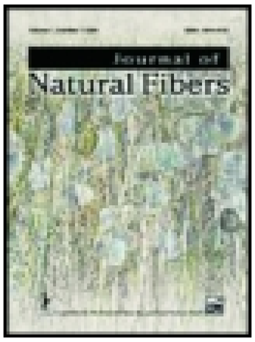

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Babatunde E. Ogunbode[a], Bemgba B. Nyakuma[b], Temitope A. Lawal[a], Jamaludin M. Yatim[c], Aliyu Abdul[d], and Moses I. Ayoosu[e]

[a]Department of Building, School of Environmental Technology, Federal University of Technology, Minna, Niger State, Nigeria; [b]Research Initiative for Sustainable Energy Technologies (RISET), Makurdi, Benue State, Nigeria; [c]Department of Structures and Materials, School of Civil Engineering, Universiti Teknologi Malaysia, Skudai, Johor, Malaysia; [d]Department of Civil Engineering Technology, College of Science And, Technology, Niger State Polytechnic, Zungeru, Niger State, Nigeria; [e]Department of Architecture, Faculty of Built Environment and Surveying, Universiti Teknologi Malaysia, Johor, Skudai, Malaysia

ABSTRACT

–The sustainable utilisation of Kenaf fibres (KF) as environmentally friendly reinforcement in mortar, concrete and polymer composites promotes the concept of a circular economy and sustainable construction. However, the hydrophilic nature of such biofibres adversely affects their structural applications as reinforcement in concrete, mortar and polymer. Therefore, the study examined the effect of alkali treatment (mercerization) on the surface sorption characteristics of KF. The effect of water sorption characteristics on the mechanical properties of surface-modified and untreated KF was also investigated in the study. Scanning electron microscopy was performed to examine the morphology and microstructure of the fibre surface along with the tensile fracture of the swollen/un-swollen fibres. The water absorption studies revealed that mercerization reduced the overall water uptake of KF by 16%, which enhanced the adhesion of the fibre surface. The findings provide the knowledge required by scientists and engineers for determining the water requirements for the design of bio-fibrous concrete composites.

摘要

可持续利用红麻纤维（KF）作为砂浆、混凝土和聚合物复合材料中的环保钢筋, 促进了循环经济和可持续建设的概念. 然而, 这种生物纤维的亲水性对其在混凝土、砂浆和聚合物中的结构应用产生了不利影响. 因此, 研究了碱处理（丝光）对KF表面吸附特性的影响. 研究了表面改性和未改性KF的吸水特性对力学性能的影响. 用扫描电子显微镜观察了纤维表面的形貌和微观结构, 以及膨胀/未膨胀纤维的拉伸断裂. 吸水性研究表明, 丝光处理使KF的总吸水率降低了16%, 从而增强了纤维表面的附着力. 这些发现为科学家和工程师提供了确定生物纤维混凝土复合材料设计需水量所需的知识.

关键词
生物纤维; 生物复合材料; 红麻纤维; 表面改性; 碱处理

KEYWORDS
Biofibres; bio-composite; kenaf fiber; surface modification; alkali treatment

Introduction

Sustainable biofibre utilization such as mortar and concrete composite reinforcement is gaining global traction (Aluko et al. 2020). Biofibres are also used as composite reinforcements in polymers, plastics,

CONTACT Bemgba B. Nyakuma bbnyax1@gmail.com Research Initiative for Sustainable Energy Technologies (RISET), Court 5 North-Bank, Makurdi, Benue State, Nigeria

The effect of water sorption characteristics on the mechanical properties of surface-modified and untreated KF was also investigated in this study. Scanning electron microscopy was performed to examine the morphology and microstructure of the fiber surface along with the tensile fracture of the swollen/un-swollen fibers. The water absorption studies revealed that mercerization reduced the overall water uptake of KF by 16%, which enhanced the adhesion of the fiber surface. The findings provide the knowledge required by scientists and engineers for determining the water requirements for the design of bio-fibrous concrete composites.

packaging, and automobiles (Alagesan, 2020; Bajwa and Bhattacharjee 2016). The most popular sources of biofibres used for composite reinforcements are oil palm empty fruit bunch (Or, Putra, and Selamat 2017), jute (Ali et al. 2020), bamboo (Krishnan, Velmurugan, and Velmurugan 2019), sisal (Pappu, Pickering, and Thakur 2019), flax (Awais et al. 2020), coconut (Bamigboye et al. 2020), hemp (Liao, Zhang, and Tang 2020), Kenaf (Nampoothiri et al. 2020).

