

Enhancement of mechanical and flame-retardant properties of Acacia hybrid through the treatment of a combination of styrene-acrylic copolymer and sodium silicate

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Nâng cao tính chất cơ học và đặc tính chống cháy của gỗ Keo lai (*Acacia hybrid*) bằng cách xử lý kết hợp chất đồng trùng hợp styrene-acrylic và natri silicat

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Từ khóa:

Biến tính gỗ, chất đồng trùng hợp styren-acrylic, chống cháy Keo lai, natri silicat, tính chất cơ học.

ABSTRACT

The mechanical and flame-retardant properties of Acacia hybrid wood were studied through the treatment of an aqueous solution containing a styrene-acrylic copolymer (SC) and sodium silicate (SS). The results showed a significant increase in the mechanical properties of SC/SS-treated wood. Specifically, the modulus of rupture (MOR) and modulus of elasticity (MOE) improved by up to 71.54% and 53.65%, respectively. The surface hardness of the cross-section and radial section also increased by 37.24% and 46.65%, respectively. The treated wood's weight percentage gain (WPG) reached up to 23.31%. The largest leaching rate (LR) of treated wood was only 8.01%, suggesting this treatment has obvious effects in fixing the SS in the wood. However, it did not cause positive bulking efficiency (BE) and anti-swelling efficiency (ASE). The incorporation of SS with SC resulted in the decomposition of polysaccharides at a lower temperature while increasing the limiting oxygen index (LOI), which was 34.95% higher than that of untreated wood, making it more difficult to ignite. Scanning electron microscopy (SEM) results showed that the SC/SS deposited in the cell lumina after polymerization, but significant structural changes occurred, including vessel and cell wall deformation due to the partial dissolution of hemicellulose and lignin from the cell wall in the alkaline solution. The results of this study demonstrate the feasibility of applying the SC/SS combination treatment to wood in practical applications.

TÓM TẮT

Tính chất cơ học và đặc tính chống cháy của gỗ Keo lai (*Acacia hybrid*) được nghiên cứu khi xử lý bằng dung dịch gồm chất đồng trùng hợp styren-acrylic (SC) và natri silicat (SS). Kết quả cho thấy tính chất cơ học của gỗ đã được xử lý bằng SC/SS đã tăng lên đáng kể. Cụ thể, độ bền uốn tĩnh (MOR) và mô đun đàn hồi uốn tĩnh (MOE) đã tăng lần lượt lên tới 71,54% và 53,65%. Độ cứng bề mặt của mặt cắt ngang và xuyên tâm cũng tăng lên lần lượt là 37,24% và 46,65%. Tỷ lệ tăng khối lượng (WPG) gỗ sau xử lý lên tới 23,31%. Tỷ lệ rửa trôi hóa chất (LR) của gỗ đã được xử lý chỉ còn 8,01%, cho thấy phương pháp xử lý này có hiệu quả rõ rệt trong việc cố định SS trong gỗ. Tuy nhiên, nó không mang lại tác dụng tích cực khi xác định sức căng vách tế bào (BE) và khả năng chống trương nở (ASE). Việc kết hợp SC và SS dẫn đến quá trình phân hủy các polysaccharide ở nhiệt độ thấp hơn đồng thời làm tăng chỉ số giới hạn oxy (LOI), cao hơn 34,95% so với gỗ chưa được xử lý, khiến gỗ khó bắt lửa hơn.

Kết quả từ kính hiển vi điện tử quét (SEM) cho thấy, SC/SS được tích tụ trong ruột tế bào gỗ sau quá trình polymer hóa, đồng thời cũng xảy ra những thay đổi đáng kể về cấu trúc hiển vi như sự biến dạng của các mạch gỗ và vách tế bào gỗ do một phần hemicellulose và lignin bị hòa tan trong môi trường kiềm. Kết quả của nghiên cứu này chứng minh tính khả thi của việc áp dụng phương pháp xử lý kết hợp SC/SS cho gỗ trong các ứng dụng thực tế.

