Effects of Saturated Steam Treatment on the Cell-Wall Mechanics and Moisture Sorption Properties of Kenaf Fibers

Wangwang Yu,[a,b] Bin Xu,[b] Bangrui Fan,[c] Zhang Yaoli,[b] and Xinzhou Wang [b,*]

To improve their hygroscopicity, kenaf fibers were thermally modified by saturated steam at 180 °C for 5, 10, 20, and 40 min. The chemical structure, cellulose crystallinity, cell-wall mechanics, and moisture sorption properties of kenaf fibers were analyzed to evaluate the modification effectiveness. Mass loss and Fourier transform infrared spectroscopy spectra (FTIR) changes indicated that the lignin content and cellulose crystallinity (C_rI) increased with the reduction in hemicellulose after a steam treatment, especially for a long duration. The increased C_rI and relative lignin content resulted in an increased elastic modulus (E_r) and hardness (H) of fiber cell walls after the steam treatment. The reduction in hydrophilic groups and increased stiffness of the cell wall after the steam treatment caused an obvious reduction in the equilibrium moisture content (EMC) at the given relative humidity (RH). It also reduced the moisture increment/decrement and sorption hysteresis during the adsorption and desorption process.

Keywords: Cell wall; Chemical component; Mechanical behavior; Nanoindentation; Wood

Contact information: a: School of Mechanical Engineering, Nanjing Institute of Industry Technology, Nanjing 210023, PR China; b: College of Materials Science and Engineering, Nanjing Forestry University, Nanjing 210037, PR China; c: Zhenghe Ruichang Artware Co., Ltd., Fujian 353600, PR China;
** Corresponding author: wxz_njfu@126.com*

INTRODUCTION

Natural fibers are the best potential substitute for synthetic fibers, *e.g.*, glass and carbon, in reinforced polymer-based composites. These fibers are primarily used for the construction and automotive industry because of their high strength to weight ratio (Singleton *et al*. 2003; Zhai *et al.* 2012; Mahjoub *et al*. 2014). Kenaf (*Hibiscus cannabinus* L.) is an annual crop plant, which has been considered the third-largest renewable source for industrial application after wood and bamboo. Kenaf shows extensive traditional applications such as cordage crop, textiles-basics attire, and fodder. Its various industrial applications include pulp and paper, medium density fiberboard (MDF), and polymer-based composites because of its wide variety of manufacturing processes and satisfactory mechanics (Nieschlag *et al.* 1960; Ali *et al.* 2014; Naili *et al.* 2017). However, some intrinsic properties of kenaf fibers (KFs) limit their use, such as high hygroscopicity and low durability (Akil *et al.* 2011; Faruk *et al.* 2012). The poor interfacial adhesion between the hygroscopic KFs and hydrophobic polymer matrix frequently results in poor bonding properties, moisture uptake, and biological attack of the fiber-based composites (Aziz *et al.* 2005; Datta and Kopczyńska 2015).

Modifications to the matrix, the fibers, or both *via* chemical and physical methods are used to improve the compatibility in KF-reinforced composites. Most of the chemical modification is generally utilized to reduce the hydroxyl groups of natural fibers by grafting

or reacting with another chemical such as acetylation, silane, alkaline, and peroxide treatments. However, it can be cost prohibitive as well as environmentally unfriendly (Huda *et al.* 2008; John *et al.* 2008; Fiore *et al.* 2015). Therefore, thermal treatment has been a focal point within the last few decades due to the simpler treatment process and lower cost. The modifications of the fiber chemical structure at a high temperature under a nitrogen atmosphere or immersion in hot oil can improve the hydrophobic properties and dimensional stability of wood, bamboo, and other natural fibers effectively (Ayrilmis *et al.* 2011; Holt *et al.* 2014; Altgen *et al.* 2016). More recently, modification of wood under high-temperature saturated steam has been introduced to produce a wood material with improved properties in a shorter time. In this method, less cellular damage occurs as wood stays moist during the steaming process (Rautkari *et al.* 2014; Kévin *et al.* 2017). This study attempts to evaluate the effectiveness of KF modification under a high-temperature saturated steam atmosphere.

