Comparative Analysis of Longitudinal Compressive and Bending Properties of Hydrothermal-Treated Juvenile and Mature Elm Wood

Da Tong, Yan Zhang, and Kuiyan Song*

The longitudinal compressive and multi-directional bending properties after hydrothermal treatment of juvenile and mature elm wood were analyzed. Wood chemical composition and X-ray diffraction analyses were conducted in order to investigate the different properties of the juvenile and mature wood. Scanning electron microscopy was used to observe the wood's microstructure during longitudinal compression. The results indicated that both juvenile and mature wood could bend multi-directionally and that their relative cellulose crystallinities increased after hydrothermal treatment. The hydrothermal-treated juvenile wood contained more hemicellulose with unstable net-linked polysaccharide and condensed lignin, higher relative crystallinities degree than did mature wood, and more spaces formed by the extractive separation of mature wood. The longitudinal compressive and bending performances of the juvenile wood were worse than those of mature wood. The relationship between variations of stress and strain was separated into two stages, both of which displayed linear increases. However, the stage after the proportional ultimate stress increased slowly and smoothly, confirming the formation of some folds in the wood cells.

Keywords: Elm; Longitudinal compression; Multi-directional bending; Variation of stress and strain

*Contact information: Key Laboratory of Bio-based Material Science and Technology, Ministry of Education, Northeast Forestry University, Harbin, Heilongjiang Province, 150040 P. R. China; *Corresponding author: skuiyan@126.com*

INTRODUCTION

Given its unique characteristics, solid wood typically can bend only in one direction and with a relatively large curvature radius before breakage (Song and Li 2009). The critical radius of curvature can be defined as the maximum radius of circular arc coinciding with the tightest part of the curve before the wood breaks. In industry, when bending to a specified extent, only 70 to 75 percentage of products have been able to achieve the desired levels of bending without breakage. For this reason, the traditional bending technology adds manufacturing costs while reducing the utilization rate of timber, slowing the development and production of curved wooden articles. The technology of longitudinal compression can improve the Single-directional bending performance of wood and make it multi-directional.

Solid wood cannot be longitudinally compressed without a suitable softening treatment. Hydrothermal treatment is considered one of the most effective softening methods for wood modification (Furuta *et al.* 2010; Hamdan *et al.* 2000; Tjeerdsma and Militz 2005). Water is a polar molecule that can easily penetrate into the wood cell wall and interact with the hydroxyl groups on the D-glucose-based pyranoid rings of cellulose non-crystalline regions and the hydroxyl groups on the hemicellulose, increasing the distance between the molecular chains and providing a bigger space for molecular motion (Navi *et al.* 2002). Upon absorption of water, the wood matrix is rearranged so as to increase the Young's modulus (Obataya *et al.* 1998). Moreover, high temperatures increase the energy of the molecular thermal motion and thus the possibility of forming new chemical bonds. A wood cell wall consists of the skeletal structure of cellulose and the amorphous matrices of hemicellulose and lignin (Li 2002). Inagaki *et al.* (2010) observed a decrease in the distance between microfibrils and an increase in the thickness of the microfibrillar crystalline region after the application of a hydrothermal treatment. Under moist and increased-temperature conditions, hemicellulose undergoes depolymerization and is cleaved into acetyl groups (Bourgois and Guyonnet 1988; Garrote *et al.* 2002; Tjeerdsma and Militz 2005). Moreover, evidence of lignin condensation has been found in hydrothermal-treated wood (Garrote *et al.* 1999). The hydrothermal softening condition varies depending on the type of timber (softwood or hardwood) because of the differences in structure, lignin content, and hemicellulose composition (Assor *et al.* 2009; Furuta *et al.* 2010; Hamdan *et al.* 2000; Nakajima *et al.* 2009). The structural characteristics of the three major chemical components of wood significantly decrease the glass transition temperature, with water acting as a plasticizer (Horvath *et al.* 2011). The mechanical properties of timber change rapidly with the manipulation of moisture and temperature (Kelley *et al.* 1987). The visco-elastic behavior of solid wood cannot completely recover after longitudinal compression (Penneru *et al.* 2006; Zhou *et al.* 2010). Thus, studies on the relationship between the permanent deformation rate (PDR) of the elastic recovery of wood and on the success rate (the percentage of specimens achieved a specified bending extent without breakage) and relatively low values of curvature radius sufficient for breakage by bending during longitudinal compression are beneficial for examining the longitudinal compression technique. Juvenile wood has a shorter fiber length and larger helix and microfiber angles in its wood cell walls than does mature wood, and thus it has lower stiffness and strength and is more easily deformed (Gorišek and Torell 1999; Passialis and Kiriazakos 2004). This study analyzes the different longitudinal compressive and bending properties of juvenile and mature wood.