Natural fibers provide excellent bonding ability, reinforcement, materials, and engineering properties for producing composites used in construction (Alsubari et al. 2021). Furthermore, the cost-effectiveness and environmental friendliness of biofibres enhances sustainable construction (Rangappa, Siengchin, and Dhakal 2020). Biofibres also reduce the aging rate, which enhances life span, durability, and extended usability of biocomposites (Chang, Mohanty, and Misra 2020). However, biofibres are susceptible to microbial and pest attacks, high water uptake, and limited thermal stability, which hamper application as reinforcement in composites (Aaliya, Sunooj, and Lackner 2021). Likewise, the diversity, heterogeneity, and inconsistent biofibre properties require in-depth characterization (Ali et al. 2020). Therefore, it is critical to examine the properties of low cost, abundant and widely cultivated biofibres such as Kenaf.

Kenaf (*Hibiscus cannabinus* L.) is a tall herbaceous plant of the *Malvaceae* family (Ayadi et al. 2017), widely cultivated in Africa and Asia for fiber, energy, and animal feeds (Lyu et al. 2020). Kenaf fiber (KF) is widely used for composite reinforcement applications due to its favorable mechanical properties (Sapiai et al. 2020b). KF is characterized by complex polymers with large hydroxyl groups that have a high affinity for water absorption, which affects composite performance (Moudood et al. 2019). Likewise, high moisture affects composite wettability, matrix adhesion, and mechanical properties (Halip et al. 2019). The study by Sapiai et al. (2020a) observed that the processing/production of KF composites is prone to structural damage such as fractures (Sapiai et al. 2020a), which prompts the need for structural/chemical modifications (Kumar and Durgam Muralidharan 2020). The outlined challenges of biofibre application in composites can be addressed by surface modifications such as mercerization (alkaline treatment) (Kumar, Muralidharan, and Sathyamurthy 2020). The process chemically cleans and modifies the biofibre surface and structure (Sahu and Gupta 2020) by removing hemicellulose, lignin, surface wax, and oils (Ouarhim et al. 2019). The process also improves fiber density, surface area, crystallinity, strength, thermal stability, and interfacial adhesion (Sahu and Gupta 2020). Hence, alkali treatment could enhance mechano-chemical properties such as biofibre-inorganic matrix bonding with Portland cement during concrete production.

Therefore, this study seeks to examine the effect of alkali treatment on KF biocomposites. The study also investigates the water-sorption mechanism and uptake characteristics of surface-modified KF as sustainable reinforcement for biocomposites. The morphology, microstructure, and tensile properties of surface-modified KFs are also characterized and their effects on water sorption highlighted. It is envisaged that the study will enhance the knowledge on biofibres and biocomposites applications in construction.

Materials and methods

Materials

The KFs were collected from the National Kenaf and Tobacco Board (NKTB-LKTN) in Kelantan, Malaysia. The KFs were obtained as curled long fibers previously subjected to a bacterial retting process. Figure 1 presents the KF used in this study, whereas the physicochemical characteristics are shown in Table 1. KF principally consists of hemicellulose, cellulose, lignin, pectin, and structural water, which are classified as sugar-based polymer components. The surface modification (or mercerization) of the KF was performed using reagent-grade sodium hydroxide (NaOH) purchased from Merck Malaysia.

figure 1. long curled kenaf fiber.

table 1. Physicochemical and mechanical characteristics of kenaf fibers.

fiber property	symbol(unit)	value/range
diameter	(m)	39.7–115.1
density	(g/cm^3)	1.2
cellulose	(%)	31–57
hemicellulose	(%)	21–23
lignin	(%)	4.79–19
pectin	(%)	2
tensile strength	(n/mm^2)	704.00
elastic modulus	e(gpa)	39.77
elongation at yield	(%)	1.77

Alkali treatment (Fiber Mercerization)

The dry KF was subjected to mercerization (alkali treatment) by immersion in 5% NaOH (sodium hydroxide) solution at 25°C for 3 hours. The process parameters and experimental procedure for the mercerization (alkali treatment) of KF in this study were selected based on the optimal conditions for the process deduced and reported by Hassan et al. (2020). The mercerization process promotes the ionization of the hydroxyl group into an alkoxide, which enhances the sorption characteristics of the KF (Sinha, Narang, and Bhattacharya 2017).

