A Comparative Analysis on the Longitudinal Compression Characteristics of Juvenile and Mature Northeast Chinese Ash (*Fraxinus mandshurica* Rupr.) Subjected to Alkaline Treatment

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Longitudinal compression can help wood form some folds on the wood cell walls after a suitable softening procedure. These folds can enhance the one- and multi-dimensional bending performances of wood. The longitudinal compression properties of alkali-treated juvenile and mature northeast Chinese ash (*Fraxinus mandshurica* Rupr.) were analyzed. Elastic and elastic-plastic stages were inferred from the longitudinal compression curves. Scanning electron microscopy images showed that some folds formed in the wood cell wall vessels and fibers. X-ray microdensitometer test results showed a decrease in the fluctuation of the wood cell wall density of the specimens. The swelling and degradation or extraction of hemicellulose, lignin, and extractive occurred after alkaline treatment. Slippage between cellulose chains and curving within a cellulose chain were inferred during longitudinal compression. Juvenile wood specimens had higher modulus of elasticity and larger variability than mature wood after alkaline treatment. This finding can be attributed to the higher extent of degradation or extraction of hemicelluloses, lignin, and extractive, as well as the smaller microfibril angle and the similar cellulose crystallinity of treated juvenile wood compared to those of mature wood specimens after alkaline treatment.

Keywords: Northeast Chinese ash; Longitudinal compression; Stress-strain Relationship; NaOH; microdensity; Relative crystallinity; Microfibrils angle

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INTRODUCTION

Northeast Chinese ash (*Fraxinus mandshurica* Rupr.) is one of the most commonly used wood species for bending furniture manufactured in northeast China. Given the unique characteristics of solid wood, both juvenile and mature wood do not bend in multiple dimensions and have small radii of curvature in one-dimensional bending. Wood is a natural macromolecule composite consisting of a cellulose skeletal structure as well as matrix composites of hemicellulose and cross-linked lignin (Fengel and Wegener 2003; Li 2002). Wood is also an orthotropic material with three different directions: radial (perpendicular to the year rings), tangential (parallel to the annual rings), and longitudinal (along the stem) (Dinwoodie 2000; Li 2002). Among these directions, the highest strength tolerance capacity is observed in the longitudinal direction. However, wood cannot be compressed at large amplitudes without an effective softening process. Hydrothermal treatment is considered to be one of the most effective softening methods for wood modification (Furuta *et al*. 2010; Hamdan *et al*. 2000;

Kutnar *et al*. 2009; Tjeerdsma and Militz 2005). Alkaline modification also reportedly induces swelling in the noncrystalline regions and deformation between microfibers. Swelling occurs because of the breakage of interlocked hydrogen bonds as well as the removal of hemicellulose and lignin from the fibers (Gassan and Bledzki 1999; Goda *et al*. 2006). Thus, a loosely bound structure can change from anisotropic and then form cross-linking. Consequently, the slippage of cellulose microfibrils easily occurs when hemicellulose and lignin are absent. Gomes *et al*. (2007) studied alkali-pretreatment fibers used in glass fiber reinforced plastics and found that the strain at break increases. The removal of hemicellulose and lignin from lignocellulose material can also enhance the tensile strength for increased crystallinity (Mwaikambo 2009; Pejic *et al*. 2008; Saha *et al*. 2010). Numerous studies have also been conducted on alkali-treated fibers with a focus on improving surface performance (Baley *et al.* 2006; Ramadevi *et al*. 2012) and fiber mercerization (Cao *et al*. 2012; Sreekala and Thomas 2003). After alkali treatment, the creep properties of jute fibers are found to be degraded, resulting in closer packing and arrangement (Ray *et al*. 2009).

Most of the molecular chains of high-molecular-weight polymers used in plastics are folded. These folds flatten when an external stress is encountered, which indicates poor strength and well-bending property. However, trees have fine, natural structures of linear high-molecular-weight polymers and high strength acquired throughout their evolution. These structures are beneficial in resisting gravity, wind, and other physical elements in the environment. If some folds exist in wood molecular chains, wood properties such as plasticity and bending performance can be improved (Song *et al*. 2004) in terms of enlarged curve radius, which is beneficial to the wood shaping industry. Longitudinal compression can help form some folds after a softening process. In this study, juvenile and mature wood properties before and after alkaline treatment, as well as the stress-strain relationships after alkaline treatment were analyzed by comparison with untreated specimens. This is a basic research for the target of multi-direction bending of wood shaping industry.