Measuring the EMC by the gravimetric method in the given RH conditions is suitable to analyze the thermal modification effectiveness on the hygroscopicity of natural fibers (Papadopoulos and Hill 2003; Hill 2006). The dynamic vapor sorption (DVS) technique can provide accurate isotherms over a wide RH range and is very effective in analyzing the moisture absorption characteristics of small-size samples like wood chips and other natural fibers (Hill *et al.* 2009; Xie *et al.* 2011; Hosseinpourpia *et al.* 2017). Furthermore, the chemical, physical, and mechanical properties of thermal treated wood have been intensively analyzed on macro and micro scales to find the interaction between these properties (Kačíková *et al.* 2013; Wang *et al.* 2018). Only limited attempts have been made for the analysis of natural fibers, especially on mechanical characterization. The nanoindentation (NI) technique facilitates the measurement of micro-scale mechanical properties of materials, which are well suited for plant cell walls (Wimmer *et al.* 1997).

The aim of this study was to assess the effectiveness of high-temperature saturated steam modification on kenaf fibers. The changes in chemical structure, cellulose crystallinity, cell-wall mechanics, and sorption properties under different modification conditions were analyzed by using FTIR, X-ray diffraction (XRD), NI, and DVS, respectively, to understand the mechanism of steam treatment on Kenaf fibers.

EXPERIMENTAL

Materials

Kenaf fibers (KFs) with an average length of 60 mm were provided by Jiangsu Yanli Automobile Decorative Parts Stock Co. Ltd. (Jiangyin, China). The fibers were conditioned at 65 ± 3% relative humidity (RH) and a temperature of 20 ± 2 °C until reaching a moisture content of approximately 10%.

Saturated Steam Treatment

The KFs were thermally modified in a pressure tank (RDW-1.5/0.5-5-D, Rongda Bolier Container Co. Ltd., Hangzhou, China) filled with saturated steam at a pressure of 10 atm (180 °C). Temperature of saturated steam was raised to 180 °C within 1 min and then kept constant for 5, 10, 20, and 40 min, respectively. After saturated steam exposure, the steam treated KFs were collected for analysis.

Mass Loss and Morphologic Characterization

The *ML* was measured using the oven-drying method, as follows,

$$ML\ (\%)\ = 100\ \times \frac{(m_0 - m_1)}{m_0} \quad (1)$$

where m_0 and m_1 are the oven-dried mass of the control and treated sample, respectively.

The morphological changes of KFs after treatment were observed by a scanning electron microscope (SEM) (TM 1000, Hitachi Inc., Tokyo, Japan). Specimens were coated with gold and mounted on aluminum substrates with conductive tape prior to the SEM examination.

FTIR and XRD Analysis

The control and treated kenaf flour were prepared for FTIR and XRD analysis. The FTIR spectra were carried out using a Vertex 80v FTIR (Bruker, Karlsruhe, Germany) in the range of 4000 to 600 cm^{-1} with an accumulation of 64 scans and a resolution of 4 cm^{-1}. The crystalline structures were analyzed by Uitima IV X-ray diffractometer (Rigaku Inc., Tokyo, Japan) using CuKα radiation with a scanning speed of 4° min^{-1} at a diffractograms range of 3 to 40° (2θ). The relative crystallinity degree (C_rI) was estimated based on the method proposed by Segal *et al.* (1959) shown in Eq. 2,

$$C_r\mathrm{I}\ (\%)\ = 100\ \times \frac{(I_{002} - I_{am})}{I_{002}} \quad (2)$$

where I_{002} and I_{am} represent the intensity of the 200 crystalline peak and diffraction of the amorphous part, respectively.

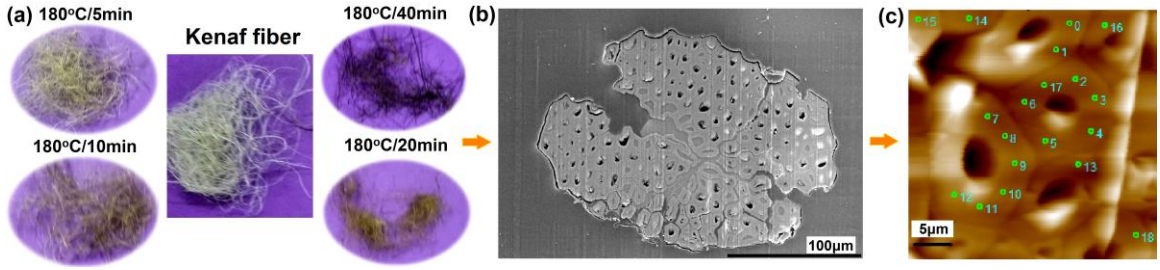

Fig. 1. Kenaf fiber sample preparation for NI test: (a) morphology photographs of kenaf samples; (b) kenaf fiber embedded in polypropylene; (c) NI test locations

Nanoindentation (NI)