Fiber – OH + NaOH Fiber – O^{-} $^{-}$ Na^{+} + H_2O + Surface Impurities

Next, the resulting fibers were washed repeatedly in distilled water to remove the NaOH solution from the surface of the fiber. The fibers were then washed with mildly acidified water to neutralize any remnants of the NaOH on the surface. Finally, the fibers were further washed with distilled water to remove the glacial acetic acid solution before air drying at room temperature.

Water sorption analysis

About 6.0 g of dried KF was used for the water sorption studies. The water absorption study was performed according to the prescribed ASTM standard (ASTM D570-98 2010). The average diameters of the untreated and treated KF were 69.8 µm and 65.4 µm, respectively. For each run, the samples were immersed in distilled water at room temperature. The increase in the weight of the samples was recorded at specific time intervals to examine the kinetics of water absorption by the polymer. This process was continued until equilibrium was attained. The percentage of water absorption, M_t at any time, t, was calculated based on Equation (1) (Najafi and Kordkheili 2011; Zabihzadeh 2010):

$$M_t(\%) = \frac{W_o - W_t}{W_o} \times 100 \tag{1}$$

The terms w_o and w_t denote the initial weight of the sample and the weight of the sample at time, t, respectively. The percentage equilibrium moisture absorption M_∞ was calculated as the average value of several measurements with no appreciable additional absorption.

Water Absorption Kinetic Analysis

The kinetic parameters n and k for sorption by KF were calculated according to Equation (3) (Mathew et al. 1995). The diffusion pattern of the biofibres was fitted to Equation (3) (Suh, Lim, and Park 2000), derived from the transport phenomena Equation (2) (Lee and Jang 1998), to determine the swelling mechanism.

$$\frac{M_t}{M_\infty} = kt^n \tag{2}$$

$$\log\left(\frac{M_t}{M_\infty}\right) = \log(k) + n\log(t) \tag{3}$$

The terms n and k are the constants, whereas M_t and M_∞ denote the water uptake or absorption at time t and the equilibrium or saturation point. The 'n' for case 1 is 0.5, case 2 is $n = 1$, whereas for case 3 it is $0.5 < n < 1$. The absorption coefficients n and k were determined from the linear regression analysis based on the slope and intercept of the plot of log M_t/M_∞ versus time, t, from experimental data. The value of "n" is an indication of the mechanism of sorption (George, Thomas, and Thomas 1999; Katoch, Sharma, and Kundu 2010). The diffusion *(D)* and sorption *(S)* coefficients were calculated from Equations (4) and (5), respectively (Crank 1979; Sombatsompop and Chaochanchaikul 2004; Thwe and Liao 2002).

$$D = \pi\left(\frac{mh}{4M_\infty}\right)^2 \tag{4}$$

Where π is 3.142, m is the initial slope of the plot of M_t versus $t^{1/2}$, and h is the average diameter of the fiber specimens. The sorption coefficient (S) is calculated as (Crank 1979):

$$S = \frac{M_\infty}{M_p} \tag{5}$$

The term M_∞ is the solvent mass uptake at equilibrium and M_p is the dry sample mass. Next, the sorption (S) and diffusion (D) coefficients were used to calculate the permeability coefficient (P) of the samples using Equation (6) (Crank 1979).

$$P = D \times S \tag{6}$$

Tensile analysis

The effects of surface modification on the mechanical properties of the Kenaf fiber were examined through tensile strength, elastic modulus, and breakpoint elongation tests. The outlined tensile tests on the Kenaf fiber were examined on the Universal Testing Machine (Instron LRX, United States) using

25 N load cell capacity at a crosshead rate of 0.05 mm/s. The fibers were mounted on a paperboard with a central window and 50 mm gauge length.

Morphology and Microstructure Analysis

The surface characteristics, cross-sections, morphology, and microstructure of the untreated and treated fibers were captured by scanning electron microscopy (SEM Model: JEOL JSM 6390LV, Japan). For each test, the SEM images of the untreated and treated KF were captured at magnifications of ×500, whereas the fracture was captured at ×1000 magnification. All tests in the study were carried out three times to ensure the accuracy and reliability of the measurements and the averaged results presented in the manuscript.

