

Effect of alkali treatment on microstructure and mechanical properties of individual bamboo fibers

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Abstract The aim of this study was to study the microstructure and mechanical properties of individual bamboo fibers after alkali treatment. The individual bamboo fibers were treated by sodium hydroxide (NaOH) solution with varying concentration (6, 8, 10, 15 and 25%) followed by rinsing and freeze-drying treatments. The alkali treated individual bamboo fibers were subsequently characterized by scanning electron microscopy, atomic force microscopy, Fourier transform infrared spectroscopy, X-ray diffraction, confocal laser scanning microscopy and tensile strength tests. The results indicated that the alkali treatment

resulted in more wrinkles and pores on the surface of bamboo fibers. Microfibril aggregates treated by 15 and 25% NaOH solution changed from a randomly interwoven structure to a granular structure. Cellulose I was transformed to cellulose II after 15 and 25% NaOH solution treatment. The alkali treatment reduced the diameter, lumen and cross sectional area of fiber, leading to the cracks in cell wall. The tensile strength and modulus of elasticity (MOE) of individual bamboo fibers decreased with alkali treatment. NaOH concentration almost did not affect the tensile strength but influenced the MOE significantly. The elongation at break of treated individual fiber increased significantly. When compared to untreated individual fibers, the elongation at break of the fiber was increased by 232 and 221% after 15 and 25% NaOH treatments, respectively. Besides, increasing alkali concentration caused the change of the fibers from brittleness to ductility, indicating that alkali treated bamboo fibers have a promising application in textile.

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Introduction

In recent decades, there has been an increasing effort to exploit the renewable raw materials to produce

sustainable products so as to reduce the use of fossil fuels, and to decrease the environmental pollution. The natural fibers as raw materials became a major alternative to synthetic fibers since they are widely abundant in nature. The natural fibers have great potential applications in composites (Lee and Wang 2006; Okubo et al. 2004), pulp (Yang et al. 2008) and textile (Liu et al. 2011; Nayak and Mishra 2016). There have been lots of researches studied on the mechanical properties of natural fiber, e.g. wood (Burgert et al. 2002; Groom et al. 2002), jute (Saha et al. 2010), kenaf and ramie (Wang et al. 2011). The fibers derived from bamboo have attracted more interests and attention due to their extraordinary mechanical performances (e.g. average tensile strength 1.43–1.69 GPa, and modulus 32–34.6 GPa, Yu et al. 2011) and plenty sources for the production of bamboo fibers. Compared with other natural fibers, such as kenaf (average tensile strength 0.98 GPa, modulus 19 GPa) and ramie (average tensile strength 1 GPa, modulus 11 GPa), bamboo fibers have better tensile strength and modulus, but lower elongation at break (bamboo 7.1%, kenaf 8.3%, ramie 8.9%) (Wang et al. 2011). In 2013, there are 1.88 billion bamboo culms consumed creating a total production value of 27.4 billion US dollar in China (SFA 2014). In Jiangxi province, China, the bamboo industry provides 0.86 million jobs linked to bamboo products including pulp, textile, composites, furniture etc., creating about 4.6 billion US dollar in 2014 reported by society of bamboo industry (<http://blog.sina.com.cn/jxzcycy09>). However, the utilization rate of bamboo material is still very low, only about 40% (Wang et al. 2016).

At present, bamboo based textiles have not yet achieved their full potential. As reported, almost all available bamboo fabric in market is made using the viscose process (Nayak and Mishra 2016). Consequently, most bamboo textiles in market are not natural fibers but come from a natural source (Liese and Köhl 2015). It was reported that natural bamboo fibers has antibacterial properties (Xing and Liu 2004). But the natural coarse and stiff quality of natural bamboo fibers restricted their applications in the field of textile, fine and soft fibers are highly desirable for the textile (Liu et al. 2011; Nayak and Mishra 2016). Many techniques have been employed successfully for chemical modification of natural fibers. The microstructures and performances of natural fibers

varied depending on different treatments such as NaOH (Das and Chakraborty 2008; Liu and Hu 2008; Ray and Sarkar 2001), $\text{HNO}_3\text{-KClO}_3$ (Liu et al. 2012), NaClO (Chen et al. 2011), benzoate (Amel et al. 2013). Alkali treatment has been considered as one of the most popular and cost-effective methods to obtain high-performance natural fibers (Liu et al. 2011). Many studies reported that the alkali treatment is important in determining the structure and properties of natural fibers, such as wood (Chen et al. 2015b; Ishikura et al. 2010; Nakano 2010), abaca (Cai et al. 2015), hemp (Kabir et al. 2013; Liu et al. 2013; Ouajai and Shanks 2005), jute (Gassan and Bledzki 1999; Ray and Sarkar 2001; Saha et al. 2010; Sinha and Rout 2008), oil palm (Chowdhury et al. 2013), cotton (Keshk 2015; Eronen et al. 2009), kenaf (Amel et al. 2013; Nitta et al. 2013) and Alfa (Borchani et al. 2015).

However, there are a limited number of researches on the bamboo fibers with alkali treatment, especially on the individual bamboo fibers. Alkali treatments with different concentrations in bamboo strips and bamboo dusts brought about changes in dimensions, morphology and mechanical properties (Chen et al. 2016; Das and Chakraborty 2006, 2008). Bamboo fiber bundles treated by NaOH with different concentrations in different times have various structures (Liu and Hu 2008). To my knowledge, there is no report on the microstructure and mechanical properties of alkali treated individual bamboo fiber, because natural individual bamboo fiber is very small (length is 1.49–2.28 mm, diameter is 12.24–17.32 μm) (Tian 2015) and much harder than many plant fibers (average hardness is almost 0.5 GPa) (Yu et al. 2011). Therefore, it is difficult to characterize the microstructure and mechanical properties of individual bamboo fibers.

To expand the application of bamboo fibers in the textile, chemical modification bamboo fibers is of great importance. In this research individual bamboo fibers (*Neosinocalamus affinis*) were isolated from bamboo strips. The individual fibers were treated by NaOH solutions with varying concentrations and were characterized by FE-SEM, AFM, FIIR, XRD, CLSM and micro tensile strength testing. The objective of this research is to investigate the effects of NaOH concentrations on the microstructure and mechanical properties of individual bamboo fibers.