1. INTRODUCTION

To reduce the logging of naturally-grown forests and protect the ecological environment of forests, the fast-growing plantation wood species such as Acacia hybrid (*Acacia mangium* × *Acacia auriculiformis*) have become the primary raw material source for Vietnam's wood industry nowadays. Nevertheless, these woods often exhibit low density, low durability, and less mechanical strength, which limits their applications and development [1, 2]. They are also more susceptible to fire hazards than denser natural woods. Chemical modification of wood offers a promising option to improve the quality and produce added value of fast-growing timber [3].

Sodium silicate (SS, also called water glass), the most commonly used silicon compound for wood modification, has been extensively reported recently [4-6]. Wood treatments with SS solutions have enhanced the wood's dimensional stability, hardness, decay, and fire resistance [7, 8]. However, the leaching of silica particles from wood during service poses a potential issue that must be addressed [9]. To mitigate this problem, some researchers have combined SS with polymerizing resins (such as glyoxal-urea, urea-formaldehyde resin, phenolic resin, melamine-formaldehyde resin, and urea-modified melamine-formaldehyde resin) to reduce the leaching of silica [9]. This approach primarily relies on the physical encapsulation mechanism of polymerized resin within the wood.

The styrene-acrylic copolymer (SC) is prepared via a batch emulsifier-free emulsion copolymerization of styrene with acrylic acid. The combination of SC and SS used to treat poplar (*Populus adenopoda* Maxim.) and radiata pine (*Pinus radiata* Don.) woods in previous research has shown evident

effectiveness in enhancing wood properties. The results revealed that this method could improve some mechanical properties and increase the ignition resistance of the woods, as evidenced by longer ignition times and higher limiting oxygen index values [10, 11]. However, using a combination of SC and SS remains relatively unexplored within the context of Acacia hybrid wood in Vietnam.

Therefore, this study aims to bridge this gap by assessing the mechanical strength, flame-retardant capabilities, and structural changes induced by the treatment of Acacia hybrid with SC and SS. The leaching rate (LR) of the agent due to water immersion was also investigated. The findings of this study are expected to contribute to the development of sustainable wood composites with enhanced fire performance, thus expanding the potential applications of Acacia hybrid in wood industries.

2. RESEARCH METHODS

2.1. Materials

The treated wood was Acacia hybrid sapwood (oven-dried density of approximately 0.45 g cm⁻³), originating in Ba Vi, Hanoi, Vietnam. Acacia hybrid wood was cut into specimens with dimensions of 10 × 20 × 20 mm³ and 150 × 6.5 × 3 mm³ ((Longitudinal (L) × Tangential (T) × Radial (R)) for determination of weight percent gain (WPG), leaching test, cell wall bulking efficiency (BE) and anti-swelling efficiency (ASE), and limiting oxygen index (LOI) test, respectively. For mechanical properties tests, wood specimens measuring 180 × 10 × 10 mm³ and 40 × 30 × 30 mm³ (L × T × R) were used to determine the bending strength and surface hardness test, respectively. All specimens were conditioned in the climate chamber (65% relative humidity and 20°C) to a moisture content of approximately 10-12% prior to treatment.

SS (white powder with a silica/alkali molar ratio of 1.03/1.0) was purchased from Fuchen Chemical Reagent Factory (Tianjin, China). SC (solid content 33%, pH 8.46, and an average molecular mass of 15000 g mol⁻¹) was obtained from Diransa San Luis S.A (Buenos Aires, Argentina). Polycarbodiimide (PC) crosslinking agent was of extra purity grade and purchased from Xirun Chemical Technology Co., Ltd. (Shanghai, China). The pH of the final treatment solutions was measured using a model HM-40X pH Meter (TOA DKK, Japan).

