

EFFECTS OF HEAT AND ALKALI TREATMENTS ON MECHANICAL PROPERTIES OF KENAF FIBERS

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Abstract

To improve mechanical properties of natural fiber in preparation of composites, heat and alkali treatments to kenaf fibers were performed and effects on mechanical properties were investigated. Compared with other heat treatment temperatures, the tensile strength of kenaf fiber at 140 °C exhibited maximum value, which could be attributed to the increase of crystallinity index of fibers after heat treatment. The result showed the fracture strain of the alkali treated fibers improved. Alkali treatment caused the elimination of hemicellulose and some impurities, and then led to the decreased crystallinity and the loosely bound structure of the fiber. This results in higher elongation at break of the treated fiber.

1 Introduction

Because of the demands of renew and recycle materials to solve the ever-expanding problem of energy shortage and the increasing environmental consciousness, there has an increasing interest concerning biodegradable materials with the focus on biomass renewable resource. Natural fibers, such as ramie, jute, abaca and bagasse [1-4], have been used as reinforcement in composites owing to their low cost, acceptable specific strength and modulus and biodegradability. Especially, kenaf fiber-reinforced composites have been applied for car parts in some automobile manufactures.

However, the drawbacks in natural fibers like high moisture absorption lead to their low mechanical properties and poor fiber-matrix adhesion in composites, and limit their applications. Hence natural fibers need to be treated to change their structural and surface properties by physical and chemical methods, and thereby improve the mechanical properties in their applications. Heat

treatment is considered to increase the crystallinity index (CrI) of the fiber to enhance the fiber strength, and alkali treatment might modify the fiber structure to obtain better mechanical properties.

The objective of this paper is thus to study the effects of heat and alkali treatments to kenaf fiber on the mechanical properties. The fibers were treated by various heat temperatures and different concentrations alkali solution, and then the tensile properties were investigated. To clarify the change of the mechanical properties, the fiber CrI and infrared spectra before and after treatment were examined by X-ray diffraction (XRD) and Fourier transform infrared spectra (FRIR), respectively.

2 Experimental

2.1 Materials

Kenaf fibers, *Hibiscus cannabinus* L., were obtained from bast of the plants. The fiber length depends primarily on the location and is generally longer than one meter. The average diameter of the fiber was about 50-80 μm in this experiment. Similar to other natural fibers, the major constituents of kenaf fibers are cellular, hemicellulose and lignin. Mechanical properties of the fibers mainly depend on the amount of cellulose as well as the microfibrillar angle.

2.2 Heat and alkali treatments

Heat treatment is a physical method, which does not change the chemical composition of the fibers. Kenaf fibers were treated in the vacuum heater at different temperatures 130, 140, 160, 190 and 220 °C for 10 h, respectively. The mechanical properties and the crystallinity index between the untreated and treated fibers were investigated.

Alkali treatment is a chemical method, which can change the constituents of fibers. Kenaf fibers were soaked in 5, 10, 15wt% NaOH solution at 25 °C

for 2 h, maintaining a liquor ratio of 20:1. The fibers were washed several times with water to remove any alkali solution sticking to the fibers surface, neutralized with dilute acetic acid and then washed again with water. Finally, the result fibers were dried at 70°C for 72 h before the examination of the tensile test.

2.3 Measurements

Tensile tests of single kenaf fibers were performed according to the standard method used for carbon fiber determination of tensile properties of the single filament specimens (ISO 11566). The tensile strength of 35 specimens of single fibers was measured for each condition.

TG analysis of the kenaf fiber was carried out using a TG-DTA 200s (Mac Science Co., Japan) under air atmosphere. The temperature profile was from 30 to 450°C at a heating rate of 10 °C/min. The amount of sample used was approximately 6 mg.

The infrared spectra of the fibers before and after alkali treatment were measured with a JIR-6500 (Jeol Dataum Ltd., Japan). Powdered fiber pelletized with potassium bromide was used for recording the spectra. Transmittance was measured over a range from 4000 to 500 cm^{-1} .

In order to assess the influence of the treatment on the fiber crystallinity, XRD analysis was applied using a Rigaku Rint 2500 diffractometer. The equatorial diffraction patterns (2θ) were recorded from 5 to 35° using Cu-K α radiation at 40 KV and 20 mA. The CrI was calculated according to the Segal empirical method [5].

$$\text{CrI} = \frac{I_{002} - I_{\text{am}}}{I_{002}} \times 100\%$$

where I_{002} is the maximum intensity of the 002 lattice reflection of the cellulose crystallographic form (I) at $2\theta=22^\circ$ and I_{am} is the intensity of diffraction of the amorphous at $2\theta=18^\circ$.