Results and discussion

Water uptake and sorption mechanism

Figure 2 shows the curves for the water absorption and sorption behavior of untreated and treated KF at 25°C ± 2 in distilled water. As observed, the percentage of the water absorbed by the fibers was plotted against the square root of the soaking time. Each datapoint denotes the mean of three samples. The spongy structure of the KF leads to initial capillary sorption, which results in a large initial uptake in all cases. The untreated and treated KF show the two-step sorption behavior typically exhibited by natural fibers in the case I Fickian sorption behavior (Sreekala and Thomas 2003). Furthermore, our findings showed that alkali treatment greatly reduced the overall water uptake of KF. Hence, the initial uptake due to capillary action was also reduced. The equilibrium percentage of water uptake for the untreated and treated fibers in distilled water at room temperature is presented in Table 2. The

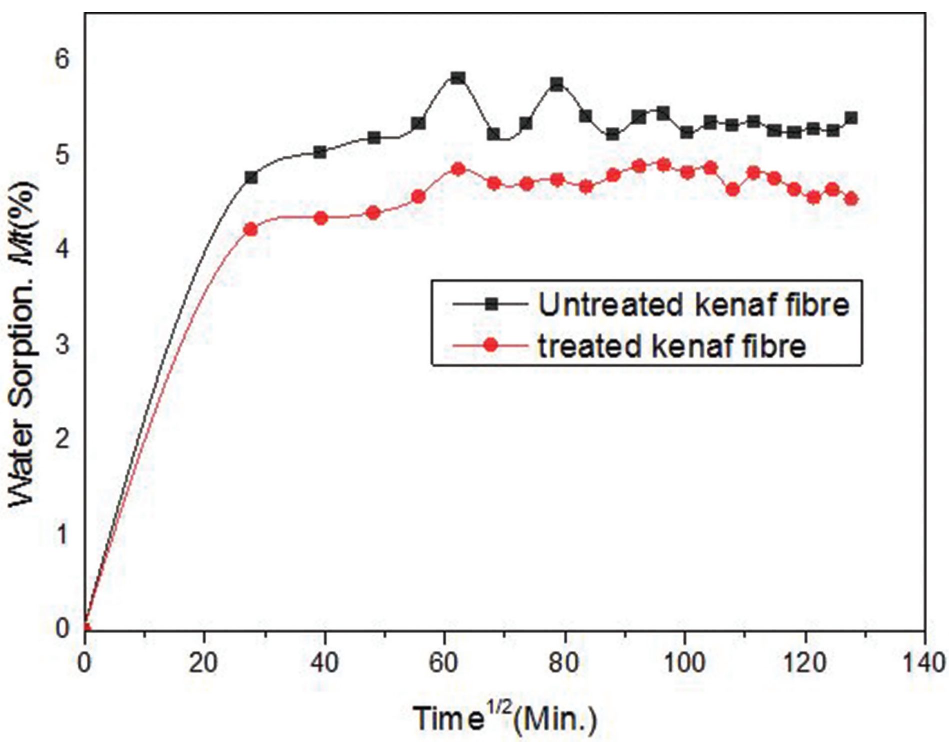

figure 2. water sorption curves for distilled water/treated and untreated kenaf fiber.

table 2. Waste absorption parameters for treated and untreated kenaf fibers.

treatment	water absorption for saturation point $M_t(\%) = \frac{W_o - W_t}{W_o} \times 100$	water uptake reduction (%)
untreated	5.81	0.00
treated	4.90	0.156

decrease in the uptake value observed for the treated fibers may be attributed to the physicochemical changes resulting from the alkali treatment of the KF, as similarly reported by Sreekala and Thomas (2003).

Figure 3 shows the morphology of the KF. As observed, the SEM micrographs show that the untreated KF (Figure 3a) is characterized by a porous structure, whereas the treated fiber (Figure 3b) has a smooth surface structure. Mercerization of natural fibers causes the leaching out of the amorphous layer and waxy cuticle on the fiber surface, which reduces the sorption characteristics and capillary action (Sreekala and Thomas 2003). Sreekala, Kumaran, and Thomas (2002) reported that mercerization also decreased the water uptake capacity of natural fibers. This is because the interstices between the microfibrils of the fiber are blocked by the linked agents, which reduces the accessibility to water. The nature of the chemical bonds formed on the fiber surface after the alkali treatment is illustrated in Figure 4.