2.2. Treatment of wood specimens

Wood specimens were oven-dried at 103°C for 24 h and then impregnated by a full-cell process under initial vacuum (-0.01 MPa, 12 h)

and pressure (0.5 MPa, 24 h) in aqueous solutions. The aqueous solutions contained 15% SS or 10% SC plus 0, 5, 10, or 15% SS (based on the total weight of the solution). In addition, 10% of the PC crosslinking agent (based on the weight of SC) was added to each solution to catalyze to form a polymeric network (Table 1) readily. The treated wood specimens were abbreviated as W_{15%SS}, W_{10%SC}, W_{10%SC+5%SS}, W_{10%SC+10%SS} and W_{10%SC+15%SS}, respectively. Untreated wood specimens (abbreviated as W_{CTRL}) served as controls. After impregnation, samples were removed from the tank and dried in air at room temperature for one week. Afterward, the specimens were cured at 80°C for 24 h and then at 110°C for 24 h.

Table 1. Test groups of wood with different treatment solutions

Labels	pH value	Treatment solution		
		SC (%)	SS (%)	PC* (%)
W _{CTRL}	—	0	0	0
W _{15%SS}	13.3	0	15	0
W _{10%SC}	8.33	10	0	10
W _{10%SC+5%SS}	11.85	10	5	10
W _{10%SC+10%SS}	12.83	10	10	10
W _{10%SC+15%SS}	13.01	10	15	10

Note: PC addition is defined based on the weight percentage of the SC.

2.3. Methods of analysis

2.3.1. Weight percentage gain (WPG)

Weight percentage gain (WPG) is an important index for testing the effects of wood impregnation and evaluating the amount of impregnator penetrating the wood. The calculation formula (Eq. 1) employed here was:

$$WPG (\%) = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

where W₁ and W₂ are the oven-dry weight of untreated and treated wood specimens, respectively. Ten replicates were used for each group in the experiments.

2.3.2. Leaching test

The leaching test was assessed by applying the method in the EN 84:1997 standard. The sample immersion was performed for 14 days

in distilled water, which changed daily at room temperature. LR of the modifier after the sample was immersed in water was determined according to Eq. 2:

$$LR (\%) = \frac{W_{bl} - W_{al}}{W_{bl}} \times 100 \quad (2)$$

where W_{bl} and W_{al} are the oven-dry weight of treated wood specimens before and after leaching test, respectively. Ten replicates were used for each group in the experiments.

2.3.3. Cell wall bulking efficiency (BE) and anti-swelling efficiency (ASE)

Cell wall bulking efficiency (BE) was assessed as a dimensional change (%) in the specimens in the dry state before and after treatment and calculated using the following formula:

$$BE (\%) = \frac{V_{at} - W_{bt}}{V_{bt}} \times 100 \quad (3)$$

where V_{at} and V_{bt} the oven-dry volume of the specimens after and before treatment, respectively (mm^3).

The dimensional stability was calculated based on the volumetric swelling coefficient (S) and the ASE. The ASE between the oven-dried and water saturation states was determined according to Hill [3]. Based on the difference in swelling between the treated and the control samples, the ASE was calculated as follows:

$$ASE (\%) = \frac{S_u - S_t}{S_u} \times 100 \quad (4)$$

where S_u is the volumetric swelling of untreated specimens and S_t is the volumetric swelling of the treated specimens. They were determined by the following equation:

$$S_u \text{ or } S_t (\%) = \frac{V_2 - V_1}{V_1} \times 100 \quad (5)$$

where V_1 is the oven-dry volume of the specimens before the test (cm^3) and V_2 is the volume of the specimens after the test (cm^3). Ten replicates per group were used in the experiments.

