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MECHANICAL PROPERTIES AND SET RECOVERY OF COMPRESSED POPLAR WITH GLYCERIN PRETREATMENT

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ABSTRACT

In order to improve the mechanical properties of low-density wood, the densified wood was fabricated. Northeast China fast-growing poplar was firstly immersed in 50% glycerin for 24 h, and then compressed under 150°C to attain 60% compression ratio with different thermal modification time (0.5, 1, and 2 h). The set recovery, modulus of elasticity (MOE), modulus of rupture (MOR) and hardness of compressed wood were tested to assess the influence of thermal modification time and wet/dry cycles on mechanical properties and set recovery of compressed poplar with glycerin pretreatment. It can be found that the thermal modification time of 1 h can be more appropriate, the first wet/dry cycle has a significant effect on mechanical properties and set recovery of compressed wood due to the dilution of glycerin during the soaking.

KEYWORDS: Glycerin pretreatment, thermal modification time, wet/dry cycles, mechanical properties, set recovery, poplar.

INTRODUCTION

The mechanical properties of wood, such as the surface hardness, MOE and MOR, are positively associated with its density (Gong et al. 2010, Rautkari et al. 2011). The density of the wood depends on the cell wall thickness and size of lumen, and the densities of cell wall are approximately the same (1500 $kg \cdot m^{-3}$) regardless of the wood species or cell type (Kellogg and Wangaard 1969). Thus, density can be increased by compressing the porous structure in the transverse direction (Laine et al. 2016), when deformations in the densification process are

largely the result of the viscous buckling of cell walls without major fracture taking place (Kutnar et al. 2008). In general, wood cannot be compressed directly, because cracking may occur. Before compression, wood needs to be softened from a rigid state to a thermoplastic flow state (Wang and Huang 2011). After compression, the densified wood has a tendency to return to its original dimensions, which is known as set recovery (Rautkari et al. 2010), therefore, it is necessary to fix permanently the compressive deformation through some pretreatment methods for uncompressed wood (Rezayati Charani et al. 2007, Li et al. 2013) and post-treatment methods for compressed wood (Laine et al. 2013, Navi and Heger 2004).

The melamine formaldehyde resin or phenol formaldehyde resin impregnation is a pretreatment method widly studied (Inoue et al. 1991, Wallstrom and Lindberg 1999, Yano et al. 2000, Ohmae et al. 2002, Inoue et al. 2007b), which contributes to dimensional stability by swelling the cell wall and forming a rigid cross-linked network upon curing. However, the high cost of resin limits its application in the wood densification processes.

The pretreatment with glycerin is also an alternative process to fabricate the densified wood. As glycerin is adsorbed to hydroxyl groups through hydrogen bonding in amorphous region, wood enters in swollen state and the molecules are easy to slip (Yan et al. 2010). The glycerin can accelerate the degradation of wood component, and form a chemical cross-linkage between molecules of the matrix constituents, which contributes to fix deformation of compressed wood (Yan et al. 2011). In addition, the stress relaxation is also aggravated by glycerin (Yan et al. 2011).

The post-treatment at high temperature after densification is one of common processes used to fix the compressive deformation (Rautkari et al. 2010, Esteves et al. 2017), due to the release of stresses stored in the cell wall polymers by their decomposition and the formation of some cohesive structures (Higashihara et al. 2004). The steam post-treatment is being receiving more attention (Ito et al. 1998, Skyba et al. 2009, Fang et al. 2012, Kutnar and Kamke 2012, Rautkari et al. 2013). However, most deal with thin wood samples due to efficient steam injection (Dwianto et al. 1999, Navi and Heger 2004, Inoue et al. 2007a). The closed steam system limits its appication to batch production. High temperature post-treatment in the absence of steam is also effective in reducing compression-set recovery, due to changes in the polar side groups on the molecular structures of cellulose, hemicelluloses, lignin, and extractives (Hillis 1984, Morsing 2000).

