

Precise Improvement of Wood Properties by Solution Quantitative Adsorption Furfurylation Based on Cell Wall Modification

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Furfurylation expands the value of wood and wood-based products in construction and engineering applications by improving its dimensional stability and lowering its moisture absorption. However, the traditional liquid phase vacuum and pressure impregnation (VPI) process faces some problems and shortcomings in industrial application, such as excessive consumption of modifiers, and inducing wood drying defects. To avoid these inherent shortcomings, a novel furfurylation method based on solution quantitative adsorption (SQA) was first applied in this study to improve the properties of wood. The results showed that the SQA furfurylation could achieve the precise modification of cell wall and avoid the deposition of furfuryl alcohol (FA) resin in the cell cavities. The scanning electron microscopy and nanoindentation results showed the preparation of ultra-stable wood materials (ASE > 70%) with low FA resin load (weight percent gain of about 20%) and high FA utilization. In addition, the SQA furfurylation could lead to the distribution of FA resin in the interior of wood, thus improving the physical properties of wood without altering the overall mechanical properties.

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INTRODUCTION

The over-consumption of non-renewable fossil resources and their negative environmental impact has led to the application of environmentally friendly and renewable resources, such as wood materials, and is high on the priority lists in many fields (Serrano-Ruiz *et al.* 2010; Liu *et al.* 2021a). Given the depletion of natural forests and timber export restrictions in many countries (Gilani and Innes 2020; Shen *et al.* 2020), fast-growing wood sources, including Chinese fir and poplar, are poised to become the major future timber resources (McEwan *et al.* 2020). Because of its fast growth rate and short rotation period, fast-growing wood is increasingly being used; however, some inherent shortcomings of fast-growing wood, such as low density, low strength, dimensional instability, and ease of biological degradation, have seriously limited wide application of these wood materials (Ermeydan *et al.* 2014; Li *et al.* 2020a; Yang *et al.* 2022). Therefore, to improve the use of fast-growing wood, various chemical modification methods have been dedicated to ameliorating its dimensional stability and hygroscopicity.

Impregnation with synthetic resin has been shown to be an efficient approach for wood modification (Anwar *et al.* 2009; Meng *et al.* 2019; Li *et al.* 2021), with its

effectiveness linked to the molecular weight of resin and its penetration into the material (Keplinger *et al.* 2015; Berglund and Burgert 2018). In recent years, furfurylation has garnered attention from academia and industries due to the abundant sources of furfuryl alcohol (FA) and its environmentally benign nature (Kong *et al.* 2018; Martin *et al.* 2021). Derived from crop straw (Gong *et al.* 2017; Zhao *et al.* 2022), FA can infiltrate wood cell walls because of its strong polarity and low molecular weight, and then it can become polymerized into hydrophobic resin *in situ* under acidic catalysis and high temperatures (Li *et al.* 2016; Zheng *et al.* 2022). As a result, the physical and mechanical properties of furfurylated wood, such as dimensional stability, hardness, compressive strength, resistance to biodegradation, and anti-weathering efficiency, have been significantly improved (Pfriem *et al.* 2012; Sejati *et al.* 2017; Thygesen *et al.* 2020). In addition, furfurylation has slight impacts on the environment due to its comparatively lower toxicity after combustion (Lande *et al.* 2004; Pilgård *et al.* 2010).

Furfurylation has been industrialized in some countries including Norway and the Netherlands in Europe (Jones and Sandberg 2020). Nonetheless, challenges persist in traditional vacuum-pressure impregnation (VPI), including the inability to store FA solution in the long term due to the presence of acidic catalyst and the difficulty in recycling waste FA solution. It is difficult to dry furfurylated bamboo, hindering industrial application of furfurylation. In addition, the loss of a large amount of modifiers in the VPI modification process has led to a low utilization rate of FA (Liu *et al.* 2024). Furthermore, the filling of resin in the wood cell cavity and the excessive swelling in the cell wall tends to damage the integrity of the cell wall and reduces the buffering effect of the material on the impact, resulting in a significant decrease in the impact resistance of wood (Westin *et al.* 2006).

In a previous study, a novel vapor phase furfurylation (VPF) technique was applied to enhance the dimensional stability of Chinese fir and poplar (Liu *et al.* 2020a). This approach successfully addressed some issues encountered in traditional VPI furfurylation by obviating the need for FA modification solution preparation, and it also showed that a part of MA in MA-esterified wood still retained catalytic properties for the resinization of FA. However, VPF furfurylation faces challenges, such as FA degradation, with prolonged heating due to imperfect vapor-based technology. Hence, there has been a need to explore more suitable wood furfurylation processes.