EXPERIMENTAL

Materials

The experimental elm wood (*Ulmus pumila* Linn.) was felled at the breast-height of trees with an average DBH (diameter at breast height) of 24 to 26 cm in the Maoershan forest region located at E127°18′0″ to 127°41′6″, N45°2′20″ to 45°18′16″, Northeast China. This kind of elm wood is moderately hard and strong. To compare the properties of juvenile and mature wood, the samples were cut to sizes of 270 mm (longitudinal) × 16 mm (tangential) × 16 mm (radial). The juvenile wood was cut near the pith, while the mature wood was cut near the bark.

Hydrothermal Treatment

The juvenile and mature wood samples were immersed in boiling water. The optimized softening methods A, B, and C, which were identified based on previous research findings, are shown in Table 1.

Table 1. The Softening Conditions of Elm Samples

Softening Condition	Experiment A	Experiment B	Experiment C
Hydrothermal Treatment Time (min)	100 to 110	120 to 130	140 to 150

Longitudinal Compression

Juvenile and mature wood samples were compressed using a universal mechanical testing machine with a mold (to avoid bending during compression) at a speed of 3 mm/min. For each compression ratio, five juvenile and five mature wood samples were respectively compressed. The mold is shown in Fig. 1. Samples with rubber shims around them were settled into the steel mold and were properly nipped with regulative bars and screws. Then longitudinal stress was given using a compression bar with cross section of 15.8 mm × 15.8mm. The proportional limits of stress, the maximum stress, and the PDR values of longitudinal compression were tested.

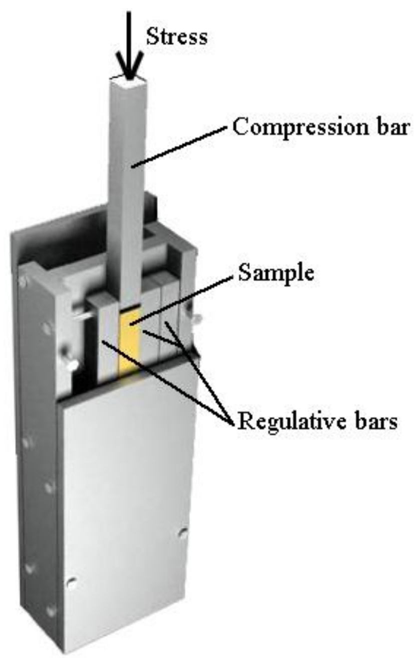


Fig. 1. Schematic version of the compression mold

Single- and Multi-directional Bending

After compression, the juvenile and mature elm wood samples were bent using the universal mechanical testing machine in order to test the minimum curvature radius of Single-directional bending to breakage without a mold and multi-directional bending with a mold.