Many attempts have been made to develop a suitable process for the densification of different wood species by assessing the improvements in mechanical properties, dimensional stability and durability (Ulker and Burdurlu 2016, Esteves et al. 2017, Kúdela et al. 2017).

In general, the pretreatment with glycerin and thermal post-treatment are used together to reduce set recovery. Though some studies have reported the mechanical properties and set recovery of compressed wood using those two treatment processes (Yan 2010, Cai et al. 2013), few studies focused on the mechanical properties and set recovery of compressed wood exposed to humid or wet condition, which may destroy the cohesive structures formed in hot treatment. Especially, as the compressed wood is soaked in the water, the glycerin may be diluted, which may affect the mechanical properties and set recovery of compressed wood.

This aim of this paper is to investigate the influence of thermal modification time during densification on mechanical properties and set recovery of compressed poplar with glycerin pretreatment. Northeast China poplar was firstly immersed in 50% glycerin for 24 h, then compressed under 150°C to attain 60% compression ratio with different thermal modification time (0.5 h, 1 h, and 2 h). In order to assess the impact of moisture changes, the compressed specimens were subjected to five cycles of water soaking and drying, and their mechanical properties and set recovery were compared with those without bearing environmental changes. An analysis of variance (ANOVA) was used to evaluate statistical significance of the parameters, and the result is considered statistically significant for $p < 0.05$.

MATERIALS AND METHODS

Materials and pretreatment

The clear specimens without knots, cracks, decay and other visible defects were made from Chinese white poplar (*Populus tomentosa*) from northeast China, with a length of 600 mm (longitudinal) and width of 160 mm (tangential). They were firstly placed in an environmental-controlled room (20°C, 65% relative humidity) until equilibrium moisture content of approximately 12% was achieved, and then planned to thickness of 45 mm (radial). The measured wood density was equal to 400 kg·m^{-3}. After that, the specimens were impregnated in 50% glycerin solution at 50°C, which is the optimal concentration to control the set recovery (Yan 2010), for 23 h and at 100°C for 1 h.

Compression process

The specimens were compressed at 150°C with a speed of 10 mm·min^{-1} along the radial direction (Fig. 1) to a target thickness of 18 mm in order to obtain 60% compression ratio (CR), which can be calculated according to Eq. 1. The wood was densified following the process shown in Fig. 2, and then platens were maintained in the same position for 0.5 h, 1 h, and 2 h, respectively, in order to investigate the influence of thermal modification time.

$$CR = \frac{t_I - t_C}{t_I} \times 100\% \quad (1)$$

where: t_I is the original thickness (mm), and t_C is the target compressed thickness (mm).

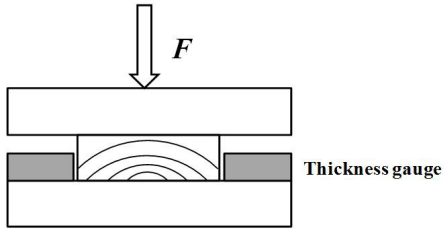

Fig. 1: Schematic diagram of the compression.

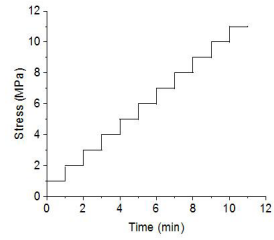

Fig. 2: Schematic diagram of the densification process.

Set recovery

To measure set recovery, the small specimens (50 mm longitudinal, 15 mm tangential) were cut from compressed wood, which had been conditioned in an environment-controlled room (20°C, 65% relative humidity) until equilibrium moisture content was achieved. Then, the specimens were dried in a convection oven at 103°C, and their thickness was measured as t_C (mm). After that, the specimens were soaked in water for 24 h and again oven-dried, and their thickness was measured as t_S (mm). The percentage of set recovery (SR) can be determined using Eq. 2:

$$SR = \left[\left(t_S - t_C \right) / \left(t_I - t_C \right) \right] \times 100\% \quad (2)$$

Wet/dry cycles

In order to assess the impact of moisture changes, a process of wet/dry cycles was performed, in which soaking in water for 24 h and again oven-drying is defined as one wet/dry cycle, and the set recovery and mechanical properties of compressed wood were measured after one, three and five wet/dry cycles.