EXPERIMENTAL

Materials

Three northeast Chinese ash logs, including juvenile and mature wood, were felled in the Maoershan forest region of northeast China. The logs had an average diameter at breast height of 24 to 26 cm and were 1.2 to 1.9 m off the ground, in order to achieve the optimal basic physical and mechanical bending properties of ash wood. From another study by the authors on northeast Chinese ash wood properties, it was revealed that the demarcation of juvenile and mature wood is around the 20^{th} year with a diameter of juvenile wood that measures approximately 9 cm to 11 cm in the central part of the trunk. To compare the properties of juvenile and mature wood, juvenile wood was cut near the pith, whereas mature wood was cut near the bark. The availability of equipment for longitudinal compression, mechanical testing, and subsequent multi-dimensional bending of the specimens were also considered. The dimensions of the specimens were 270 mm (longitudinal) × 16 mm (radial) × 16 mm (tangential). The specimens were selected such that they had no macroscopic defects, including decay, twill, clip skin, and knots. The surface grain slope angle, which was the measured angle between wood grain and the axial direction on radial section, was less than 10°. Twenty specimens of juvenile

and mature wood, respectively were selected. The specimens were equilibrated at the same room environment.

Alkaline Treatment

Fifteen experimental juvenile and mature ash specimens were respectively dipped in 3000 mL of alkaline solution at room temperature and normal pressure for 240 to 300 min. The alkaline solution was pre-made at one time and consisted of sodium hydroxide (NaOH) and a modifier (Urea). The concentration of sodium hydroxide was 6 to 7 wt%. The modifier had a concentration of 10 to 15 wt%. The compression test was conducted immediately after the specimens were taken out of the solution.

Longitudinal Compression

The longitudinal compression of 10 experimental juvenile and mature ash wood specimens respectively was carried out using a universal mechanical testing machine equipped with a longitudinal compression mold (Fig. 1) to avoid the bending of specimens. The compression speed was set at 2 mm/min to achieve the compression proportional limit stress and maximum stress.

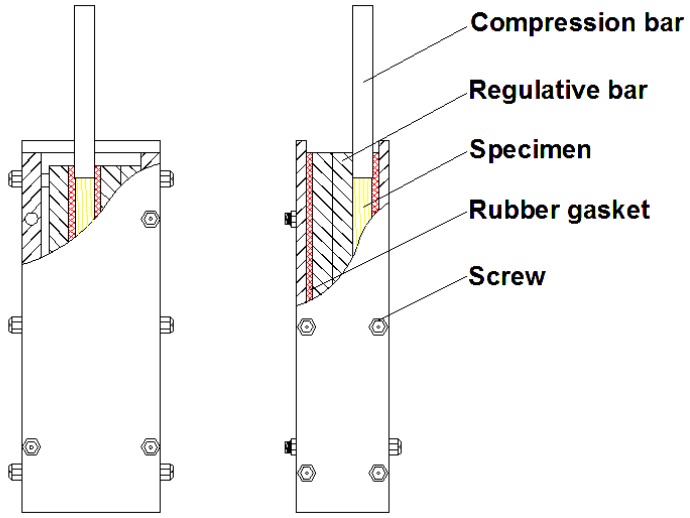

Fig. 1. Schematic version of the compression mold

Scanning Electron Microscopy (SEM)

The structural changes of experimental juvenile and mature ash specimens were observed using a JSM-5610LV scanning electron microscope (SEM Company, Japan). Three experimental juvenile and mature ash specimens were respectively selected. Then they were put into an electric thermostatic drying oven. To avoid dimensional changes caused by moisture variation, the weighted mean moisture content of compressed and uncompressed specimens was controlled at 8.15%. For the same reason, slices for wood anatomy analysis were cut into 10 mm (longitudinal) × 10 mm (radial) × 3 mm (tangential) from the center of these specimens. Each sample was coated with approximately 10 to 20 nm gold before examination.

Wood Microdensity Analysis

Normal and three experimental alkali-treated compressed juvenile and mature ash wood samples were respectively cut in the center of the specimens with the following

dimensions: 2 mm (radial) × 4 mm (tangential), and longitudinal length after compression. The weighted average moisture of samples was 8.26%. The microdensities of juvenile and mature samples were tested using an X-ray microdensitometer (SOF-TEX model, Japan) (Knapic *et al.* 2007). The test conditions are as follows: speed, 1.0 mm/s; voltage, 23 kV; and current, 15 mA.