Analysis of Chemical Composition of Wood

The content variations in lignin, holocellulose, and hot water extractions before and after the softening treatment were tested according to the Chinese Standard GB-2677

(Jiang *et al*. 2004; Wei *et al*. 2008; Zhou *et al*. 2009). The cellulose content was tested using the nitric acid-ethanol method (Tan *et al*. 2008). The hemicellulose content was determined as the holocellulose content without the nitrocellulose content. Each component content was a percentage composition from an individual process following the above standard and method. The wood material is comprised of cellulose, hemicellulose, lignin, and extractives, in addition to moisture, among which cellulose was evaluated by use of the nitrocellulose content. The variation extent was calculated as follows:

$$Variation\ extent = \frac{Untreated\ sample\ content - Treated\ sample\ content}{Untreated\ sample\ content} \times 100\% \quad (1)$$

X-ray Diffraction (XRD) Analysis

An X-ray diffractometer (Model: D/MAX2200), which was manufactured by the Japanese Rigaku Corporation, was used to determine the relative cellulose crystallinity of the treated and untreated samples. The test conditions were as follows: scanning angle 3° to 60°, speed 4°/min, step length 0.02°, voltage 40 kV, and current 30 mA. The elm samples had sizes of 100 to 120 mesh. The data were calculated using the Segal method (Segal *et al*. 1959; Troedec *et al*. 2008) as follows,

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

where C_rI is the percentage of relative crystallinity, I_{002} is the highest intensity of the lattice diffraction angle (approximately 22°), and I_{am} is the scattering intensity, where 2θ is close to 18° at the non-crystalline background.

Scanning Electron Microscopy (SEM) Structural Observation

Scanning electron microscopy (SEM) structures of the compressed and uncompressed samples were observed using a JSM-5610LV scanning electron microscope (JEOL Company, Japan). The average moisture content of the compressed and uncompressed samples was identified at 8.15% in order to avoid size changes due to water absorption. Slice testing was conducted from the centre of the compressed and uncompressed samples.

RESULTS AND DISCUSSION

Longitudinal Compression Ratio and PDR

The test data in Table 2 indicated that when softening method A was adopted, in the process of longitudinal compression, the longitudinal compression ratio reached 21% and the average PDR values of the juvenile and mature wood samples were 3.66% and 3.59%, respectively. When the longitudinal compression ratio was increased to 22%, the longitudinal compression success rates of the juvenile and mature wood samples reached 20% and 60%, respectively. Meanwhile, when the longitudinal compression ratio was increased to 23%, both the juvenile and mature wood samples failed to become compressed because of the irregular deformation or drastic decrease of mechanical strength.

When softening method C was used, only the mature wood samples were compressed to 21%, and the average PDR reached 4.64%, while the juvenile wood samples were crushed. When the juvenile wood samples were compressed to 80% of their original length, the success rate of longitudinal compression was 60%; however, when the mature wood samples were compressed to 78% of their original length, the success rate of the mature wood reached 40%.

When method B was used, the longitudinal compression ratio reached 23%, and the juvenile and mature wood samples obtained average PDR values of 5.26% and 5.23%, respectively. When the longitudinal compression ratio was increased to 24%, the success rates of the juvenile and mature wood samples reached 20% and 60%, respectively.

Table 2. Longitudinal Compression Ratio and PDR

Compre-ssion Ratio %	Compre-ssion Values (mm)	Samples Part	Softening Method A			Softening Method B			Softening Method C		
			PDR %	S (mm)	CV %	PDR %	S (mm)	CV %	PDR %	S (mm)	CV %
19	51.30	Mature	3.01	0.21	2.59	4.01	0.12	2.99	4.04	0.36	8.91
		Juvenile	3.08	0.35	4.21	4.07	0.29	7.13	4.06	0.56	13.80
21	56.70	Mature	3.59	0.28	2.89	4.47	0.32	7.16	4.64	0.25	5.39
		Juvenile	3.66	0.59	5.98	4.54	0.54	11.90		—	
23	62.10	Mature		—		5.23	0.29	5.55		—	
		Juvenile		—		5.26	0.49	9.32			

S = the standard deviation; CV = coefficient of variation; "—" indicates the longitudinal compressed samples did not achieve success and the blank means the longitudinal compressive proceeding was not measured.