3. Results and discussion

3.1. Thermogravimetric analysis of kenaf fiber

The thermal stability of the kenaf fibers was studied to assess the possibility of their being used as reinforcement. Fig. 1 shows the TG curve of the kenaf fibers. Similar to other natural fibers, the kenaf fibers showed three stages of weight loss. The first stage was due to water evaporation and continued up to about 120 °C. The second stage started at about 218 °C, this stage resulted from the

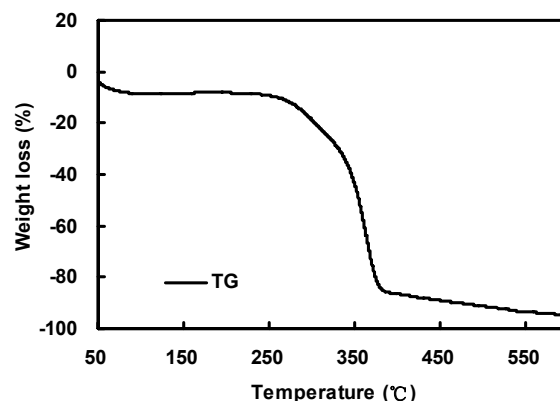


Fig. 1. TG curve of the kenaf fiber.

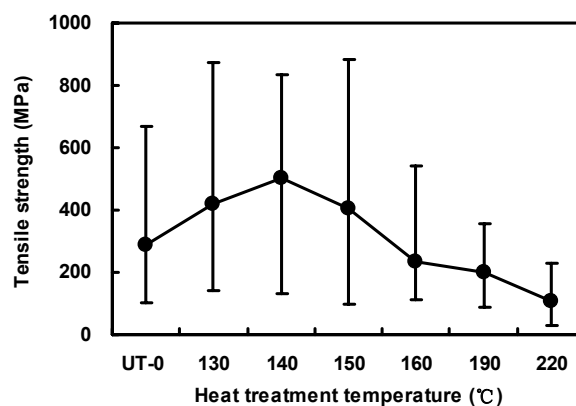


Fig. 2. Tensile strength of untreated and treated fibers. (UT: Untreated)

generation of noncombustible gases such as CO_2 and formic and acetic acids. The third stage began at about 330 °C. This occurred because of the pyrolysis and generation of combustible gases. TG analysis indicates that the kenaf fibers were thermally stable below 218 °C and that, as such, the fibers could be effectively used as reinforcement when the molding temperature was set under this temperature.

3.2 Effect of heat treatment on tensile strength of kenaf fiber

Fig. 2 shows the tensile strength of the fibers before and after heat treatment. Compared with the untreated fiber, the tensile strength increased at heat temperature 130 and 140 °C, while decreased over 140 °C. The maximum value was found at 140 °C. With increasing heat treatment temperature, the appearance color of the fibers became brown and black gradually by the observation of naked eye, and moreover the fibers became easily brittle and broken. This is considered the thermal degradation of the fiber could occur in light of the TG analysis, especially at 220 °C.

Table 1. CrI of untreated and heat treated kenaf fiber

	Untreated	Heat treatment temperature (°C)				
		130	140	160	190	220
CrI of kenaf fiber (%)	48.7	51.2	55.8	49.0	43.0	32.3

Table 2. Tensile properties of untreated and alkali treated kenaf fiber

Kenaf fiber	Fiber diameter (mm)	Tensile strength (MPa)	Young's modulus (GPa)	Fracture strain (%)
Untreated	71.9	289	25.5	1.45
5% alkali treated	56.2	455	26.4	1.41
10% alkali treated	52.9	261	25.6	2.20
15% alkali treated	58.6	213	24.2	4.70

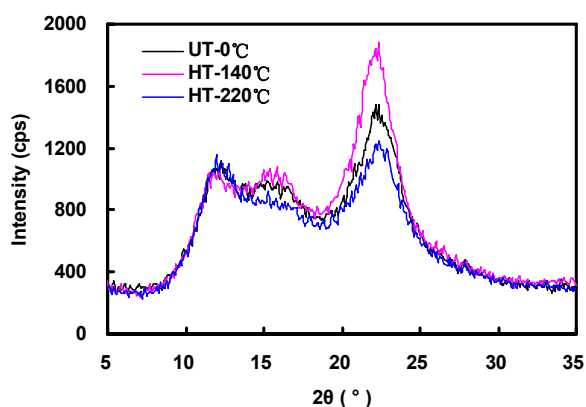


Fig. 3. X-ray diffraction patterns of untreated and treated fibers. (HT: Heat Treatment)

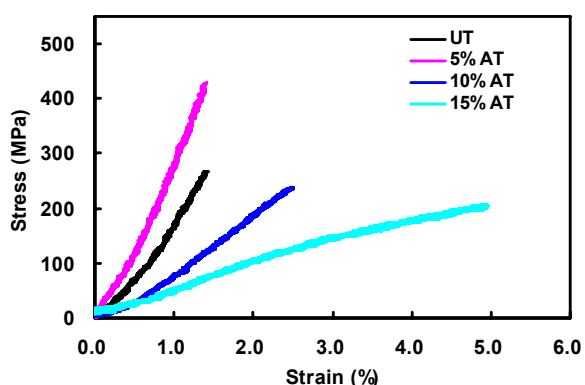


Fig. 4. Typical stress-strain diagrams of untreated and alkali treated fiber. (AT: Alkali Treatment)

