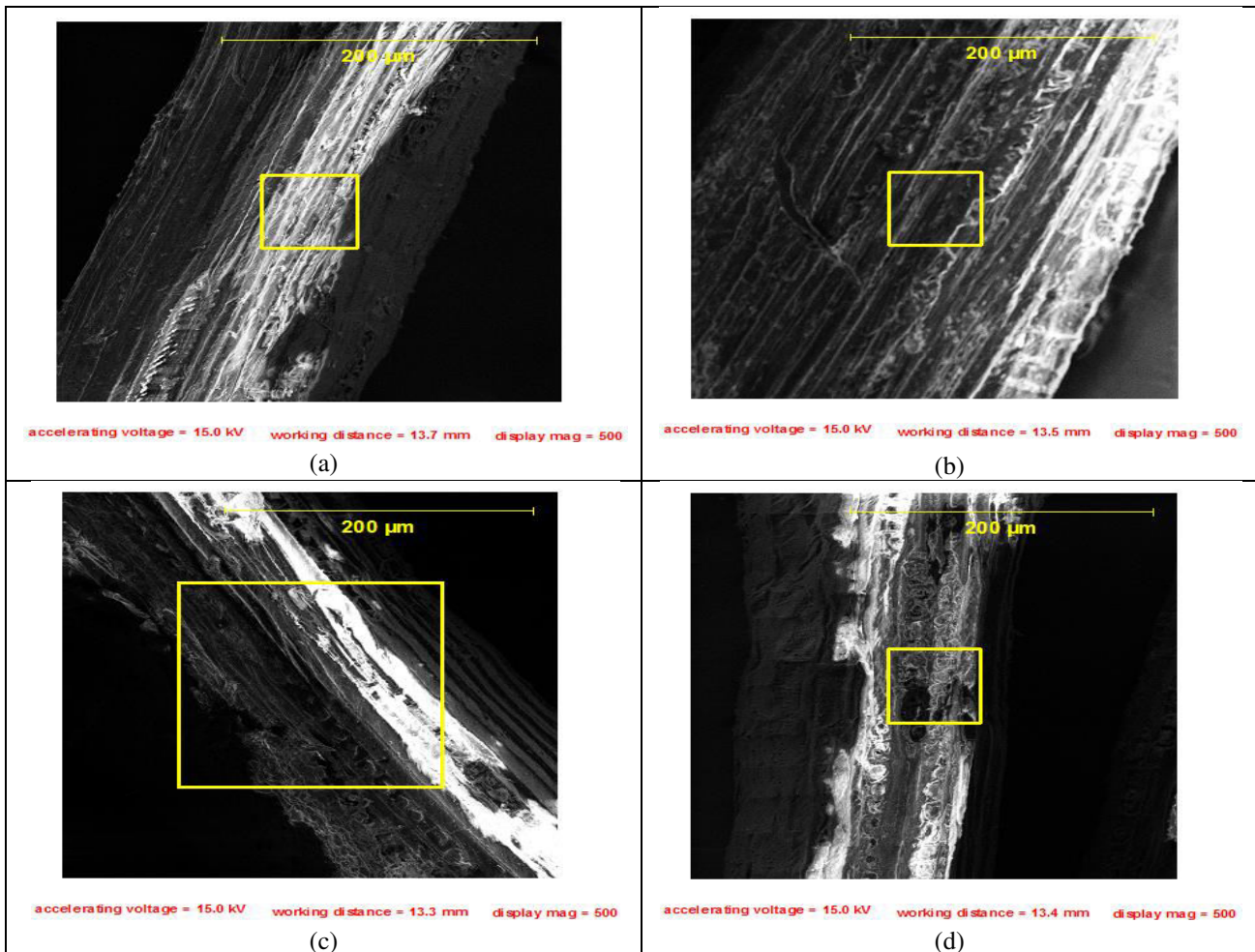


Figure-1. SEM of *C. populnea* fibers: (a) untreated (b) modified with NaOH (c) modified with SLS (d) modified with EDTA