Water transport mechanism

Table 3 shows the values of the coefficients of the water transport mechanism of the KF examined in this study. Our findings indicate that alkali treatment significantly influences the diffusion behavior of KF, which is due to surface modification. The mechanism of moisture sorption in biofibres is ascribed to the diffusion of water molecules between the micro-gaps in the polymer chains (Ahmad et al. 2011; Dhakal, Zhang, and Richardson 2007; Saleem et al. 2016). Based on this theory, there are three known cases of diffusion behavior (Ayrilmis and Kaymakci 2013; Kushwaha and Kumar 2009). Case 1 or Fickian diffusion, is the rate of diffusion that is markedly below the polymer segment mobility. The equilibrium inside the polymer is attained rapidly and maintained independently of time (n = 0.5). Case 2 is relaxation control, in which the penetrant mobility is much greater than other relaxation processes. This type of diffusion is characterized by the development of a boundary between the swollen outer part and the inner glassy core of the polymer. The boundary advances at a constant velocity, and the core diminishes in size until an equilibrium penetrant concentration is reached within the polymer (n = 1). Case 3 or non-Fickian diffusion occurs when anomalous diffusion occurs at a comparable juncture between the penetrant mobility and segment relaxation of the polymer. It is also described as the intermediate behavior between cases 1 and 2 diffusions ($0.5 < n < 1$). The three cases of diffusion can be distinguished theoretically by the shape of the sorption curve, which is represented by Equation 3.

Table 3 presents the values of the n and k diffusion coefficients, the sorption coefficient, and permeability of KF (treated/untreated) in distilled water at 25°C. The value of n (0.004 for the treated fiber) is closer to 0.5. Therefore, the water and moisture absorption approaches in the case of diffusion behavior is typically termed less Fickian diffusion. Our findings show that the diffusion and sorption of moisture are higher in the untreated KF (Table 3). Furthermore, a major decrease in the diffusion coefficient, sorption coefficient, and permeability coefficient values was observed after treatment of the fiber. The diffusion coefficient (D) is the most important parameter of Fickian's model, which shows

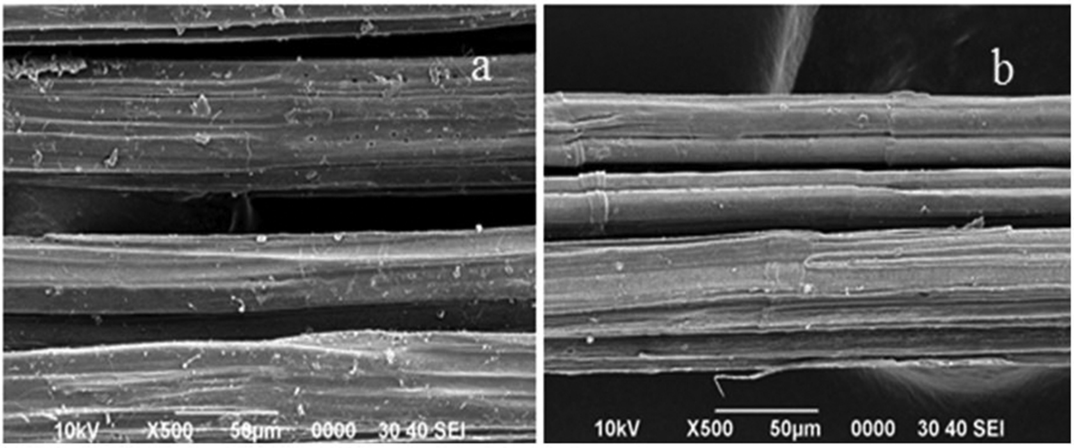

figure 3. sem micrographs of kenaf fiber (a) untreated (b) treated by mercerization.

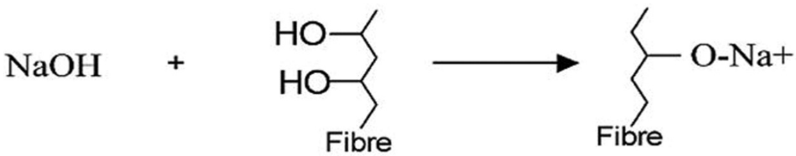

figure 4. chemical surface modification after alkali treatment of kenaf fibers.

the penetration ability of water molecules within the fiber. Typically, the free hydroxyl groups on the surface are modified during alkali treatment, which promotes hydrophobicity along with decreased water uptake and diffusion coefficient of the treated fiber. In contrast, the amorphous waxy material binds the water molecules to the fiber surface, which facilitates the water sorption and the higher diffusion coefficient of the untreated fiber. Typically, moisture is absorbed in the amorphous region of the fiber. The decrease in the diffusion coefficient of the treated fiber highlights the transformations of the amorphous region.