Wood Chemical Composition Analysis

Based on Chinese Standard GB-2677 (Jiang *et al*. 2004; Zhou *et al*. 2009), the holocellulose, lignin, and extractive contents of five juvenile and mature ash wood were tested before and after alkaline treatment. The cellulose content corresponding to the nitrocellulose content was tested by the nitric acid-ethanol method (Tan *et al*. 2008). The hemicellulose content was calculated based on the holocellulose content without the nitrocellulose content. The percent change was calculated as follows:

$$Percent\ change = \frac{Untreated\ specimen\ content - Treated\ specimen\ content}{Untreated\ specimen\ content} \times 100\% \quad (1)$$

X-ray Diffraction (XRD) Analysis

The cellulose relative crystallinity and wood microfibrillar angle of five alkali-treated and untreated specimens were determined using an X-ray diffractometer (D/MAX 2200, Rigaku, Japan) with Ni-filtered Cu-Kα radiation at a voltage of 40 kV and a current of 30 mA. Scattered radiation was detected within the range of 5° to 50° at a speed of 4°/min and step length of 0.02°. Juvenile and mature ash specimens were milled through 100 to 120 meshes. The data were calculated by the Segal method (Segal *et al*. 1959) using Equation 2,

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

where C_rI is the percentage of relative crystallinity, I_{002} is the highest lattice diffraction angle intensity, and I_{am} is the scattering intensity; 2θ is almost 18° at the non-crystalline background.

The wood microfibrillar angle was determined using the M50 method (Ruan *et al.* 1982), namely the peak width at half of the I_{002} peak height, through the software JADE 5.0.

RESULTS AND DISCUSSION

Stress-Strain Relationship

The juvenile and mature ash wood specimens exhibited different characteristics when they were longitudinally compressed to their maximum capacity after an alkaline softening process. Figure 2(a) is based on a former compression strength test parallel to the grain; the data came from ash trees felled in the same place. This finding may indicate that the stress-strain relationship of the compression strength test parallel to grain of juvenile wood had lower compression strength than mature wood, which had a higher slope. Compared with the compression strength test parallel to grain, the much longer alkali-treated specimens, in Fig. 2(b), showed different curve features. The curves presented two obvious parts with different slopes. At the beginning of compression

(elastic region), the stress sharply increased almost linearly with increased strain. When reaching the proportional ultimate stress δ_p (inflection point of the curves), with increased strain, the stress slightly increased compared with the early stage. Both modulus of elasticity (MOE) and stress of juvenile wood were higher than those of mature wood, and those (expect the maximum stress) of juvenile wood are significantly different from mature wood to different extent, as shown in Table 1, which was opposite to the results of the untreated compression strength test. The accessible strain of longitudinal compression after alkaline treatment increased, and the required stress to achieve the yield point decreased to almost 10 MPa. Beyond the proportional ultimate stress δ_p, with increased stress, the specimens of compression strength test parallel to grain broke, and a long period of elastic-plastic deformation of the alkali-treated specimens was observed. The folds gradually formed and increased in ash vessel walls and wood fibers, as indicated by the elastic-plastic region (Fig. 3). The experimental study showed that closer cutting sections into the wood pith resulted in higher stress required for longitudinal compression with higher values of stress deviation after alkaline treatment. This finding revealed the poor stability of longitudinally compressed juvenile wood after alkaline treatment. The standard deviations of both proportional ultimate stress and maximum stress of juvenile wood were higher than those of mature wood after alkaline treatment.