3.2 FTIR spectroscopic analysis of *C. populnea* fibers

The FTIR spectrum of unmodified and modified *C. populnea* fibers are shown in Figure -2. The peak of the absorption bands corresponds to various functional groups are presented in Table-1. Absorption band peak of -OH stretching vibration (Figure-2a) in unmodified *C. populnea* fibers shifted to 3281.7 and 3531.8 cm^{-1} , 3486.6 and 3269.1 cm^{-1} , and 3505.8 cm^{-1} when modified with NaOH, SLS and EDTA, respectively. The absorption peak was found for C - O of unmodified *C. populnea* fibers shifted to 1139.1, 1131.9 and 1109.7 cm^{-1} when modified with NaOH, SLS and EDTA, respectively. There is disappearance of acryl group of cellulose with absorption peak of 1255.4 cm^{-1} when modified with NaOH, SLS and EDTA, respectively. The absorption peak of C=C from

ring vibration of lignin shifted to 2360.2, 2355.7 and 2363.5 cm^{-1} , respectively, when modified with NaOH, SLS and EDTA. It can also be observed that there is shift in absorption peak from 1645 cm^{-1} to 1646.4, 1647.3 and 1646.6 cm^{-1} which characterized -OH from the water content when modified with NaOH, SLS and EDTA. There is appearance of C=C aromatics symmetric rings from lignin correspond to peak 1538.6 cm^{-1} when fiber treated with EDTA. The absorption peak of 2905.5 cm^{-1} correspond to -CH₂ stretching of polysaccharides and wax shifted to 2904.6 and 2909.9 cm^{-1} for SLS and EDTA treatment, respectively, but disappeared when treated with NaOH. The shift in absorption peak with disappearance and appearance of functional groups characterized active participation of the functional group in the reaction with change in mechanical properties.

