

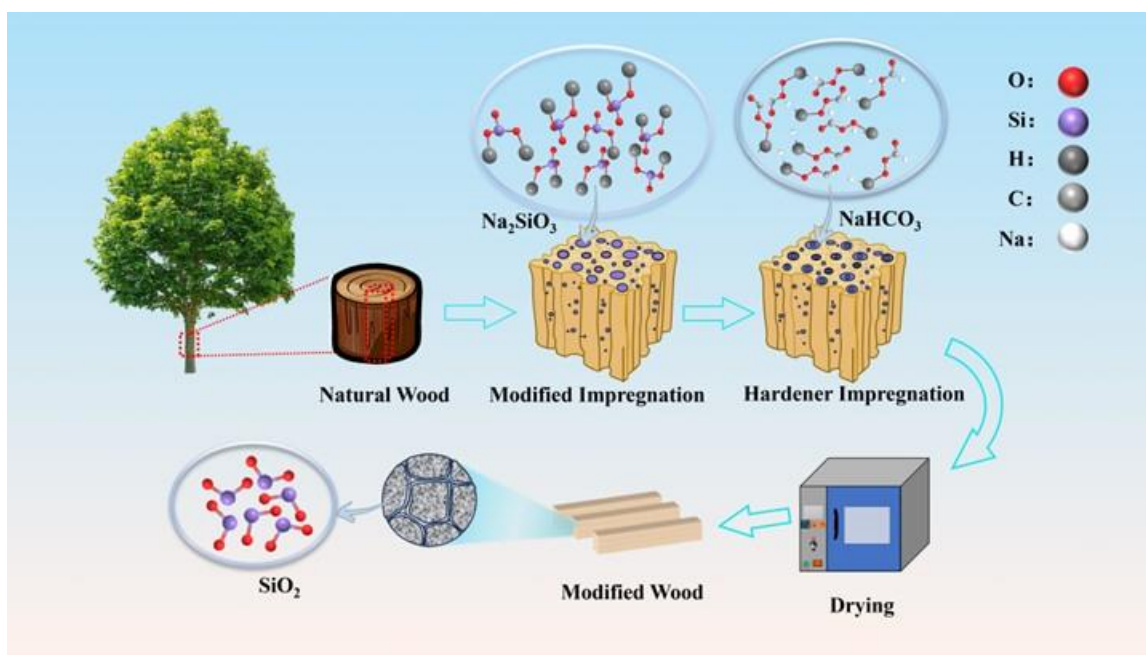
Enhancing Rubberwood Properties via Sodium Silicate Modification: A Study on Mechanical and Thermal Stability

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DOI: 10.15376/biores.20.3.6033-6053

GRAPHICAL ABSTRACT



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Rubberwood (RW), a commercially valuable timber species widely used for mid-to-high-end wood products in Yunnan, was modified through full-cell impregnation with sodium silicate (SS) solutions at varying concentrations (10 to 30%). The treatment significantly improved the wood's performance, overcoming challenges such as achieving optimal impregnation while preserving its integrity. Comprehensive analysis indicated that a 20% sodium silicate solution provided the most effective modification. This optimal treatment increased compressive strength by 15% (78.8 MPa), increased modulus of elasticity by 35.7% (1900 MPa), and reduced water absorption by 13.3% (103.5%) compared to untreated samples. Microstructural analysis confirmed optimal impregnation at 20%, with Fourier Transform Infrared (FTIR) spectroscopy revealing Si-O-Si peaks and X-ray photoelectron spectroscopy (XPS) indicating the presence of silicon, confirming the successful penetration of sodium silicate and silica formation. Furthermore, X-ray diffraction (XRD) analysis indicated that there was no alteration in the position of the cellulose diffraction peaks, which demonstrated that the sodium silicate impregnation treatment did not destroy its crystalline structure. This modification enhanced the mechanical properties and thermal stability of rubberwood while providing an eco-friendly alternative to traditional chemical treatments. Sodium silicate, mildly toxic and abundant, offers a sustainable solution for improving wood quality in various applications.

DOI: 10.15376/biores.20.3.6033-6053

Keywords: Rubberwood; Sodium silicate modification; Dimensional stability; Chemical bonding

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INTRODUCTION

Rubberwood (*Hevea brasiliensis*) is a tropical economic crop that originated in the Amazon region, and 70% of global rubber plantations are concentrated in Malaysia, Indonesia, Thailand, and other regions (Yang *et al.* 2023). In China, rubberwood trees are primarily cultivated in Yunnan and Hainan Province. Rubberwood is known for its appealing color, distinct texture, and excellent physical and mechanical properties. Accordingly, it is widely used in building materials, furniture manufacturing, aerospace, and other fields (Cao *et al.* 2020). However, the high sugar and starch content characteristic of rubberwood (substantially exceeding that of most commercial timber species) renders it particularly susceptible to microbial colonization under humid conditions. Such colonization leads to significant biodeterioration risks including mold formation, insect

infestation, and fungal decay – all of which substantially compromise its processing stability and end-use performance (Cherelli *et al.* 2023).

In recent years, numerous experts and scholars, both domestically and internationally, have engaged in extensive research on wood modification treatments. Wood modification is usually divided into physical modification and chemical modification. Currently, heat treatment is a commonly used method of physical modification. Chotikhun *et al.* (2020) investigated the alterations in the physical and mechanical properties of rubberwood subjected to microwave heat treatment. The results showed that the dimensional stability of the samples was improved with microwave heating, while the color change was minimal, thereby demonstrating the efficacy of microwaves in the heat treatment of rubberwood. Similarly, Patcharawijit *et al.* (2019) studied the changes of mechanical properties, surface color, durability, and cell wall constituents of rubberwood that was treated with steam at temperatures ranging from 140 to 180 °C for durations of 1 to 3 h. The results showed that after superheated steam treatment, the mechanical strength was improved, the hygroscopicity was reduced, and the corrosion resistance and termite resistance were enhanced.

Heat treatment is an environmentally friendly modification method. Compared to chemical modification, it is simpler to perform and does not produce pollution (Park *et al.* 2024). However, heat treatment can cause certain damage to the mechanical properties of wood. The process alters the cell wall structure, leading to increased brittleness and potential reductions in tensile strength, compressive strength, and bending strength. Excessive heating may also compromise wood hardness due to thermal degradation of cell wall polymers (Srivaro *et al.* 2019; Ali *et al.* 2022).

Compared to physical modification, chemical modification methods can more effectively enhance the decay resistance and mechanical properties of wood. As an important chemical modification approach, wood silicification significantly improves strength, dimensional stability, and decay resistance while preserving the natural appearance and texture of wood. The silicification process typically does not generate harmful volatile substances such as formaldehyde and is environmentally friendly, making it highly regarded in both academia and industry (Ismail and Abdul Khalil 2000; Jiang *et al.* 2022). Wang *et al.* (2024a) employed in situ polymerization of silica sol and phenolic resin combined with vacuum-pressure impregnation and thermal treatment, significantly enhancing the physical-mechanical properties of poplar wood. Additionally, Zheng *et al.* (2019) investigated the effects of zinc chloride-silicone oil treatment on Chinese fir, demonstrating that the modified wood exhibited markedly improved dimensional stability and decay resistance. Hung (Hung and Wu, 2018) prepared SiO₂ using the sol-gel method and analyzed the effect of SiO₂ content on the creep behavior of wood. The results showed that when the concentration was 20%, the fracture modulus of the modified wood was significantly higher than that of the natural wood. SiO₂ infiltrated into the cell walls and intercellular spaces of the wood, significantly enhancing its creep resistance.

