Flame Retardancy and Physical-mechanical Properties of Poplar Veneers Impregnated by Calcium Carbonate

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Fast-growing poplar (*Populus tomentosa* Carr) can produce wood veneers, but their poor quality restricts their application in construction and building. Modification of wood has the potential to improve its properties. In this study, poplar veneers were impregnated with calcium carbonate ($CaCO_3$) to reinforce their performance. The results showed that $CaCO_3$ was uniformly distributed in cell lumens in impregnated veneers. After impregnation, the maximum weight gain rate was up to 41.4%, and water uptake decreased from 6.82% to 0.94%. The hardness increased from 7.6 to 10.0 MPa, and the extent of wear fell from 0.91% to 0.05%. The ignition time was prolonged, and the heat release rate and total heat release were low. Experimental results demonstrated that $CaCO_3$ improved the physical-mechanical properties and flame retardancy of poplar veneers.

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INTRODUCTION

Since 2001, the planted forest resources in China have increased by more than 7.0 $\times 10^7$ hm^2, ranking first in the world (NFGA 2019). The planted forests can provide about 2.3 $\times 10^7$ m^3 of wood per year, effectively solving the conflict between the demand and supply of timber. *Populus tomentosa* Carr., one of the leading fast-growing tree species, has been a widespread species in paper and wood products, with its advantages of wide distribution, variety, adaptability, and a short growth cycle in China (Xi *et al.* 2009). Fast-growing and high yielding presents advantages in short-rotation, but these attributes are traditionally assumed to come at the price of lower wood quality (Barnett and Jeronimidis 2003). Poplar wood has a relatively low density and strength (Xi *et al.* 2009), but it is good at machining, bonding, and finishing properties, making it well-suited for producing veneers and veneer-related construction materials (Guélou *et al.* 2021).

The processing and use of poplar veneers are strongly influenced by wood defects such as soft texture and low density (Zhou *et al.* 2017; Guélou *et al.* 2022). In addition, most poplar wood is hygroscopic and highly sensitive to water (Dong *et al.* 2016). Modifying poplar wood is essential to overcome the drawbacks (Xu *et al.* 2017). Currently, research on wood modification focuses on two aspects. The first is a combination of graft polymerization and distribution mechanism of monomers in wood. The second approach explores modification techniques (Zelinka *et al.* 2022). However, most modifications involve harmful chemicals or reagents, which pose environmental concerns (Zhu *et al.* 2016). More durable and environmentally friendly poplar veneers are needed.

Introducing inorganic materials into a lignocellulose matrix improves the performance of composites without causing environmental problems (Gao *et al.* 2015; Guan *et al.* 2022). These approaches provide an opportunity to incorporate different minerals into wood for novel combinations of material properties (Merk *et al.* 2016). Only a few minerals, including calcium carbonate ($CaCO_3$), can be incorporated through chemical strategies into wood structure (Klaithong *et al.* 2013; Liu *et al.* 2015; Liang *et al.* 2023). $CaCO_3$ can increase wood dimensional stability and fire resistance without reducing its physical and mechanical properties (Moya *et al.* 2020). For example, the compression strength and elasticity modulus of $CaCO_3$/ *Paulownia* wood composites were 44.2% and 53.3% higher than those of the original wood (Huang *et al.* 2018). Interestingly, the results of introducing $CaCO_3$ in wood do not follow a “the more the better” relationship. Moya *et al.* (2020) found that more $CaCO_3$ leads to an increase in moisture absorption, from 12% to 18%. It is difficult for $CaCO_3$ to enter wood cell lumen directly. Generally, the method of alternately impregnating wood with sodium carbonate (Na_2CO_3) or sodium bicarbonate and calcium chloride ($CaCl_2$) is used, and chemical reactions occur between the two compounds in the cell cavity, giving rise to in *in-situ* mineral formation (Moya *et al.* 2020; Liang *et al.* 2023). Vacuum or/and pressure impregnation can be used to improve impregnation (Guan *et al.* 2022). However, it is difficult for minerals to form covalent bonds with the wood structure (Merk *et al.* 2016).

This paper examined a $CaCO_3$ deposition strategy in poplar veneers, which involves alternating impregnation with calcium chloride ($CaCl_2$) in ethanol and sodium carbonate (Na_2CO_3) in water. N-dodecyl-N,N-dimethylglycine ($C_{16}H_{33}NO_2$) was used as a zwitterionic surfactant in the impregnation. The performances of impregnated veneers were studied, including flame retardancy and physical-mechanical properties.