As can be seen in Table 2, the mature wood generally had a higher success rate than did the juvenile wood, while the average PDR value of the juvenile wood samples was slightly higher than that of the mature wood samples. However, the variation coefficient of the juvenile wood samples was higher than that of the mature wood samples, indicating that the PDR value of the juvenile wood samples after longitudinal compression was unstable. The analysis above showed that, aside from the softening methods and the compression ratio, the effects of the longitudinal compression also depended on the properties of the wood.

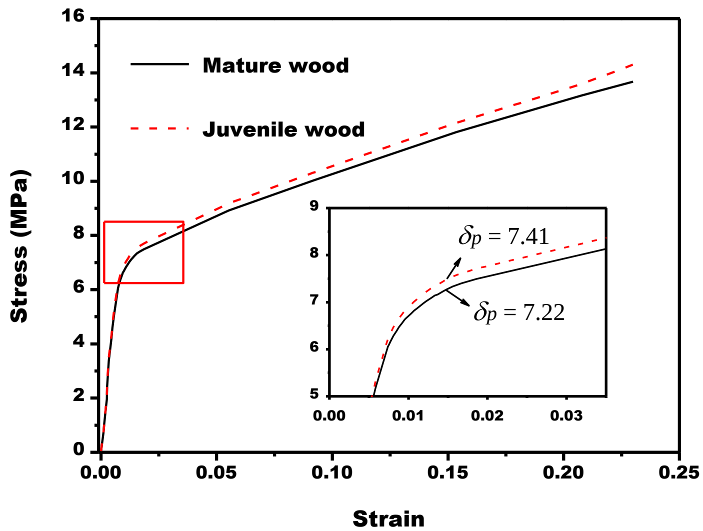
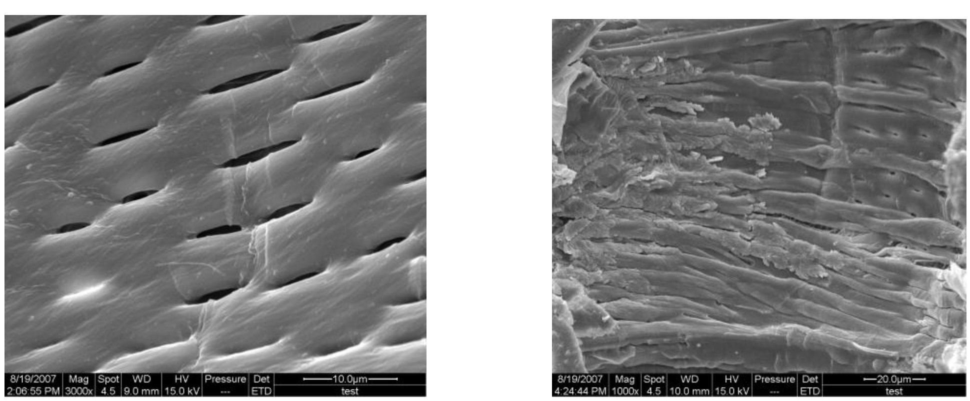


Fig. 2. Relationship between longitudinal compressive stress and strain after hydrothermal treatment

Stress and Strain Variations

After application of softening method B, juvenile and mature elm wood samples were selected to be tested for maximum longitudinal compressive properties. The stress and strain variations of the samples were also subsequently analyzed.

Figure 2 shows the relationship between stress and strain variations. As can be seen in Fig. 2, at the early stage of longitudinal compression, the stress increased significantly in a linear manner with the increase in strain. At δ_p (proportional ultimate stress), the stress of hydrothermal-treated juvenile and mature wood increased slowly and smoothly, indicating the formation of some folds in the wood cells. It can be inferred that the folds influenced stress and strain, resulting in the difference between stress and strain in non-softening longitudinally compressed wood. Figure 3 confirms such an inference. After longitudinal compression, the pits on the cell wall became long and thin, and some folds appeared.