However, the field of wood modification still faces several challenges. Firstly, different modification methods exhibit inherent limitations in enhancing wood properties, particularly concerning treatment uniformity and long-term durability. While thermal modification is simple and environmentally benign, it often compromises mechanical strength. Conversely, conventional chemical treatments may introduce environmental pollution and safety hazards. Consequently, developing modification strategies that effectively improve wood performance without adverse effects remains a critical research priority.

Sodium silicate is a representative of inorganic silicate modification, which is widely used, low cost, low toxicity, and has unique advantages in wood modification (Hung and Wu 2018). This method has significantly enhanced the performance of wood, improving its impact resistance and durability while effectively reducing the emission of harmful small molecules, such as formaldehyde. As a result, this advancement fosters a healthier and safer living environment for consumers. The improved properties of the wood have elevated its popularity as an eco-friendly material, enhancing both its sustainability and aesthetic appeal (Götze *et al.* 2008). Compared with other wood modification techniques, sodium silicate can effectively penetrate wood cells, offering not only simple operation and low cost but also preservation of wood's natural characteristics. Although acetylation improves water resistance, it requires high temperature and pressure, involves higher costs, and may affect wood's mechanical properties (Augustina *et al.* 2023). Polymer impregnation may influence wood permeability and appearance. In comparison, the sodium silicate treatment process is environmentally friendly and low-cost, while improving the physicochemical properties of wood, demonstrating high comprehensive cost-effectiveness.

In contrast to modification methods in previous studies, sodium silicate was selected for this research as the modifying agent and vacuum-pressure impregnation was used to investigate the effects of different sodium silicate solution concentrations on rubberwood properties. The study aimed to provide a novel technical pathway for high-value utilization of wood resources. Through comprehensive analysis of physical-mechanical properties, chemical composition, and microscopic morphology of modified rubberwood, the goal was to determine an optimal modification concentration to achieve maximum performance enhancement.

EXPERIMENTAL

Materials

Rubberwood (*Hevea brasiliensis*) was obtained from Xishuangbanna, Yunnan, and was derived from trees that were over 15 years old. The average oven-dry density of the selected rubberwood samples was $621 \pm 63 \text{ kg/m}^3$. Wood exhibiting a straight grain and devoid of significant visible defects was selected and processed into samples measuring $20 \text{ mm} \times 20 \text{ mm} \times 300 \text{ mm}$ ($R \times T \times L$). The samples were divided into four groups of 12 pieces each and were placed in a constant temperature and humidity box at a temperature of 20°C and a humidity of 65%, until the change in mass did not exceed 0.1% after every 24 h.

Sodium silicate (SS), industrial grade with a modulus of 3.4, was purchased from Huifeng New Materials Co., Ltd. Zhengzhou, China. Three concentrations of sodium silicate solution were prepared: 10% (solid-liquid ratio of 1:9), 20% (solid-liquid ratio of 1:4), and 30% (solid-liquid ratio of 3:7). Ultrapure water and SS were mixed evenly in a heat-collecting magnetic stirrer at a constant temperature of 40°C ; after cooling, the viscosity of the three solutions was measured using a viscometer.

Sodium bicarbonate, analytical grade, was purchased from Guangdong Guanghua Technology Co., Ltd., Shantou, China.

Distilled water was made in the laboratory.

Preparation of Impregnated Modified Wood

The test samples were conditioned in a climate chamber (HD-E702, Haida Instrument Co., Ltd.) to achieve a moisture content ranging from approximately 8 to 12%. The balanced sample was placed in an impregnation tank, which was sealed and then subjected to impregnation modification. Figure 1 illustrates the impregnation procedure.

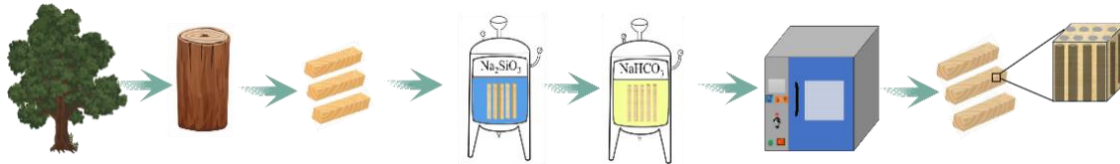


Fig. 1. Preparation of sodium silicate-impregnated modified rubberwood

First, the impregnation process began by evacuating the tank to -0.08 MPa for 20 min to remove air from both the chamber and wood cells. During this stage, sodium silicate solution (Component A) was introduced to submerge all specimens before releasing the vacuum.

Subsequently, the system was then pressurized to 0.4 MPa for 40 min to drive Component A penetration throughout the wood microstructure. After depressurization and solution discharge, the tank was re-evacuated (-0.08 MPa, 20 min) to introduce the curing agent (Component B). A final pressurization (0.4 MPa, 40 min) ensured uniform Component B distribution and reaction with the silicate. Treated samples were removed, surface-wiped, and prepared for curing.

Finally, the modified samples were put into an electric heating convection-drying chamber (Model 101A-2E, Shanghai Experimental Instrument Factory Co., Ltd.) and subjected to a stepwise temperature ($60\text{ }^{\circ}\text{C}/4\text{h} \rightarrow 80\text{ }^{\circ}\text{C}/1.5\text{h} \rightarrow 120\text{ }^{\circ}\text{C}/0.5\text{h}$) increase method for curing (where the $60\text{ }^{\circ}\text{C}$ pretreatment facilitates complete solution penetration and initial gelation, the $80\text{ }^{\circ}\text{C}$ main treatment promotes silanol group condensation, and the subsequent $120\text{ }^{\circ}\text{C}$ short-term treatment completes the formation of a three-dimensional Si-O-Si network structure while preventing thermal degradation of the rubberwood matrix). Subsequently, the specimen was dried at $103 \pm 2\text{ }^{\circ}\text{C}$ until it was completely dry. After drying, the specimen was removed and placed in a drying dish to cool to room temperature.

Rubberwood specimens were impregnated with sodium silicate solutions at three concentration levels (10%, 20%, and 30%), yielding the following sample designations: 10% RW-SS (10% sodium silicate-impregnated rubberwood), 20% RW-SS, 30% RW-SS.

Water Absorption Analysis

According to the GB/T 1927.7-2021 standard for measuring wood water absorption, both the material and modified material samples were placed in a blower-drying oven at $103\text{ }^{\circ}\text{C}$ until completely dry. After drying, the samples were weighed and then submerged in a beaker filled with distilled water, secured with stainless steel wire to ensure they were immersed at least 5 cm below the water surface. The samples were first weighed after 6 h, followed by additional weighings at 24, 48, and 96 h, with further weighings every 96 h until the samples reached saturation. Once saturated, the wood blocks were dried again and weighed to calculate the weight leaching rate. The dimensions of the test samples were $20\text{ mm} \times 20\text{ mm} \times 20\text{ mm}$ ($R \times T \times L$), with six samples tested in each group. Water absorption (WA) was calculated as follows,

$$WA = \frac{m - m_0}{m_0} \times 100\% \quad (1)$$

where m is the mass of the sample after water absorption (g), and m_0 is the dry mass of the sample (g).