2.3.4. Mechanical properties

The bending properties, including modulus of rupture (MOR) and modulus of elasticity (MOE), were evaluated at a test speed of 1.5 mm min^{-1} following DIN 52186 (1978) standards using an electromechanical universal testing machine (INSTRON 50 KN, America). Surface hardness was measured with an integrated probe of the Time TH210 hardness meter (Beijing TIME High Technology Ltd., Beijing, China) and expressed as Shore D hardness according to the ASTM D2240 method. At least ten replicates were used per treatment and test. Before testing, the samples were conditioned at 20°C and 65% RH.

2.3.5. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was conducted using a thermogravimetric analyzer

(Pyris 6 TGA; PerkinElmer Inc, Wellesley, United States) in an air atmosphere from 40 to 700°C with a heating rate of $10^\circ\text{C min}^{-1}$. Approximately 3-5 mg of each sample was analyzed. Each treatment was performed in triplicate, and the residual mass (RM) and its derivative (DTG) curves were recorded.

2.3.6. Limiting oxygen index (LOI) test

LOIs of treated and untreated specimens were determined using a JF-3 oxygen index instrument (Jiangning Analysis Instrument Factory, Nanjing, China) with sheet dimensions ($135 \text{ mm} \times 6.5 \text{ mm} \times 3 \text{ mm}$) according to ASTM D2863-2010. The LOI values obtained by this test were the average of six replicates and were recorded in percentage.

2.3.7. Microscopic analysis

The microscopic structure of untreated and treated wood was examined with a scanning electron microscope (SEM, FEI Quanta 200, Holland) at an acceleration voltage of 5-10 kV. Use a microtome blade to carefully cut the $3 \times 5 \times 5 \text{ mm}^3$ ($L \times T \times R$) small samples, taken from the middle part for slice processing. Before microscopic observations, all specimens were adhered onto aluminum stubs, dried at 80°C overnight, and then sputter-coated with a layer of gold (20 nm).

3. RESULTS AND DISCUSSION

3.1. Weight percent gain (WPG)

The WPGs of 15% SS-treated and 10% SC-treated specimens in the presence of SS were increased before leaching (Fig. 1a) due to the incorporation of SS or SC/SS into wood [12, 13]. According to Fig. 1a, the WPGs of 15% SS- and 10% SC-treated wood were 17.75% and 14.47%, respectively. For SC/SS treated specimens, as the concentration of SS increased, the WPG of the treated wood increased linearly, ranging from 18.24% to 23.31%. The SS or SC/SS incorporated in wood was previously shown to be primarily localized in the lumens rather than in the cell walls, as indicated by the absence of

cell wall bulking after treatment [10, 13]. It can be seen that among the two reagents, SS caused a greater WPG of the Acacia hybrid, and the addition of SC further improved the WPG relative to impregnation with SS alone. Therefore, the density accordingly increased, which also was well verified by the mechanical properties described below.

3.2. Leaching testing

As shown in Fig. 1b, 15% SS-treated wood had a notably high LR, reaching up to 13.96% (as demonstrated in our previous research). After the SC was introduced to the wood, the LR of wood samples decreased compared to those

treated by SS alone. The LR of the W_{10%SC+15%SS} was highest at only 8.01%. Wood treated with SS/SC still retains a relatively high WPG after leaching (Fig. 1a). At 10% SC plus 15% SS, the retained WPGs treated wood reached up to 13.43%, which suggests SS could be fixed in the cell lumen and approx. 43% of them have been kept. This process may be due to the mechanism of physical encapsulation of polymerized SC in wood. SC molecules would encapsulate silicon particles, fixing them in the cell lumens. Therefore, the SC achieved the effective fixation of SS as expected.