EXPERIMENTAL

Materials

The poplar veneers of 50 mm× 50 mm × 2 mm (no insect holes, knots, and other defects) were obtained from a plywood mill in Harbin, China. They were conditioned at 20 °C and 65% RH before impregnating.

$CaCl_2$, Na_2CO_3, and $C_{16}H_{33}NO_2$ were supplied by Tianjin Chemical Reagent Co., Ltd. and were used to prepare solutions in absolute ethanol and deionized water, respectively.

Veneer Impregnation

Veneers were placed in a tank for the impregnation process. First, the tank was vacuumed by a vacuum pump. Then, $CaCl_2 \cdot C_{16}H_{33}NO_2$ solution was siphoned into the tank using the negative pressure, and the veneers thus submerged in solution. The negative pressure was then released and an air compressor was used to pressurize the tank. After 1 h of impregnation in $CaCl_2 \cdot C_{16}H_{33}NO_2$, the veneers were removed and rinsed with ethanol. This was followed by the same vacuum-pressurize impregnation in aqueous Na_2CO_3 solution for 1 h. Finally, all veneers were removed from Na_2CO_3 solutions, oven-dried at 120 °C for 6 h, and then cooled to room temperature. The ratio of the mixed solution is shown in Table 1.

Table 1. The Level of Factors in Orthogonal Experiment

Na_2CO_3 (mol/L)	$CaCl_2$ (mol/L)	$C_{16}H_{33}NO_2$ (g)
0	0	0
0.15	0.2	0.05
0.25	0.3	0.25
0.35	0.4	0.45

Micromorphologies Observation

The environmental scanning electron microscope (SEM, QUANTA2000, FEI Company, OR, USA) characterized the micromorphologies of veneers. Gold was sprayed on the sputtering coating machine (Bal-Tec/Leica SCD005, Leica microsystem AG, Wetzlar, Germany). The wall structure of wood cells was observed, and the morphologies and distribution of $CaCO_3$ in the cells were studied.

Performance Test

The weight percent gain (WPG, %) of veneers was calculated as Eq. 1,

$$\mathrm{WPG} = \frac{W_2 - W_1}{W_1} \times 100 \qquad (1)$$

where W_1 is oven-dried weight before impregnation (g), and W_2 is oven-dried weight after impregnation (g). Veneers were immersed in deionized water at room temperature, and the weight was measured after 1 h. The water uptake (WU, %) was calculated as follows,

$$\mathrm{WU} = \frac{W_4 - W_3}{W_3} \times 100 \qquad (2)$$

where W_3 is the weight before immersion (g), and W_4 is the weight after immersion (g).

The hardness (MPa) and wear of veneers were tested by a universal testing machine (CMT6305, Shandong Wanchen Testing Machine Co., Ltd, Jinan, China) and operated according to Chinese standard GB/T17657-2013 (2013). A steel ball with a diameter of 5 mm was selected for the hardness test, and its movement speed and step length were 4.5 mm/min and 1 mm. The sandpaper with 180# was used in the wear test to grind each veneer for 50R. The wear rate (WR, %) was calculated as in Eq. 2,

$$\mathrm{WR} = \frac{W_6 - W_5}{W_6} \times 100 \qquad (3)$$

where W_5 is weight before wearing (g), and W_6 weight after wearing (g).

An X-ray diffractometer (XRD, D/max-2200VPC, Rigaku Corporation, Tokyo, Japan) was used to measure the phase structure and crystallinity of $CaCO_3$ in the impregnated veneers. The Scanning range is 3° to 90°, maximum power is 2kW, rated voltage is 20 to 60 kV, and rated current is 12 to 50 mA.

A cone calorimeter (ISO 5660-1, FTT Limited, East Grinstead, U.K.) was used to determine the flame retardancy of veneers based on the heat release rate.

Each treatment was tested five times in each assay.

Data Analysis

Multiple comparisons were used to compare performance parameters between design levels. Analysis of variance was used to estimate the effects of $CaCl_2$, $NaCO_3$, $C_{16}H_{33}NO_2$, and their interaction on performance parameters. Statistical significance (a p-value equal to 0.05) was determined using the F-test.