Swelling Analysis

According to GB/T 1934.2-2009 “Method for Determination of the Swelling of Wood,” rubberwood untreated and modified specimens were placed in an oven and dried at 103 °C until reaching absolute dryness. The radial (L_{r0}), tangential (L_{t0}), and longitudinal (L_{l0}) dimensions were measured. The specimens were then immersed in distilled water until dimensional stabilization was achieved (the difference between two consecutive measurements was less than 0.02 mm). After stabilization, the radial (L_{r1}), tangential (L_{t1}), and longitudinal (L_{l1}) dimensions were measured again. Swelling ratio (α) was calculated as follows,

$$\alpha = \frac{(L_{r1} \times L_{t1} \times L_{l1}) - (L_{r0} \times L_{t0} \times L_{l0})}{L_{r0} \times L_{t0} \times L_{l0}} \times 100\% \quad (2)$$

Density Analysis

According to GB/T 1933.2-2009 “Method for Determination of the Density of Wood,” rubberwood untreated and modified specimens were placed in an oven and dried at 103 °C until reaching absolute dryness. The samples were weighed (m) and their dimensions in radial (L_{r0}), tangential (L_{t0}), and longitudinal (L_{l0}) directions were measured. Density (ρ) was calculated as follows,

$$\rho = \frac{m_0}{L_{r0} \times L_{t0} \times L_{l0}} \times 100\% \quad (3)$$

Mechanical Properties

In accordance with GB/T 1927.11-2022, the bending strength of wood was determined using a universal testing machine (UTM4000, Sansi Zongheng, Shenzhen, China). The dimensions of the samples were 20 mm × 20 mm × 300 mm (R × T × L), with a span of 240 mm. Each test group comprised five samples, and the loading rate was set at 5 mm/min.

In accordance with GB/T 1927.9-2021, the compressive strength of wood was determined using a universal testing machine. The dimensions of the samples are specified as 20 mm × 20 mm × 30 mm (R × T × L). Each test group comprises five samples, and the loading rate is set at 3 mm/min.

The micro-mechanical properties of the cell walls of the specimen were tested using a nanoindenter (iMicro model, KLA, USA). The Continuous Stiffness Measurement (CSM) method was used to measure dynamic stiffness, thereby obtaining hardness and modulus values that continuously vary with indentation depth. A small block measuring 5 mm × 5 mm × 10 mm (T × R × L) was cut from a specific region of the sample and fixed onto an ultra-thin slicing machine. The testing surface was smoothed using a glass knife until it was flat, with a surface roughness of less than 10 nm. After preparation, the small block was adhered to the sample stage using a hot melt glue stick, ensuring that it was flattened and any air bubbles were removed. The sample stage was then loaded onto the sample tray, and the test chamber door was closed to begin the measurement.

Field Emission Scanning Electron Microscope (FESEM) Analysis

The microstructural characteristics of the sample were examined utilizing a FESEM (Regulus-8100, Hitachi, Japan). The sample was sectioned into 10 μm thin slices, air-dried, and subsequently affixed to conductive adhesive, to enhance surface conductivity. The sample was subjected to vacuum gold coating before being analyzed under the microscope to observe the cross-sectional tissue structure.

Fourier Transform Infrared Spectra (FT-IR) Analysis

Infrared spectroscopy analysis was performed on rubberwood that had been thoroughly dried both before and after modification. The wood was then ground into a fine powder, and samples with a particle size of 160 mesh were selected for further analysis. These samples were compressed into transparent pellets using the potassium bromide (KBr) method for FT-IR testing. Changes in the functional groups before and after modification were assessed using a Fourier transform infrared spectrometer (Nicolet iS50, Thermo Fisher, USA). The infrared spectral range was set between 500 and 4000 cm^{-1} , with a resolution of 4 cm^{-1} and a total of 32 scans.

X-ray Diffraction (XRD) Analysis

The specimens before and after modification were dried to absolute dryness and then crushed with a pulveriser to screen the specimens with a particle size of 160 mesh. The samples were tested using an X-ray diffractometer (Rigaku H12, Ultima N, Japan) with a scanning range of $2\theta = 5$ to 60° , a step size of 0.02° , and a scanning speed adjusted to $8^\circ/\text{min}$. Relative crystallinity was calculated using the Segal method,

$$C_r I = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (4)$$

where CrI is the relative crystallinity (%), I_{002} denotes the maximum diffraction peak intensity of the main crystalline peak 002, and I_{am} denotes the scattering intensity of the amorphous background diffraction at a 2θ angle of 18° .

X-ray Photoelectron Spectroscopy (XPS)

After thoroughly drying the rubberwood to absolute dryness, both before and after modification, it was crushed using a grinder, and samples with a particle size of 160 mesh were selected. The chemical composition and relative content of various elements in both the rubberwood material and the modified material were analyzed using X-ray photoelectron spectroscopy (XPS, K-Alpha+, Thermo Fisher Scientific, USA).

Differential Scanning Calorimetry (DSC)

The thermal storage capacity of the wood was evaluated using a differential scanning calorimeter (DSC). Rubberwood samples, both before and after modification, were ground into a fine powder using a grinder. A sample weighing between 5 and 10 mg was placed in an aluminum crucible, with an empty crucible serving as the reference. The temperature was gradually increased from ambient conditions to 350°C at a rate of 10°C per min, followed by a cooling phase down to 30°C . Both the protective and testing atmospheres for the samples were maintained under nitrogen with a flow rate of 20 mL/min.

RESULTS AND DISCUSSION

Material Color and Water Absorption

As shown in Fig. 2a, no noticeable visual color change was observed. Rubberwood modified with sodium silicate effectively preserved both the visual appearance and environmental characteristics of the wood. Table 1 presents the leaching rates of rubberwood modified with three different concentrations of sodium silicate. As can be seen from Table 2, the p-values were less than 0.01, indicating that water glass impregnation modification had a significant effect on the weight leaching rate of rubberwood. Higher concentrations of sodium silicate led to increased leaching rates, which correlates with the color changes in the leachate shown in Fig. 2b.