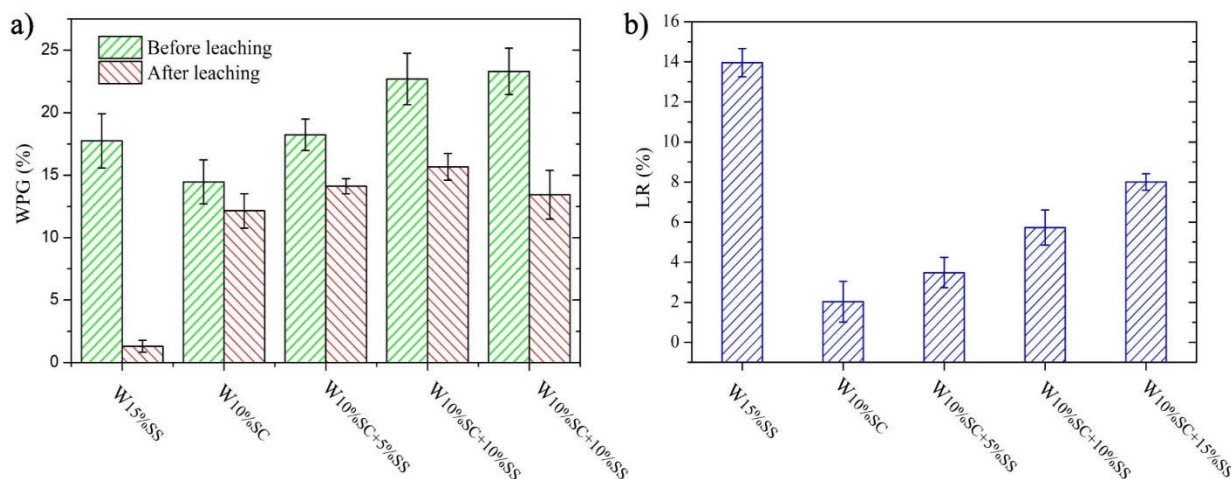


Figure 1. Weight percent gain (WPG) (a) and leaching rate (LR) (b) of Acacia hybrid wood treated with 15% SS or 10% SC and varying concentrations of SS
 Error bars show standard error (StD) based on 10 replicates

3.3. Cell wall bulking efficiency (BE) and dimensional stability (ASE)

Treatments with 15% SS or 10% SC and varying concentrations of SS caused a negative BE of Acacia hybrid wood (Fig. 2), which indicates SC/SS contraction and cell wall degradation. The cell wall adhering to the inclusions will be pulled toward the lumen so that the cell will be shrunk [14]. When treating wood in an alkaline environment, some of the ester bonds in hemicellulose and lignin are hydrolyzed [15]. That caused the

microstructure shrinkage of treated wood (as analyzed in our previous study), and therefore BE was decreased. The BE of treated wood dropped to -18.87%, further evidenced by the high cell wall shrinkage in the Acacia hybrid, as observed in Fig. 7. The negative BE also indirect proof that the SC cannot penetrate cell walls sufficiently because of its large molecular mass (15 000 g mol⁻¹). Meanwhile, SS was mainly distributed in the cell lumen and did not have any reaction to the cell wall.

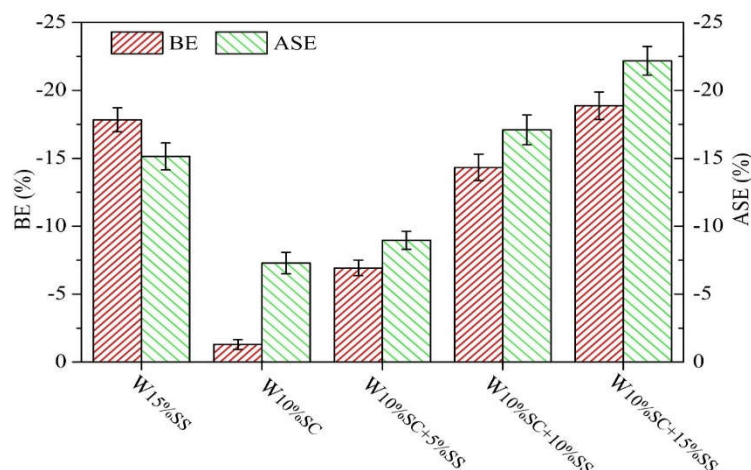


Figure 2. Bulking efficiency (BE) and anti-swelling efficiency (ASE) of Acacia hybrid wood untreated and treated with 15% SS or 10% SC and varying concentrations of SS

Error bars show StD based on 10 replicates

The ASE also showed a negative value and decreased with increasing SS concentration (Fig. 2). Accordingly, treatment with the SC/SS did not improve dimensional stability. The degradation of the cell walls was severe, which can mainly be explained by the high pH of treatment solutions [15].