During furfurylation, weight percentage gain (WPG) plays an important role in the improvement of material properties, and the various WPG values have different influence in material properties. While an appropriate WPG can improve the dimensional stability of bamboo, an excessive WPG will destroy its dimensional stability (Dong *et al.* 2021; Li *et al.* 2021; Liu *et al.* 2021b). To avoid problems in the VPI furfurylation and the excessive use of FA resin, a two-step furfurylation procedure based on solution quantitative adsorption (SQA) was applied to improve wood properties in this study for the first time. According to a previous study, the dimensional stability and durability of bamboo can be improved by surface furfurylation *via* a simple soaking treatment at room temperature based on the fact that FA is easily adsorbed by woody materials (Liu *et al.* 2020b). This finding raises the question of whether the permeability of FA can be improved by extending the storage time. In this study, maleic anhydride (MA) was introduced into wood cell walls as a furfurylation catalyst in the first step. In the second step, quantitative FA liquid was absorbed by soaking and subsequently infiltrated into wood cell walls by means of standing at room temperature over several days. This simple furfurylation process aims to enhance wood properties, including dimensional stability, through quantitative FA liquid adsorption

while completely circumventing FA liquid degradation due to the absence of heating during soaking and standing.

EXPERIMENTAL

Materials

Chinese fir (*Cunninghamia lanceolata*) and poplar (*Populus* spp.) woods were obtained from a wood factory in Hunan Province in China. The suitable cubic samples for different tests were prepared according to Table 1, and all the samples were dried under 103 °C until a constant weight (recorded as m_0) was attained. Furfuryl alcohol (FA; CAS: 98-00-0, density: about 1.1285 g/mL at 25 °C) used was of industrial grade and bought from Shanghai Jiubang Chemical Co., Ltd. (Shanghai, China). Maleic anhydride (MA, purity $\geq 99.5\%$) was obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). All chemicals were used without further purification.

Table 1. Basic Information about the Samples

Items		Size (T × R × L)	Number of Samples
Physical Properties	WPG	20 mm × 20 mm × 20 mm	8
	ASE		
	EMC		
Mechanical Properties	CS	20 mm × 20 mm × 30 mm	8
	MOR	10 mm × 10 mm × 160 mm	10
	MOE		
Characterization		20 mm × 20 mm × 20 mm	8

Note: T means tangential direction; R means radial direction; L means longitudinal direction; WPG is weight percentage gain; ASE is anti-swelling efficiency; EMC is equilibrium moisture content; CS is parallel-to-grain compressive strength; MOR is modulus of rupture; MOE is modulus of elasticity

Solution Quantitative Adsorption for Furfurylation of Wood Cubes

In this study, SQA was introduced for wood furfurylation. In the first step, wood samples were placed in a custom-built reactor (PF-2) and subjected to high vacuum for 10 minutes. Then they were placed into a 4.5 wt% MA solution, with 10 minutes of vacuum application, followed by 1.0 MPa pressure impregnation at room temperature for 40 min. In the second step, the oven-dried MA-esterified samples were immersed in pure FA liquid for quantitative adsorption until the target adsorption amount was reached (about 25% in this study). Then, all the samples were wrapped in aluminum foil and placed in a closed reaction vessel for autonomous penetration for 20 days. After the storage, the samples were cured at 105 °C for 20 h and then oven-dried at 60 °C for 20 h.

Analysis of physical and mechanical properties

The WPG and solidification ratio (SR) of furfurylated wood were determined using Eqs. 1 and 2, respectively,

$$WPG = \frac{(m_2 - m_1)}{m_0} \times 100\% \quad (1)$$

$$SR = \frac{(m_2 - m_1)}{(m_3 - m_1)} \times 100\% \quad (2)$$

where m_0 and m_1 (g) are the oven-dried masses of wood before treatment and after MA solution impregnation, respectively, m_2 (g) is oven-dried mass of samples after SQA furfurylation and removal of free FA, and m_3 (g) is the oven-dried mass of samples that contains free FA after SQA furfurylation.

The anti-swelling efficiency (ASE) was used to judge the dimensional stability of wood samples. Both the ASE and equilibrium moisture content (EMC) were tested following the procedures of the previous study (Liu *et al.* 2020b). The modulus of elasticity (MOE), modulus of rupture (MOR), and parallel-to-grain compressive strength (CS) of wood were tested according to the Chinese national standard GB/T 15780-1995 (1995) and calculated using the method of Li *et al.* (2023), while work of fracture (WF) was calculated via Eq. 3,

$$WF = \frac{\int_0^{S^n} F dS}{bh} \quad (3)$$

where F is the load (N) during the three-point bending test, S is the displacement (mm) of the pressure head in the test, h is the thickness (mm) of the samples, and b is the width (mm) of the samples.