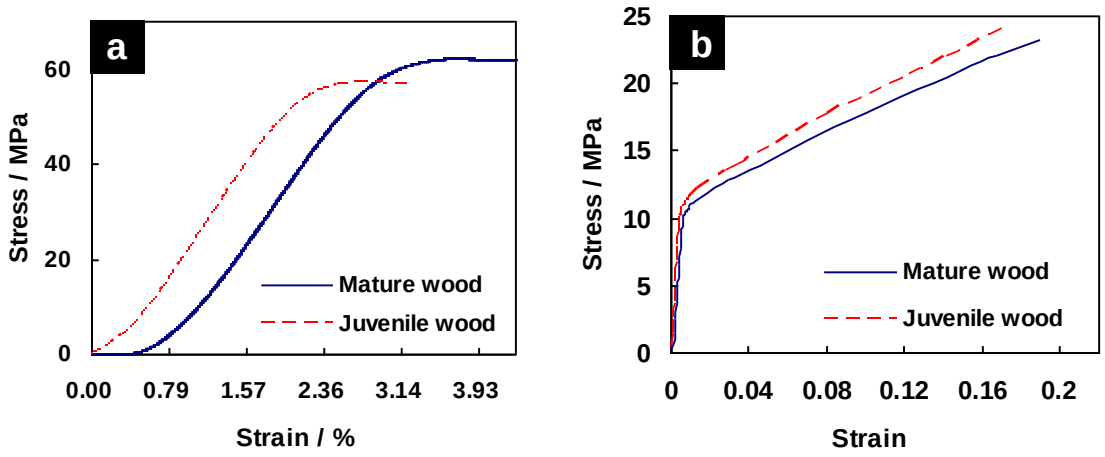

Fig. 2. Stress and strain relationship of longitudinally compressed ash specimens: (a) compression strength test parallel to grain (Untreated); (b) alkali-treated

Table 1. The Main Mechanical Properties of Alkali-treated Ash Samples after Longitudinal Compression

Specimens	MOE (MPa)		Proportional ultimate Stress δ_p (MPa)	Maximum stress δ_s (MPa)
	Elastic region	Elastic-plastic region		
Juvenile wood	2160	81	11.42±0.29	25.58±0.36
Mature wood	1829	79	10.85±0.27	25.35±0.33
ANOVA	***	***	*	

ANOVA is the analysis of variance between juvenile and mature wood. ***p＜0.01; **p＜0.05; *p＜0.1; blank means insignificant.

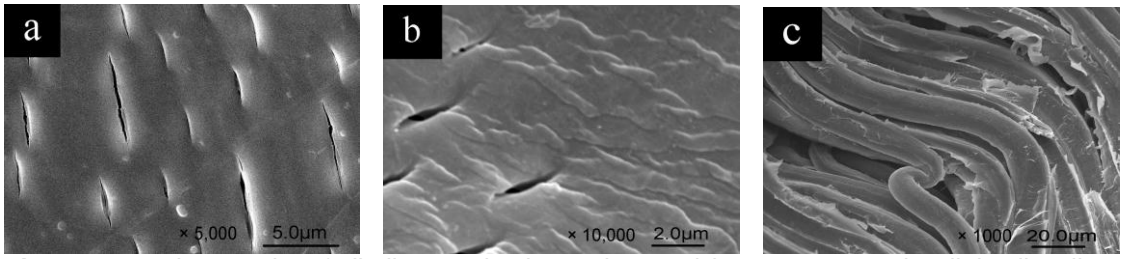

Fig. 3. SEM micrographs of alkali-treated ash specimens: (a) Uncompressed radial cell wall vessel; (b) Compressed radial cell wall vessel; and (c) Compressed radial wood fibers

Wood Microdensity and Microfibrillar Angle Analysis

Juvenile and mature wood had different stress-strain relationships before and after alkaline treatment based on analyses of macrostructural and microstructural changes. From the studies of Mwaikambo (2009) and Wang *et al.* (2001), wood density and micro-fibrillar angle are known as the two main factors affecting the wood longitudinal strength. Figure 4 shows the wood microdensity changes of juvenile and mature wood. The density of both juvenile and mature wood increased after longitudinal compression, whereas the amplitude of fluctuation decreased, which indicated that the thin walls were more compressive than the thick walls. After alkaline treatment, juvenile and mature wood had enhanced flexibility, and they were uniformly compressed along the length of samples instead of over-densification at two ends of the samples. The longitudinal compression process did not change the lower average density of juvenile wood in comparison to mature wood. Therefore, wood density did not influence the characteristic changes of juvenile and mature wood during longitudinal compression.