Table 1. Weight Leaching Rates of Three Concentrations of Modified Materials

Sample	10% RW-SS	20% RW-SS	30% RW-SS
Leaching efficiency	2.2	3.18	3.58

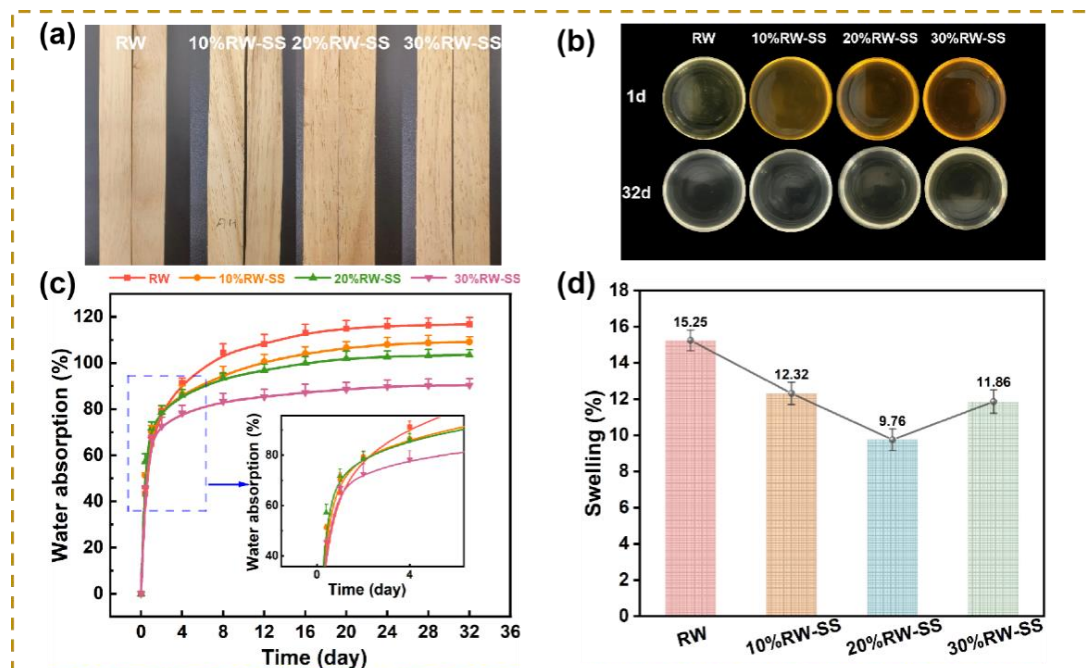


Fig. 2. (a) Surface of untreated and modified wood samples, (b) Comparison of the leachate color on Day 1 and Day 32, (c) Water absorption, (d) Swelling percentage

Table 2. Analysis of Variance (ANOVA) Results of the Weight Leaching Rates Sodium Silicate-Modified Rubberwood

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (df)	Mean Square (MS)	F	p-value	F crit
Between Groups	2.869689	2	1.434844	22.91677	0.001551	5.143253
Within Groups	0.375667	6	0.062611			
Total	3.245356	8				

Notes: p-value ≤ 0.01

As shown in Fig. 2c, the water absorption of the wood samples reflected the impact of different sodium silicate concentrations. Water absorption increased significantly within the first 24 hours, reached near-saturation by Day 8, followed by a gradual stabilization. The unmodified wood had a water absorption of 67.6%, while the 30% RW-SS sample, which exhibited the most effective modification for water absorption, reached 68%. This indicates that, under short-term soaking conditions, the sodium silicate impregnation modification did not result in a significant improvement in water resistance. The unmodified wood exhibited a water absorption of 104.4%, whereas the 10% RW-SS, 20% RW-SS, and 30% RW-SS treated specimens demonstrated significantly reduced absorption percentages of 94.8%, 93.5%, and 83.4%, corresponding to reductions of 9.2%, 10.4%, and 20.1% compared to the control, respectively. Following 8 days of immersion, the increasing rate of water absorption was markedly reduced in all specimens, suggesting that the internal pore structure of the wood had achieved a relatively stable moisture equilibrium state due to the effective pore-filling by sodium silicate gels. During the subsequent water absorption process, it is observed that as the soaking time was increased, the rate of increase in water absorption gradually diminished. The trend of water absorption across different concentrations of modified wood remained relatively consistent, with the rate of change in sample mass exhibiting stability. At this juncture, the water absorption of the unmodified wood was recorded at 116.8%, whereas the water absorption for the 30% RW-SS modified material was 90.3%, reflecting a reduction of 22.7% in comparison to the unmodified material. The water absorption percentages for the 10% RW-SS and 20% RW-SS modified materials were 109.1% and 103.5%, respectively. The modification effect of 30% sodium silicate was particularly pronounced, demonstrating a significant enhancement in water absorption relative to untreated wood.

Figure 2d presents the swelling ratios of untreated and sodium silicate-modified wood. A lower swelling ratio indicates better dimensional stability of the wood (Hartwig-Nair *et al.* 2024). The wet expansion rate of the untreated material reached as high as 15.2%. After modification with sodium silicate at concentrations of 10% to 30%, the wet expansion percentages decreased to 12.3%, 9.8% and 11.9%, respectively. Among these, the material modified with 20% sodium silicate concentration exhibited the best dimensional stability. The minimum swelling percentage (9.8%) was achieved at a sodium silicate concentration of 20%, which represents a 36% reduction compared to the unmodified wood. This result shows an improvement over the findings of Baar (Baar *et al.* 2020), who reported a reduction of only 25%. Notably, this trend does not fully align with the water absorption behavior, suggesting a complex interaction between modification effects and hygroscopic properties.

When the concentration was 20%, the reduction of water absorption and combined with the analysis of the lowest data of wet swelling rate, due to the effective penetration of the water glass modifier into the wood structure, filling the internal pores, thus reducing the porosity and water transport (Zhou *et al.* 2020). Furthermore, the modifier chemically bonds with certain hydroxyl (-OH) groups, thereby decreasing the number of moisture adsorption sites and resulting in a lower water absorption rate (Li *et al.* 2021). This suggests that the moderate concentration of sodium silicate was able to form a uniformly distributed silicate network in the interior of the wood and thereby effectively inhibit the moisture-absorption and swelling of the cell wall. However, the 30% sodium silicate-modified sample exhibited the lowest water absorption but showed a significantly reduced swelling ratio compared to the 20% sample. Although increased viscosity at higher concentrations hindered solution penetration, the 30% treatment formed a denser surface barrier layer that

effectively restricted rapid water uptake despite uneven internal distribution. In contrast, the 20% concentration achieved more uniform internal filling but likely developed a less effective surface barrier, resulting in slightly inferior water absorption performance while maintaining better overall dimensional stability through balanced penetration and deposition effects.

Mechanical Properties

According to the mechanical property data of sodium silicate modified rubberwood shown in Fig. 3(a), combined with the analysis of density change in Fig. 3(b), it can be concluded that sodium silicate modification significantly improved the mechanical properties of rubberwood. The modification process involving sodium silicate results in the deposition of a substantial quantity of silicate on the cell walls and within the cell cavities of the wood.

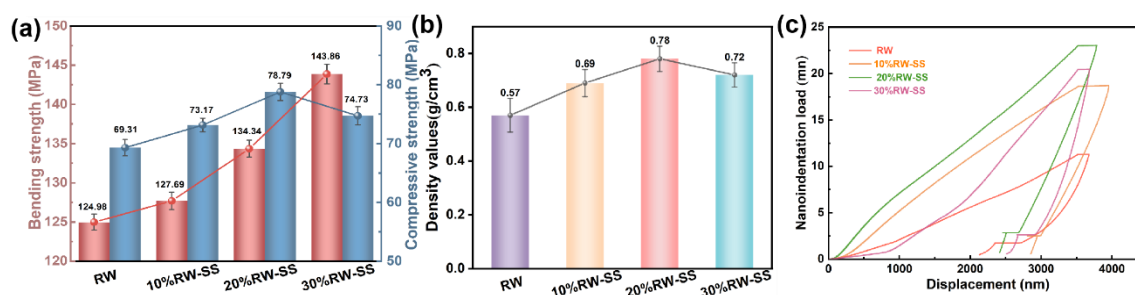


Fig. 3. The bending and compressive properties of both unmodified material and sodium silicate-impregnated modified rubberwood (a), density values (c), nanoindentation load-displacement curve (b)

This modification not only preserves the structural integrity of the wood fibers but also significantly enhances the cells capacity to withstand external forces, leading to a marked increase in both the bending elastic modulus and bending strength (Żurawska *et al.* 2002; Bi *et al.* 2022; Zhang *et al.* 2023). As shown in Tables 3 and 4, the p-value was less than 0.01, indicating that the sodium silicate impregnation modification had a significant effect on the bending and compressive strength of rubberwood. The density, bending strength, and compressive strength of unmodified rubberwood were 0.57 g/cm³, 125 MPa and 69.3 MPa, respectively. After modification with sodium silicate, the density of the wood increased, and the bending strength and compressive strength showed different degrees of improvement. This enhancement can be attributed to the silica particles formed from the reaction between the curing agent sodium bicarbonate and sodium silicate, which fill the pores of the wood.