Characterizations

Three samples were taken from untreated and furfurylated wood groups for structural and chemical analysis. The micro-morphology of wood was characterized by scanning electron microscopy (SEM; ESEM-XL 30, FEI Company, Hillsboro, OR, USA). The mechanical properties of wood cell walls were measured by nanoindentation using a Triboindenter (Hysitron, Minneapolis, MN, USA) with a Berkovich diamond tip. The SEM and nanoindentation were used to study the spatial distribution of FA resin in furfurylated wood. The detailed characterization process was completed according to Liu *et al.* (2020a). The schematic of the sampling procedure for this analysis is shown in Figs. 1a and 1c, the indentation modulus and hardness were calculated according to the procedure described by Li *et al.* (2023).

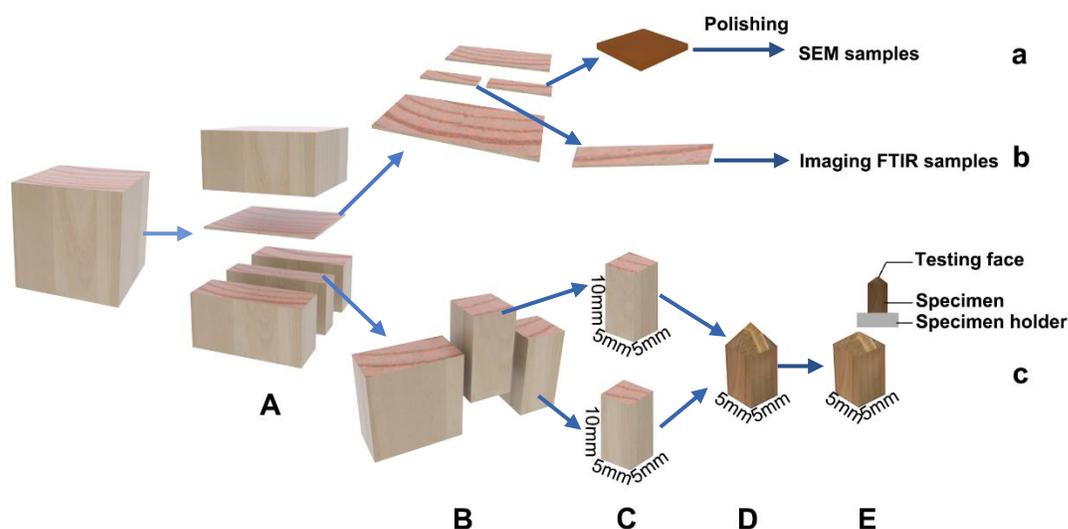


Fig. 1. Scheme of preparation of wood samples for characterizations. The sample cubes were divided (A), and then they were sawn up into the samples (B); with further processing, the samples for SEM (a), FTIR imaging (b), and nanoindentation test (c) were prepared.

To examine the penetration of FA resin in furfurylated wood after the SQA furfurylation, the infrared spectra of the samples in different regions were obtained by imaging *via* Fourier transform infrared spectroscopy (FTIR) microscopy (Perkin Elmer Inc., Shelton, CT, USA) and the preparation of test specimens are shown in Fig. 1b, the detailed characterization process refers to some previous studies by Li *et al.* (2020b).

RESULTS AND DISCUSSION

Figure 2 illustrates the process of furfurylation of wood samples using SQA. In this study, MA was first injected into the wood cell wall under vacuum. Part of the MA would have been esterified with the hydroxyl groups in the cell wall components, which affected the material properties, while the remaining was deposited on the surface or inside the cell wall (as shown in Fig. 3a) that could be used as a catalyst for FA curing. Then, FA liquid was adsorbed by wood samples after simple soaking treatment *via* SQA. The FA monomer was adsorbed by wood cells spontaneously due to the presence of hydroxyethyl (-CH₂OH) groups during the storage stage through differences in pressure and content levels (as shown in Figs. 2b and 2c). From previous studies, it has been demonstrated that FA can be cured well by using a two-step method without preparing an FA modifier solution (Liu *et al.* 2020a), so that FA monomers would then self-polymerize *in situ* to form FA resin (Lande *et al.* 2004), and the FA resin would be present at a gradually reducing level in the wood from outside to the inside due to the difference in FA concentrations, as shown in Fig. 2d.