Experimental

Samples preparation

Raw materials were obtained from bamboo (*Neosinocalamus affinis*) grown in Chengdu, Sichuan Province, China. Bamboo was taken from the culm where was 2 m high from the ground with an initial moisture content of 8–12%. Bamboo was cut into small strips (30 mm longitudinally and 2 × 2 mm in cross section). Individual bamboo fibers were isolated by immersing the small bamboo strips in a chemical solution (one part 30% hydrogen peroxide and one part glacial acetic acid) at 65 °C for 18.5 h (Wang et al. 2011). Individual bamboo fibers were washed with deionized water to neutrality and then one part was dipped in 6, 8, 10, 15 and 25% NaOH solution at room temperature (23 ± 2 °C) for 2 h maintaining fiber weight to alkali volume ratio of 1:100 (Das and Chakraborty 2008; Zhang 2011). After the treatment, individual bamboo fibers were thoroughly washed using deionized water in order to remove NaOH from the fibers until the pH was 7. Both the untreated and treated fiber suspension were frozen in liquid nitrogen followed by drying for 24 h in a freeze dryer.

Surface morphology

The morphology of the freeze-dried fibers was selected randomly to observe with a field emission scanning electron microscope (FE-SEM, XL30 ESEM FEG, FEI Company, OR, USA). The microfibrillated aggregates on the bamboo fiber surfaces were imaged by Icon atomic force microscope (AFM, Burker Corporation, CA, USA). The specimens were scanned under tapping mode using sharp tapping mode probes with a tip radius 8 nm. The length of the cantilever was 125 μm, the spring constant was 42 N/M and the resonant frequency was 320 kHz. The samples were scanned at room temperature of 23 ± 2 °C and ambient humidity of 35 ± 5 %. Images were captured as 512 × 512 pixel images. Real time scanning was performed with scan rates 1.0 Hz, scan angle 0°, and tapping frequencies ranging from 340–380 kHz. Images were obtained in the phase mode, where the phase lag was used to determine differences in materials, as presented by Fahlén and Salmén (2005). Ten specimens for each treatment were

selected and scanned, and untreated fiber specimen was characterized as control.

Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR spectra of treated and untreated bamboo fibers were obtained in Nexus 670 spectrometer (Thermo Nicolet, CA, USA) within the range of 4000–500 cm^{-1} , with a resolution of 4 cm^{-1} and 64 scans. The KBr pellet consisting of KBr and fibers selected randomly with a weight ratio of 100:1 was prepared prior to the measurement. The FTIR analysis for each sample was performed in triplicate.

X-ray diffraction (XRD) analysis

The crystal structure of the treated and untreated fibers selected randomly was characterized using a Panalytical X-ray Diffractometer (Panalytical Corporation, Almelo, Netherlands). The X-ray diffraction patterns of the treated and untreated fibers were obtained in the diffractometer with a $\text{CuK}\alpha$ radiation source (X-ray wavelength $\lambda = 0.154178$ nm) with a detector placed on a goniometer scanning the range from 5° to 45°. The XRD analysis for each sample was performed in triplicate.

Confocal laser scanning microscopy (CLSM) analysis

The areas of cross-section of bamboo cell wall were observed with a confocal laser scanning microscopy (CLSM; Zeiss, LSM 510 Meta, Germany). Prior to CLSM observation, the fibers were dyed with acridine orange solution. Axio vision software (Zeiss, Germany) was used to calculate the fiber area (Yu et al. 2011; Wang et al. 2011). Fifteen specimens for each treatment were tested.

Mechanical properties test

Mechanical properties test of the individual bamboo fibers was conducted in accordance with the procedure described in Chen et al. (2011). First, individual bamboo fibers were selected with length more than 2 mm. Then fibers were glued on an organic, channelled glass plate with one droplet of glue at each end,

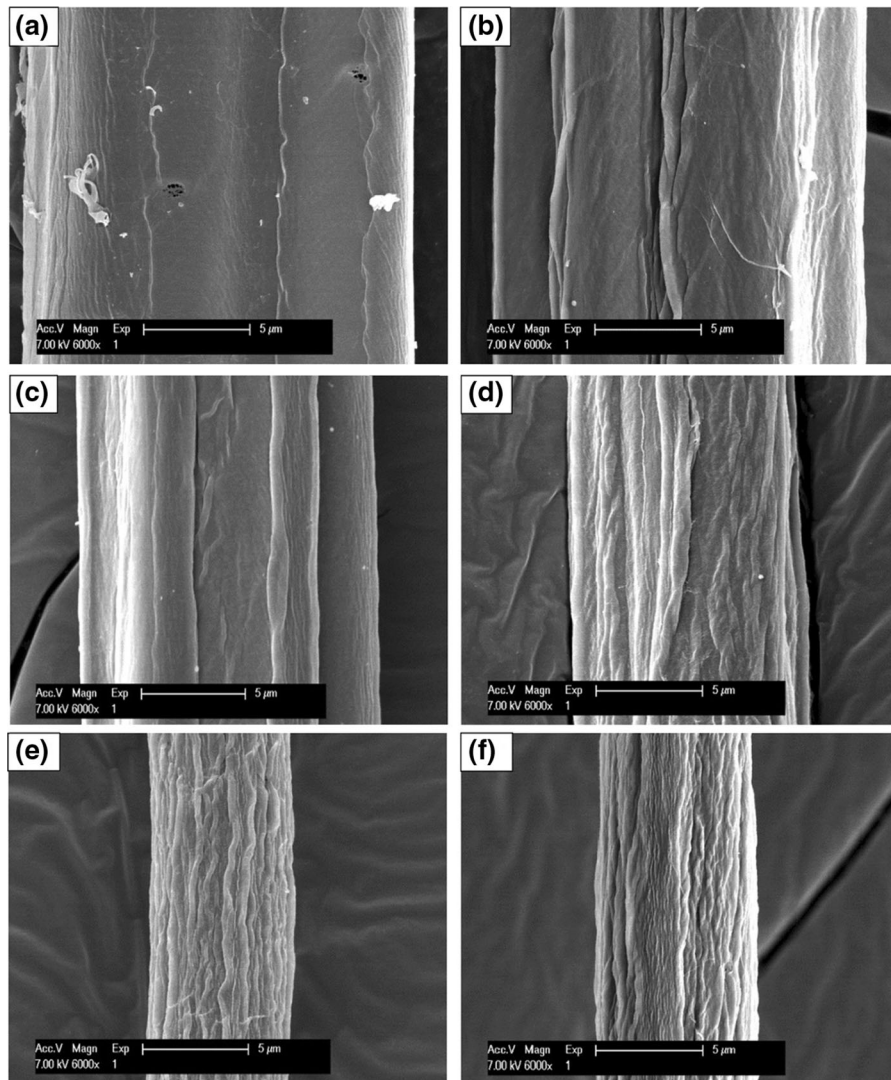


Fig. 1 SEM images of the surface morphology of individual bamboo fiber treated by NaOH solutions with different concentrations (**a** untreated; **b** 6%; **c** 8%; **d** 10%; **e** 15%; **f** 25%)

and then placed in ambient condition (25 ± 2 °C, $35 \pm 5\%$ RH) for at least 24 h. The tensile strength test of individual fibers was conducted at a high-resolution commercial mechanical tester with the measurement accuracy of 0.005 N (Microtester 5848, Instron, MA, USA). A constant strain rate of 48 $\mu\text{m}/\text{min}$ was set at 25 °C and 20% RH. The fibers were removed from the tensile apparatus immediately upon failure and stored for subsequent CLSM imaging of cross-sectional area for tensile modulus and strength calculations. Typical failures of individual fibers were observed in FE-SEM and CLSM, respectively. Fifteen specimens for each treatment were tested.