Table 3. Analysis of Variance (ANOVA) Results of the Bending Strength of Unmodified and Sodium Silicate-Modified Rubberwood

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (df)	Mean Square (MS)	F	p-value	F crit
Between Groups	635.2387	3	211.7462	7.792103	0.009276	4.066181
Within Groups	217.3957	8	27.17446			
Total	852.6344	11				

Notes: p-value ≤ 0.01

Table 4. ANOVA Results of the Compressive Strength of Unmodified and Sodium Silicate-Modified Rubberwood

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (df)	Mean Square (MS)	F	p-value	F crit
Between Groups	138.4538	3	46.15127	10.17024	0.004184	4.066181
Within Groups	36.30299	8	4.537874			
Total	174.7568	11				

Notes: p-value ≤ 0.01

This filling action provided a degree of reinforcement to the cellulose and hemicellulose, thereby augmenting the bending and compressive properties (Mahltig *et al.* 2008). As illustrated in Fig. 3a, there was a positive correlation between the concentration of sodium silicate and the bending strength of rubberwood. Specifically, an increase in sodium silicate concentration corresponded to an enhancement in the bending strength of rubberwood. At a sodium silicate concentration of 30%, the bending strength reached a peak value of 144 MPa, representing a 15% improvement relative to the unmodified material. In terms of compressive strength, the maximum value was observed at 20% sodium silicate concentration, with an increase of 13.7% compared to the unmodified material, when the density also reached a maximum value of 0.78 g/cm³. This is closely related to the formation of a uniform and dense filling network of sodium silicate inside the wood, a structure that effectively enhances the overall load-bearing capacity of the cell wall. However, when the concentration was elevated to 30%, a decrease of 5.2% in compressive strength was noted in comparison to the 20% sodium silicate modified wood. This decrease may be attributed to the high concentration of sodium silicate solution and the high solid content; such an increase may hinder the penetration and migration capabilities of the modifier, ultimately leading to a reduced amount of sodium silicate in the cell cavities and pores of the rubberwood.

The hardness of wood is significantly influenced by the microscopic structure of its cell walls. In recent years, researchers have increasingly employed nanoindentation technology to assess the hardness of wood cell walls, thereby facilitating a more precise understanding of their physical properties (Wu *et al.* 2019; Golovin *et al.* 2023). As illustrated in Fig. 3c, during the loading process, the extent of penetration of impregnated modified wood was lower than that of unmodified wood. The maximum load that rubberwood can withstand was measured at 11.3 mN. Following impregnation with sodium silicate solutions at concentrations of 10%, 20%, and 30%, the maximum load capacity exhibited a trend of initial increase followed by a decrease, with recorded values of 18.5, 23 and 20.5 mN, respectively. This trend indicates that the resistance of rubberwood to external forces was enhanced after modification. Furthermore, when the indentation depth ranged from 0 to 3300 nm, the load increased sharply with the increasing indentation depth. Upon reaching its maximum value, the load entered a holding stage, which was subsequently followed by the unloading phase. The data presented in Table 5 indicate that the hardness of the unmodified rubberwood cell wall was measured at 40 MPa, while the elastic modulus was recorded at 1400 MPa. By comparing the mechanical properties of rubberwood cell wall before and after modification, sodium silicate can effectively enhance the elastic modulus and hardness of cell wall. Upon impregnation with sodium silicate solutions of varying concentrations, the hardness of the cell wall exhibited a trend characterized by an initial increase followed by a subsequent decrease as the concentration

of sodium silicate rises. The peak hardness was observed in the cell wall modified with a 20% sodium silicate solution, achieving a value of 100 MPa. Concurrently, the elastic modulus also demonstrated an upward trend with increasing sodium silicate concentration, attaining a maximum value of 2400 MPa at a 30% concentration of RW-SS. A comparative assessment of the variations in cell wall hardness, elastic modulus, and load suggests that the impregnation of rubberwood with sodium silicate is instrumental in enhancing its mechanical properties.

Table 5. Indentation Depth, Hardness, and Elastic Modulus of Unmodified Material and Sodium Silicate-Modified Rubberwood

Sample	Depth of Impression (nm)	Hardness (MPa)	Elasticity Modulus (MPa)
RW	3668	40	1400
10% RW-SS	3896	80	1700
20% RW-SS	3744	100	1900
30% RW-SS	3663	80	2400

Microscopic Morphology Map

To investigate the distribution of sodium silicate within rubberwood, field emission scanning electron microscopy (FESEM) was employed to analyze the internal morphology of both the rubberwood and the modified material. Figure 4 illustrates the microscopic morphology of the cross-section of rubberwood, with images I-IV representing the microscopic morphology of the rubberwood material, 10% RW-SS, 20% RW-SS, and 30% RW-SS at a magnification of 500 times, respectively. Images V-VIII depict the microscopic morphology of the same materials at a magnification of 5000 times.

As shown in Fig. 4I, only a small number of vessel elements in the rubberwood contained starch granules, while the majority of the vessel elements appeared almost hollow. After the sodium silicate impregnation treatment, the cell walls of the rubberwood underwent some deformation and shrinkage. The occurrence of this phenomenon may be due to the partial dissolution of hemicellulose in the cell walls during treatment with an alkaline solution under high pH (11 to 13) conditions (Nguyen *et al.* 2019; Xu *et al.* 2020; Nguyen *et al.* 2024). The 20% sodium silicate modification exhibited the best impregnation and filling effect. After drying and curing, it accumulated in the wood's lumens, making the lumens appear almost completely filled, with little to no visible hollowness. Yang *et al.* (2008) found that within the concentration range of 15% to 55%, the concentration of sodium silicate is positively correlated with viscosity the higher the concentration, the greater the viscosity.