RESULTS AND DISCUSSION

Distribution and Morphologies of $CaCO_3$ in Poplar Veneers

The unimpregnated veneers showed well-defined fibers, vessel elements, and their pits on the wood section (Fig. 1A). In contrast, the impregnated veneers showed large amounts of $CaCO_3$ particles filling the cell lumen (Fig. 1B, C) and pit chambers (Fig. 1D). Poplar has a higher porosity than other hardwoods (Chen *et al.* 2013). Under negative pressure, the air in the wood pores is extracted. If $CaCl_2$ solution is applied simultaneously, the solution rapidly penetrates the wood pores. After wood drying, $CaCl_2$ solution deposits in the pores. When carbonic acid enters poplar veneers as a solution, the two solutions react with ions to form inorganic residues in wood pores and deposits. Wood is incubated successively with salt solutions. $CaCO_3$ formation is dominated by high supersaturation (Merk *et al.* 2016). As shown in Fig. 1, inorganic particles of $CaCO_3$ were evenly distributed in wood fibers, vessel elements and their pits. Most of them were in a nanometer scale of size with square, columnar, or diamond shapes. Most crystal forms of $CaCO_3$ were assumed to be calcite. Some of them huddled together and showed spherical shapes.

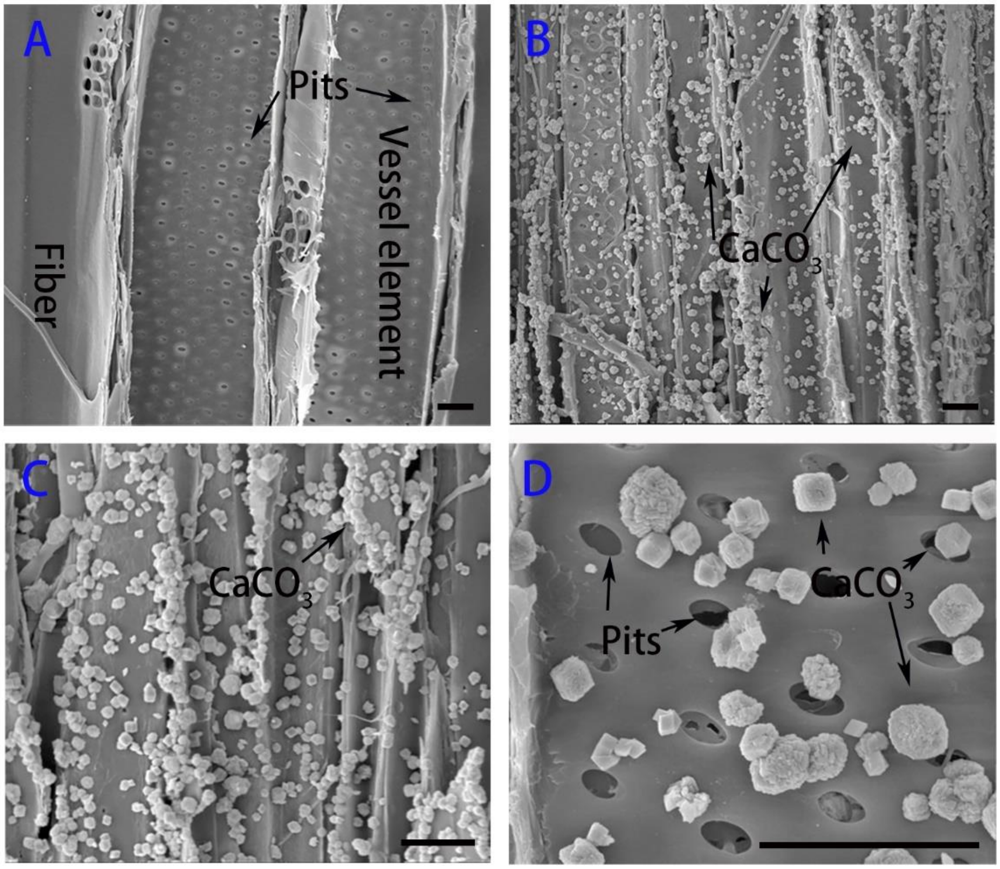

Fig. 1. SEM images of poplar veneers unimpregnated (A) and impregnated by $CaCO_3$ (B, C, and D). Scale bar=20 μm