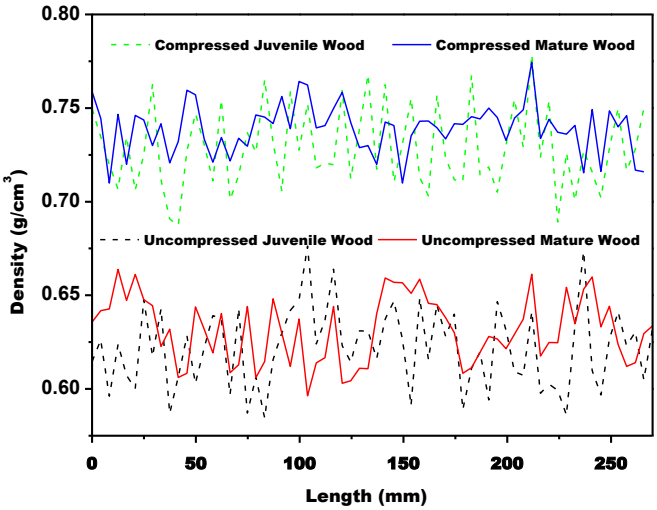

Fig. 4. Wood micro density of compressed alkali-treated and the normal uncompressed juvenile and mature ash specimens

Microfibrillar angles were significantly decreased in juvenile and mature wood after alkaline treatment (Table 2). Juvenile wood of normal specimens had a larger microfibrillar angle and displayed lower anti-compression strength when longitudinally compressed because of the higher energy absorption (Reiterer *et al.* 2001; Yoshihara 2009). However, the microfibrillar angle of juvenile wood was smaller than that of mature wood after alkaline treatment; micro-fibrillar angle is a main influencing factor of MOE (Gindl and Schoberl 2004; Nairn 2006); thus, compression of juvenile wood was

difficult because of the smaller microfibrillar angle. Therefore, the microfibrillar angle had a higher contribution to the longitudinal compression influence than the wood density in the same wood species. Wood chemical compositions and cellulose crystallinity were tested in order to explain the changes in the microfibrillar angle of juvenile and mature wood.

Table 2. Microfibrillar Angle of Alkali-treated and Untreated Juvenile and Mature Ash Wood

Specimens	Untreated (°)	Treated (°)	Variation Rate (%)	ANOVA
Juvenile wood	11.69	9.17	21.56	***
Mature wood	10.99	9.24	15.92	***

ANOVA is the analysis of variance between alkali-treated and untreated specimens. ***$p < 0.001$

Wood Chemical Composition Analysis

Table 3 shows results of the chemical compositions of juvenile and mature wood before and after alkaline softening treatment. Both juvenile and mature wood's relative lignin, hemicellulose, nitrocellulose, and extractive contents decreased after alkaline treatment. However, juvenile wood underwent a more obvious change rate than mature wood. Alkali treatment caused the extractive content to decrease proportionally at large amplitudes. Juvenile wood has higher lignin content but lower cellulose and hemi-cellulose content than mature wood. On the contrary, the percent change of lignin content, cellulose content, and hemicellulose content of juvenile wood was larger than that of mature wood. When the ash specimens were dipped into alkali solution, the contents of hemicellulose, lignin, and cellulose in the amorphous region and parts of cellulose in the crystalline region were degraded (Venkateshappa *et al.* 2010). Thus, it can be concluded that the cellulose amorphous region of juvenile wood was larger than that of mature wood.

The hydration effect of sodium causes the easy penetration of water into the amorphous region of microfibers, which caused more active peeling reaction of juvenile wood. The absence of hemicellulose and lignin exposed cellulose in the alkali solution; then cellulose was reacted with the sodium hydroxide and generated alkali cellulose. When the alkali cellulose combined with sodium, water penetrated the cellulose molecules, which accelerated the swelling of cellulose and weakened the inner binding force among the amorphous region of cellulose. The polar functional groups of C=O and $-NH_2$ in urea easily formed hydrogen groups with cellulose molecules, which increased the polarity of the cellulose molecule, thus increasing the swelling or even the dissolution of cellulose (Han *et al.* 2008; Zhang 2011; Zhou and Zhang 2000). The existing water molecules also induced microcellulose fiber rearrangement in the amorphous region, which decreased the microfibrillar angle and produced more spaces between microfiber chains, making the structure less dense and less rigid (Nishino *et al.* 1995; Pickering *et al.* 2007).

The more lignin, hemicellulose, and extractive contents that were decreased in juvenile wood, the better the arrangement that was in the amorphous region. So the microfibrillar angle of juvenile wood has a larger variation rate than mature wood. On the other hand, the lignin and hemicellulose contents of juvenile wood were still larger than those of mature wood after alkaline treatment. The cross-linked lignin and hemicellulose will also increase the resistance during longitudinal compression (Hatakeyama and Hatakeyama 2010; Navi and Stanzl-Tschegg 2009; Terashima *et al.* 2009).