To ascertain the effect of heat treatment on tensile property of the fiber, the XRD analyses of the fibers were carried out. The result is given in Fig. 3 and the CrI was calculated according to the Segal empirical method described in the experimental section as presented in Table 1. As seen from Fig. 3, kenaf fiber exhibited a typical cellulose I pattern, a

well defended peak at $2\theta=22^\circ$. The reflections peak at 22° corresponds to the 002 crystallographic plane of the cellulose I lattice. Maximum value of the intensity is clearly observed in the fiber treated at 140°C . Table 1 presents the similar result and reveals that the CrI of the treated fiber at 140°C was higher than that of at other heat temperatures and the untreated fiber. The increase of the CrI indicates the improvement in the cellulose structure and finally contributes to enhancing the tensile strength of the treated fiber. However, the CrI of the fibers treated at higher temperature, 190 and 220°C , the fiber CrI decreased in comparison with that of the untreated. This lowered the tensile strength of the fiber, along with the thermal degradation of the fiber.

3.3 Effect of alkali treatment on tensile properties of kenaf fiber

The fibers were treated with different concentrations alkali solution and the tensile properties of untreated and treated fiber were investigated. The result is given in Table 2. After alkali treatment, all the diameter of the treated fiber decreased. In the case of 10 and 15% NaOH solution, the tensile strength decreased, Young's modulus changed slightly and fracture strain increased drastically, which were two to three times higher than that of the untreated. This is considered the modification of the fiber structure occurred due to alkali treatment, and finally led to influencing the tensile properties of the treated fiber. However, the tensile strength increased, young's modulus and fracture strain almost did not vary, when the fiber treated with 5% NaOH solution. Fig. 4 displays typical stress-strain diagrams of untreated and the treated kenaf fibers. Similar to the mentioned above, alkali treatment brings on a dramatic increase in

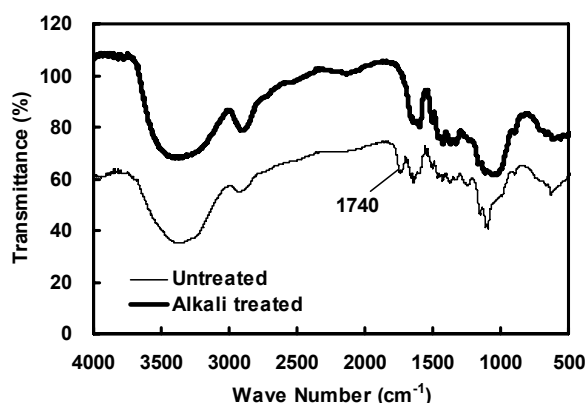


Fig. 5. FTIR spectra of the untreated and treated kenaf fiber.

fracture strain of the treated fiber. Except the fiber treated with 5% solution, the slopes of the stress-strain relation in the treated fibers are obviously lowered and their behaviors were not linear. This is attributed to the elimination of hemicellulose after alkali treatment.

FTIR spectra analyses of the untreated and 10% alkali treated fibers are shown in Fig. 5. Although the occurrence of majority peaks did not change, it is noted that the absorption peak around 1740 cm^{-1} , corresponding to the C=O stretching of hemicellulose in the untreated fiber, was almost missing in the spectrum of the alkali treated fiber, indicating the elimination of hemicellulose occurred by alkali treatment. Furthermore, there was no change in the peak around 1515 cm^{-1} , a result of aromatic skeletal ring vibration of lignin, which shows no structural change of the lignin component in the kenaf fibers after alkali treatment.

Kenaf fibers mainly consists of crystalline cellulose surrounded and cemented together with hemicellulose, lignin, waxy materials and other impurities, and can be considered as a bundle of microfibrils. The arrangement of microfibrils is a determining factor of the mechanical properties in the fiber. Microfibrils are made up of long cellulose chains and hemicellulose [6].

The FTIR analyses clarified the elimination of hemicellulose by alkali treatment, which results in the untreated fiber bundle broken down into smaller ones. This phenomenon of fibrillation is the reason why the fiber diameter of the treated fiber decreased. During alkali treatment, cellulose molecular chains in a microfibril also lose their crystalline structure partly, and the alignment of the microfibrils is destroyed and the overall crystallinity reduces. The decreased crystallinity and the disorder of the microfibrils alignment then decrease the stiffness of

the fiber, and thus improve the fracture strain of the treated fiber. In addition, alkali treatment removes the impurities, waxy materials, lignin as well as hemicellulose, which is responsible for an easy deformation of the cellular networks. The extensive hydrogen bonding network may be broken and the ordered structural arrangement of cellulose may loose. Consequently, higher elongation at break is observed for the alkali treated fiber because of the decreased crystallinity in cellulose and the loosely bound structure of the microfibrils within the fiber. Same observation on ramie fiber has been reported by Goda et al [2].

4. Conclusions

For purpose of improving mechanical properties of natural fiber in preparation of composites, heat and alkali treatment to kenaf fibers were performed and effects on tensile properties were investigated. The obtained results are:

- 1) The tensile strength of the kenaf fiber treated at heat temperature $140\text{ }^{\circ}\text{C}$ showed the highest value.
- 2) The fracture strain of the fiber treated with 10 and 15% alkali solution improved.

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