(a) (b)

Fig. 3. Compressed elm wood showing the wall of vessel (a) Radial section; (b) Tangential section

As can be seen in Table 3 and Fig. 2, the modulus of elasticity (MOE) and stress of the juvenile wood were higher than those of the mature wood. The test results indicated that the stress and longitudinal compression requirements, including the deviation, became higher as the sampling locations became closer to the wood pith. In the elastic compressive region, the standard deviations of the stress and strain of juvenile wood were higher than those of mature wood. Moreover, in each region, the relational correlation of juvenile wood was lower than that of mature wood.

Table 3. The Main Mechanical Indicators of Elm Samples after Longitudinal Compression

Samples Part	MOE (MPa)	Proportional Ultimate Stress and Correlation Coefficient		Maximum Stress and Correlation Coefficient	
		σ_P (MPa)	γ	σ_S (MPa)	γ
Mature	831.28	7.22	0.9857	13.83	0.9943
Juvenile	888.08	7.41	0.9808	14.43	0.9923

Single- and Multi-directional Curvature Radii

From the effects of the softening, it was found that the longitudinal compression ratio and the PDR were smaller for the samples that adopted methods A and C than those for the samples that adopted method B. When method B was used along with the same longitudinal compression ratio, a smaller minimum curvature radius to breakage was obtained despite the PDR value of the juvenile wood samples being higher than that of the mature wood samples. However, the mature and juvenile wood samples had minimum curvature radii of 48 and 49 mm, respectively, and average minimum curvature radii of 50.2 and 53.6 mm, respectively, with large standard deviations and variation coefficients, as shown in Table 4. When softening method A was used, the single and multi-directional curvature radii of both wood samples reached their maximum values. However, the curvature radius was at a minimum when method B was used. During the subsequent bending process, the folds on one side of the wood cell wall gradually flattened, resulting in a smaller curvature radius and the probability of multi-directional bending. In summary, the curvature radii of the mature wood samples were smaller than those of the juvenile samples.

Table 4. Bending Curvature Radii of the Hydrothermal-treated and -Untreated Elm Samples

Test Items	Samples Part	Untreated			Softening Method A			Softening Method B			Softening Method C		
		Mean (mm)	S (mm)	CV %	Mean (mm)	S (mm)	CV %	Mean (mm)	S (mm)	CV %	Mean (mm)	S (mm)	CV %
Single-directional Curvature Radius	Mature	72.8	0.26	0.35	55.8	0.19	0.34	50.2	0.13	0.26	53.6	0.15	0.28
	Juvenile	75. 6	0.27	0.36	57.2	0.22	0.38	53.6	0.20	0.37	56.8	0.28	0.48
Multi-directional Curvature Radius	Mature		—		42.8	0.15	0.36	39.2	0.13	0.32	41.5	0.16	0.38
	Juvenile				44.6	0.18	0.41	40.8	0.15	0.36	43.8	0.19	0.44

The Single-directional bend was processed without a mold, while the multi-directional bend was processed with a mold. "—" indicates the longitudinal compressed samples did not achieve success. The mean is the arithmetic mean of sample values. S = standard deviation; CV = coefficient of variation.