Table 3. Chemical Composition Changes of Ash Specimens Before and After Alkaline Treatment

Wood chemical composition test		Untreated specimens indexes (%)	Treated specimens indexes (%)	Percent change (%)
Moisture content	Juvenile wood	5.94	5.85	1.52
	Mature wood	5.81	5.98	-2.93
Lignin content	Juvenile wood	27.37	25.52	6.76
	Mature wood	25.20	23.85	5.36
Nitrocellulose content	Juvenile wood	48.25	46.57	3.48
	Mature wood	49.81	48.24	3.15
Hemicellulose content	Juvenile wood	28.49	27.12	4.81
	Mature wood	29.95	29.21	2.47
Extractive content	Juvenile wood	2.85	0.88	69.12
	Mature wood	2.21	0.72	67.42

Under the longitudinal compression stress, folds formation in the elastic-plastic region were observable in Fig. 3, which is an indication that slipping or curling could have occurred in wood cellulose chains. The schematics of these compression-related phenomena are shown in Fig. 5.

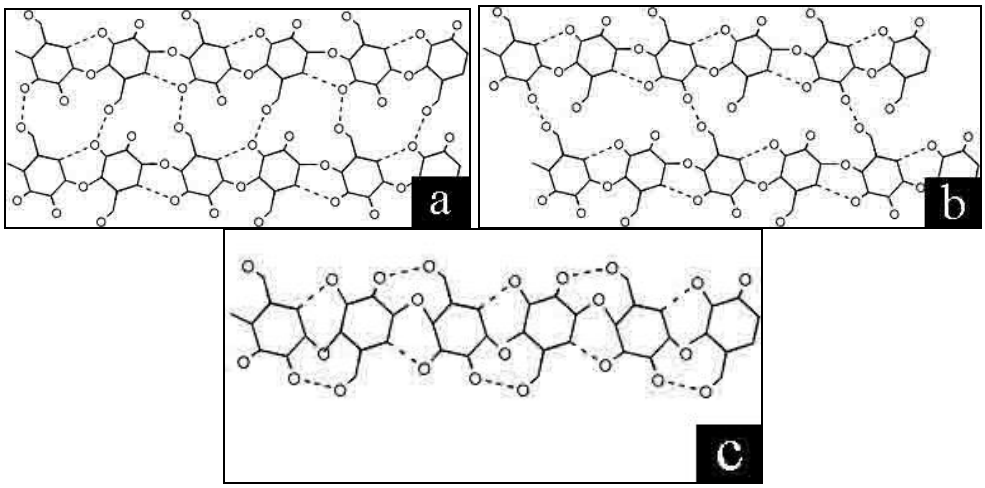

Fig. 5. Changing schematics of wood cellulose chains before and after longitudinal compression: (a) Uncompressed cellulose chains; (b) Slipping among cellulose chains; and (c) Curling within a cellulose chain

The distance between the swelled cellulose chains was increased. When longitudinal compressive load was applied, slipping occurred between cellulose chains; the old hydrogen bonds broke and new hydrogen bonds formed with a movement in position (Zhang 2011), as shown in Fig. 5(b). With hemicellulose degradation and dissolution, the distance between cellulose chains decreased and the hydrogen bond strength increased (Korpela 2002). Curling within a cellulose chain (Fig. 5(c)) also occurred. The distance decreased, and new hydrogen bonds were formed between each D-

glucose elements (Hinterstoisser *et al*. 2003; Zhang 2011); at the same time, the angle of β-1,4-glycosidic bonds increased, which increased the unsteadiness. Immediately after depressurization, parts of the unstable tension originating from the increased bond angle were released. Parts of new chemical bonds were broken until a balance was achieved between new hydrogen bond energy and unstable inner bond angle tension. Permanent plastic deformation occurred for both juvenile and mature wood. The higher hemicellulose and lignin degradation of juvenile wood led to more spaces. Subsequently, more hydrogen groups were exposed and new hydrogen bonds formed and deformed between cellulose chains.