The KFs were embedded in polypropylene to prepare the samples for NI testing, and the transverse section of the samples were polished by ultramicrotomy (Leica MZ6, Wetzlar, Germany) until the average roughness of the surface was less than 10 nm, as shown in Fig. 1. The NI test was operated on a Hysitron TriboIndenter (Eden Prairie, MN, USA) after all samples were equilibrated at 20 °C and 50 ± 3% RH in the chamber. Testing was conducted on the secondary cell wall layer of kenaf fibers with the load function including loading, holding at the peak load of 400 μN, and unloading for 5 s. Approximately 30 indents were analyzed on six cell walls of KFs at different treatment conditions. According to the method introduced by Oliver and Pharr (1992), the hardness (H) and reduced elastic modulus (E_r) were obtained by Eqs. 3 and 4, respectively,

$$H = \frac{P_{max}}{A} \quad (3)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \quad (4)$$

where P_{max} is the peak load, A is the projected contact area of the tips at peak load, S is the initial unloading stiffness, and β is a correction factor correlated to indenter geometry (β = 1.034).

Determination of Dynamic Water Vapor Sorption

The moisture sorption properties of KFs were analyzed with a DVS Intrinsic device (Surface Measurement Systems Inc., London, UK) at 25 °C. Approximately 5 mg of 20-mesh flour was ground from the control, and treated KFs were used for each measurement. As shown in Fig. 2, the relative humidity (RH) of the test atmosphere ranged from 0 to 90% in the step of 10% and then decreased from 90 to 0% RH in a reverse order. According to the method introduced by Hosseinpourpia *et al.* (2016), the target RH constant was maintained once the mass change of the test sample was less than 0.002% min^{-1}. The sample mass data was collected every 60 s at each given RH step to analyze the effects of steam treatment on the hygroscopicity of KFs.

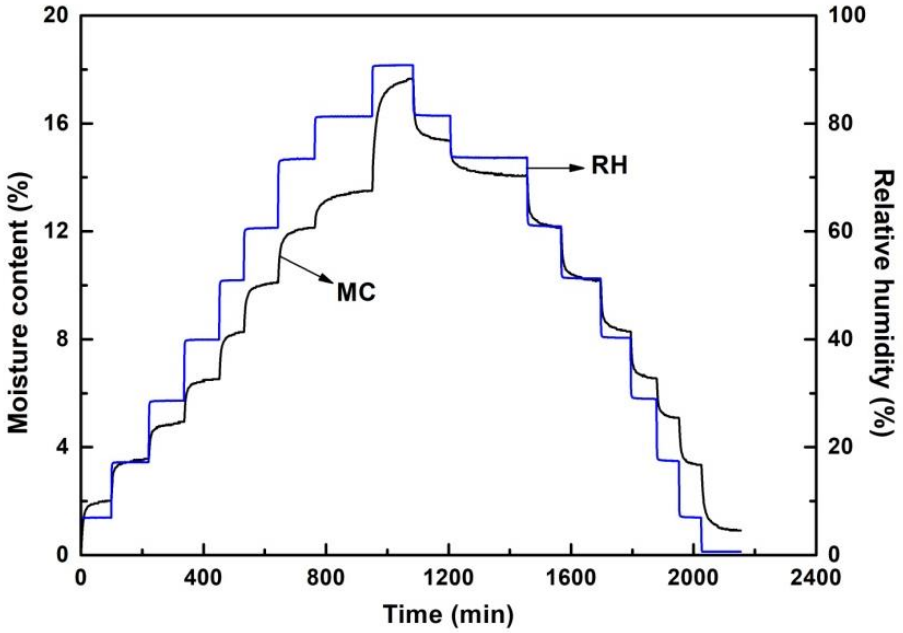

Fig. 2. Plot showing data from a typical DVS isotherm run for kenaf fiber

RESULTS AND DISCUSSION

Mass Loss and Surface morphology

The mass loss of kenaf fibers increased from 4.9% to 15.3% as the steaming time increased at 180 °C (Fig. 3). The obvious mass loss is due to hemicellulose degradation as the temperature increased up to 180 °C (Hakkou *et al.* 2006; Özlemet *et al.* 2017). The surface microstructural changes of KFs after steam treatment observed by SEM showed that the untreated KFs represent the bundle with a relatively smoother and more compact surface in comparison to the treated fibers, as shown in Fig. 3 (a-e). The treated fibers looked jagged and the surface was rough and porous. Specifically, a large amount of single

fiber splitting phenomena occurred after steaming at 180 °C for 40 min, which may be attributed to the degradation of wood polymers.

