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EFFECTS OF MEDIUM-LOW TEMPERATURE HYDROTHERMAL TREATMENT ON MICROSTRUCTURE AND DIMENSIONAL STABILITY OF CHINESE SWEETGUM WOOD

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ABSTRACT

To investigate the changes of microstructure and dimensional stability during hydrothermal treatment, the Chinese sweetgum (*Liquidambar formosana* Hance) wood samples were treated in a numerical show constant temperature water bath with temperature of 60, 80 and 100°C for 4 h. The dry shrinkage rate and water absorption of untreated and treated samples were measured. Scanning electron microscopy (SEM) was selected to observe and investigate the changes of wood microstructure, which caused by hydrothermal treatment. The results showed that dry shrinkage rate increased from 4.92% to 7.00% and 9.62% to 10.12% in tangential direction and radial direction, respectively. However, the shrinkage rate difference (SRD) as an index to evaluate possibility of wood deformation, decreased from 1.96 to 1.45, which meant the shape stability of treated samples improved. The water absorption increased from 93.15% to 112.11%. From the results of SEM, the most sediment on aspirated pits were removed and pit membrane was ruptured after treatment. It had positive effect on moisture migration and wood permeability. It is maybe the reason of the variation of water absorption and dry shrinkage rate.

KEYWORDS: Chinese sweetgum, medium-low temperature, hydrothermal treatment, microstructure, dimensional stability.

INTRODUCTION

Chinese sweetgum (*Liquidambar formosana* Hance) as an important fast-growing native tree species has many advantages of short growth cycle, rich growing stock, and delicate structure. It can be used for construction, furniture, decoration and other wood product. However, its shortcoming of interlocked grain, uneven shrinkage, hard to dry and low shape stability have negative effects on industrial utilization. It is urgent to be improved by some efficient techniques. Hydrothermal treatment is one of the recent technologies used to improve the durability, wood permeability and dimensional stability of wood (Ding et al. 2017, Esteves and Pereira 2009, Raul Pelaez-Samaniego et al. 2013, Yao et al. 2018).

During hydrothermal treatment, the changes in both the chemical composition and components' ratio, as well as wood microstructure changes, occur simultaneously (Jiang et al. 2017, Reinprecht et al. 2013, Skyba et al. 2009, Unsal et al. 2010). Biziks et al. (2013) treated the birch wood (*Betula pendula*) samples in a thermal regime (140, 160, 180°C) for 1 h and investigated the changes of microstructure by means of scanning electron microscopy (SEM). Significant changes of areas and linear sizes of the birch wood cells were achieved. After treatment at 180°C, voids and cracks are formed between fibers, thus leading to a decline in the mechanical properties of the wood. Rovshandeh et al. (2007) studied the effects of hydrothermal treatment on the dimensional stability of beech wood. The results indicated that best anti-swelling efficiency value was achieved at 170°C (treat temperature) within 1 hour (treat time). Mirzaei et al. (2012) modified the beech wood in a reactor at temperatures of 130 and 150°C for a holding time of 30 minutes. The block shear testing method was applied to determine the bond shear strength of treated wood. The results showed a reduction in bond shear strength and shear strength parallel to grain due to hydrothermal treatment. Saliman et al. (2017) applied response surface methodology to research the effects of hydrothermal treatment parameters on decay resistance of oil palm wood. They found that the weight loss of the treated oil palm wood caused by white rot fungus reduced as the treatment temperature and time increased. Buffered medium showed insignificant effect on improvement of decay resistance of the oil palm wood. Two most common hydrothermal pre-treatments for wood-mild steam explosion and hot water extraction were also applied to treat industrially-cut wood chips of Norway spruce (*Picea abies* (L.) Karst.). The results show that short hot water extraction treatments lead to significant variations in the local composition within the wood chips, while steam explosion accomplishes a comparably more even removal of hemicelluloses due to the adjective mass transport during the explosion step (Wojtasz-Mucha et al. 2017).