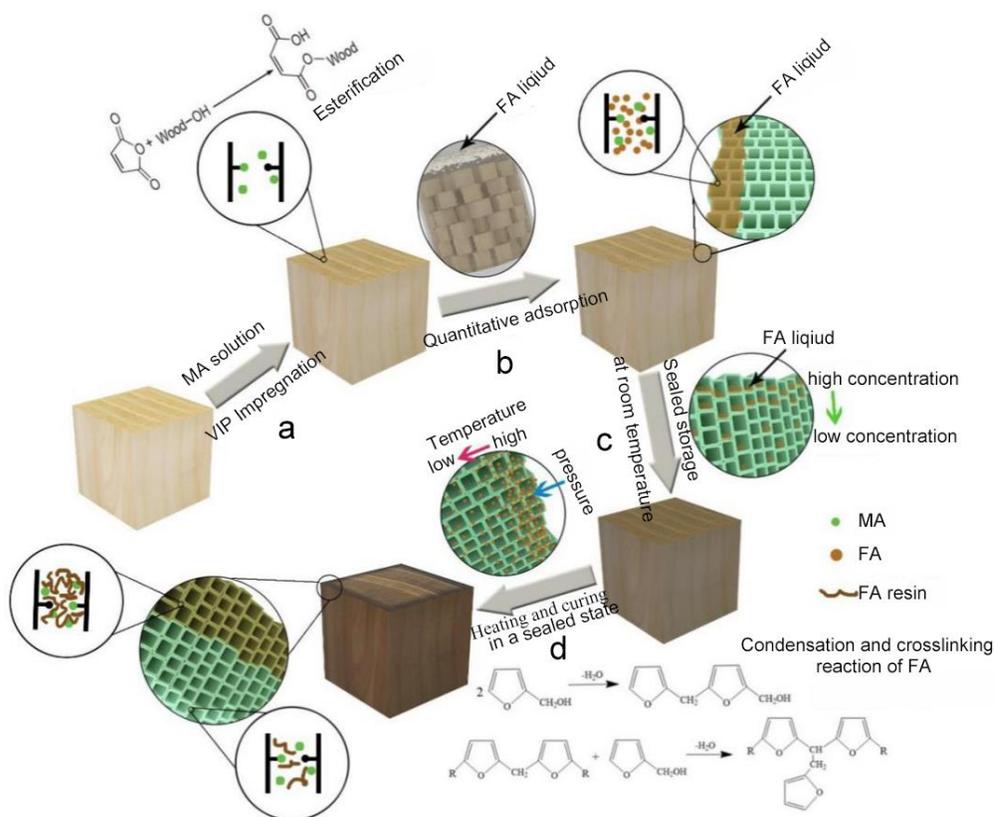


Fig. 2. Schematic illustration showing the fabrication process of furfurylated wood *via* solution quantitative adsorption

Physical and Mechanical Properties

The physical and mechanical properties of Chinese fir and poplar before and after the SQA furfurylation are presented in Fig. 3. The WPG of furfurylated Chinese fir and poplar were 22.0% and 21.8%, respectively, and the SR could reach 82.4% and 85.0%, which is 70% higher than that of VPI modification (Li *et al.* 2020, Liu *et al.* 2020a). These results indicated that the SQA furfurylation could improve the utilization efficiency of FA. On the one hand, the infiltration and solidification of FA in the sample after quantitative adsorption were carried out in the same closed environment, which avoids the loss of FA in the process of sample transfer and any spilling during the process of curing. On the other hand, there is no water involved in the adsorption and curing process, which avoids drying of the sample. In these processes of the VPI furfurylation, FA will be lost with the outward movement of water, resulting in the reduction of the SR. Furthermore, the volume of Chinese fir and poplar samples modified by the SQA furfurylation increased about 8.97% and 9.16%, respectively, which is primarily attributable to cell wall bulking with FA resin (Verma *et al.* 2014; Yang *et al.* 2022). In addition, the weight of MA-esterified Chinese fir and poplar increased by 6.54% and 4.23%, respectively, and the ASE values were 41.2% and 35.3%, respectively, while the EMC decreased by 14.8% and 18.6%. The positive changes in dimensional stability and hygroscopicity of wood were mainly due to the esterification of carboxyl group in MA with hydroxyl group in wood components, which reduces the hydrophilic groups in the wood and decreases the hygroscopic capacity of wood (Li *et al.* 2012). At the same time, the intermediates formed in the reaction produce a bridging effect in the wood cell wall and increase the dimensional stability (Sejati *et al.* 2017).