Results and discussion

Surface morphology

The surface morphologies of individual bamboo fibers treated by NaOH solutions with varying concentrations are presented in Figs. 1 and 2.

With increasing NaOH concentration, the diameter of individual bamboo fiber decreased as shown in Fig. 1, the similar phenomenon was also reported for fruit fibers (Boopathi et al. 2012). The average diameters (13 individual fibers measured for each type) were 16.46 ± 4.47 , 15.23 ± 1.33 , 11.47 ± 3.55 , $9.75 \pm$

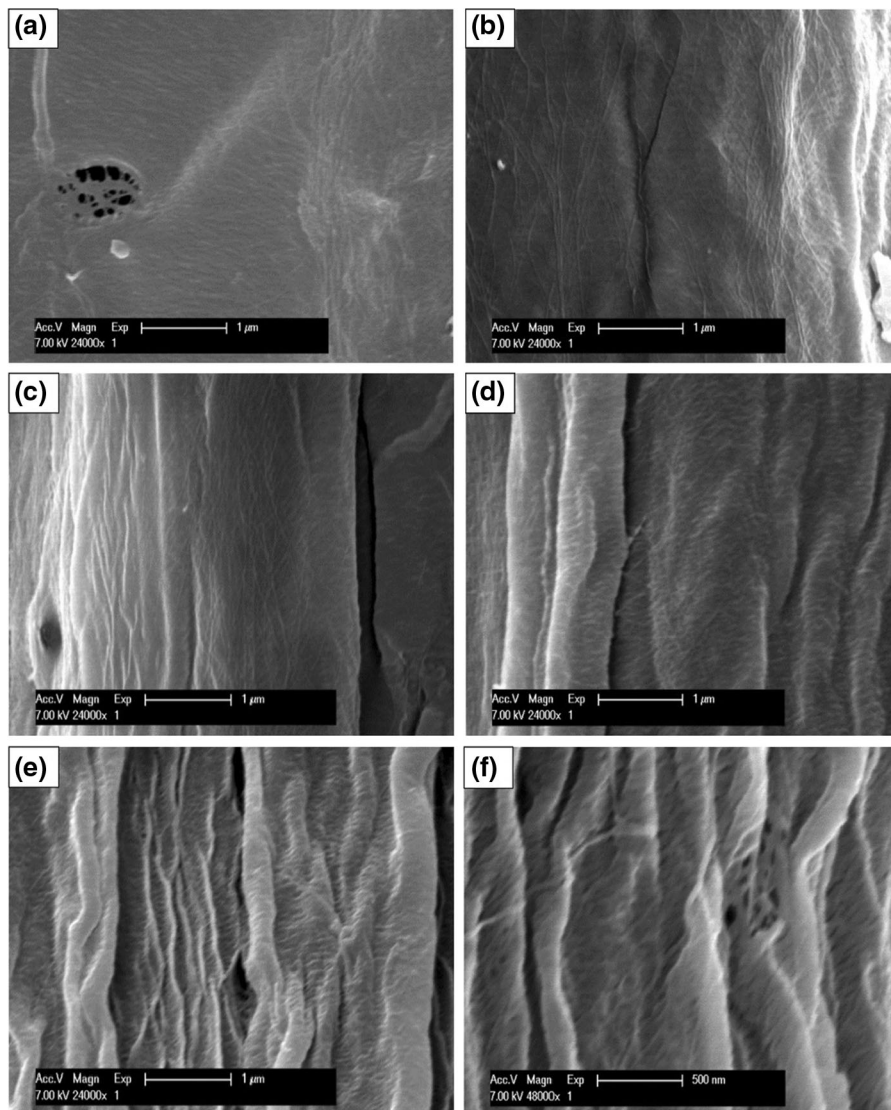


Fig. 2 Higher-magnification SEM images of the surface morphology of individual bamboo fiber treated by NaOH solutions with different concentrations (**a** untreated; **b** 6%; **c** 8%; **d** 10%; **e** 15%; **f** 25%)

4.10, 7.42 ± 2.05 , $8.02 \pm 1.57 \mu\text{m}$ for individual bamboo fibers treated by 0, 6, 8, 10, 15 and 25% alkali solution, respectively, while the average lengths (30 individual fibers measured for each type) were 2.65 ± 0.64 , 2.75 ± 0.55 , 2.99 ± 0.64 , 3.07 ± 0.65 , 2.37 ± 0.49 , $2.59 \pm 0.56 \text{ mm}$ for individual bamboo fibers. The diameter decreased with increasing alkali concentration, while the length increase at low alkali concentration and then decreased with high alkali concentration. For the dimension change of fiber, it possibly resulted from the loss of matrix and microfibrils aggregations with alkali treatment.

Meanwhile the wrinkle on the surface was increasingly apparent and the microfibril aggregates became more distinguishing. The untreated fibers surface was smooth. The orientation of cellulose microfibril aggregates was not obvious as shown in Figs. 1a and 2a but can be observed distinctly in Figs. 1b–d and 2b–d.

Furthermore, the cellulose microfibril aggregates not only can be seen clearly, but also some pores were observed between microfibril aggregates in Fig. 2e, f at high magnification.