Tensile properties

Untreated kenaf fiber

Table 4 presents the tensile properties of the KF in the swollen and desorbed stages. As observed, the tensile strength of the untreated KF decreased upon sorption up to about 10%. For the elongation at break of the swollen fibers, a noticeable increase was observed compared to the virgin un-soaked fiber. The stress-strain characteristics of the KF in the desorbed and the swollen stage are also described in Figure 5. The swollen and desorbed fibers show a slope change at higher elongations compared to the virgin fibers. Three-stage behavior was observed on the swollen and desorbed fibers. Initially, a crystalline behavior was noticed, then an elastic behavior and finally a crystalline stress-strain behavior. Furthermore, the crystallinity of KF was reduced by the water sorption characteristics of

table 3. water sorption parameters for treated and untreated kenaf fibers.

surface treatment	n	k (g/g min^2)	$d\times 10^{-9}$ (cm^2 s^{-1})	s (g/g)	$p\times 10^{-9}$ (cm^2 s^{-1})
untreated	0.003	0.590	8.200	0.964	7.905
treated	0.004	0.561	7.970	0.813	6.480

the fiber. The tested tensile modulus of the fibers caused a substantial variation in the fiber sorption characteristics. The modulus elongation of the untreated (5.29%) and treated (3.87%) KF decreased upon sorption. In a similar trend, Mannan and Talukder (1997); Sreekala and Thomas (2003) investigated the stress-strain behavior and tensile properties of oil palms and jute fibers. The authors demonstrated that the fibers undergo two-stage elastic elongations due to the amorphous and crystalline phases of the fibers. The findings also highlight the influence of the chemical structure, cellulose content, and microfibrillar angle on the enhancement of the mechanical performance of the fibers. The three-dimensional network of the fiber structure is cemented by lignin and hemicelluloses, while the high elongation of the fibers is due to the amorphous phase. On sorption, water molecules enter into the spaces between the cellulose fibrils and microfibrils. The micropores of the fibers aid the retention of moisture on to the fibers. The presence of water molecules destroys the lignin-cellulose network, thereby decreasing the strength properties. The amorphous phase becomes more active in the swollen stage, which accounts for the high elongation behavior. The structural failure of the fibers on the application of stress is clearly shown in Figure 6. On inspecting the captured image, a fractured surface was observed along with irregular features on the fiber. As the applied stress increases, the weak primary cell wall collapses, and the cohesion of cells occurs, leading to fiber failure.

Treated kenaf fiber

Table 4 shows that alkali treatment decreased the tensile strength of the fibers, which is due to breakage or disintegration of the bond structures of the non-cellulosic materials. Furthermore, the process sharply increases the elongation properties of the treated fiber. The tensile modulus of the fiber also showed enhancement upon the alkali treatment. The enhancement of the KF tensile properties may be ascribed to the modifications of the cellulose region on the fiber surface. The tensile stress-strain behavior of the treated KF in the un-swollen and swollen stage is presented in Figure 7. The slope change of the stress–strain curves after the initial linear sorption shows that the fiber becomes more elastic after treatment. Furthermore, the untreated and treated KF exhibited brittle behavior, which is due to the crystalline nature of the fiber. The alkali treatment enhanced the crystallinity of the fiber due to increased molecular interactions. Typically, fiber crystallinity is dependent on structural regularity, cellular arrangement, and secondary interactions (Sreekala and Thomas 2003). The elongation of the fiber also increased upon alkali treatment as observed in the un-swollen stage. However, the stress of the treated fibers increased linearly without significant slope change during the swollen stage. The elongation at break of the fibers was also considerably higher upon sorption. Finally, the surface modification significantly altered the fibrillar structure, slope changes, and fracture patterns due to the removal of the amorphous components in the fibers.