The XRD patterns of poplar veneers had two prominent peaks that showed large amounts of cellulose (Fig. 2). Compared to unimpregnated veneers (Fig.2A), $CaCO_3$-impregnated veneers showed different crystal planes at many peak positions (Fig. 2B, C, D). The peak angle mainly contained $CaCO_3$. The crystal plane index showed that the peak area of about 29.5 was the largest, indicating that the main crystal form of $CaCO_3$ was calcite (Ma *et al.* 2012). The amount and type of $CaCO_3$ produced were not the same in veneers with different CO_3^{2-} molar ratios. With the increase of molar ratio, the amount of $CaCO_3$ increased, and the crystalline form was more concentrated and abundant. However, the maximum peak intensity of calcite occurred in impregnated veneers with CO_3^{2-} molar ratios of 0.25 (Fig. 2 C, Table 2). The results suggested that the CO_3^{2-} molar ratios of 0.25 had the advantage of preparing highly crystalline products.

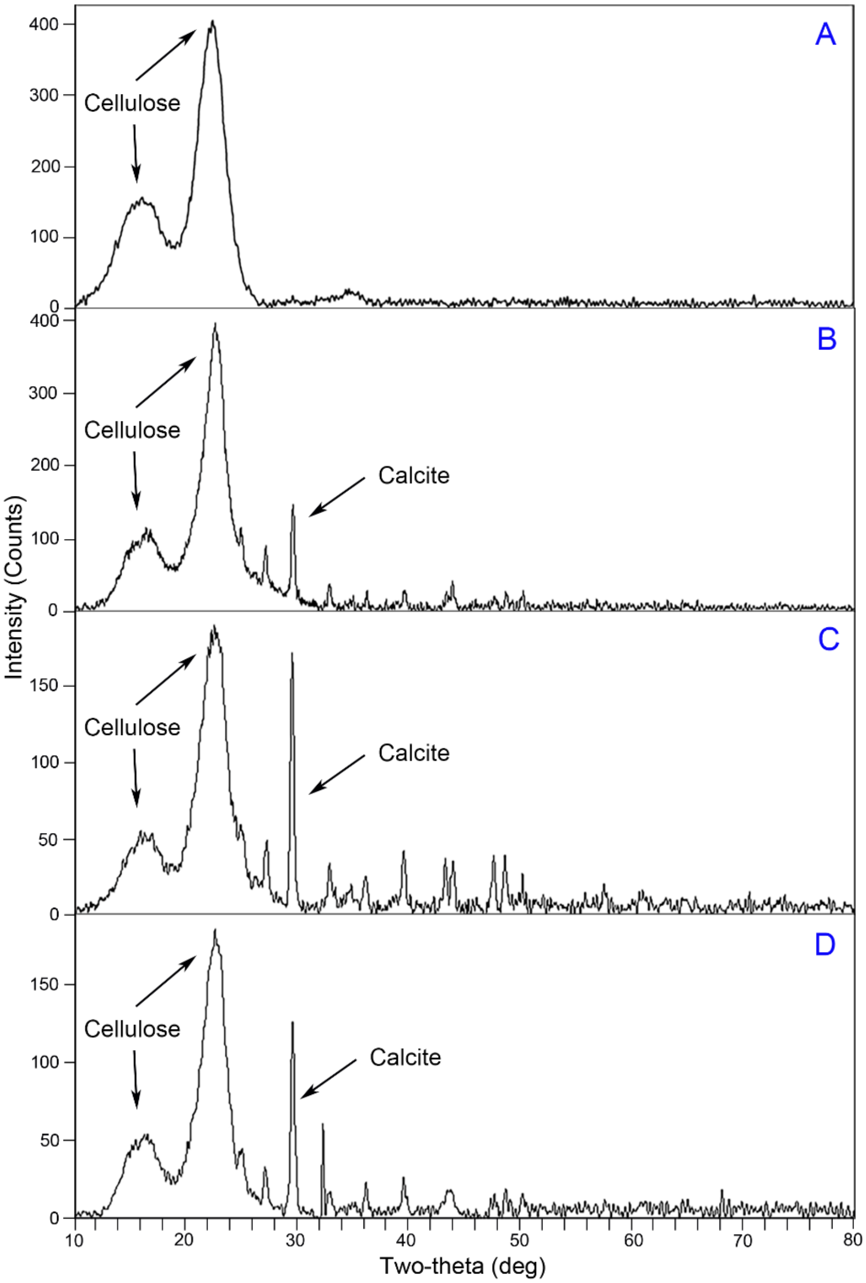

Fig. 2. XRD patterns of poplar veneers unimpregnated (A) and impregnated by CO_3^{2-} molar ratio 0.15 (B), 0.25 (C), and 0.35 (D), respectively