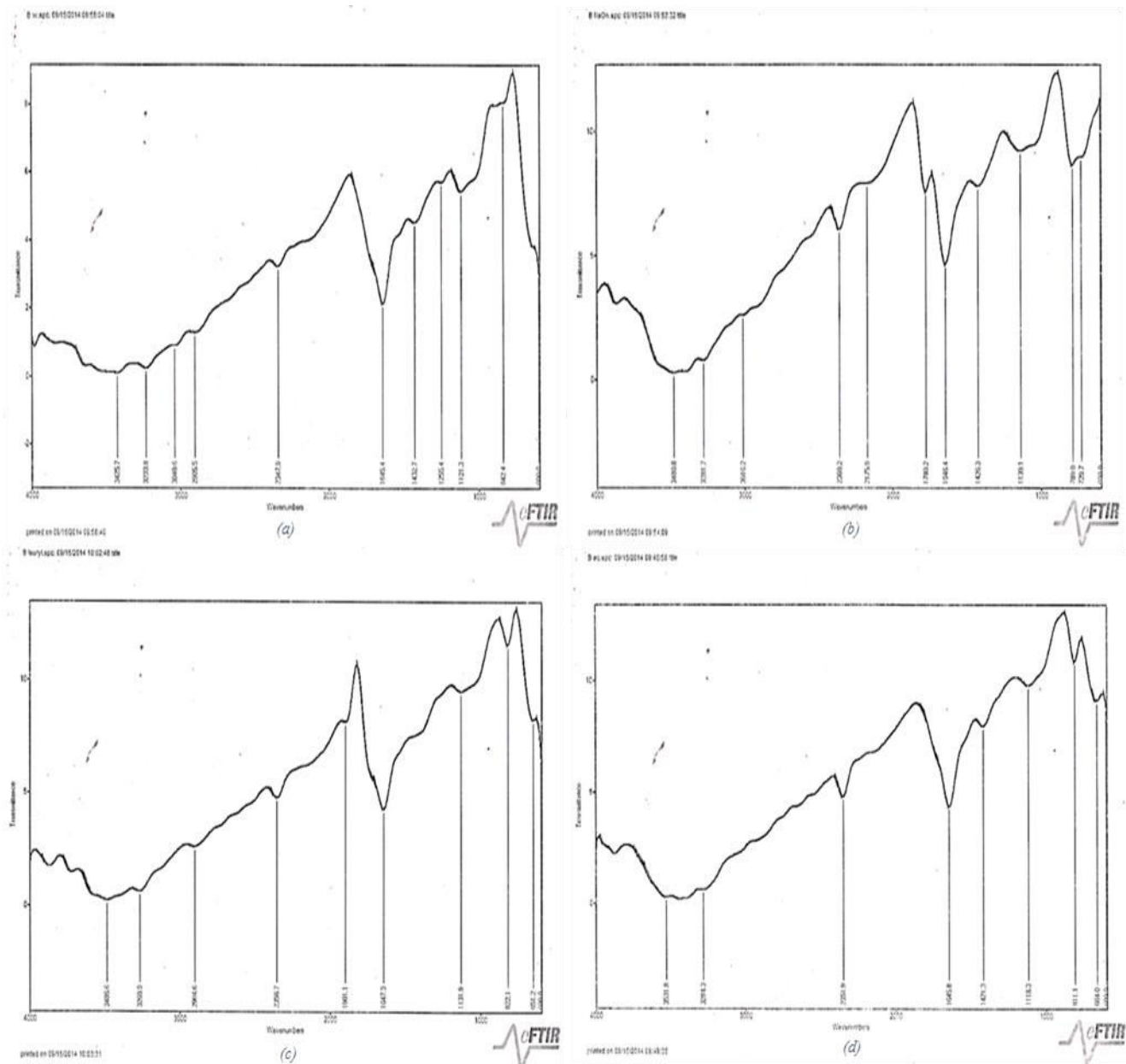




Figure-2. FTIR of *C. populnea* fibers with (a) unmodified (b) modified with NaOH (c) modified with SLS (d) modified with EDTA.

Table-1. FT-IR spectral data of unmodified white *C. populnea* fibers.

Wave number (cm ⁻¹)	Functional group
3425.7 - 3253.8	v (-OH) broad, strong band from the cellulose, hemicellulose and lignin of <i>C. populnea</i> fiber
~2905.5	v (C-H) in aromatic rings and alkanes
~2347.9	v (C=C) aromatic skeletal ring vibration due to lignin
~1645	-OH from water content of the fiber
~1432.7	δ (C-H) from pectins, lignins and hemicellulose
~1255.4	δ (C-OH) out-of-plane
~1121.3	v (C-OH) secondary alcohol
~842.4	phenyl ring substitution band from lignin

Table-2 shows the tensile properties of the *C. populnea* fibers based on chemical treatment used for modification. It can be observed that NaOH, SLS and EDTA treatment, respectively, increased the tensile strength by 159.99, 298.46 and 250.86 % of untreated *C. populnea* fibers. This indicated that all chemical treatments used really improved load carrying capacity of the fiber, though SLS treatment gave highest tensile strength. It can be deduced that SLS treated *C. populnea* fiber can be employed where high reinforcement is needed. 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 *C. populnea* fibers by 584.34, 458.82 and 1629.85 % of untreated *C. populnea* fibers. EDTA gave the highest stiffness effect. This is similar to the report of [19]. Moreover, it can be deduced that the NaOH treated *C. populnea* fiber gave highest elongation with about 548.29% of the untreated *C. populnea* fibers as well as

that of EDTA treatment with about 544.58% of the untreated *C. populnea* fibers and SLS gave least elongation with about 172.01% of the untreated *C. populnea* fibers. This is to say that all treatments improved ductility of *C. populnea* fibers. It can be observed that the treated *C. populnea* fibers with NaOH, SLS and EDTA, respectively, required about 457.53, 303.15 and 218.54 % energy of untreated *C. populnea* fibers for fracture to occur. This reveals that the application of *C. populnea* fibers in composite applications should be based on most favourable properties of the chemical treatments, although all treatment improved tensile properties of *C. populnea* fibers. The improved properties of *C. populnea* fibers may be attributed to removal of impurities from the *C. populnea* fiber surfaces, thereby, causes a reduction in diameter of the fiber. This is better for improvement of interlocking between matrix and fiber as reported by [20-22].

Table-2. Tensile properties of *C. populnea* fiber at treatment conditions.

Fiber sample	C (%)	t (mins)	pH	T _{sa} (MPa)	T _m (MPa)	Elongation (%)	Energy at break
CP	0	0	7±0.1	20.5249	966.75	2.01536	0.00793
CP _{NaOH}	15	20.94	12.6±0.2	53.363	5647.63	11.05	0.04288
CP _{SLS}	5.84	20.22	7.6±0.2	81.783	5402.4	3.46656	0.02404
CP _{EDTA}	5	150	6.6±0.2	72.01384	15756.54	10.9752	0.01733

CP represent *C. populnea* fiber, subscript NaOH, SLS and EDTA represent chemical treatment used

The aspect ratio, density and water absorption for unmodified and modified *C. populnea* fibers at treatment conditions are presented in Table-3. It can be observed that the aspect ratio of modified *C. populnea* fiber with NaOH, SLS and EDTA, respectively, found to be higher compared with that of untreated fibers due to reduction in fibers diameter. The increased aspect ratio helps in fiber dispersion and compatibility with matrix. This is in agreement with the report of many researchers that NaOH,