3.4. Mechanical properties

3.4.1. Bending properties

The MOR and MOE of Acacia hybrid wood were significantly increased after treatment with 15% SS or combined SC/SS (Fig. 3). For SC/SS-treated wood, the W_{10%SC+15%SS} specimen exhibited the highest MOR and MOE values, were 150.44±12.75 MPa and 11.47±0.73 GPa,

respectively. The results indicate that they were about 71.54 and 53.65% higher than those of the W_{CTRL} specimen (Fig. 3a, b). These improvements may be due to the reinforcement effect of the SC inclusions and the SS depositions into the cell lumina. After impregnation, a large amount of inorganic SS or SC/SS entered the lumina as a filling, acting as a supporting material. The stiff inclusions share the applied load with the cell walls through the interfaces, and thus improving its mechanical properties [16, 17]. The same principle leads to an increase in compressive strength and surface hardness.

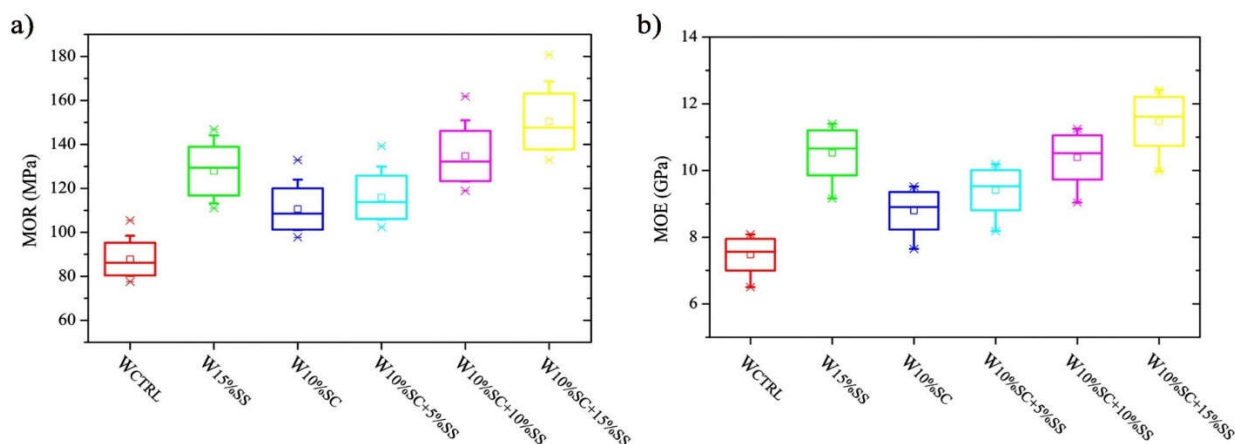


Figure 3. Modulus of rupture (MOR) (a) and modulus of elasticity (MOE) (b) of Acacia hybrid wood untreated and treated with 15% SS or 10% SC and varying concentrations of SS. The bottom and top edges of the box are standard deviation, the band in the box is 50th percentile, x-symbol at the end of box chart represents the 1th percentile (bottom end) and the 99th percentile (top end), the small square in the box represents the mean, and the whiskers out of box chart represent the outliers