XRD Analysis

With the absence of hemicellulose and lignin, the microfibers rearranged and new chemical bonds were formed. Consequently, the proportion of the cellulose amorphous region decreased and the cellulose relative crystallinity increased. This finding was confirmed by the XRD results in Table 4. The relative crystallinity of juvenile and mature wood significantly increased after alkaline treatment. Due to the greater reduction of lignin, hemicellulose, and extractive in juvenile wood compared to mature wood, more spaces appeared among the juvenile cellulose amorphous region, and then more hydrogen bonds reformed between the cellulose chains. Therefore, the juvenile wood variation rate was much higher than that of mature wood. Figure 6 shows the XRD pattern of juvenile and mature wood.

Table 4. The Relative Crystallinity Degree of Ash Specimens Before and After Alkaline Treatment

Specimens	Untreated (%)	Treated (%)	Variation Rate (%)	ANOVA
Juvenile wood	36.71	54.64	48.84	***
Mature wood	41.01	55.69	35.80	***

ANOVA is the analysis of variance between alkali-treated and untreated specimens. ***p＜0.001

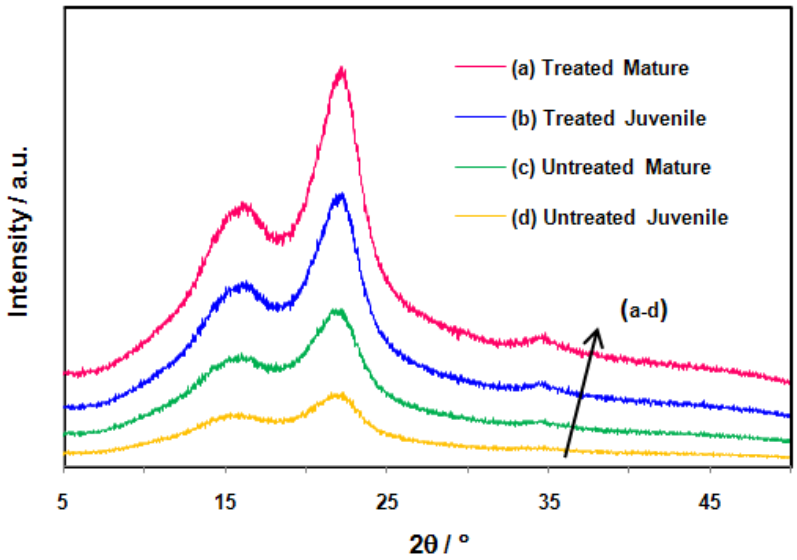

Fig. 6. XRD pattern of juvenile wood and mature before and after alkaline treatment

The relative crystallinity of both types of wood increased without crystal transformation, and all the curves showed the strongest peak near 22.4°. Juvenile wood had a higher proportion of cellulose amorphous region (Li 2002); thus, more hydrogen bonds reformed with the rearrangement of cellulose chains because there was more degradation of lignin and hemicellulose (Nishino *et al.* 1995). Microscopically, the length of the crystallinity region increased. In Table 4, the variation rate of juvenile wood was larger than that of mature wood. Cellulose was the main source of strength (Li 2002); therefore, the greater change of microcellulose crystallinity contributed to the larger increase of stiffness in juvenile wood. But the cellulose crystallinity of juvenile wood and mature wood was approximately the same after alkaline treatment. Macroscopically, the cellulose crystallinity should not be an important factor contributing to the higher MOE of juvenile wood during longitudinal compression.

CONCLUSIONS

1. The longitudinal compression stress-strain relationship of both juvenile and mature ash wood treated with alkali consists of two regions, namely, elastic and plastic regions, with less stress required after alkaline treatment during longitudinal compression. The folds were formed in the linear plastic region on the wood cell wall. The wood cell wall was uniformly compressed along the length of samples instead of over-densification at the two ends. The juvenile and mature wood specimens were uniformly compressed longitudinally.
2. Alkali-treated juvenile wood had a higher MOE and proportional ultimate stress δ_p than mature wood. But the standard deviation of juvenile wood was higher than that of mature wood, which revealed the instability of alkali-treated juvenile wood.
3. The hemicellulose and lignin contents of alkali-treated juvenile wood in the amorphous region were drastically decreased, leading to larger spaces between microfibers, rearrangement of microfibers, and formation of new hydrogen bonds between microfibers. Consequently, the microfibrillar angle of juvenile wood decreased and the relative crystallinity increased at a larger degree of variation than that of mature wood.

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