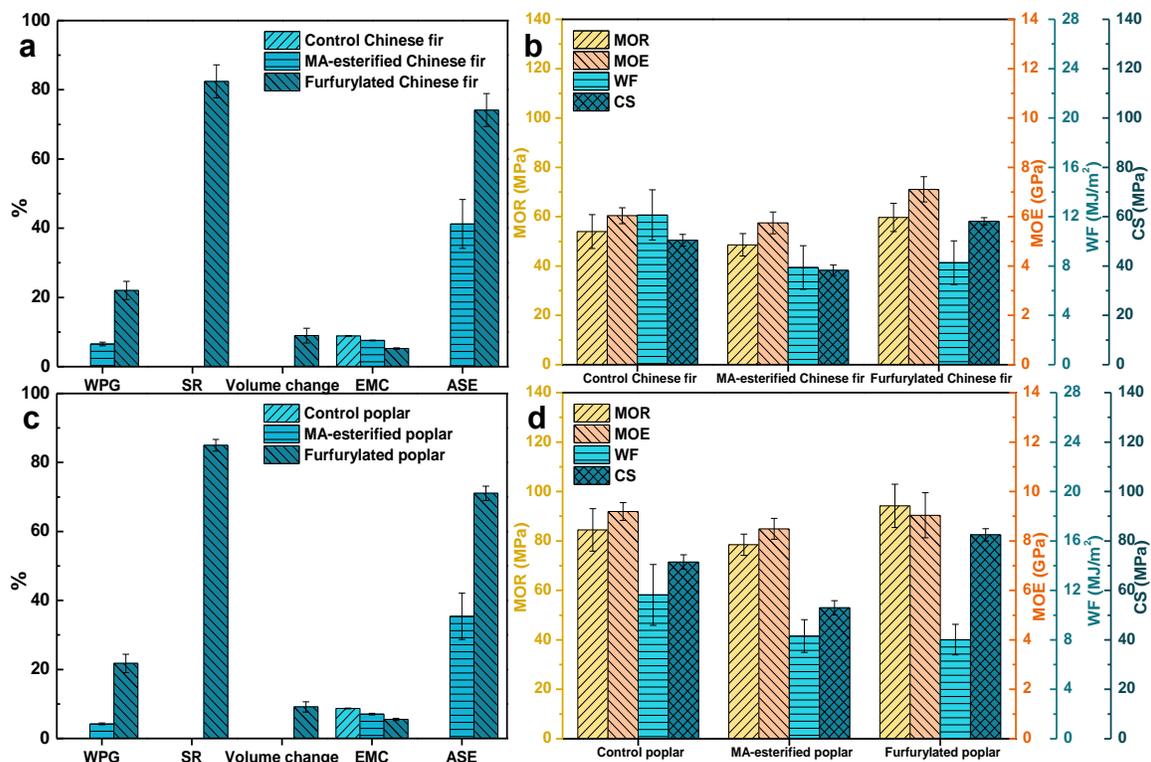


Fig. 3. The changes in physical and mechanical properties of Chinese fir (a, b) and poplar (c, d) during SQA furfurylation process

As presented in Figs. 3a and 3c, the ASE values of furfurylated Chinese fir and poplar samples were about 74.1% and 71.1%, respectively, indicating that the SQA strongly improved the dimensional stability of wood. Meanwhile, the dimensional stability of furfurylated samples increased by 79.8% and 101.4% compared with that after MA treatment. This is mainly attributable to the polymerization of FA monomer in the cell wall, which tended to swell the cell wall, making it maintain a permanent expansion state, thus reducing its shrinkage and expansion ability after water absorption (Dong *et al.* 2015; Yang *et al.* 2019). The filling of FA increased the volume of the cell wall, reducing the space for water absorption and expansion of the cell wall (Lande *et al.* 2004). Furthermore, the decrease of the affinity of furfurylated wood samples for water was also reflected in the decrease of EMC. The EMC of furfurylated Chinese fir and poplar was about 5.24% and 5.52%, respectively, which represents a 41.1% and 36.6% reduction of moisture content when compared to the untreated sample, and also decreased by 30.8% and 22.0%, respectively, compared with the MA-esterified samples. The decrease in hydrophilic properties of furfurylated wood can be attributed to the hydrophobic FA resin that filled the cell wall, which reduces the water storage capacity of the cell wall and also prevents water from binding with hydrophilic groups on the cell wall (Yao *et al.* 2017; Thygesen *et al.* 2021).

As a type of bulk bamboo modification, the SQA furfurylation also resulted in changes of mechanical properties (shown in Figs. 3b and 3d). Compared with untreated wood, there was a slight increase in MOR, MOE, and CS of furfurylated Chinese fir, which were increased by 10.6%, 17.7%, and 15.2%, respectively. For furfurylated poplar, the MOR and CS were increased by 11.5% and 15.3%, respectively, with slight change in MOE. The mechanical properties improvement was mainly caused by the swelling of FA resin on the cell walls, which can produce more supporting effects on the cell wall and even on the cell cavity to offset the adverse effects caused by acid modifiers, but also can help to strengthen the structure of wood, thereby increasing the compressive collapse resistance of wood (Li *et al.* 2016; Kong *et al.* 2018). This explanation was confirmed by the fact that the CS values of furfurylated Chinese fir and poplar were 55.6% and 52.0% higher than those of MA-esterified Chinese fir and poplar, respectively. However, due to the degradation of wood cell wall components in acidic and heated environments, as well as the expansion of the cell wall by FA resin, the cell wall structure was more easily destroyed, thus reducing the toughness of the material (Lande *et al.* 2008; Liu *et al.* 2021a; Yang *et al.* 2022). This was directly reflected in the WF of furfurylated Chinese fir and poplar that decreased 31.9% and 31.1%, respectively, while the decreases of MA-esterified Chinese fir and poplar were 28.6% and 35.1% respectively. Furthermore, the SQA furfurylation reduced the influence of FA resin swelling on the material toughness by reducing the rate of FA resin, resulting about 40% to 90% lower than the reduction in material toughness caused by VPI furfurylation (Liu *et al.* 2021a; Yang *et al.* 2022).