It indicated that matrix in the cell wall were removed after alkali treatment. In our previous

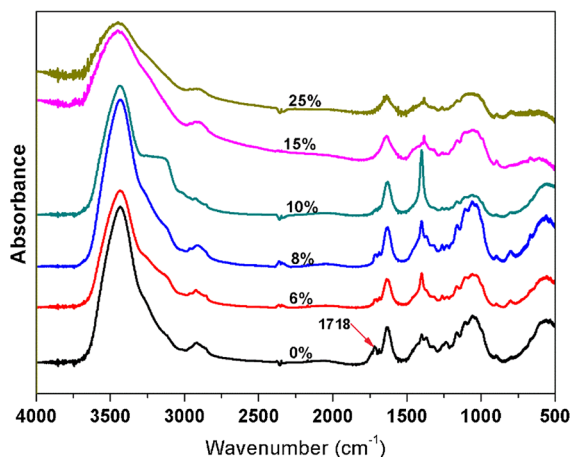


Fig. 3 FT-IR spectra of bamboo fibers treated by NaOH solutions with different concentrations

research, the isolated method used in this paper can remove the lignin of bamboo fiber (Chen et al. 2013). In addition, FT-IR indicated the absence of the peaks at 1510 and 1423 cm^{-1} in Fig. 3 assigned to aromatic skeletal vibration and aromatic skeletal vibration combined with C–H in plane deformations, respectively (Qin et al. 2004; Tian 2015), suggesting lignin in bamboo fibers have been removed during the isolation process. There was no lignin in the isolated individual fibers, either treated by alkali or untreated.

However, as shown in Fig. 3, there was peaks at 1718 cm^{-1} in fiber untreated and treated by alkali with concentration at 6 and 8%, respectively. The band at 1718 cm^{-1} was attributed to C=O stretching vibration in hemicellulose presenting in bamboo fibers (Das and Chakraborty 2006). There was still hemicellulose in fiber treated by 6 and 8% NaOH solution.

As the concentration of alkali increased to 10, 15 and 25% respectively, the bands at 1718 cm^{-1} disappeared, which indicated hemicellulose in bamboo fiber was almost removed. It is widely reported that alkali treatment can remove hemicellulose at certain NaOH concentrations (Ariawan et al. 2015; Ouajai and Shanks 2005; Rajulu et al. 2003; Saha et al. 2010; Zuluaga et al. 2009). In our case, it seems the NaOH concentration at 10% is adequate to remove all the hemicellulose that exists in the individual bamboo fibers.

In addition, microfibrils were prone to aggregate easily when the cell wall lost lignin and hemicellulose, which might be partly responsible for the reduction in

the diameter of fiber with the alkali treatment at higher concentration.

It showed that alkali concentration is an important factor affecting the surface morphology of bamboo fibers. The higher concentration tended to cause more cracks as shown in Figs. 1 and 2, leading to a rougher surface of bamboo fibers. The similar phenomena were also reported on other plant fibers after alkali treatment (Liu et al. 2013; Saha et al. 2010; Sinha and Rout 2008).

The phase images, obtained by AFM, of individual bamboo fiber treated with various NaOH concentrations are shown in Figs. 4 and 5. It can be found that cellulose microfibril aggregates in cell wall varied depending on NaOH concentrations.

The microfibril aggregates in surface of bamboo fiber treated by low concentration (6 and 8%) showed a randomly interwoven structure in Figs. 4b, c and 5b, c, which is similar with the untreated ones in Figs. 4a and 5a.

However, the microfibril aggregates began to change when the NaOH concentration increased up to 10% as shown in Figs. 4d and 5d. In the case of 15 and 25% NaOH treatment, the randomly interwoven structure of cellulose microfibril aggregates disappeared. Granular structure was observed, which might be due to the transformation of cellulose I to cellulose II (Das and Chakraborty 2006; Liu and Hu 2008) or the damage of fine fiber structure as the alkali concentration increased more than 10% (Cai et al. 2015). Microfibrils were swollen and more granular in cellulose II than in cellulose I (Eronen et al. 2009). In addition, the transformation of cellulose I into cellulose II was irreversible at both the crystallographic level and the morphological level (Dinnad et al. 2002).

X-ray diffraction

The XRD patterns of bamboo fibers treated with various NaOH concentrations are shown in Fig. 6.

In Fig. 6, two characteristic peaks in the XRD pattern of untreated bamboo fiber were observed at $2\theta = 16.01^\circ$ and 22.25° , which correspond to typical cellulose I with crystallinities having preferred orientation along the fiber axis (indicated by reduced tensile near 34.7°) (French 2014). The similar XRD patterns of the 6, 8 and 10% alkali treated fibers were observed as the untreated one, showing the similar cellulose I