Physical-mechanical Properties of Poplar Veneers

After $CaCO_3$ mineralization, the weight increase of veneers was obvious. The observed weight gain was consistent with what was expected based on the concentrations of the two solutions and the expected amounts impregnated into the wood. The maximum weight gain rate was up to 41.4% (Fig. 3A). The results of variance analysis showed that the effects of $NaCO_3$, $CaCl_2$, $C_{16}H_{33}NO_2$, and their interaction on WPG were significant ($P<0.01$, Table 3). WPG showed an upward trend with increases in $NaCO_3$ and $CaCl_2$ (Fig. 4A, B). The effect of $C_{16}H_{33}NO_2$ was quite unique (Fig. 4C). With the increase of $C_{16}H_{33}NO_2$, the WPG first increased, reached the maximum value with $C_{16}H_{33}NO_2$ being 0.25g, and then decreased. As a zwitterionic surfactant, $C_{16}H_{33}NO_2$ played a key role in modifying the $CaCO_3$ produced to disperse well in veneers, avoid aggregation, and adhere to wood cell walls. It should be noted that, in addition to $CaCO_3$, the impregnated veneers probably contained a small number of by-products, such as NaCl and unreacted salts.

Table 2. XRD Data Analysis

$C0_3^{2-}$ molar ratio	Peak position angle (°)	Crystal size (nm)	Half height and width	Peak height	Peak area/integral strength
0.35	29.502	233	0.371	163	3268
0.25	29.585	212	0.322	130	2282
0.15	29.550	223	0.389	127	2017