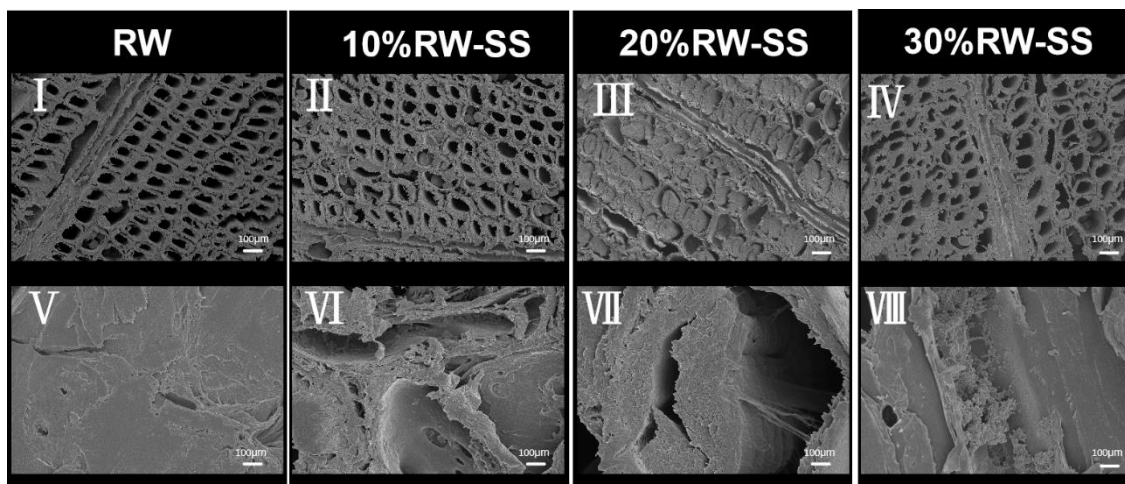


Fig. 4. The microstructure of the material, specifically the sodium silicate-modified rubberwood. I-IV are 500x microscopic morphology images, V-VIII are 5000x microscopic morphology images.

Table 6. Viscosity Measurements of Three Sodium Silicate Solutions

Solution	10%SS	20%SS	30%SS
Viscosity (mPa.s)	3.5	5.3	9.3

As shown in Table 6, the viscosities of sodium silicate solutions at concentrations of 10%, 20%, and 30% were 3.5, 5.3, and 9.3 mPa·s, respectively. As the concentration increased, the viscosity also increased. In Fig. 4VIII, a layer of solid sodium silicate is observed to adhere to the thin walls surrounding the lumens of rubberwood treated with the 30% concentration modifier. However, the filling effect was not as effective as with the 20% concentration. This may be due to the higher viscosity and lower permeability of the 30% sodium silicate solution, which resulted in less impregnation of sodium silicate into the rubberwood and consequently less deposition within the lumens. Combined with the analysis of the water absorption tests, the impregnation effect of the 30% concentration modifier was suboptimal, but it provided the greatest improvement in water absorption. This suggests that sodium silicate deposited primarily on the surface area of the sample, resulting in surface modification rather than significant internal impregnation.

Fourier Transform Infrared (FT-IR) Analysis

Figure 5 presents the FTIR spectra of both unmodified rubberwood and rubberwood modified with sodium silicate. Compared to the untreated rubberwood, the intensity of some absorbance peaks changed after impregnation with sodium silicate. Specifically, the absorbance peak observed around 3300 cm^{-1} is associated with the stretching vibration of -OH groups. Notably, compared with the untreated material, the vibration peak of hydroxyl group shifted to the direction of low wave number, indicating the change of some hydroxyl groups, which may be related to the modification effect of sodium silicate. This observation further confirms that sodium silicate has successfully impregnated into the rubberwood and interacted with the wood components.

The peak at 2919 cm^{-1} corresponds to the C-H stretching vibration, a functional group present in all three main components of rubberwood (Wang *et al.* 2024). After modification, the intensity of this peak was weakened, indicating that sodium silicate induced partial decomposition and degradation of the wood. Furthermore, the intensity of

the absorbance peak near 1737 cm^{-1} diminished, signifying damage to the acetyl groups within the hemicellulose of the wood. This phenomenon can be attributed to the strong alkalinity of sodium silicate, which catalyzes the hydrolysis of carboxylic esters, resulting in the elimination of carboxylic acids (Kuai *et al.* 2022). This indicates that the modifier can enter the wood cell wall, which is consistent with the FSEM observation that sodium silicate is distributed inside the wood.

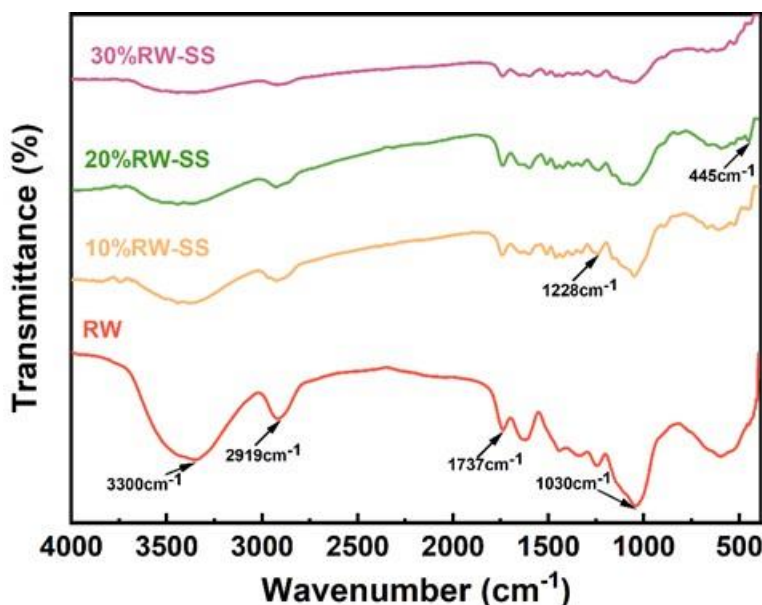


Fig. 5. FT-IR spectra of both unmodified material and sodium silicate-modified rubberwood

Additionally, the absorbance peak of lignin phenolic ether at 1228 cm^{-1} was weakened, and lignin phenolic ether was consumed, which indicates that hemicellulose had changed. The variations observed in the cellulose C-O stretching vibration peak, located near 1030 cm^{-1} , were notably significant, suggesting alterations in the structural composition of cellulose. The modification of cellulose may enhance the fiber bonding within the wood, thereby promoting the structural stability of the wood and improving its overall mechanical properties. In addition, the intensity of the absorbance peak decreased at 898 cm^{-1} , which is the unique C-H stretching vibration peak of cellulose or hemicellulose (Pandey and Nagveni 2007; Qu *et al.* 2019; Li *et al.* 2020). This indicates that interactions occurred between the impregnating liquid and the three primary components of rubberwood. The modified sample, following impregnation, exhibited characteristic peaks of silica, which are identified at approximately 810 cm^{-1} for the bending vibration of Si-O-Si and around 445 cm^{-1} for the symmetric stretching vibration of Si-O-Si (Liu *et al.* 2011). These findings suggest that sodium silicate had penetrated the wood and subsequently formed silica upon drying and curing. In summary, the FTIR analysis revealed significant alterations in the chemical structure of sodium silicate-modified rubberwood, which exhibits direct correlations with the improved mechanical and physical properties of the treated wood.

X-ray Diffraction (XRD) Analysis

As illustrated in Fig. 6, under different treatment conditions, the shape and position of the cellulose characteristic peaks remained largely consistent, with only variations in the

diffraction peak intensity. This observation suggests that the modification with sodium silicate does not alter the crystal structure of the cellulose, but rather it results in changes to the relative crystallinity. There were significant diffraction peaks at 18° , 22° , and 34° , which correspond to the cellulose crystalline planes (101), (002), and (040), respectively. The peaks at approximately $2\theta=22^\circ$ and $2\theta=34^\circ$ correspond to the diffraction peaks of the cellulose crystalline planes (Zhang *et al.* 2019). After modification, a marked reduction in the intensity of the diffraction peaks for the (101) and (002) crystalline planes is evident.