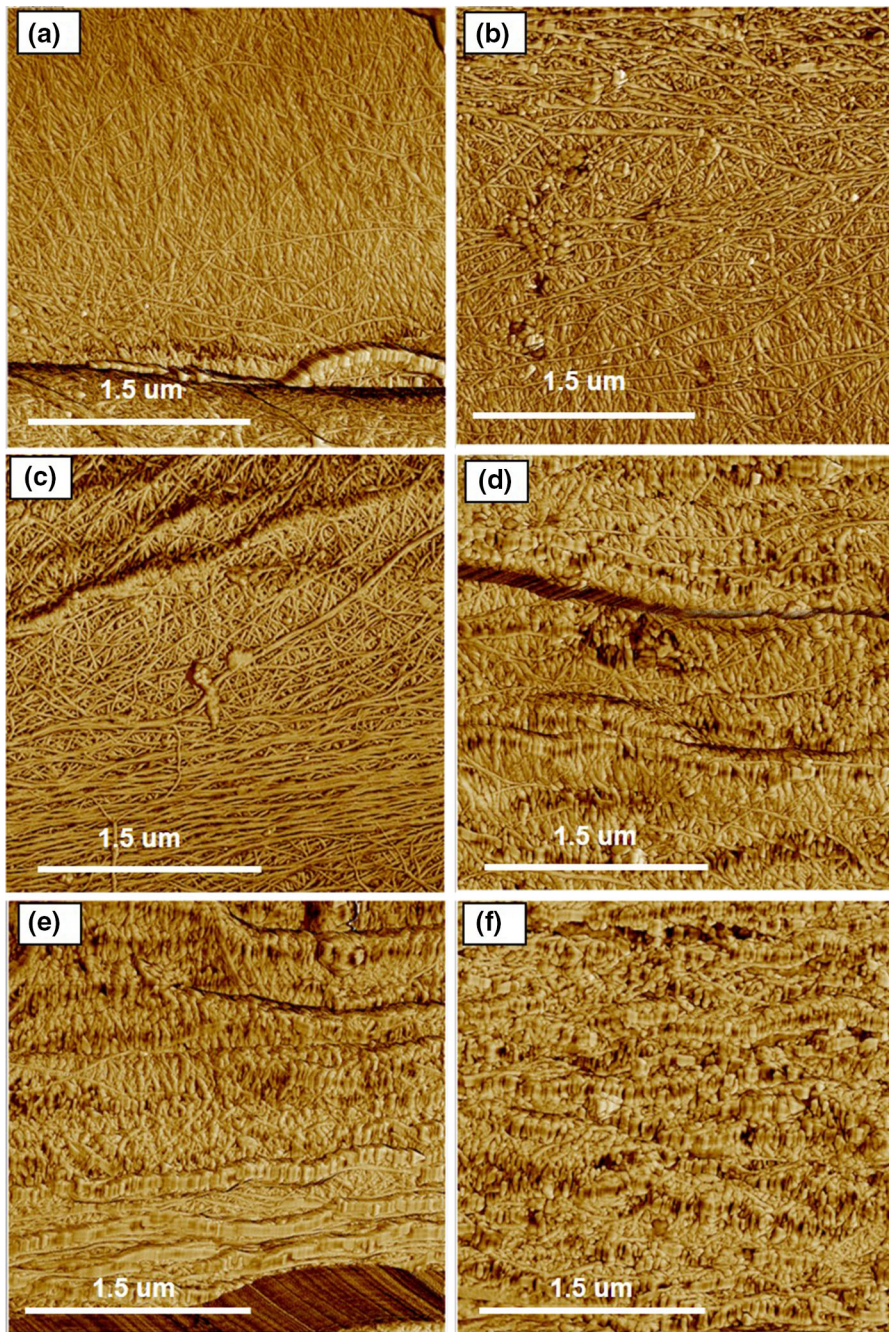


Fig. 4 AFM images of the arrangement of microfibrils of bamboo fiber treated by NaOH solutions with different concentrations (a untreated; b 6%; c 8%; d 10%; e 15%; f 25%)

structure. As reported by Sun et al. (2014), for poplar treated by $\text{Ca}(\text{OH})_2$ solution at low concentration, there was no transition of cellulose I into II occurred and the relative carbohydrate was not significantly altered.

However, when the NaOH concentration increased up to 15 and 25%, XRD patterns changed, the peaks at $2\theta = 12.25^\circ$, 20.2° and 21.8° correspond to (1–10), (110) and (020) planes of typical cellulose II with preferred orientation of the crystallites along the fiber

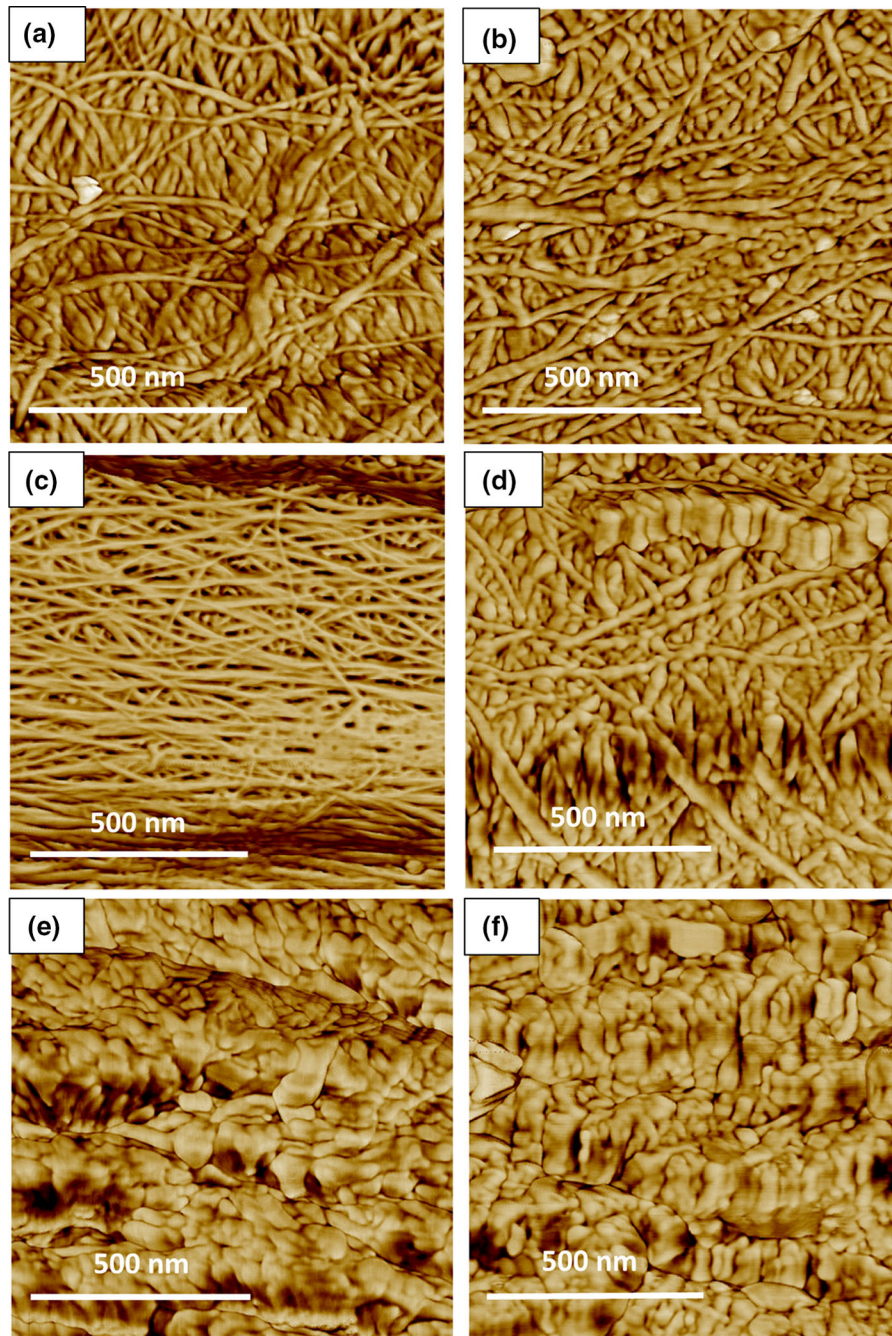


Fig. 5 Higher-resolution AFM images of the arrangement of microfibrils of bamboo fiber treated by NaOH solutions with different concentrations (**a** untreated; **b** 6%; **c** 8%; **d** 10%; **e** 15%; **f** 25%)

axis (indicated by no peak near 34.7°), respectively (French 2014). This finding is consistent with the phenomenon reported by Das and Chakraborty (2006), Eronen et al. (2009), and Liu and Hu (2008). Therefore, when NaOH concentration exceeded 15%, cellulose II

was the major component in the fiber (El Oudiani et al. 2011). The chains in cellulose I organized in sheets packed in a “parallel-up” fashion (Nishiyama et al. 2002, 2003), while cellulose II structure is antiparallel (Langan et al. 1999). In compact cell wall, microfibrils