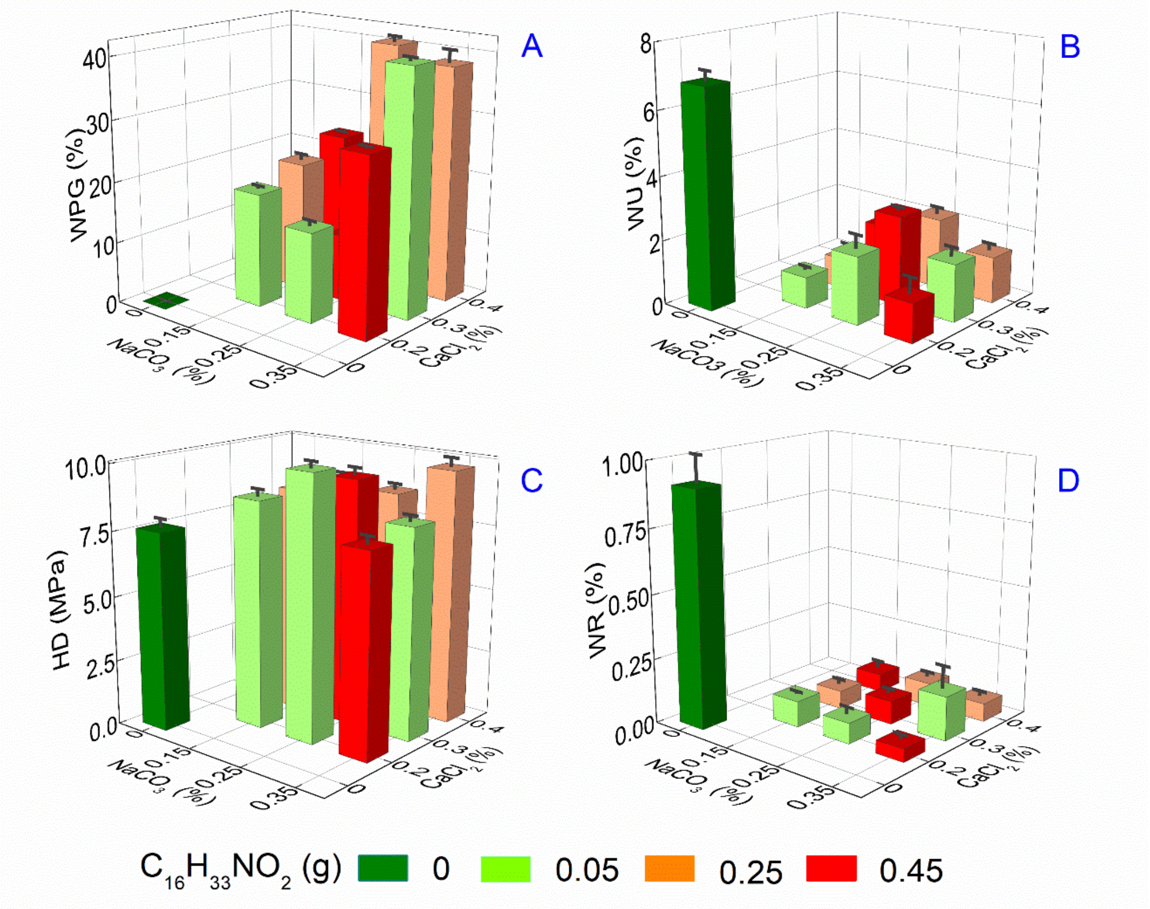

Fig. 3. Statistic of the physical and mechanical properties of unimpregnated and impregnated poplar veneers. WRG, weight percent gain; WD, wood density; WU, water uptake; HD, hardness; WR, wear rate

After being impregnated with $CaCO_3$, the maximum WU reduction of veneers was 5.88% (Fig. 3B). The remarkable decrease in WU is because many veneer pores are occupied by deposited $CaCO_3$, resulting in the inability to absorb more water. Peşman and Tufan (2016) have acquired similar results for $CaCO_3$-reinforced cellulose-high-density polyethylene composites. The results of variance analysis showed that $NaCO_3$, $CaCl_2$, and $C_{16}H_{33}NO_2$ significantly affected WU, but not by their interaction (Table 3). With the increase of $NaCO_3$ and $CaCl_2$ concentration, WU showed first an upward trend and then a downward trend (Fig. 4D, E). Although the WU of all impregnated veneers was significantly low compared with the unimpregnated veneers, there was no significant difference between the three impregnated levels of $C_{16}H_{33}NO_2$ (Fig. 4F).

Table 3. Analysis of Variance of $NaCO_3$, $CaCl_2$, $C_{16}H_{33}NO_2$ and their Interaction on Physical and Mechanical Properties

Dependent	$NaCO_3$		$CaCl_2$		$C_{16}H_{33}NO_2$		$NaCO_3$ × $CaCl_2$ × $C_{16}H_{33}NO_2$	
	F	p	F	p	F	p	F	p
WRG	719.47	0.00	316.24	0.00	215.18	0.00	337.97	0.00
WD	41.14	0.00	7.86	0.00	31.43	0.00	88.17	0.00
WU	34.49	0.00	7.00	0.01	5.28	0.01	0.37	0.69
HD	19.94	0.00	1.44	0.26	4.69	0.02	51.48	0.00
WR	0.26	0.77	2.58	0.10	3.57	0.04	0.60	0.56

Note: WRG, weight percent gain; WD, wood density; WU, water uptake; HD, hardness; WR, wear rate; *F*, variance test value; *p*, significance level.