Distribution of FA Resin

To determine the distribution of FA resin after SQA furfurylation, the micromorphology of wood was observed with SEM, as shown in Fig. 4. Figures 4a and 4b show the microstructures of the untreated and furfurylated Chinese fir and poplar cross-sections. The micromorphology images showed that the cell wall of the furfurylated wood remained intact after SQA furfurylation, while the cell wall of the modified wood was seriously broken after VPI furfurylation (Li *et al.* 2020b) due to the overfilling of FA resin, which led to the increase of cell wall brittleness. This result revealed that the increase of

brittleness of wood after SQA furfurylation was much lower than that after VPI furfurylation. In addition, there was no visible FA resin deposited in the cell lumens at different positions of the furfurylated wood, and the cell wall remained porous (Figs. 4c1 and c2), indicating that the improvement of physical mechanics of wood by SQA furfurylation was not achieved by filling the cell lumens with FA resin.

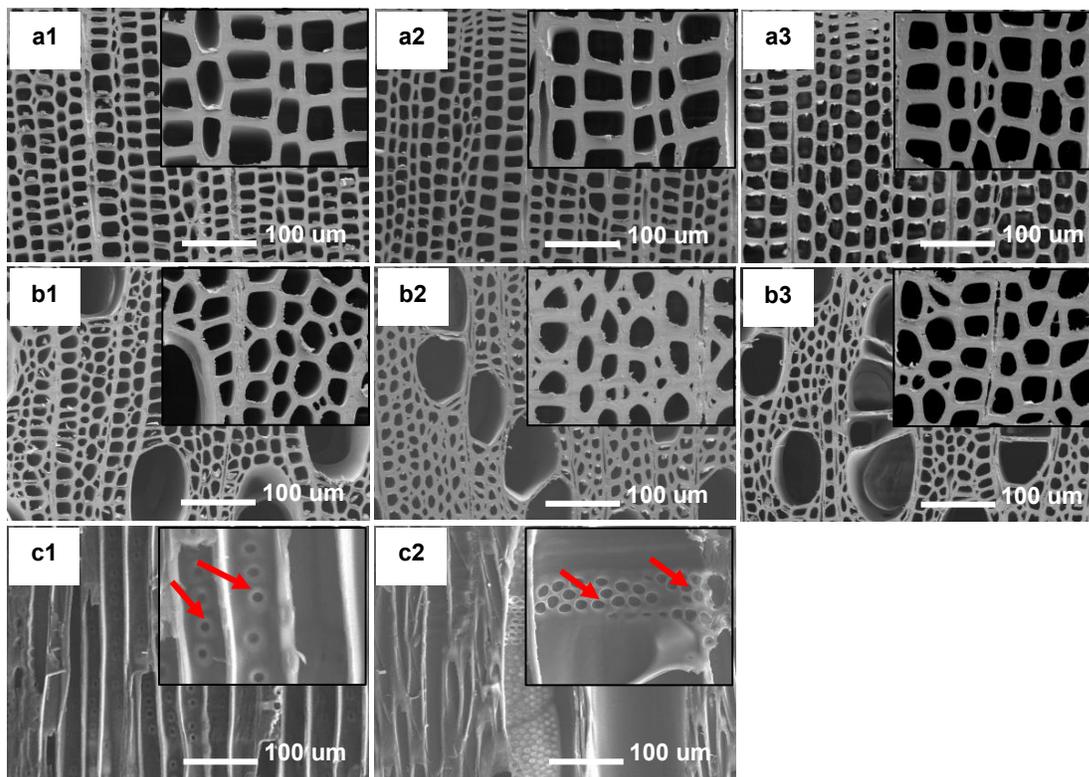


Fig. 4. SEM images of untreated and furfurylated wood: Untreated Chinese fir (a1), the region near to the surface (a2), and the center (a3) of furfurylated Chinese fir; untreated poplar (b1), the region taken from the surface (b2) and the center (b3) of furfurylated poplar; SEM images of longitudinal sections of the region taken from the surfaces of furfurylated Chinese fir (c1) and poplar (c2)