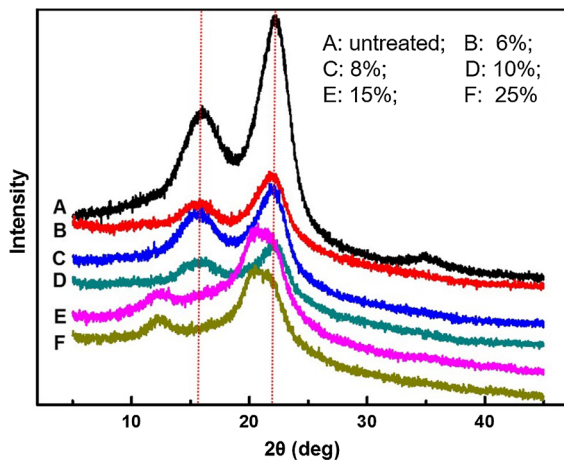


Fig. 6 XRD patterns of bamboo fiber treated by NaOH solutions with different concentrations

of opposite polarity co-existed side by side in native cellulose system. In an alkali treatment, during the swelling process, neighboring cellulose chains belonging to microfibrils of opposite polarity would get close enough to meet each other. At washing and drying step, the cellulose chains would recrystallize into antiparallel form which is thermodynamically more stable (Dinnad et al. 2002; Nishimura and Sarko 1987; Okano and Sarko 1985; Wang and Chen 1996).

The patterns of untreated fibers showed that there was only cellulose I in bamboo fiber, the alkali treatment led to different lattice transformation when the concentration increased. Lattice transformation from cellulose I to cellulose II have been reported occurring in many alkali treated plant fibers such as wood (Borysiak and Doczekalska 2005; Ishikura et al. 2010; Nakano and Nakano 2014), ramie (Kobayashi et al. 2011), abaca (Cai et al. 2015), Alfa (Borchani et al. 2015).

CLSM analysis

The cross-sectional area and cross sections of cell wall in individual bamboo fiber are shown in Table 1 and Fig. 7, respectively. It indicated that different alkali treatment significantly influenced the lumen and cross sectional area of cell wall of the fibers.

The cross-sectional area of all the treated individual bamboo fibers were smaller than that in untreated one that might be due to partial removal of amorphous materials in fiber as a result of alkali treatment.

The areas of individual bamboo fiber treated by 6 and 8% NaOH solution were slightly smaller in

Table 1 Cross-sectional area of bamboo cell wall

Treatment	Area (μm^2) (CV)
A (untreated)	108.48 (0.26)
B (6%)	103.58 (0.27)
C (8%)	107.54 (0.26)
D (10%)	88.08 (0.29)
E (15%)	90.23 (0.32)
F (25%)	105.53 (0.27)

comparison with the untreated one. Furthermore, after alkali treatment with 10% NaOH solution, the cross-sectional area of individual bamboo fiber decreased to be the minimum. While increasing NaOH concentration up to 15 and 25%, the cross-sectional areas increased when compared with fibers with 10% NaOH treatment. It might be attributed to transformation of cellulose I to cellulose II.

Lumens in cross sections of bamboo fibers treated by both 6 and 8% NaOH solution were observed, which was similar with the one untreated.

However, when NaOH concentration increased to 10, 15 and 25%, the lumens disappeared and a crack appeared between different layers. Bamboo fiber has a polylamellate structure of alternating broad and narrow lamellae which is different from that in wood cell wall (Liese and Köhl 2015). The removal of cementing materials during alkali treatment led to better packing of cellulose microfibrils (Das and Chakraborty 2008). The aggregation of cellulose microfibrils may result in various stresses in different layers in bamboo cell wall and thus created cracks between layers as shown in Fig. 6d–f.

Mechanical properties

In this study, it was found that mechanical properties were significantly affected by the alkali treatment due to the change of fiber structure.

The typical stress–strain curves of untreated individual bamboo fibers and fibers treated by NaOH with various concentrations are shown in Fig. 8.

The stress–strain curves of individual bamboo fibers treated with 6 and 8% NaOH are similar to the untreated one. However, the stress–strain curves of 10, 15 and 25% NaOH treated fibers appeared differently in comparison of the untreated one.

The tensile strength, the modulus of elasticity (MOE) and the elongation at break are summarized in Table 2.