These earlier published articles have revealed the efforts of hydrothermal treatment on wood chemical composition and components' ratio and microstructure changes, etc. (Jose-Vicente et al. 2016, Percin et al. 2016). In most industrialized processes today, heat treatment involves temperatures between 150 and 260°C for times ranging from a few minutes to several hours.

In this study, a medium-low temperature hydrothermal treatment was applied as pre-treatment for Chinese sweetgum wood drying process, to improve the drying performance and drying speed. SEM was used as a suitable method for observing the microstructure changes and investigating the effects of pre-treatment parameters on microstructure changes. The effects of medium-low temperature hydrothermal treatment on shape stability, dry shrinkage rate and water absorption were also investigated in this paper.

MATERIALS AND METHODS

Materials

The age of Chinese sweetgum tree used to make samples was about 20-24 years old and harvested in Xuancheng, Anhui, China. All the samples were defect-free and cut to the size of 200×100×20 mm (L×T×R).

Experimental setup and procedure

The hydrothermal treatment was carried out in a numerical show constant temperature water bath. Treatment temperatures were stated as 60, 80 or 100°C. The mentioned medium-low temperature hydrothermal treatment consisted of three technological stages:

1. temperature increase up to the stated treatment temperature (60, 80 or 100°C),
2. holding at the stated treatment temperature for 4 hours,
3. cooling.

Then, the dry shrinkage rate, water absorption and microstructure changes of Chinese sweet gum wood were tested as follows.

Dry shrinkage rate testing

The specimens for dry shrinkage rate testing with the size of 20×20×20 mm were cut from the raw samples (200×100×20 mm). Then, these small specimens were sorted into 3 treatment groups and 1 control group, and 20 samples were tested for each group. Dry shrinkage rate (SR) and dry shrinkage rate difference (SRD) of each sample were compared and calculated according to the following Eqs. 1 and 2:

$$SR = \frac{L_0 - L}{L_0} \times 100 \qquad (\%) \tag{1}$$

where: L_0 - the length of samples in saturated-water phase,
L - the length of samples after oven dry.

$$SRD = \frac{SR_T}{SR_R} \tag{2}$$

where: SR_T and SR_R are dry shrinkage rate in tangential direction and radial direction, respectively.

Water absorption testing

The water absorption testing was accomplished according to Chinese standard (GB 1934.1-2009), the values of water absorption were calculated according to the Eq. 3:

$$Water\ absorption = \frac{m_0 - m}{m} \times 100 \qquad (\%) \qquad (3)$$

where: m_0 - the weight of samples in saturated-water phase,
m - the oven dry weight of samples.

Obtaining and investigation of SEM images

The samples were oven dried and coated with gold using a sputter coater and then dried. After drying, the samples were directly placed in a Scanning Electron Microscope (SEM) chamber, and cross-section micro images of treated and untreated Chinese sweetgum wood samples were obtained by using an SEM (FEG-XL30, FEI, America). All the cross-section micro images were viewed at 3500 magnifications.

RESULTS AND DISCUSSION

The samples for these four groups were tested and the results of dry shrinkage rate and water absorption were shown in Tab. 1.

Tab. 1: Results of dry shrinkage rate and water absorption tests.

Number	Temperature (°C)	SRR (%)	SRT (%)	SRD	Water absorption (%)
1	Untreated samples	4.92	9.62	1.96	93.15
2	60	5.11	9.67	1.89	98.23
3	80	6.02	9.77	1.62	105.12
4	100	7.00	10.12	1.45	112.11