Mechanical properties

Before testing, all specimens were firstly conditioned in an environment-controlled room (20°C, 65% relative humidity) until equilibrium moisture content was achieved. The three-point bending tests for 6 replicates of specimens were performed to measure the modulus of elasticity (MOE) and modulus of rupture (MOR) of compressed wood according to ASTM D143 – 14 (2015). The specimens (Fig. 3) with 312 mm in length were cut from compressed wood, and the test span is 252 mm in order to meet a minimum span-to-depth ratio of 14 (ASTM 2015). MOE (MPa) and MOR (MPa) were calculated according to Eq. 3 and Eq. 4 (ASTM 2014):

$$\mathrm{MOE} = \frac{pl^3}{4bd^3\Delta} \quad \text{(MPa)} \tag{3}$$

$$\mathrm{MOR} = \frac{3P_{\max}l}{2bd^2} \quad \text{(MPa)} \tag{4}$$

where: p is the increment of applied load below proportional limit (N), P_{max} is maximum load (N), l is the span between the two supports (mm), b is the width of specimen (mm), d is the height of specimen (mm), and Δ is the increment of deflection corresponding to p (mm).

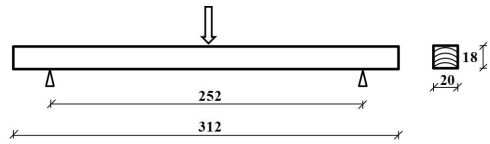

Fig. 3: Schematic diagram of the bending test.

Four replicates of specimens with 150 mm in length and 60 mm in width cut from the compressed wood were used to measure Brinell hardness (HB) according to standard EN 1534 (2000) with minor modifications, similar to that carried out by Rautkari et al. (2011). Six points on each sample were tested. A steel ball of 10 mm diameter (D) is intended in the wood surface at such a rate that maximum load (F) of 1 kN is reached in 15 s, and then the load is held for 25 s, finally the load is released over a period of 15 s. Because it is difficult to measure accurately the diameter of the indentation, therefore, the maximum depth of the indentation (h_{max}) is adopted, which was measured automatically by the universal testing equipment. The HB is calculated according to Eq. 5:

$$\mathrm{HB} = \frac{F}{\pi D h_{\max}} \quad \text{(MPa)} \tag{5}$$

Hardness recovery (HR) was also measured using Eq. 6, which represents the percentage of wood recovery directly after indentation:

$$\mathrm{HR} = \frac{h_{\max} - h_r}{h_{\max}} \times 100\% \tag{6}$$

where: h_r (mm) is the recovered indentation after releasing the load.

RESULTS AND DISCUSSION

Influence of thermal modification time

The set recovery of compressed wood with different thermal modification time is illustrated in Fig. 4. With the increase of thermal modification time, the set recovery decreases. The set recovery for compressed wood with the thermal modification of 1 h and 2 h is much less than that for specimens with the thermal modification time of 0.5 h. The set recovery at various thermal modification times (p = 0.000), shows highly significant differences, which has also been found in previous studies (Yan et al. 2010).

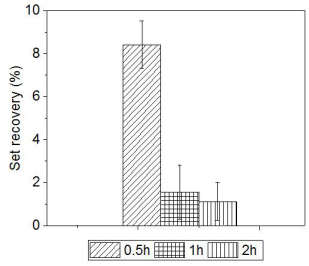

Fig. 4: Set recovery of compressed wood with different thermal modification time.

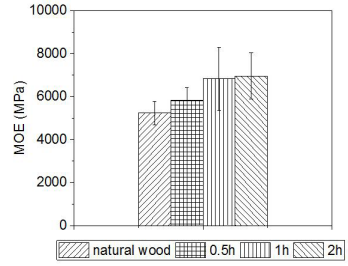

Fig. 5: MOE of specimens.