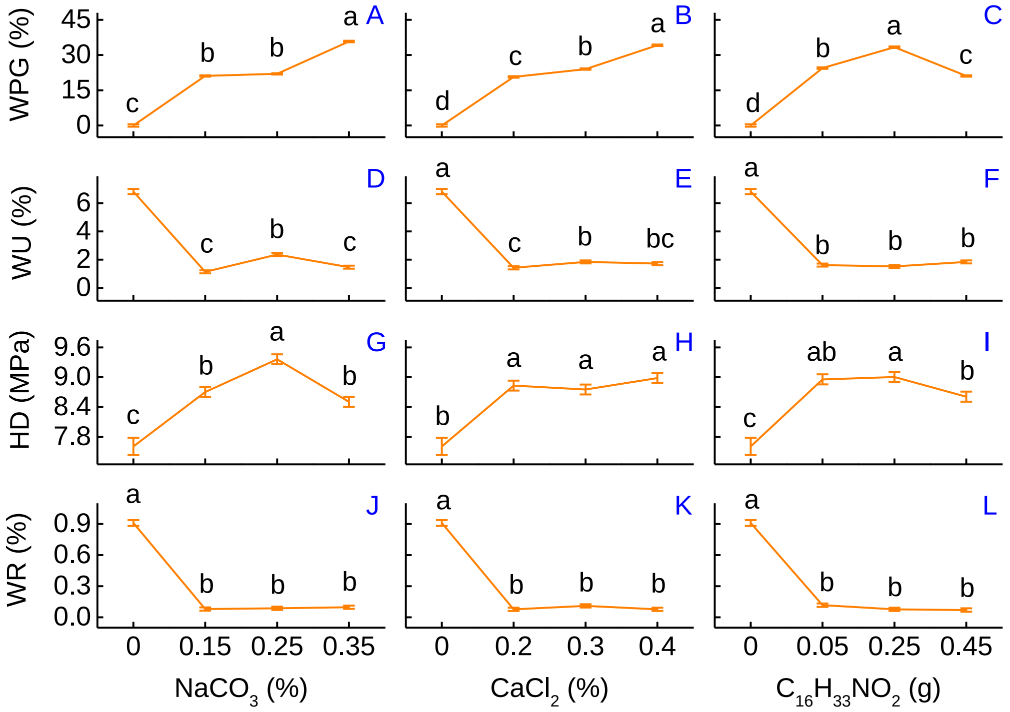

Fig. 4. Multiple comparisons of the physical and mechanical properties of poplar veneers among the factor Levels. Different lowercase letters above the bars indicate a significant difference among the levels ($p<0.05$). WRG, weight percent gain; WU, water uptake; HD, hardness; WR, wear rate

The hardness increased from 7.61 MPa for unimpregnated veneers to a maximum value of 10.06 MPa for impregnated veneers (Fig. 3C). Huang *et al.* (2018) also found that the mechanical properties of wood impregnated with $CaCO_3$ were improved. The effects of $NaCO_3$, $C_{16}H_{33}NO_2$ and their interaction on hardness were significant (Table 3). With the increase of $NaCO_3$ and $C_{16}H_{33}NO_2$, hardness first showed an upward trend, then a downward trend (Fig. 4 G, I). The results suggested that the $NaCO_3$ concentration of 0.25% and the $C_{16}H_{33}NO_2$ mass of 0.25 g were appropriate for producing very tough veneers. The hardness decrease of veneers impregnated with 0.35% $NaCO_3$ and 0.45 g $C_{16}H_{33}NO_2$ may be due to the aggregation of $CaCO_3$ generated when its concentration is too high in the liquid (Zhao *et al.* 2009). The aggregation is not conducive to producing nano-effect for $CaCO_3$ and hinders further improvement of veneer hardness.

$CaCO_3$ addition significantly reduced WR from 0.91% for unimpregnated veneers to a minimum of 0.05% for veneers impregnated with 0.35% $NaCO_3$, 0.2% $CaCl_2$, and 0.45g $C_{16}H_{33}NO_2$ (Fig. 3D). However, the WR differences of impregnated veneers were not significant among the impregnating levels of $NaCO_3$, $CaCl_2$, and $C_{16}H_{33}NO_2$, respectively (Fig. 4J, K, L). The results showed that the wear resistance of the veneers was improved due to $CaCO_3$ impregnation. The reason is that sodium carbonate reacts with calcium chloride to form $CaCO_3$. The ability of calcium carbonate to improve wear resistance has also been verified in plastics, rubber, and other materials (Chen *et al.* 2005; Wen *et al.* 2017; Mohsenzadeh *et al.* 2019). An important reason is the high wear resistance of $CaCO_3$ itself (Zeng 2010).

Flame Retardancy of Poplar Veneers

Flammability is an inherent defect of wood. Unimpregnated poplar veneers quickly burned, accompanied by a large amount of black smoke, and finally burned into grey-white ash (Fig. 5A). The final residue showed that it was the organic but not inorganic substance in poplar veneers completely combusted. The impregnated veneers burned slowly, with little or no smoke generated, and they finally burned into black ash (Fig. 5B). The results showed that adding $CaCO_3$ inhibited the combustion of organic substances in wood. Huang *et al*. (2018) obtained similar results for $CaCO_3$-reinforced *Paulownia* wood.