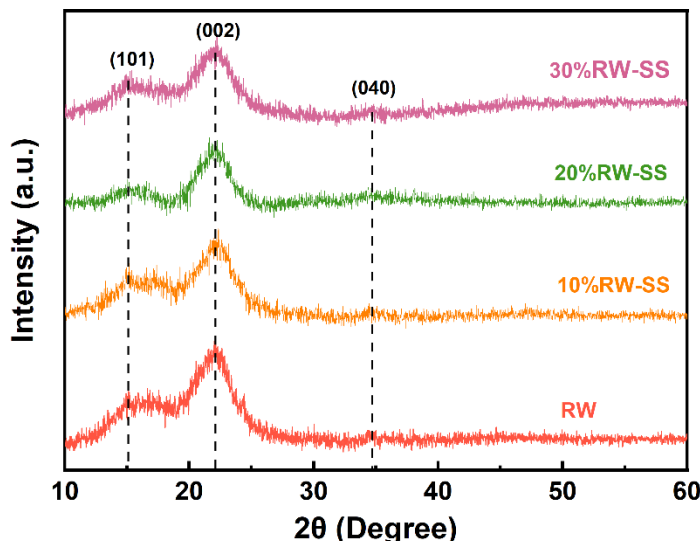


Fig. 6. The XRD patterns of materials, including sodium silicate-modified rubberwood

The crystal structure of cellulose in the cell wall is an important factor influencing its crystal size, unit cell parameters, and crystallinity. Relative crystallinity refers to the proportion of the crystalline region in the microfibrils to the whole cellulose (Liao *et al.* 2024). The degree of crystallinity in wood is intricately linked to its dimensional stability, hardness, tensile strength, and density (Cai *et al.* 2018). The relative crystallinity of cellulose in the untreated wood was 63.1%. In contrast, the crystallinity of cellulose modified with 10% RW-SS was recorded at 56.2%, while the crystallinities for 20% RW-SS and 30% RW-SS were observed to have increased to 90.1% and 72.4%, respectively. The crystallinity of the modified wood increased, but the crystallographic structure remained unchanged, indicating that the modifying agent did not disrupt the internal physical structure of the wood. Instead, it enhanced the interchain bonding of the cellulose molecules through interactions with the wood's cellulose. This increased crystallinity may be attributed to the interaction between sodium silicate and the wood cellulose, forming a more stable chemical structure, which in turn improved the mechanical properties of the wood. This observation further corroborates the formation of silica crystallization within the rubberwood following the impregnation modification process.

X-ray Photoelectron Spectroscopy (XPS) Analysis

As illustrated in Fig. 7, the unmodified rubberwood predominantly comprised carbon (C) and oxygen (O) elements. Following modification through sodium silicate impregnation, the presence of C and O elements persisted, while peaks corresponding to silicon (Si) and sodium (Na) elements also emerged.

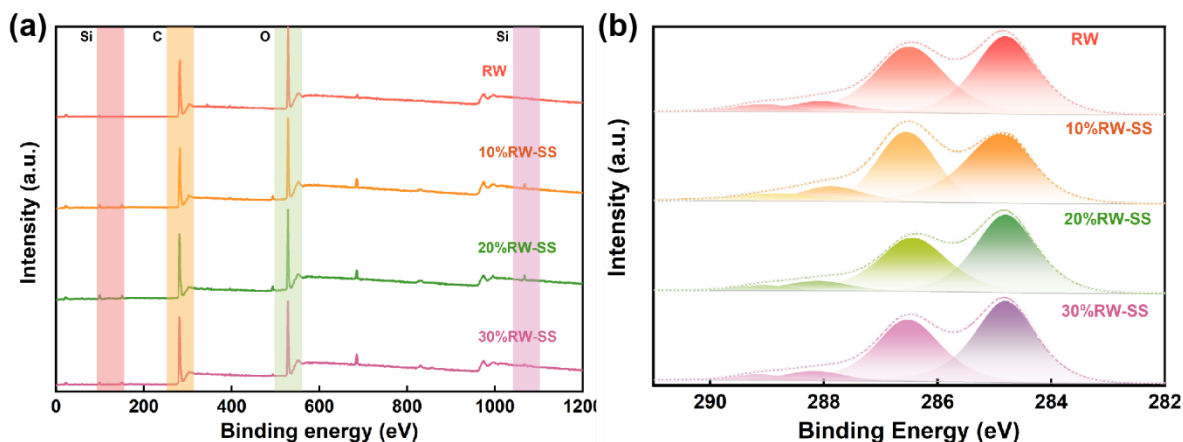


Fig. 7. The wide scan XPS spectra of both unmodified material and sodium silicate-modified rubberwood (a), narrow scan of the carbon (C) element (b)

The peaks for C and O elements across all sample groups exhibited relatively strong intensities, with their electron binding energies ranging from 279.28 to 298.08 eV and 525.28 to 545.08 eV, respectively. There was a significant increase in the peaks for C and O elements post-modification. The peaks for Si and Na elements were observed at binding energies of 95.28 to 110.08 eV and 1062.28 to 1079.08 eV (Zhang *et al.* 2023). Both elements originated from sodium silicate. Therefore, the changes in these peaks indicate the successful penetration of the sodium silicate modifier into the rubberwood. Furthermore, as the concentration of the sodium silicate solution increased, the peaks exhibited a trend of initially increasing followed by a subsequent decrease.

Table 7. The Relative Content of Elements of Various Chemical States of Materials, Including Sodium Silicate-Modified Rubberwood

Sample	C1 (%)	C2 (%)	C3 (%)	C4 (%)	C	O	Na	Si
RW	44.62	43.21	8.26	3.91	69.21	30.79	/	/
10% RW-SS	46.69	36.18	8.90	8.23	65.74	30.4	1.04	2.82
20% RW-SS	50.40	39.56	6.85	3.18	65.58	30.03	1.12	3.27
30% RW-SS	51.73	39.85	5.25	3.17	67.28	29.79	0.86	2.06

In Table 7, the relative content of Na and Si elements was the highest in 20% RW-SS, and the content of these two elements in 30% RW-SS was not as high as that in 10% RW-SS and 20% RW-SS. This also confirms that the filling effect of 30% RW-SS observed in the microstructure images was poor. This may be due to its high viscosity, which makes it difficult to impregnate into the wood. To elucidate the alterations in the composition of the sodium silicate-modified rubberwood cell walls, peak fitting analysis was conducted on the C element. In unmodified rubberwood, the contents of C1, C2, C3, and C4 were 44.62%, 43.21%, 8.26%, and 3.91%, respectively. Clearly, C1 and C2 were the predominant forms in which carbon atoms were present (Meng *et al.* 2016; Li *et al.* 2021). The decreased C2 content in modified wood indicates a reduction in carbon atoms bonded to -OH groups. This phenomenon is attributed to crosslinking reactions between sodium silicate and hydroxyl groups within the wood matrix, leading to the formation of new chemical bonds (Si-O-C) that enhance the structural stability of the modified wood. This change corresponds to the shift of the peak at 3723 cm^{-1} in the FTIR spectrum towards lower wavenumbers. The decrease in O content post-modification is ascribed to sodium

silicate deposition, its reaction with hydroxyl (-OH) groups, and alterations in the wood's surface chemical structure. The contents of Na and Si elements exhibited a characteristic trend of initial increase followed by decrease with rising sodium silicate concentration, reaching their peak levels at the 20% concentration. This optimal concentration enables sodium silicate to most effectively interact with the wood surface, forming stable chemical bonds, which leads to an increase in the content of Na and Si. Based on the above analysis, it can be concluded that the modifier not only physically fills and densifies the wood, but also penetrates the cell walls of the wood, forming a strong chemical bond with the main components of the wood.