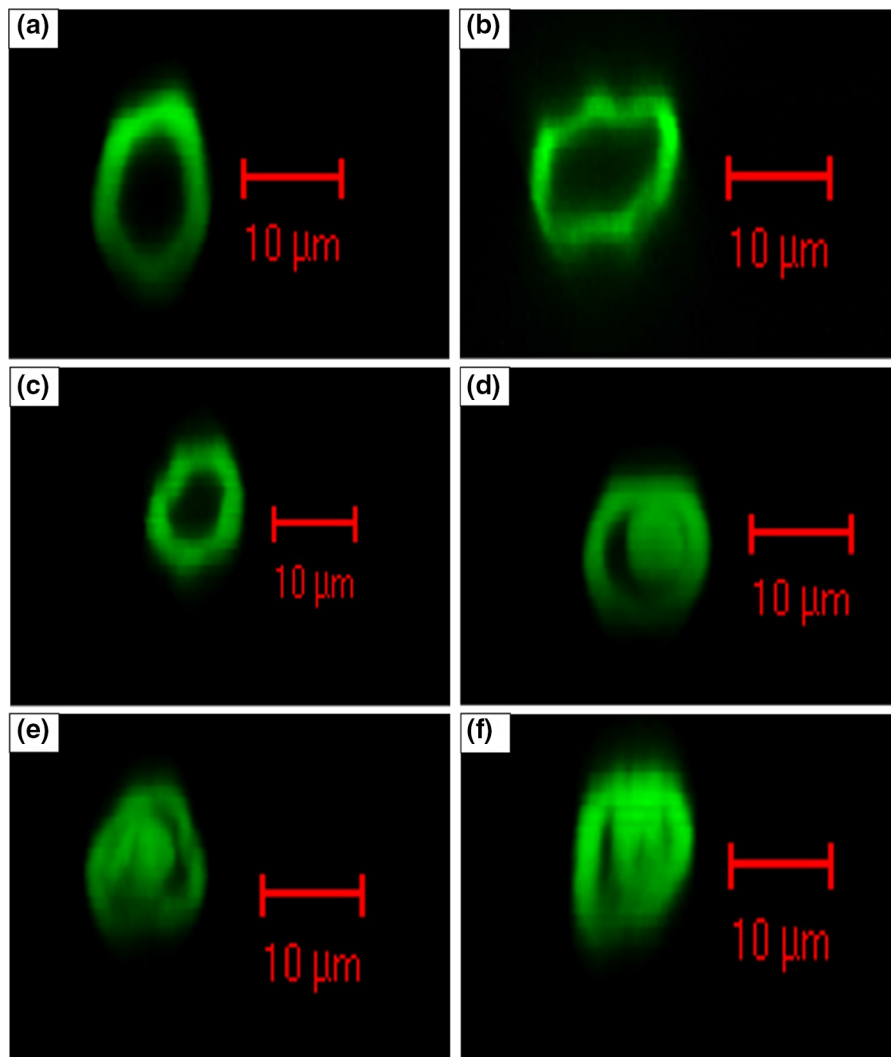


Fig. 7 Typical CLSM images of the cross section of individual bamboo fibers treated by NaOH solutions with different concentrations (a untreated; b 6%; c 8%; d 10%; e 15%; f 25%)

The tensile strength of all alkali treated individual bamboo fibers decreased as compared to untreated fiber as shown in Table 2. That might be attributed to the damage and the change in the structure of fine fiber. When concentrations were at 6, 8 and 10%, the alkali treatment removed hemicellulose gradually. While increasing concentration to 15 and 25%, the treatment not only removed the hemicellulose, but also resulted in cellulose transforming from I to II.

MOE of all treated individual bamboo fibers was lower than that of untreated fiber. With increasing NaOH concentration, MOE decreased significantly.

In the case of fibers treated by 6, 8 and 10%, the removal of matrix materials was the main cause of the reduction in the modulus (Fahlén and Salmén 2005; Zhang 2011). For individual bamboo fibers treated by 15 and 25%, the combined effects of the removal of hemicellulose and the transformation of cellulose I to II led to the bigger reduction in the MOE, because the MOE of cellulose I (around 140 GPa) is higher than that of cellulose II (90 GPa) (Northolt et al. 2001).

The elongations at break of all treated individual bamboo fibers were higher than the untreated one. While the NaOH concentration increased to 10%, the elongation at break began to increase significantly.

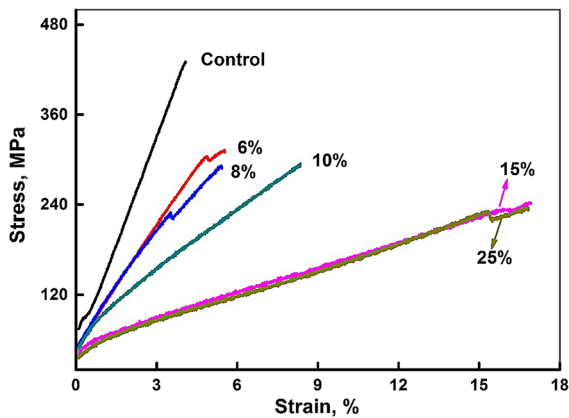


Fig. 8 Typical tensile stress–strain curves of individual bamboo fibers treated by NaOH solutions with different concentrations

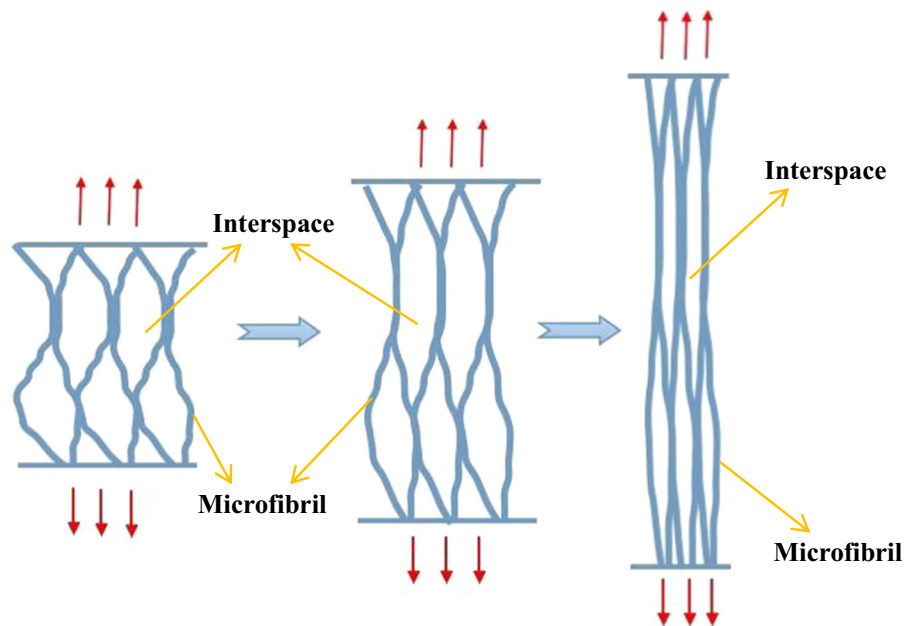
Once the concentration increased to 15 and 25%, the elongation at break increased by 232 and 221%, respectively when compared with untreated fiber. The elongations at break of individual bamboo fibers treated were much higher than that of bamboo fiber bundles (2.61%) which can be used for making yarn (Chen et al. 2015a). It indicated that bamboo fibers had a potentiality to making soft and fine yarn for textile with good quality.

As reported by Salmén (2004) that cellulose microfibril aggregates were filled with the matrix consisting of lignin and hemicellulose. The isolation process and alkali treatment removed matrix in the cell wall resulting in lots of interspace as shown in Fig. 9. When the fiber was pulled, the interspaces first

Table 2 The data of tensile strength, modulus of elasticity and elongation at the break

Sample	Tensile strength (GPa) (CV)	Modulus of elasticity (GPa) (CV)	Elongation at break (%) (CV)
A (untreated)	0.83 (0.57)	14.48 (0.49)	5.85 (0.15)
B (6%)	0.64 (0.40)	10.64 (0.40)	6.53 (0.22)
C (8%)	0.68 (0.63)	11.61 (0.56)	6.06 (0.17)
D (10%)	0.59 (0.31)	8.89 (0.39)	8.21 (0.25)
E (15%)	0.59 (0.39)	4.22 (0.50)	19.42 (0.21)
F (25%)	0.61 (0.19)	4.85 (0.44)	18.76 (0.30)