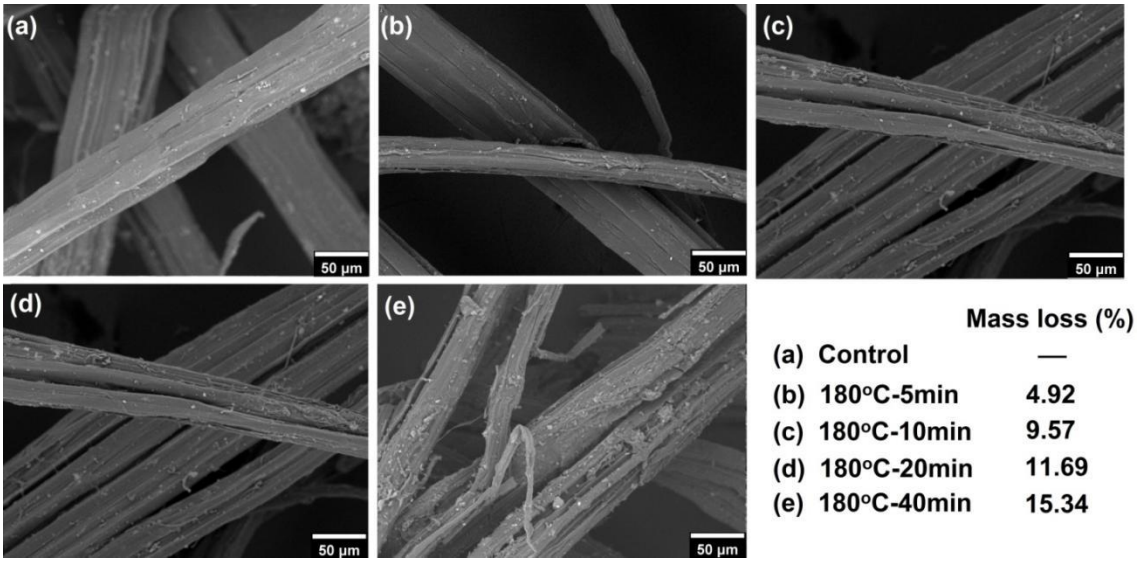

Fig. 3. Mass loss and morphology photographs of the control and treated kenaf fibers

FTIR Analysis

The chemical changes of KFs during steam treatment were confirmed by the FTIR analysis. As shown in Fig. 4, some modifications occurred on the chemical structure of treated KFs compared with the control sample.

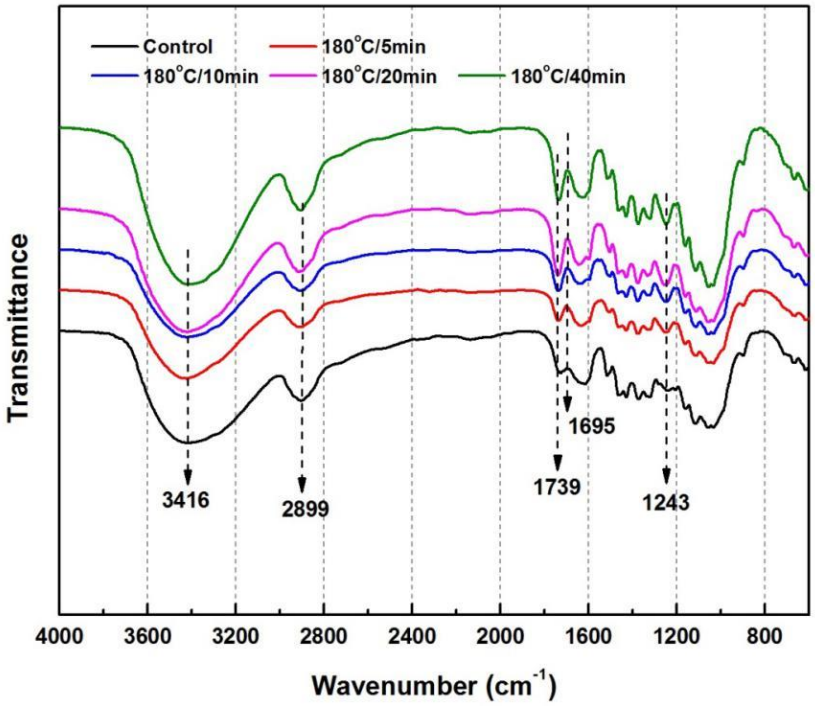

Fig. 4. FTIR spectra of the control and treated kenaf fibers

The peak intensity at 1733 cm^{-1} assigned to the C=O stretching vibrations of the acetyl groups in hemicellulose decreased with the increased steaming time, especially after treatments at 180 °C for 20 and 40 min, indicating the breaking of acetyl groups in hemicelluloses (Gèrardin *et al.* 2007; Popescu *et al*. 2011; Esteves *et al*. 2013). The peak at 1695 cm^{-1} and 1243 cm^{-1} refers to the C=C stretching linked to the aromatic skeleton and alkyl-aryl-ether bonds in the lignin. They presented an intensity increment with the increased treatment time. This may indicate an increase of the relative lignin content in fiber cell walls due to the loss of polysaccharide material and the potential "pseudolignin", which could be formed by polymerization of polysaccharide degradation products (Guo *et al*. 2017).