Differential Scanning Calorimetry (DSC) Analysis

Figure 8 presents the DSC curves for unmodified wood and modified wood with sodium silicate at varying concentrations, with a primary focus on the thermal stability of rubberwood subjected to different treatment concentrations.

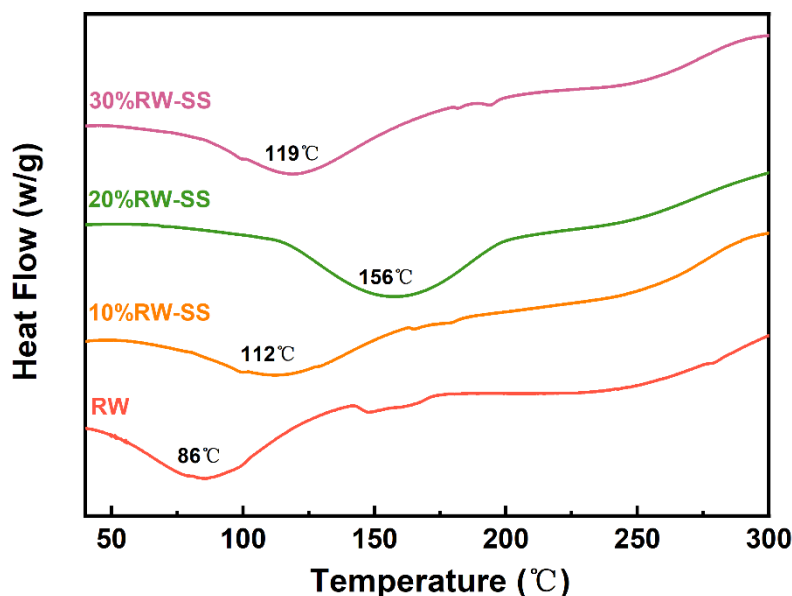


Fig. 8. The DSC curves for both unmodified material and sodium silicate-modified rubberwood

The impregnation of wood with sodium silicate solution has been shown to enhance its thermal stability (Liu *et al.* 2023; Bi *et al.* 2023). Following modification, the glass transition temperatures for the 10% RW-SS, 20% RW-SS, and 30% RW-SS samples were within the ranges of 80 to 85, 110 to 115, and 85 to 90 °C, respectively. As the temperature rose, rubberwood exhibited a prominent endothermic peak, with the peak position varying across the samples. The highest endothermic peak for the unmodified rubberwood occurred at 86 °C, which is likely associated with the evaporation of moisture in the wood (Che *et al.* 2018). As the temperature increased, the modified wood exhibited a notable endothermic peak, with the position of this peak shifting to higher temperatures. The maximum endothermic peak temperatures for 10% RW-SS and 30% RW-SS were recorded at 112 and 119 °C, respectively, reflecting an increase of approximately 30 °C in comparison to the unmodified wood. The endothermic peak for 20% RW-SS was observed at 156 °C, representing a significant increase of 70 °C relative to the unmodified wood, thereby indicating a pronounced modification effect. The endothermic peak demonstrated a trend of initially increasing and subsequently decreasing with the rise in the concentration

of the modifier. This phenomenon may be attributed to the impregnation of sodium silicate into the wood, which forms silica gel upon drying and curing. As free moisture evaporates and the silica gel undergoes dehydration, it condenses and solidifies within the vessels, resulting in the formation of a silica network structure that exhibits enhanced heat resistance (Zhang *et al.* 2020; Yang *et al.* 2021), thereby improving the thermal stability of rubberwood. The observed decrease in the temperature of the endothermic peak position for 30% RW-SS, in comparison to 20% RW-SS, suggests that the quantity of sodium silicate impregnated into the rubberwood was relatively low. This observation is further corroborated by microscopic morphology images, which indicate that the amount of modifier impregnated into the pores of the rubberwood is less than that observed in the 20% RW-SS samples.

CONCLUSIONS

This article has examined rubberwood (RW) as the primary subject of research, utilizing sodium silicate (SS) with a module value of 3.4 as the modifying agent. The sodium silicate solution was introduced into the wood *via* a vacuum pressure impregnation modification technique. A comparative analysis was performed using 10% RW-SS, 20% RW-SS, and 30% RW-SS in relation to the untreated rubberwood. The conclusions were made based on assessments of physical and mechanical properties, chemical analyses, and observations of the microstructure.

1. It was found that sodium silicate impregnation modification can improve the mechanical properties of rubberwood to a certain extent. Specifically, a concentration of 20% RW-SS yielded the most significant improvement in compressive strength, exhibiting an increase of 13.7% relative to untreated wood. Conversely, the optimal modification effect on bending strength was achieved with a concentration of 30% RW-SS, resulting in a 15% increase.
2. Field emission scanning electron microscopy (FESEM) images showed that the 20% RW-SS sample exhibited the best pore-filling effect among those tested. In contrast, the pore-filling effects for rubberwood impregnated with sodium silicate solutions at 10% or 30% concentrations were less significant.
3. The water absorption and swelling tests indicated that, compared to the untreated samples, the 30% RW-SS modification significantly reduced water absorption by 22.7%, while the 20% RW-SS modification reduced swelling by 36%. This further suggests that the 20% sodium metasilicate concentration effectively reduced moisture absorption and swelling through uniform penetration and chemical bonding, improving dimensional stability. In contrast, the 30% concentration forms a dense surface barrier due to its high viscosity, which, despite insufficient penetration, significantly reduced water absorption.
4. Based on the analyses using Fourier transform infrared (FTIR), X-ray diffraction (XRD), and X-ray photoelectron spectrometry (XPS), it can be concluded that following the impregnation of sodium silicate and subsequent curing treatment, silica was generated within the wood. This process led to interactions with the three major components of rubberwood, culminating in the formation of stable chemical bonds.

5. Sodium silicate impregnation significantly enhanced the thermal stability of rubberwood. Higher concentrations improve heat resistance, with optimal modification observed at 20% RW-SS, showing a 70 °C increase in heat absorption peak compared to untreated wood.

This study systematically evaluated the concentration-dependent effects of sodium silicate modification on rubberwood properties, demonstrating that 20% concentration exhibited optimal performance enhancement potential. Further investigations should focus on assessing the environmental stability of silicate gels in modified wood under cyclic hygrothermal conditions, including long-term durability testing across diverse environmental exposures (*e.g.*, thermal cycling, humidity fluctuations, and UV radiation), as well as comparative performance evaluations of sodium silicate against alternative inorganic modifiers (such as potassium silicate or geopolymer-based systems). Additionally, future work should prioritize optimizing the impregnation-curing process to enhance treatment efficiency and mechanical performance, while developing sustainable protocols for waste liquid treatment; concurrently, cost-effective modification strategies should be established through comprehensive techno-economic analysis, coupled with the development of nano-reinforced silicate systems to minimize chemical consumption without compromising wood functionalization efficacy.

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Article submitted: January 7, 2025; Peer review completed: March 22, 2025; Revisions accepted: April 25, 2025; Published: June 6, 2025.
DOI: 10.15376/biores.20.3.6033-6053