Conclusion

The present study examined and highlighted the effects of fiber surface modification on the water uptake behavior of Kenaf fibers (KF). Based on the findings, the following conclusions were drawn;

- The treated and untreated KF exhibited a two-step sorption behavior, while the diffusion mechanism of the system was found to be less Fickian.

table 4. Tensile properties of untreated/treated kenaf fibers in virgin and swollen states.

surface treatment	fiber sorption state	tensile strength (n/mm^2)	elongation at break (%)	tensile elastic modulus (n/mm^2)
untreated fiber	un-swollen	751 (105)	1.85 (0.0033)	40594(1800)
	swollen	678 (95)	5.29 (0.0094)	13329 (591)
treated fiber	un-swollen	704(91)	1.77 (0.0033)	39774 (1700)
	swollen	534(69)	3.87 (0.0072)	2108 (531)

note: values in parentheses denotes standard deviation

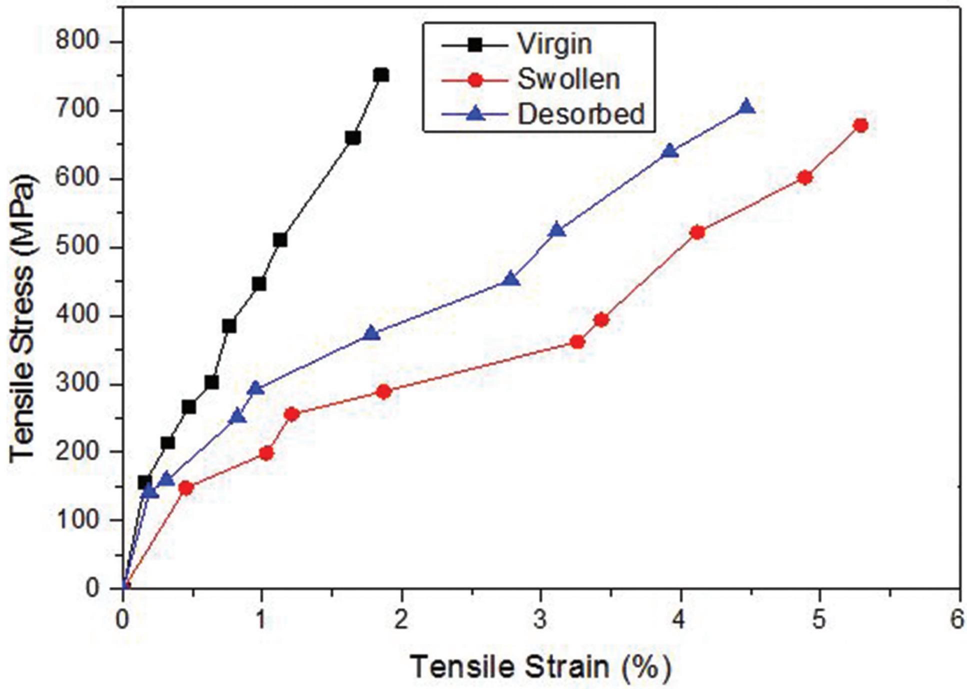

figure 5. stress-strain behavior of untreated kenaf fiber at various modified phases.