SLS and EDTA, respectively, enhanced the aspect ratio of fibers, thereby, caused reduction in diameter of the fibers through removal of amorphous constituents and increase in rough surface topography [19, 20, 23]. It can also be deduced that NaOH, SLS and EDTA reduced the density of unmodified *C. populnea* fibers by 23.29, 54.95 and 37.24 %, respectively. This is in agreement with the report of Nikoghosyan *et al* [24]. It can also be observed that NaOH, SLS and EDTA treatments, respectively, reduced



the water sorption by 7.86, 15.16 and 57.34 % of unmodified *C. populnea* fiber at saturation point as shown in Figure-3. This means that the chemical treatments

reduce the hydrophilic character of *C. populnea* fibers. This stays in agreement with the report of many researchers [19, 20].

Table-3. Some physical properties of *C. populnea* fibers at treatment conditions.

Fiber sample	C (%)	t (mins)	pH	d_f (mm)	L_f (mm)	A_r	ρ_f (g/cm ³)	W_s (%)
CP	0	0	7±0.1	0.11	100	909.091	0.717	325.58
CP _{NaOH}	15	20.94	12.6±0.2	0.104	100	961.538	0.55	300
CP _{SLS}	5.84	20.22	7.6±0.2	0.095	100	1052.63	0.323	276.19
CP _{EDTA}	5	150	6.7±0.2	0.1	100	1000	0.45	138.89

CP represent *C. populnea* fiber, subscript NaOH, SLS and EDTA represent chemical treatment used

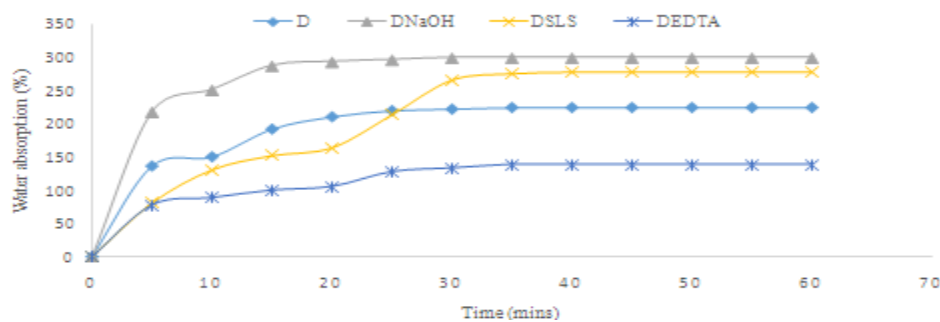


Figure-3. Water absorption of *C. populnea* based on exposure time.

Water sorption behaviour of *C. populnea* fibers using Peleg's model

Peleg's model (Equation 4) of water absorption for unmodified and modified *C. populnea* fibers and numerical results are presented in Table-3, while the graphical plot results are shown in Figures-4. It can be observed that the Peleg's model accurately describes the water absorption kinetics of unmodified and modified *C. populnea* fibers due to high value of R^2 . However, the

treatment of *C. populnea* fibers with NaOH reduced the value of k_1 by about half of unmodified *C. populnea* fibers while EDTA and SLS treatment, respectively, more than doubled and thrice of unmodified *C. populnea* fibers. It can be observed that k_1 increases with increased k_2 for NaOH and EDTA modified *C. populnea* fibers while k_1 increases with reduced k_2 for SLS modified *C. populnea* fibers.

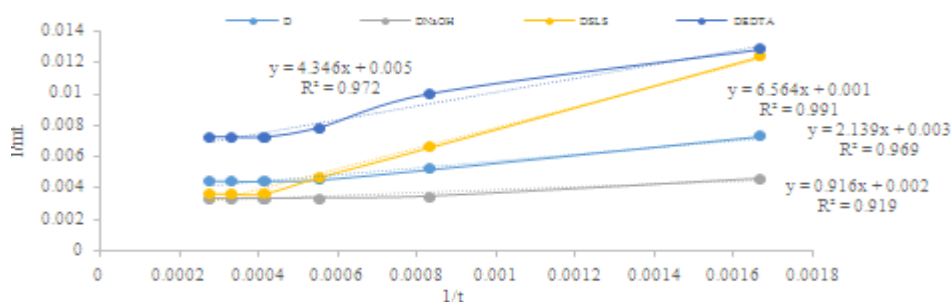


Figure-4. Simulation of water sorption of *C. populnea* fibers using Peleg's model.