Analysis of Chemical Composition of Wood

The chemical components of the juvenile and mature elm wood samples before and after hydrothermal treatment were analyzed for a high compressive MOE and for the large minimum curvature radius for breakage of juvenile wood. In Table 5, it is demonstrated that the lignin content and the moisture content were almost always increased by all three treatments. However, hemicellulose, nitrocellulose, and hot water extractive contents both of juvenile and mature wood samples were decreased, especially hot water extractive after hydro-thermal treatment. Water acted as a plasticizer and penetrated into the wood cell wall, which caused swelling of the wood. Lignin underwent *β*-O-4 bond cleavage and condensation (Assor *et al.* 2009; Kutnar and Šerek 2008; Mamoňová *et al.* 2002), which rendered it more cross-linked and less mobile. Hydrothermal treatments, for both juvenile and mature wood samples, increased the lignin contents, mainly due to extensive loss of hemicellulose and extractive content. The heterocyclic ether bonds of hemicellulose were sufficient to be the most susceptible ones, which in turn led to oligosaccharide generation and acetyl groups splitting from the hemicellulose fraction (Garrote *et al.* 1999, 2001). The hemicellulose content of juvenile wood samples was higher even with a larger degradation amount than that for mature ones. Moreover the hemicellulose consisted of saccharidic molecular units with many side chains, which increased the net-linked structures. Thus, the process of juvenile wood longitudinal compression was more difficult than that for the mature wood. The extractive content of the juvenile wood samples was higher than that of the mature wood samples; however, the extractives of mature wood samples decreased at a larger rate, which could indicate that more spaces were formed between the celluloses units in the amorphous region, and less restraint existed during longitudinal compression. Slippage took place (Hofstetter *et al.* 2005) more easily in mature wood samples. Therefore, juvenile wood samples were harder to compress compared to mature wood samples after hydrothermal treatment. The net-linked structure of lignin and hemicellulose increased juvenile wood samples' instability of PDR during longitudinal compression. Similarly, a larger curvature radius of juvenile wood was found in the subsequent bending process.

In general, cellulose acts as the main reinforced structure of wood. It seems that mature wood samples should be difficult to longitudinally compress due to the higher crystalline cellulose. The relative crystallinities of cellulose are a significant impact factor to wood samples; thus X-ray diffraction tests were conducted as follow.

Table 5. Changes in Chemical Composition of the Hydrothermal-treated and -Untreated Elm Samples

Wood Chemical Composition Test		Untreated %	Softening Method A		Softening Method B		Softening Method C	
			Index %	Variation Extent %	Index %	Variation Extent %	Index %	Variation Extent %
Moisture Content	Mature	5.63	5.45	3.20	5.85	-3.91	5.74	-1.95
	Juvenile	5.53	5.74	-3.80	5.62	-1.63	5.61	-1.45
Lignin Content	Mature	26.42	26.63	-0.79	27.28	-3.26	27.88	-5.53
	Juvenile	24.39	24.78	-1.60	25.15	-3.12	25.35	-3.94
Nitrocellulose Content	Mature	45.53	44.87	1.45	44.43	2.42	45.27	0.57
	Juvenile	44.54	43.82	1.62	43.49	2.36	43.33	2.72
Hemicellulose Content	Mature	37.08	36.43	1.75	35.84	3.34	35.76	3.56
	Juvenile	39.00	37.82	3.03	37.59	3.62	37.37	4.18
Hot water Extractive Content	Mature	1.73	1.14	34.10	0.71	58.96	0.54	68.79
	Juvenile	2.33	1.63	30.04	1.02	56.22	0.78	66.52

XRD Test

Results of XRD analysis (Table 6 and Fig. 4) showed that the relative crystallinities of both the juvenile and mature wood samples were enhanced without crystal transformation, as confirmed by the unchanged diffraction peaks near 22°20' (Li 2009). This enhancement was caused by the rearrangement of cellulose chains and reformation of hydrogen bonds between cellulose chains in the amorphous region with lignin concentration and hemicellulose degradation. Moreover with the increased processing, hydrogen bonds were rearranged, and new hydrogen bonds were formed, extending from the initial crystalline domains. These new hydrogen bonds increased the length of the crystalline region, consequently increasing the strength of the diffraction peaks. However, the crystallinity of the juvenile wood samples increased significantly after hydrothermal treatment. Therefore, compared with mature wood, juvenile wood contained a higher MOE and higher stress during the longitudinal compression and had larger minimum curvature radii for breakage after hydro-thermal treatment.