XRD Analysis

The X-ray diffractograms of the control and steam-treated KFs in Fig. 5 show that the intensity of the (002) reflection after steam treatment increased gradually. The relative crystallinity degree (C_rI) of the control and treated samples was calculated and confirmed that steam treated samples have higher C_rI compared with the control. The C_rI increased from 60.6% for the control sample to the maximum values of 73.9% for the samples treated at 180 °C for 40 min, a total increase of approximately 22%. Among woody components, only cellulose is crystalline, while hemicellulose and lignin are non-crystalline (Poletto *et al*. 2012). An increase in C_rI after steam treatment is probably due to the degradation of hemicellulose. The rearrangement of the ordered crystalline structure and the crystallization in quasicrystalline ("paracrystalline") regions of cellulose may be another reason for the increase in C_rI after steam treatment (Okon *et al.* 2017).

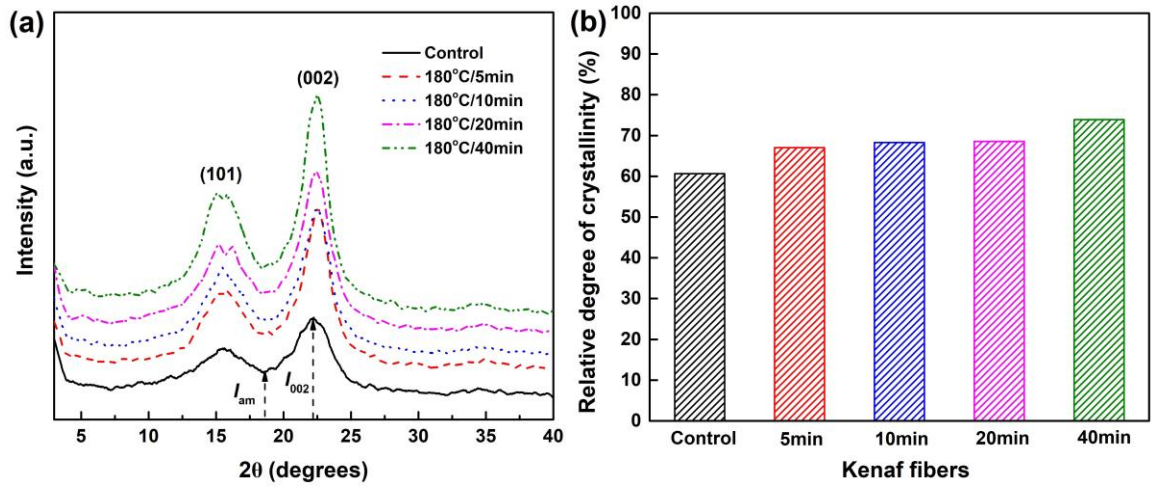

Fig. 5. XRD spectra of the control and treated kenaf fibers

Cell Wall Mechanics

Figure 6a shows the typical indentation load-depth curves of the fiber cell walls. The values of elastic modulus, hardness of the control, and steam treated fiber cell walls are summarized in Fig. 6b. Steam treatment had a positive effect on the cell wall mechanics of KFs. The mechanical properties of wood cell walls are prone to be affected by the chemical composition and the crystalline structure of the cell wall (Yu *et al.* 2011; Li *et al.* 2013).