The density of compressed wood can be up to 1000 kg·m^{-3}. The MOE and MOR of compressed wood with different thermal modification time and those of natural wood are illustrated in Figs. 5 and 6.

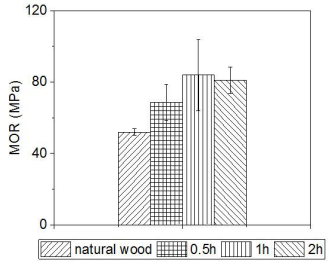

Fig. 6: MOR of specimens.

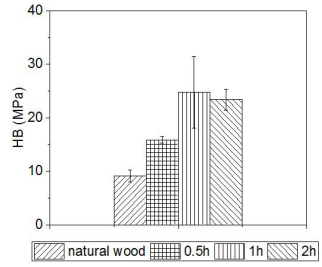

Fig. 7: Brinell Hardness of specimens.

The MOE (p = 0.183 > 0.05) and MOR (p = 0.156 > 0.05) at various thermal modification times, are not statistically significant. However, in previous studies, Yan et al. (2010) found that the MOE at various thermal modification times (0.5 h and 1 h), has significant differences.

The HB and HR of specimens are illustrated in Figs. 7 and 8. The HB (p = 0.000) and HR (p = 0.000) at various thermal modification times, show highly significant differences. The set recovery of compressed wood after wet/dry cycles is illustrated in Fig. 9. The Brinell hardness is highest for specimens with the thermal modification time 1 h. A long thermal modification time may result in the degradation of wood components, which will decrease mechanical properties of wood. The observation is consistent with the previous studies (Yan et al. 2010, Laine et al. 2016). It can be seen that the HR is lowest for the specimens with the thermal modification time of 1 h. These results can imply that the thermal modification time of 1 h can be more appropriate.

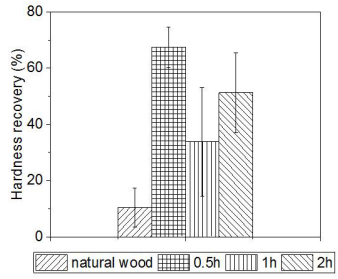

Fig. 8: Hardness recovery of specimens.

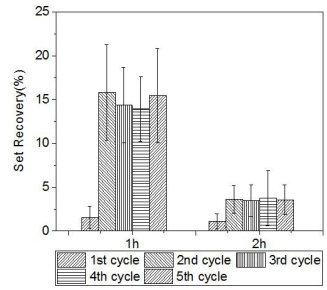

Fig. 9: Set recovery of compressed wood after different wet/dry cycles.

Influence of wet/dry cycles

Tab. 1 summarizes the *p*-value to present the statistical significance of wet/dry cycle with respect to density, set recovery and mechanical properties of compressed wood. At different wet/dry cycles, only the density, set recovery and HR of compressed wood with the thermal modification time of 1 h have statistical significance; the density and HB of compressed wood with the thermal modification time of 2 h are statistically significant.

Tab. 1: Statistical significance ANOVA, p-value. Units (-).

Thermal modification time	Density	Set recovery	MOE	MOR	HB	HR
1 h	0.014	0.000	0.469	0.535	0.984	0.008
2 h	0.000	0.200	0.589	0.286	0.032	0.084

After the second wet/dry cycle, the set recovery of compressed wood increases significantly. From the second wet/dry cycle, the set recovery changes rarely. For wood compressed under saturated steam, the set recovery reaches a plateau after the third cycle (Kutnar and Kamke 2012).

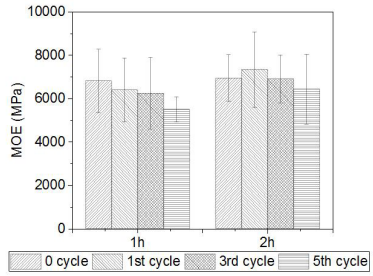

Fig. 10: MOE of compressed wood after different wet/dry cycles.