Table 6. The Relative Degree of Crystallinity of the Hydrothermal-treated and -Untreated Elm Samples

Samples Part	Untreated %	Softening Method A %		Softening Method B %		Softening Method C %	
		Relative Crystallinity	Changing Rate	Relative Crystallinity	Changing Rate	Relative Crystallinity	Changing Rate
Mature	39.99	40.27	0.70	40.43	1.10	40.55	1.40
Juvenile	37.83	39.58	4.63	40.64	7.43	40.89	8.09

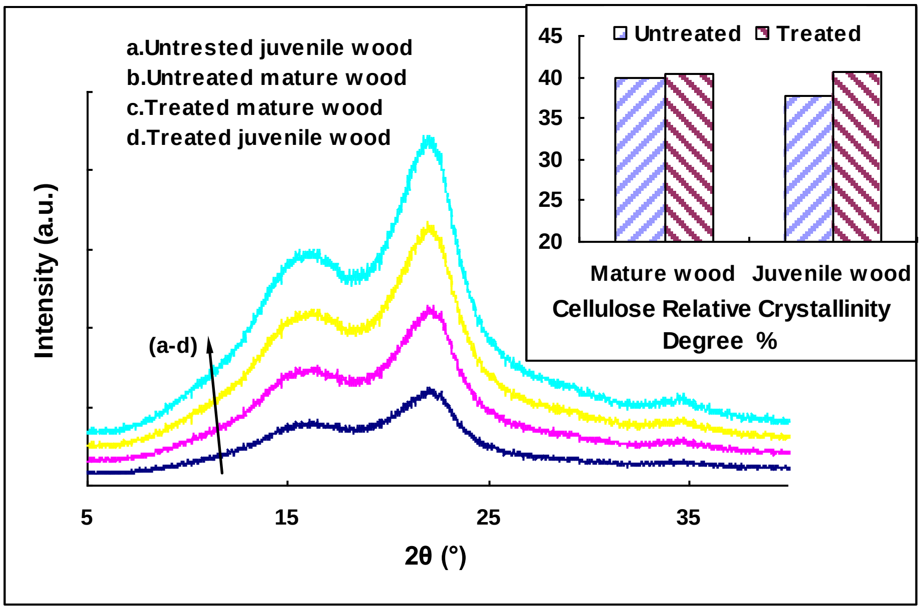


Fig. 4. XRD pattern of juvenile and mature elm wood before and after hydrothermal treatment

CONCLUSIONS

1. The relative crystallinities of the hydrothermal-treated juvenile and mature elm wood samples increased, the hemicellulose underwent degradation, the lignin became highly concentrated and remained in a viscous state at high temperatures, and extracts were produced. Under such conditions, the ratio of longitudinal compression to PDR increased significantly. Moreover, the minimum values of single- and multi-directional curvature radii before breakage decreased significantly.
2. Hydrothermal-treated juvenile wood was more difficult to compress longitudinally and bend multi-directionally than was mature wood. These phenomena can be attributed to the net-linked structures of condensed lignin and abundant side chains of hemicellulose and higher relative crystallinity in hydrothermal-treated juvenile wood compared to hydrothermal-treated mature wood.
3. The stress-strain relations indicated that both the juvenile and mature wood samples displayed elastic-like deformation and exhibited a linear relationship during the initial stage of longitudinal compression. After hydrothermal treatment, proportional

ultimate stress δ_p, stress, and strain slowly and smoothly increased in a linear manner, as a consequence of the formation of folds in the wood cells.

ACKNOWLEDGMENTS

The authors are grateful for the support of the National Natural Science Foundation of China, Grant. No. 31070489 and the Technological Project in Heilongjiang Province, Grant. No. GA09B202-07.

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Article submitted: July 29, 2012; Peer review completed: Sept. 25, 2012; Revised version received: Nov. 24, 2012; Accepted: Nov. 25, 2012; Published: November 29, 2012.