The results indicated that the dry shrinkage rate in tangential direction and radial direction increased with the treated temperature increased. The dry shrinkage rate increased from 4.92% to 7.00% and 9.62% to 10.12% in tangential direction and radial direction, respectively. The reason for this result may be because of the sediment and inclusion dissolved out during medium-low temperature hydrothermal treatment (Zhao 2016). It is can be seen from Fig. 1 and Fig. 2, and the results of this paper also had a good agreement with the results of some published articles (Zhao 2016, Lin 2014). In addition, the decrease of sediment and inclusion in wood has a positive effect on the moisture migration and wood permeability. The result was indicated by the increasing of water absorption. After treatment, the water absorption increased from 93.15% to 112.11% and it increased with the treatment temperature increasing. The SRD as an index to evaluate possibility of wood shape stability, was measured and it decreased from 1.96 to 1.45. It meant that the shape stability of Chinese sweetgum improved after treatment (Aydemir et al. 2011, Kentaro and Hiroyuki 2007). The microstructures of untreated and treated wood samples were shown in Figs. 1 and 2, respectively. The changes were obviously shown in these figures. Most sediment on aspirated pits was removed and pit membrane was ruptured after treatment. It was positive for improving the moisture migration and drying performance.

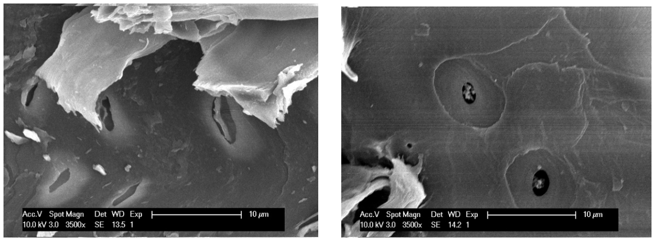

Fig. 1: SEM images of untreated Chinese sweetgum wood.

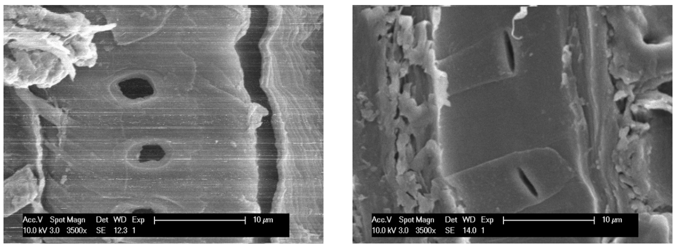

Fig. 2: SEM images of treated Chinese sweetgum wood (treated with the temperature of 100 and time of 4h).

Normally, the hydrothermal treatment has negative effect on the mechanical property of wood (Sandberg et al. 2013). To avoid this defect, the medium-low temperature hydrothermal treatment was applied to wood modification. As a natural fiber material, the morphological and wall cavity ratio of wood fibers have significant effects on mechanical property of wood. In Tab. 2, the results showed that the diameters of fibers did not have significant changes and the wall cavity ratio changed only from 0.38 to 0.41. In a certain extent, the medium-low temperature hydrothermal treatment, which reported in this paper, has negligible effects on the mechanical property of wood.

Tab. 2: Morphological and wall cavity ratio of Chinese sweetgum wood fibers.

Samples	Fibre length (µm)	Fibre width (µm)	Ratio of length to width	Diameter (µm)	Double-wall thickness(µm)	Wall cavity ratio
Untreated	1475.50	23.60	62.52	26.22	9.93	0.38
Treated	1697.30	26.60	63.81	25.95	10.71	0.41

In order to reveal the effect of pre-treatment on drying performance of Chinese sweetgum, the same drying conditions were applied for pre-treated and untreated samples. The average moisture content deviation in thickness direction and the residual stress of pre-treated samples were lower than them of untreated samples (Liu 2017).

CONCLUSIONS

The Chinese sweetgum wood was treated by medium-low temperature hydrothermal treatment in this study and the main conclusions were shown as follows:

1. The shape stability was improved by medium-low hydrothermal treatment. The SRD as an index to evaluate possibility of wood shape stability, decreased from 1.96 to 1.45.
2. The microstructure was changed by treatment. Due to the sediment falling off and inclusion dissolving out, most sediments on aspirated pits were removed and pit membrane was ruptured after treatment. It has positive effect on moisture migration and wood permeability.
3. The diameters of fibers did not have significant changes and the wall cavity ratio changed only from 0.38 to 0.41.

Based on the conclusions above, the medium-low temperature hydrothermal treatment could be applied as a kind of pre-treatment for Chinese sweetgum wood drying process to improve the moisture migration and wood permeability.

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