Water sorption and diffusion behaviour of *C. populnea* fibers using Fickian's model

The water sorption rate constant and index of *C. populnea* fibers using Fickian model (equation 4) are determined from Figure-5. The diffusivity coefficient of both unmodified and modified *C. populnea* fibers using

NaOH, SLS and EDTA are presented in Table-4. It can be deduced that unmodified and modified *C. populnea* fibers with NaOH and EDTA treatments, respectively, are less Fickian behaviour since $n < 0.5$. This is due to water penetration rate is much more below the fiber relaxation rate. SLS modified *Cissus populnea* fiber is Non - Fickian



diffusion in nature since $n = 0.5233$ ($0.5 < n < 1.0$). This is in agreement with the report of Gierszewska – Druzynska and Ostrowska – Czubenko [25]. Moreover, the experimental results obtained are fit for all treatments due to high value of R^2 with the generated model as shown in the Figure 5. It reveals that NaOH treatments reduced the sorption rate constant (k) with increase in sorption index (n) and vice versa for the case of SLS and EDTA treatments. This might be due to shrinkage in fiber, porosity or void creation and level of reduction in

hemicellulose and lignin content which makes the food gum fiber to become more hydrophobic in nature. From evaluated water diffusion coefficient, it can be observed that the water sorption rate constant increases with decrease in water diffusion coefficient (D_{wf}) of *C. populnea* fibers when modified with NaOH and vice versa for the case of SLS and EDTA modified *C. populnea* fibers. This seemed to follow a definite profile that, diffusivity of water into *C. populnea* fibers is inversely proportional to water sorption rate constant.

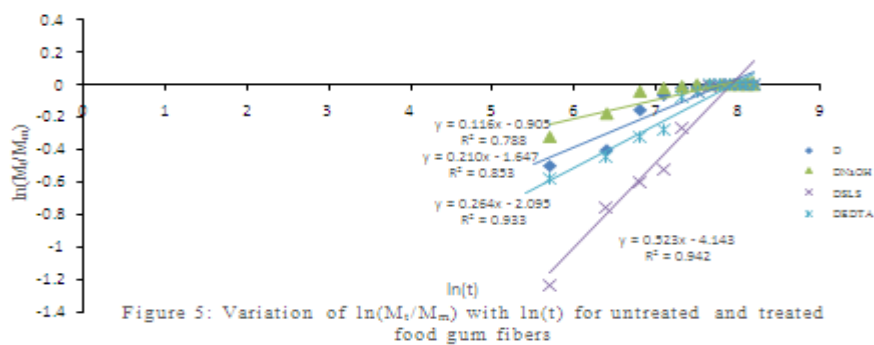


Table-4. Water diffusion behaviour of *C. populnea* fibers using Fickian model.

Fiber sample	C (%)	t (mins)	pH	d_f (μm)	n	k	D_{wf} (mm^2/s)	R^2
CP	0	0	7 ± 0.1	0.11	0.2106	0.19249213	$1.88\text{E}-07$	0.8538
CP_{NaOH}	15	24	12.6 ± 0.2	0.104	0.1164	0.40454189	$5.60\text{E}-08$	0.7887
CP_{SLS}	5.84	20.22	7.6 ± 0.2	0.095	0.5233	0.01587357	$5.92\text{E}-07$	0.9424
CP_{EDTA}	5	150	6.7 ± 0.2	0.1	0.2645	0.12303333	$2.53\text{E}-07$	0.9333

CP represent *C. populnea* fiber, subscript NaOH, SLS and EDTA represent chemical treatment used

4. CONCLUSIONS

Based on the results obtained, it can be drawn that the morphological change and active participation of functional groups of *C. populnea* fibers with modified chemical agents based on SEM and FTIR analysis attributed to removal of hemicellulose, lignin and other impurities, thereby improves tensile properties of *C. populnea* fibers. Hence the chemical treatments improved the aspect ratio with reduced density and water absorption with improved hydrophobicity of *C. populnea* fibers. Moreover, the water absorption kinetics is more fit with Peleg's model compared with power law expression. NaOH and EDTA modified *C. populnea* fibers exhibits Less - Fickian water diffusion behaviour while SLS modified *C. populnea* fibers exhibits Non - Fickian behaviour. Based on NaOH, SLS and EDTA treatments of *C. populnea* fibers, the water diffusion coefficient is inversely proportional to water absorption rate constant.

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