Fig. 9 The model of individual bamboo fiber under load after removing the matrix



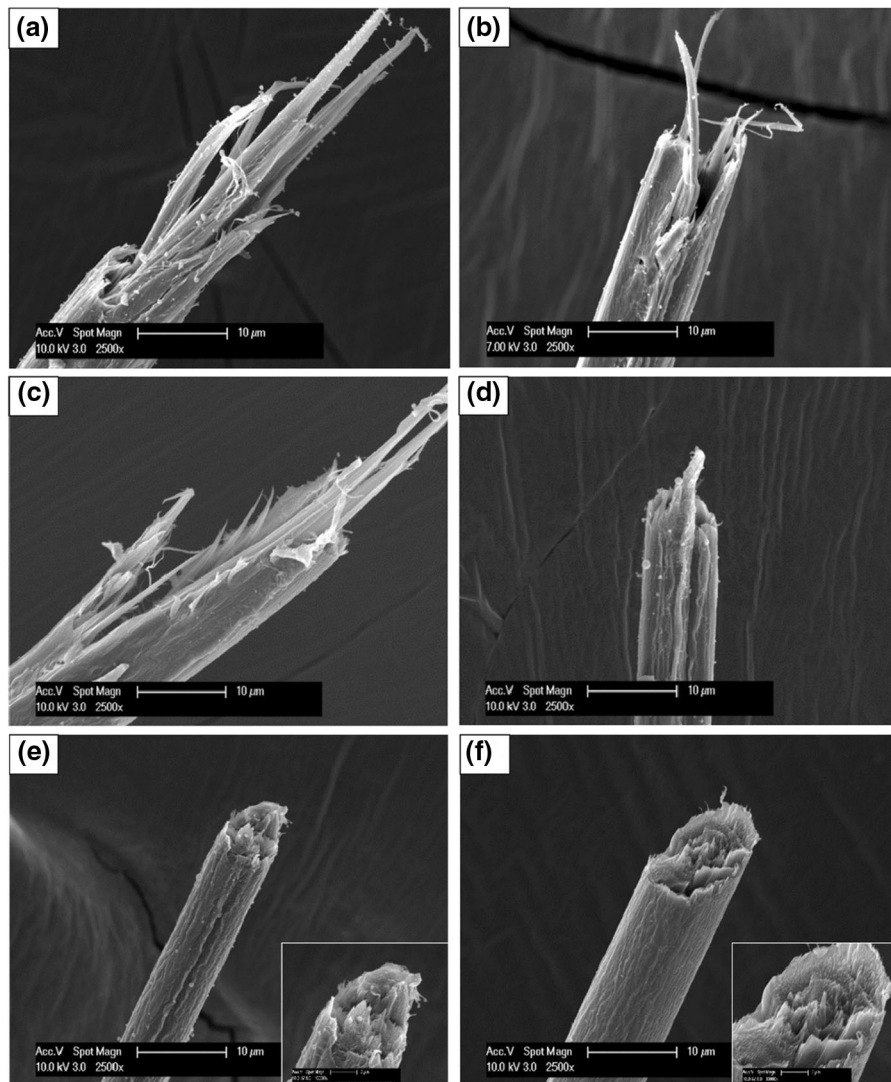


Fig. 10 FE-SEM images of the fracture after the tensile testing of individual bamboo fiber treated by NaOH solutions with different concentrations (**a** untreated; **b** 6%; **c** 8%; **d** 10%; **e** 15%; **e** 25%)

changed to be small, then loading on the microfibril which led to much higher elongations at break of the alkali treated samples. The analysis of FT-IR indicated that NaOH concentration at 10% is adequate to remove all the hemicellulose in the individual bamboo fibers. While the elongations at break treated by 15 and 25% NaOH solution were much higher than that treated by 10% NaOH solution. When the concentration increased to 15 and 25%, not only removal of hemicellulose happened but also transition of cellulose happened. Both accounted for the increase of elongation at break of individual bamboo fiber. As

reported by other researchers, the alkali treatment can soften the fibers dramatically (Gassan and Bledzki 1999; Liu et al. 2013).

The typical fractures of individual bamboo fibers during the tensile testing are shown in Fig. 10. For alkali treated bamboo fibers, chemical and structural changes resulted in the difference in the fracture. The fractures of individual bamboo fiber treated by 6 and 8% were similar to that of untreated fibers presenting as a brush. This type of fracture resulted from the cutting alongside with the microfibril or the microfibrils were pulled away from each other (Rowell 1984).

It suggested that most of the fracture happened in the weak interface between microfibrils. The fractures of fibers with 15 and 25% NaOH treatment were neat and uniform which indicated the fracture mainly happened in microfibril instead of between the microfibrils as happened in the untreated fibers (Rowell 1984). This fracture form was called broken in pieces by Rowell. In addition, the holes resulted from loss of lignin and hemicellulose in the alkali treatment helped to form brittle fracture surface. Microfibrils slippage probably happened due to the decreasing bonding when losing the matrix in cell wall. While for 10% NaOH treated fibers, the fracture was between the “brush type” and the “uniform type”.

Conclusions

The effect of alkali concentration on microstructure and mechanical properties of individual bamboo fibers were investigated. The results obtained are as follows:

1. Alkali treatment changed the surface morphology and the microfibril aggregates in cell wall. The alkali treatment resulted in more wrinkles in the surface of bamboo fibers when compared with the untreated ones. The 15 and 25% NaOH treatment caused the change of the microfibril aggregates from a randomly interwoven structure to a granular structure.
2. The transformation of cellulose I to II depended on alkali concentration. For concentrations of 6, 8 and 10%, the alkali treatment did not affect the cellulose. But cellulose I transformed to cellulose II when NaOH concentration increased up to 15 and 25%.
3. The lumens of individual bamboo fiber collapsed and cracks appeared between layers in cell wall after 10, 15 and 25% NaOH treatments. The diameter and cross sectional area of individual bamboo fiber treated was smaller than that of untreated fiber but changed different with increasing concentrations. Diameter decreased with increasing concentrations. However the cross sectional area of fibers treated by 10% NaOH was minimum. When the alkali concentration exceeding 10%, the cross sectional areas of treated fibers increased with increasing

concentration because of cellulose lattice transformation and microfibril swollen.

4. The tensile strength and MOE of individual bamboo fibers decreased after treated by the alkali. Alkali concentration almost did not affect the tensile strength but influenced MOE significantly. For NaOH treated fibers, the elongations at break were higher than the untreated one. When with strong alkali treatment (10, 15 and 25%), the elongation at break increased significantly, especially at 15 and 25%, the elongation at break increased by 232 and 221% comparing with the untreated fiber, respectively. Alkali treatment caused individual bamboo fibers from brittle to ductile with increasing NaOH concentrations.

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