3.4.2. Surface hardness

Wood's hardness is wood's ability to resist other rigid objects. The cross-section hardness of the wood is much larger than the other two. There is no significant difference between radial and tangential directions [18]. Therefore, Fig. 4 only exhibits the cross-and radial sections hardness of the Acacia hybrid treated with 15% SS or SC/SS. After treatment, the surface hardness of treated wood significantly increased in both the cross-section and radial section. The $W_{15\%SS}$ specimen of the Acacia

hybrid showed 25.99 and 40.76% higher hardness values than those of the W_{CTRL} cross-section (Fig. 4a) and radial section (Fig. 4b), respectively. Besides, the hardness of the wood gradually increased with the increase of the concentration of SS incorporated into the 10% SC for wood treatment. The hardness value for the $W_{10\%SC+15\%SS}$ specimen was 37.24 and 46.65% higher than those of the W_{CTRL} cross-section (Fig. 4a) and radial section (Fig. 4b), respectively.

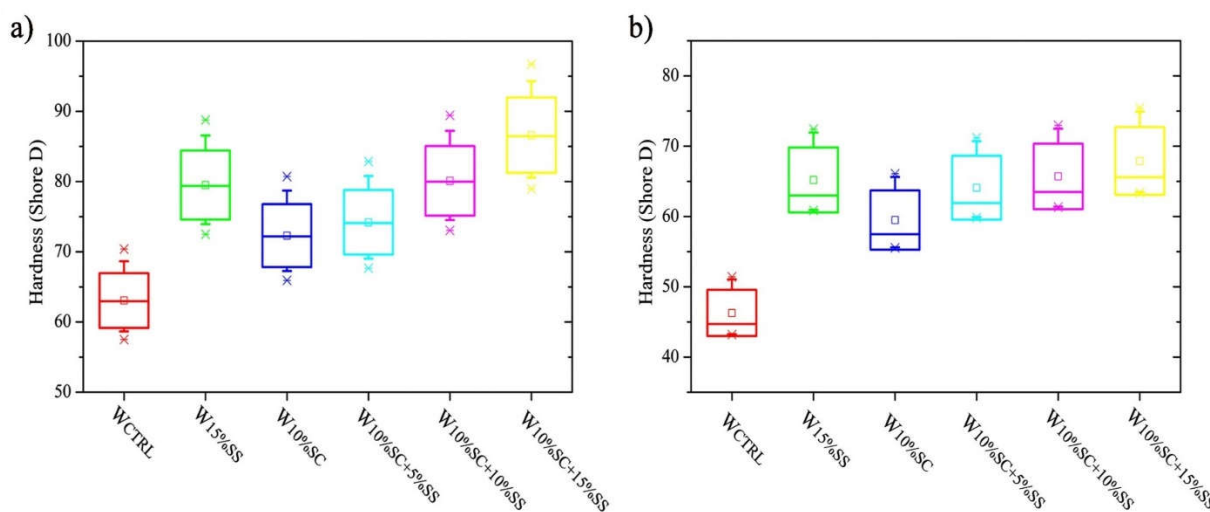


Figure 4. Surface hardness of Acacia hybrid wood untreated and treated with 15% SS or 10% SC and varying concentrations of SS in cross-section (a) and radial section (b) respectively

The meaning of box chart is same as Fig. 3

The enhanced hardness of the treated wood can probably be attributed to the SC (partially cross-linked with the PC crosslinking agent), which can stiffen the wood matrix and the stiffness of its inclusions in the cell lumina [19]. Additionally, as the SS was dispersed in the SC and impregnated into the wood under pressure, it may be retained in the SC or deposited in the wood cell lumina and subsequently polymerize, thereby increasing the wood's hardness (as indicated above) and allowing better load transfer.

3.5. Thermogravimetric analysis (TGA)

When the temperature was increased to approximately 140°C, the untreated and treated wood samples had a mass loss of 3-5% (Fig. 5a, b) due to the evaporation of free water

from the specimens. Compared to the untreated wood, Acacia hybrid wood treated with 10% SAC alone showed a similar RM curve (Fig. 5a), characterized by a highest DTG peak at 385°C (Fig. 5b) [20].