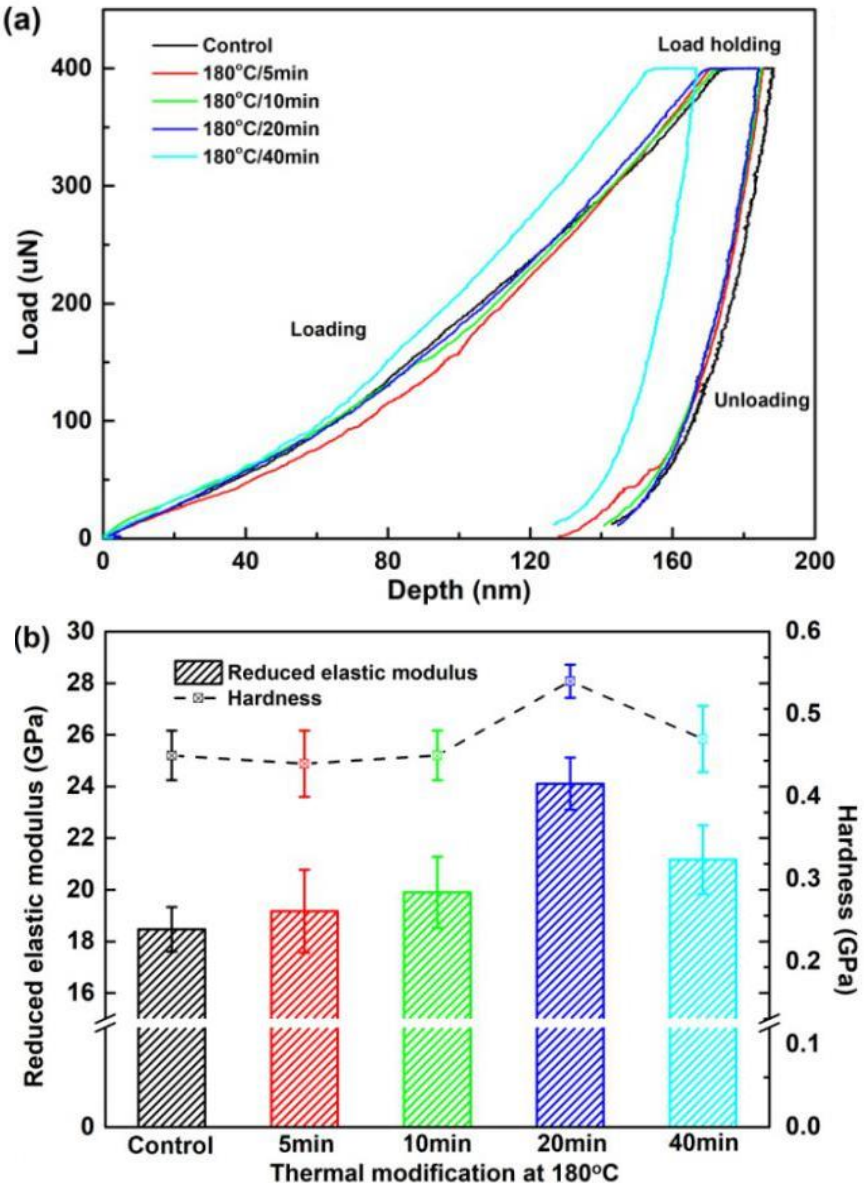

Fig. 6. Cell wall mechanics analysis by nanoindentation. (a) Typical indentation load-depth curves of cell walls; (b) reduced elastic modulus and hardness of kenaf fibers

The cell wall treated at 180 °C for 5 and 10 min, exhibited a slight increase on E_r and H, while the results of samples treated for 20 min indicated a significant increase in the stiffness. For instance, the E_r and H values of the cell wall treated at 180 °C for 20 min increased by approximately 30% and 20% when compared to the control. This reaction can be interpreted as a result of the increase of the relative lignin content and the possible cross-linking reactions with furfural induced by thermal degradation of hemicelluloses (Gindl and Schoberl 2004; Guo *et al.* 2017). Furthermore, the increased crystallinity index of cellulose may be another potent factor. However, a decreasing trend occurred on the cell wall treated at 180 °C for long periods, *e.g.*, the E_r and H of the fiber cell wall after steaming for 40 min decreased by approximately 12% and 13%, respectively. The results obtained by ML, SEM, FTIR, and XRD analysis indicated that the severe treatment at 180 °C for 40 min not only can induce the degradation of amorphous materials such as hemicelluloses

but can also damage the cellulose, which mainly dominates the elastic modulus of the cell wall (Wang *et al.* 2016).

Water Vapor Sorption Properties

Figure 7 shows that the equilibrium moisture content (EMC) of the control and treated KFs increased with the increasing RH, which can be attributed to more hydroxyl groups and the new sorption sites created accompanied with the swelling of cell wall polymers (Engelund *et al.* 2013; Hosseinpourpia *et al.* 2017). Compared with the control, the EMC of steam treated fibers decreased at each step of RH during both the adsorption and desorption as the increasing mass loss. The EMC of fibers treated at 180 °C for 5, 10, 20, and 40 min decreased by 9, 18, 30, and 32% when compared to the EMC of the control at an RH of 90%, respectively. The reduced EMC might be attributed to the reduction in the hemicelluloses with numerous free hydroxyl groups after the steam treatment.