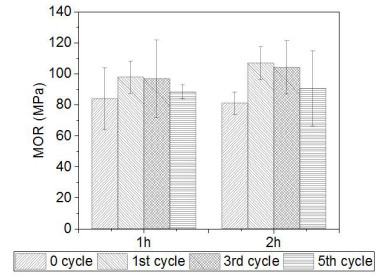

Fig. 11: MOR of compressed wood after different wet/dry cycles.

As shown in Figs. 10 and 11, after the first wet/dry cycle, the MOE and MOR decrease gradually with the increase of wet/dry cycles. For wood compressed under saturated steam, the wet/dry cycle also causes a decrease in the MOE and MOR (Kutnar and Kamke 2012).

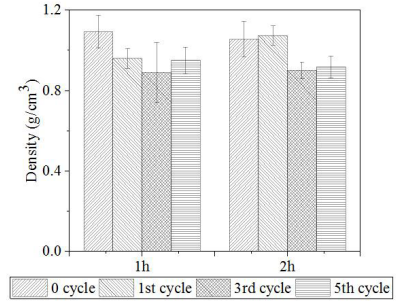

Fig. 12: Density of compressed wood after different wet/dry cycles.

Density of glycerinis equal to 1261 kg·m^{-3}, which is greater than that of compressed wood. Due to the dilution of glycerin during the soaking, the density of compressed wood decreases (Fig. 12), therefore, the HB of compressed wood also decreases. After the first wet/dry cycle, the HB of compressed wood with the thermal modification time of 1 h has nearly no change, and HB of compressed wood with the thermal modification time of 2 h decreases gradually with the increase of wet/dry cycle as shown in Fig. 13. Fig. 14 illustrates the HR of compressed wood compressed after different wet/dry cycles, and the first wet/dry cycle significantly results in the increase of HR.

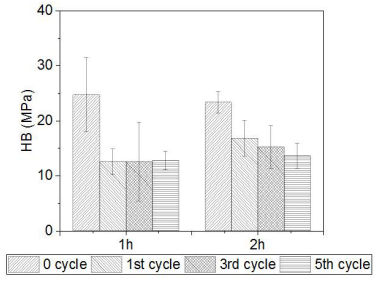

Fig. 13: Brinell Hardness of compressed wood after different wet/dry cycles

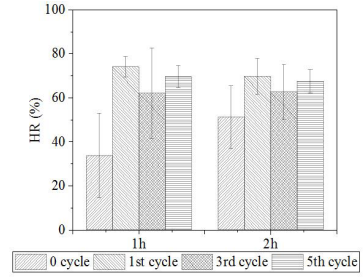

Fig. 14: Hardness recovery of compressed wood after different wet/dry cycles

CONCLUSIONS

This paper presented the influence of thermal modification time and wet/dry cycles on set recovery and mechanical properties of compressed poplar with glycerin pretreatment. The set recovery decreases with the increase of the thermal modification time, however, the set recovery of compressed wood with the thermal modification time of 1 h is just slightly higher than that of 2 h. The MOE and MOR at various thermal modification times are not statistically significant. For compressed wood with the thermal modification time of 1 h, the HB is highest and the HR is lowest. In conclusion, the thermal modification time of 1 h is suitable for compressed wood used in normal condition.

After the second wet/dry cycle, the set recovery of compressed wood clearly increases, and from the second wet/dry cycle, the set recovery of compressed wood changes rarely. The set

recovery of compressed wood with the thermal modification time of 1 h is obviously higher than that of 2 h. Thus, compressed wood with the thermal modification time of 2 h is more appropriate for humid or wet condition, due to the favorable deformation fixation.

After the first wet/dry cycle, the MOE and MOR of compressed wood decrease with the increase of wet/dry cycles. The first wet/dry cycle significantly results in the decrease of the HB and the increase of HR. Therefore, the first wet/dry cycle has significant effect on mechanical properties of compressed wood due to the dilution of glycerin during the soaking.

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