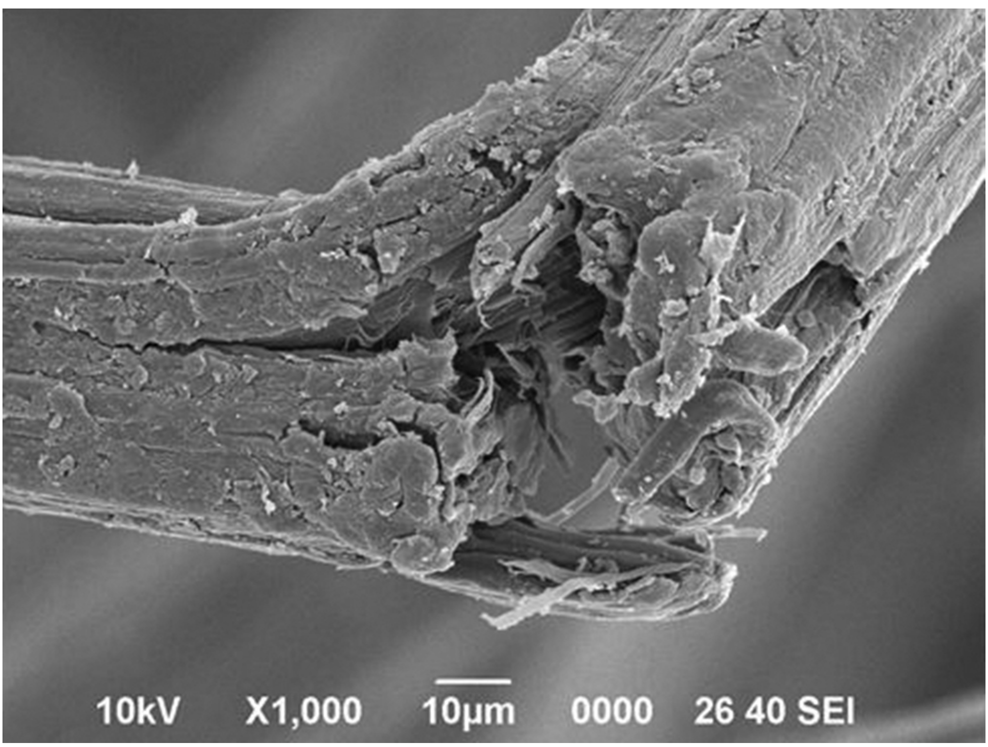

figure 6. sem micrograph of kenaf fiber tensile fracture.

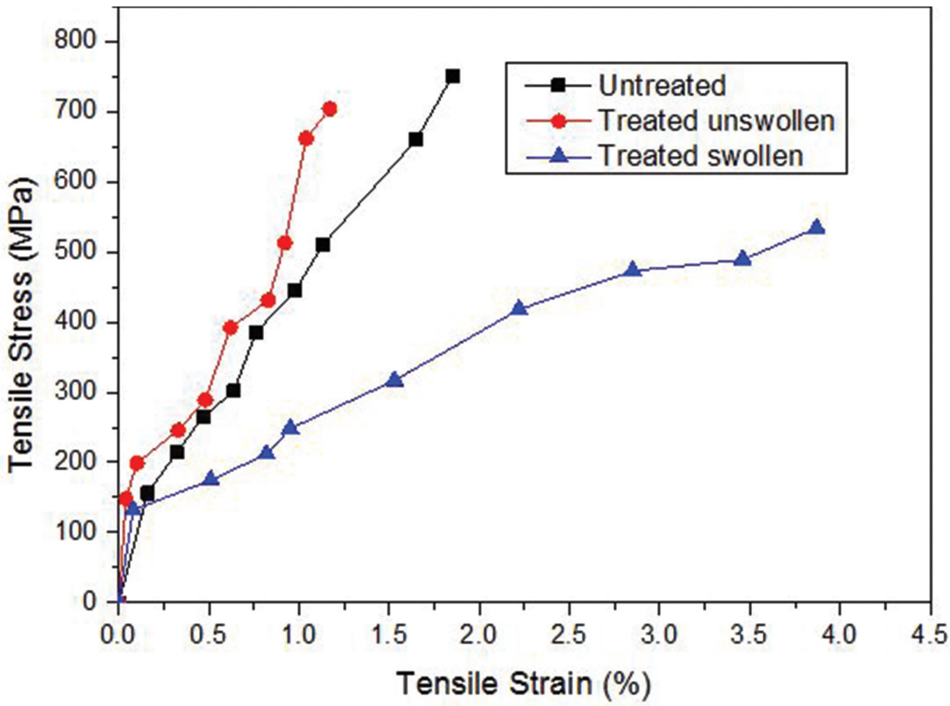

figure 7. stress-strain behavior of treated kenaf fiber in normal and swollen phases.

- The kinetic parameters showed that the diffusion coefficient, sorption coefficient, and permeability coefficient also decreased upon alkali treatment.
- The alkali treatment also reduced the water uptake, tensile strength, and stiffness (swollen stage) of KF, but enhanced the strain to break and tensile modulus due to the physicochemical changes during modifications.
- The alkali treatment reduced the overall water uptake of the KF by ~16%, while the moisture absorption values 5.81% and 4.90% were determined for the untreated and treated KF, respectively.
- The mechanical performance of the KF decreased on water sorption but reversed after desorption showing a modulus decline on sorption/desorption.

Hence, it is envisaged that findings will avail concrete scientists and engineers with critical information on water uptake requirements for producing Kenaf biofibre mortar and/or the design of concrete composites.

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ORCID

Bemgba B. Nyakuma http://orcid.org/0000-0001-5388-7950

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