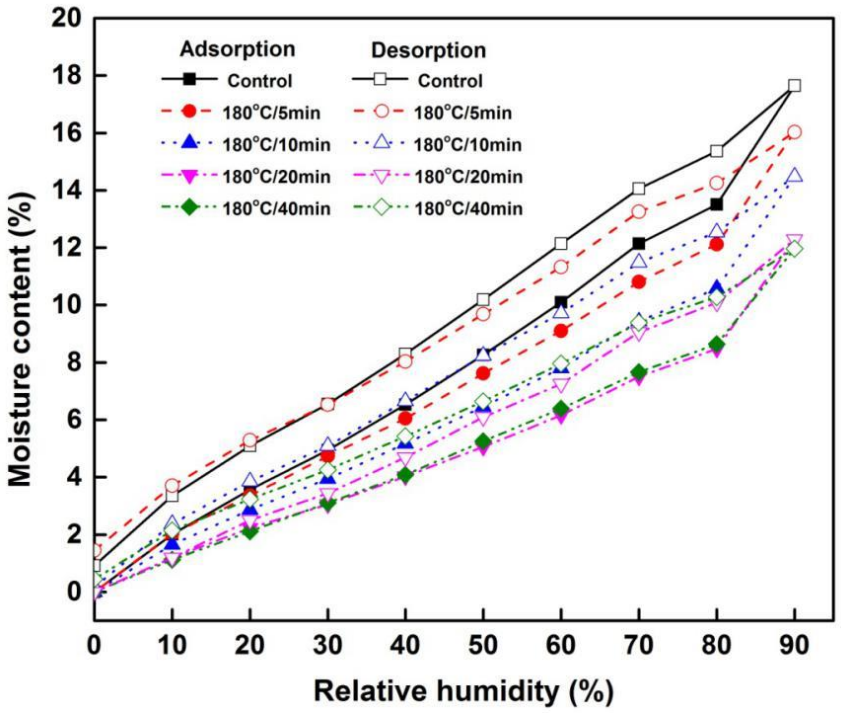

Fig. 7. Moisture adsorption and desorption behavior of kenaf fibers

The moisture increments of the control and treated KFs decreased below 20% RH (Fig. 8), which agreed with Skaar (1988) that the isotherm process is dominated by monolayer water adsorption, which involves bonding with the hydroxyl groups at a low RH. When the RH increased from 30% to 90%, the moisture increments gradually increased, especially when the RH was above 70%. The moisture increment increase resulted from the increased swelling of the cell wall matrix due to the plasticization effect at the higher RH range (Engelund *et al.* 2013; Hosseinpourpia *et al.* 2016). During the desorption process, the moisture content of fibers decreased quickly at first (from 90 to approximately 20% RH) and slowly toward the end (from 20 to 10% RH). The increase of moisture desorption decrement at the RH range below 20% may be attributed to the increased stiffness as the cell wall becomes drier. Compared to the control, both the moisture increments and decrement of modified kenaf fibers decreased during the entire

RH range, and these reductions were more pronounced. The reduced flexibility of the cell wall polymers after steaming above 20 min may make a positive contribution to this phenomenon (Hill *et al.* 2012).