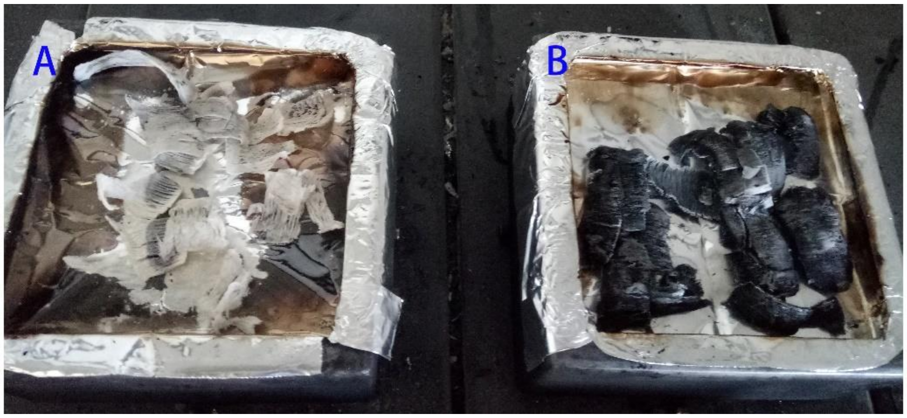

Fig. 5. Ashes of poplar veneers unimpregnated (A) and impregnated by CaCO3 (B)

The results showed that the flame retardancy of impregnated veneers was better than that of unimpregnated veneers. When the veneer burns, $CaCO_3$ may become a barrier on the surface of the cell wall to reduce the diffusion of oxygen and other flammable pyrolysis products in the veneer. Moreover, $CaCO_3$ decomposes into calcium oxide and carbon dioxide, thereby consuming heat. This reaction appeared to take place at about 37 minutes (Fig. 6). The released carbon dioxide can dilute oxygen concentration on the surface of the veneer, thereby slowing down or even preventing the combustion of the veneer. The ignition time, heat release rate (HRR), and total heat release rate (THR) of impregnated veneers were investigated to study the flame retardancy of the veneers. Generally, the longer the ignition time, the lower the heat release and the better the thermal flame retardance effect. Compared to unimpregnated veneers, impregnated veneers showed a long ignition time and low HRR and THR. The characteristic curves of HRR and THR showed the same trends in impregnated and unimpregnated poplar veneers (Fig. 6). Still, they were different from previous studies for *Paulownia* wood reinforced with $CaCO_3$ (Huang *et al.* 2018). Different species of wood may be the main reason for the difference. The peak of the HRR and the inflection point of THR appeared earlier for impregnated veneers than for unimpregnated veneers.

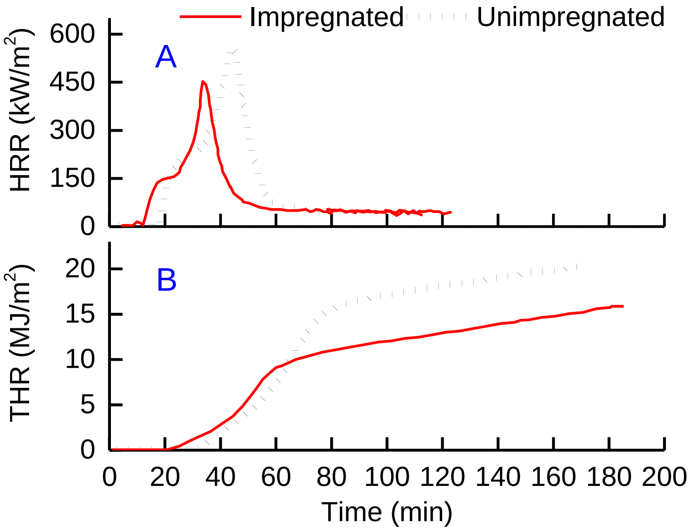

Fig. 6. Heat release rate (HRR) and total heat release (THR) of poplar veneers unimpregnated (A) and impregnated (B) with $CaCO_3$

CONCLUSIONS

1. $CaCO_3$-impregnated fast-growing poplar veneers were prepared by an inorganic reaction. N-dodecyl-N,N-dimethylglycine ($C_{16}H_{33}NO_2$) was used as a zwitterionic surfactant to modify the $CaCO_3$ produced in poplar veneers to make it disperse nicely in the pore spaces of veneers and prevent aggregation.
2. $CaCO_3$ was successfully inserted deep into the wood structure, which improved the flame retardancy and physical-mechanical properties of poplar veneers. The main crystal form of $CaCO_3$ deposited in wood cells was calcite.
3. The performances of impregnated veneers were affected by $NaCO_3$, $CaCl_2$ and $C_{16}H_{33}NO_2$. Optimized treatment was introduced as the concentration of $NaCO_3$ (0.25%), $CaCl_2$ (0.2%), and $C_{16}H_{33}NO_2$ 0.2 g.

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