To further investigate the presence and curing degree of FA in the wood cell wall, the micro-mechanical properties of the cell wall before and after treatments were measured by nanoindentation (as shown in Fig. 5). Figure 5a shows that the mean indentation modulus values of the furfurylated Chinese fir and poplar in the outside regions were 21.0 and 19.7 GPa, respectively, which represented increases of 51.0% and 53.1% compared with untreated samples. Meanwhile, the average indentation modulus of the cell wall in the inside region area of the two kinds of furfurylated wood also increased by 9.84% and 10.87%, respectively. In addition, the hardness of cell wall in outer and inner regions of the furfurylated Chinese fir increased by 37.8% and 7.87%, respectively, compared with that of the untreated control sample. At the same time, similar changes can be observed in the modified poplar, which were increased by 40.9% and 16.2%, respectively. The results of nanoindentation test showed that the mechanical properties of the cell wall had been greatly enhanced by SQA furfurylation, which confirmed that the FA monomer could successfully penetrate the wood cell wall (Liu *et al.* 2021a) and cure well under the catalysis of MA during the modification process (Lande *et al.* 2004). In addition, the results

of nanoindentation also revealed that the distribution of FA in the furfurylated wood after SQA furfurylation was uneven, and the content of FA resin may gradually decrease from the outside to the inside.

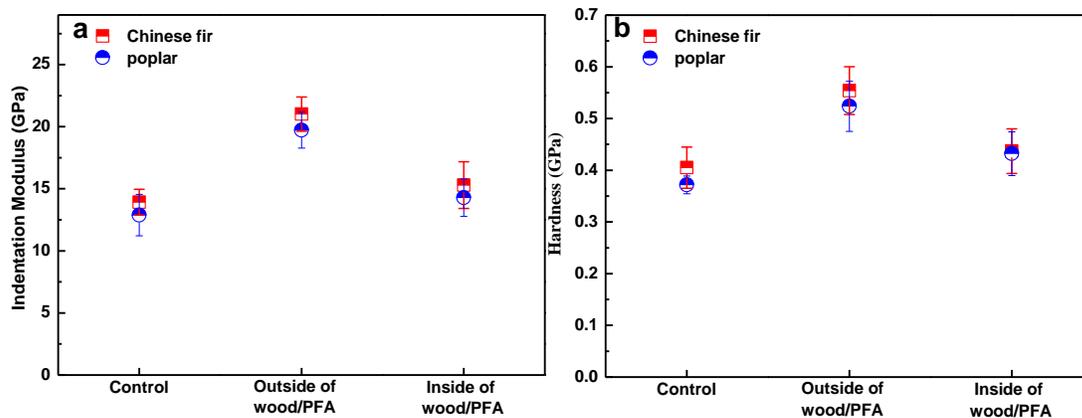


Fig. 5. The changes of mechanical properties of cell walls after SQA furfurylation; a: indentation modulus of untreated and furfurylated Chinese fir and poplar cell walls; b: hardness of untreated and furfurylated Chinese fir and poplar cell walls