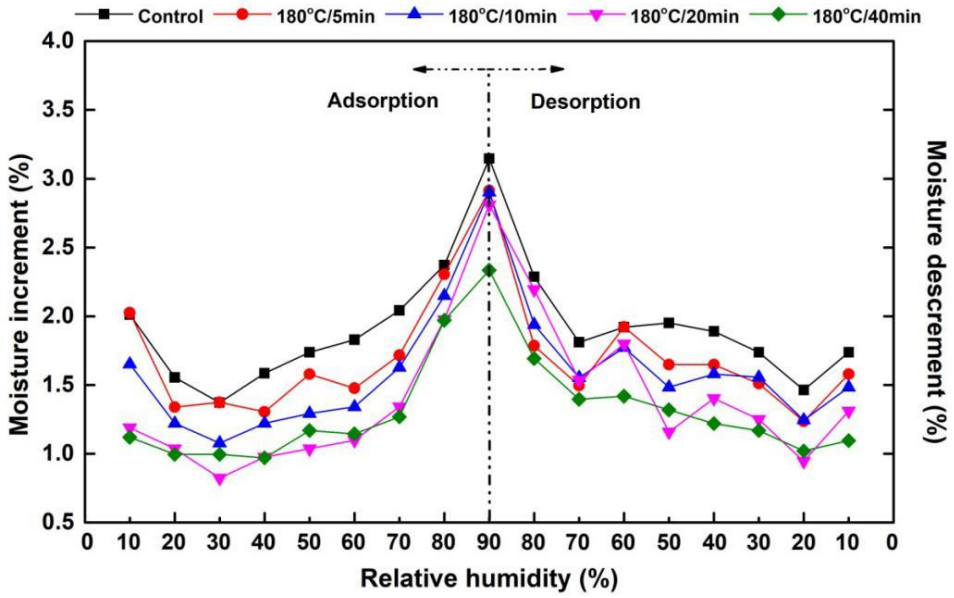

Fig. 8. Moisture content increment and decrement of kenaf fibers at given RH step.

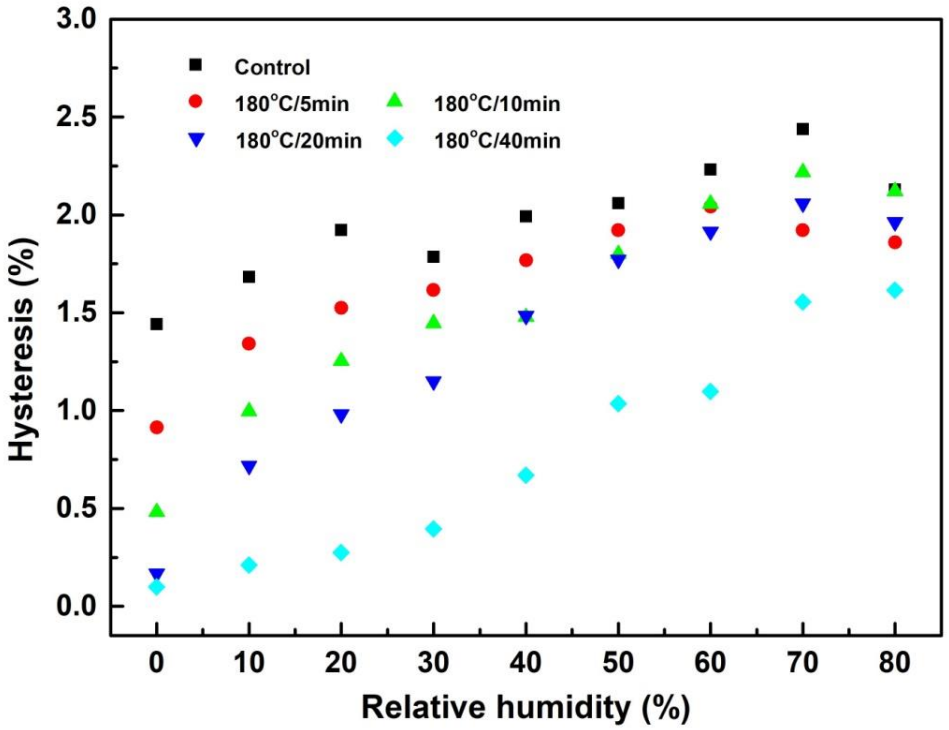

Fig. 9. Hysteresis of the control and treated kenaf fibers during the sorption process

The sorption hysteresis for the control and treated samples at the RH range of 0 to 90% is given in Fig. 9. As expected, steam treatment resulted in a decrease in hysteresis

for Kenaf fibers. Sorption hysteresis generally occurs in a glassy matrix during the sorption process and the sorption hysteresis processes are controlled by the molecular relaxation phenomena (Hill *et al.* 2012; Zhan *et al.* 2019). Steam modification increased the stiffness of the fiber cell wall and could thus increase the time delay due to cell wall polymer matrix relaxation. When compared to the control, the reduction in hysteresis of the treated fibers can be attributed to the reduced swelling degree of the cell wall with a lower moisture content.

CONCLUSIONS

1. Steam treatment of kenaf fibers at 180 °C caused an evident mass loss mainly due to the hemicellulose degradation, leading to the reduction of hydroxyl groups. Consequently, the EMC of treated fibers decreases at the RH range from 0 to 90%.
2. The condensation of lignin accompanied with the degradation of hemicelluloses and the increased cellulose crystallinity during steam treatment at 180 °C for 20 min made the greatest positive contribution to the cell wall mechanics of kenaf fibers.
3. The moisture increment/decrement and sorption hysteresis during the adsorption and desorption processes decreased after steam treatment because of the lower EMC and the improved stiffness of the fiber cell wall.
4. A severe steam treatment condition (*e*180 °C/40 min) has the potential to reduce the cell wall mechanics of kenaf fibers due to the possible degradation of the crystalline materials.

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