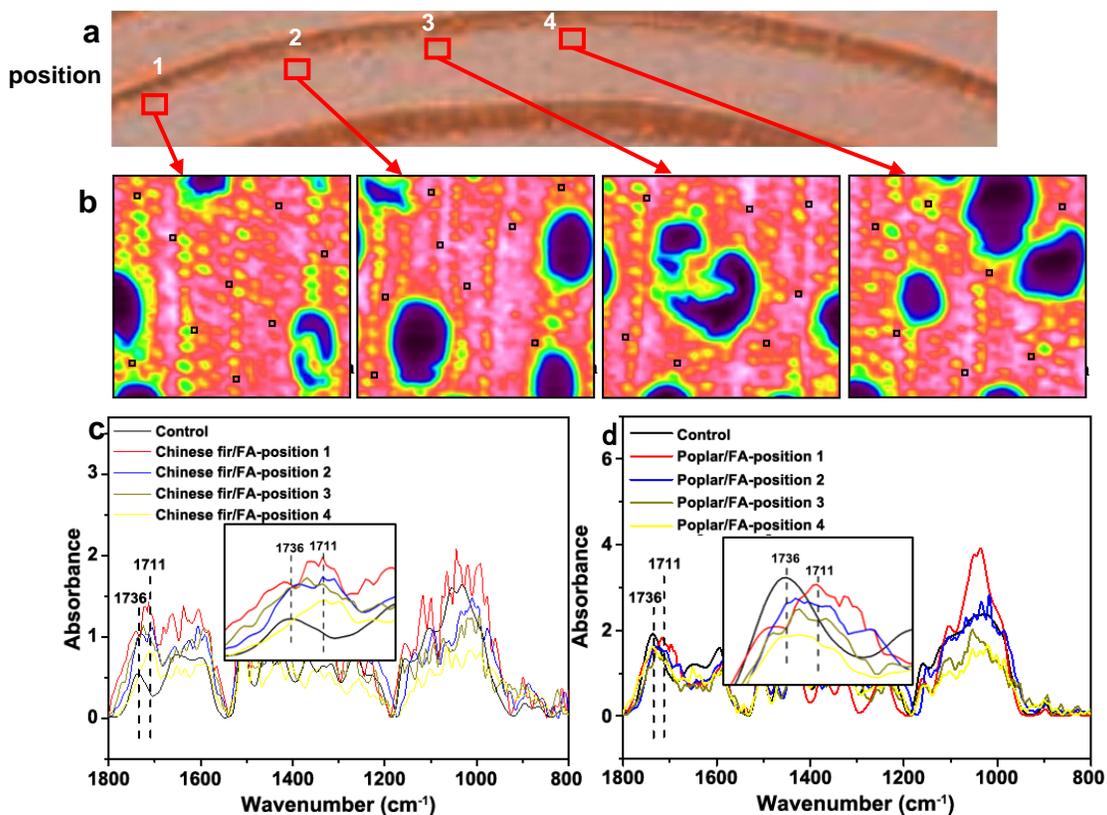


Fig. 6. The cell wall components in different regions of the furfurylated wood were characterized at four sampling sites with approximate uniform distribution from outside to inside (a) and corrected full-spectral IR absorption image at absorption band 1711 cm⁻¹ of furfurylated poplar (b), the small black empty box indicates the positions for measurement of cell wall spectra; FTIR spectra of cell walls of Chinese fir (c) and poplar (d) in the fingerprint regions before and after furfurylation

Penetration Depth of FA Resin

To observe the presence of FA resin more directly in the modified wood cell wall, FTIR, a technique for the detailed analysis of functional groups in plant cell walls (Guo *et al.* 2015), was used to visualize the changes of cell wall components and chemical structure. At the same time, four evenly distributed positions from the outside to the inside of the modified sample were tested to characterize the penetration depth of FA resin after SQA furfurylation.

Figure 6c shows that the infrared spectrum of the cell wall of furfurylated Chinese fir at different positions had stronger absorption intensity between the region of 1700 to 1750 cm^{-1} compared with the control sample, and the characteristic peak of FA resin appeared at 1711 cm^{-1} , which belongs to the stretching vibrations of γ -diketones $-\text{C}=\text{O}$ formed from FA resin hydrolyzed furan rings (Pranger and Tannenbaum 2008; Oishi *et al.* 2013). The appearance of the characteristic peaks can also be clearly observed in the infrared spectra of the cell wall of furfurylated poplar. In addition, a new shoulder peak appeared at 1562 cm^{-1} , which was attributed to the conjugated $\text{C}=\text{C}$ species and skeletal vibrations of 2,5-disubstituted furan rings (Ahmad *et al.* 2013; Liu *et al.* 2021a). Furthermore, by comparing the intensity of the characteristic peaks of FA resin at different regions, it was found that the content of FA resin in the modified wood samples after SQA furfurylation showed a high distribution outside and low distribution inside, which was consistent with the results of nanoindentation. The chemical changes directly showed the presence of FA resin in the furfurylated Chinese fir and poplar wood cell walls, indicating that FA could enter the wood cell wall and complete resinization successfully after the SQA furfurylation treatment.

CONCLUSIONS

In view of the shortage of storage and sustainable utilization of furfuryl alcohol (FA) solution in traditional furfurylation, a convenient new method for the furfurylation based on solution quantitative absorption (SQA) was proposed and demonstrated in this study.

1. Compared with the traditional vacuum and pressure impregnation (VPI) furfurylation, SQA furfurylation could substantially improve the utilization of FA where its solidification ratio (SR) could reach more than 80%.
2. The hygroscopicity of wood could be greatly reduced and the equilibrium moisture content (EMC) values of furfurylated Chinese fir and poplar were reduced 35% to 45%. Meanwhile, the dimensional stability was remarkably improved, with the anti-swelling efficiency (ASE) reaching more than 70%.
3. In addition, FA could penetrate the interior of the wood and present an uneven distribution after SQA furfurylation, which shows noteworthy gradual decrease from the outer regions to the inner regions, leading to the improvement in the physical properties of the furfurylated wood without causing substantial loss of mechanical properties. With this new approach, wood materials could be precisely furfurylated according to different modification purposes, thus expanding the application of wood in construction and engineering.

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Conflicts of Interest

The authors declare no conflict of interest.

Author Contributions

Minghui Liu: Data curation, writing of original draft, conceptualization, funding acquisition, review and editing, formal analysis, and project administration; Yong Yang: Software, funding acquisition, and investigation; Yuhan Liu: Funding acquisition, review and editing; and Linghua Yao: Conceptualization, funding acquisition.

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