Incorporation of SS in 10% SC resulted in decomposition of polysaccharide at a lower temperature (DTG peak at 300°C, Fig. 5b), which can be attributed to alkaline-accelerated pyrolysis of wood. The changes in SS concentration did not substantially influence the thermal pyrolysis behavior due to the comparable pH value of the treatment solutions (Table 1). Further decomposition took place above 400°C (Fig. 5b), primarily due to lignin degradation [21].

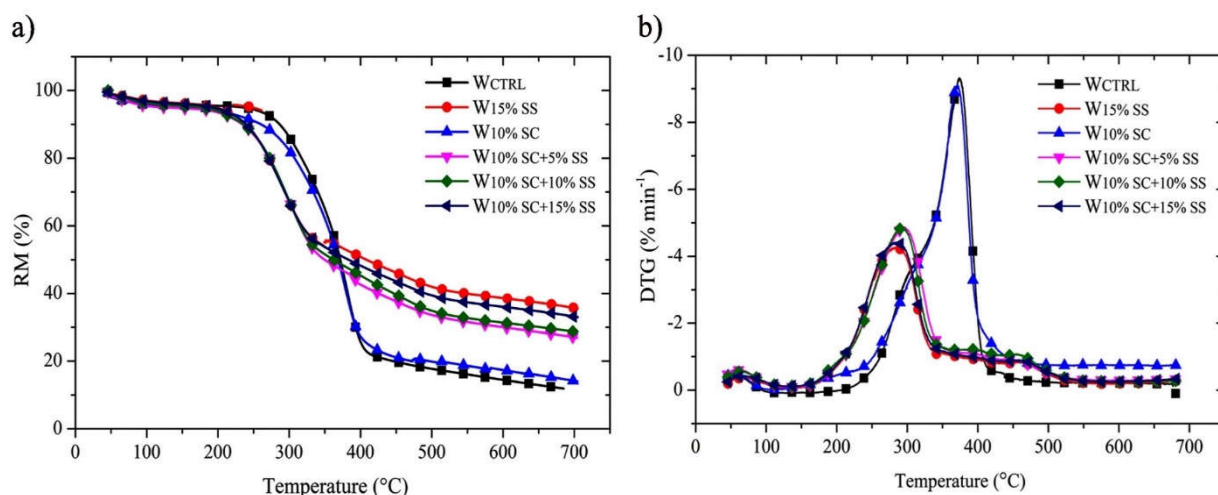


Figure 5. Change in residual mass (RM) (a) and its derivative thermogravimetric curves (DTG) (b) of Acacia hybrid wood untreated and treated with 15% SS or 10% SC and varying concentrations of SS

W_{10%SC+SS} exhibited a residual mass of 15-20% higher than W_{CTRL} (10% residual mass) after the test. The higher residual mass can be attributed to the presence of thermally stable SS and increased charcoal. Assuming that SS in wood does not decompose, W_{10%SC+SS} obtained approximately 15% residual wood charcoal, which is higher than the 10% of the W_{CTRL}. The increased wood charcoal yield can be explained by the alkaline-catalyzed pyrolysis forming charcoal at the lower temperature.

3.6. Limiting oxygen index (LOI) test

Flame resistance properties of the wood specimens were examined by measuring their

LOI. As shown in Fig. 6, treatment with SC did not substantially influence the LOI value. LOI gradually increased after SC/SS were incorporated, indicating improved thermal stability and fire resistance. The LOI of wood gradually increased when the increased concentration of SS was combined with 10% SC for wood treatment. The LOI value of W_{10%SC+15%SS} was 34.95% higher than that of untreated wood. This demonstrates that the incorporation of SS in wood facilitates the delay of wood combustion, which can be attributed to both the coating effect and the alkaline-catalyzed charring effect by the SS [22].

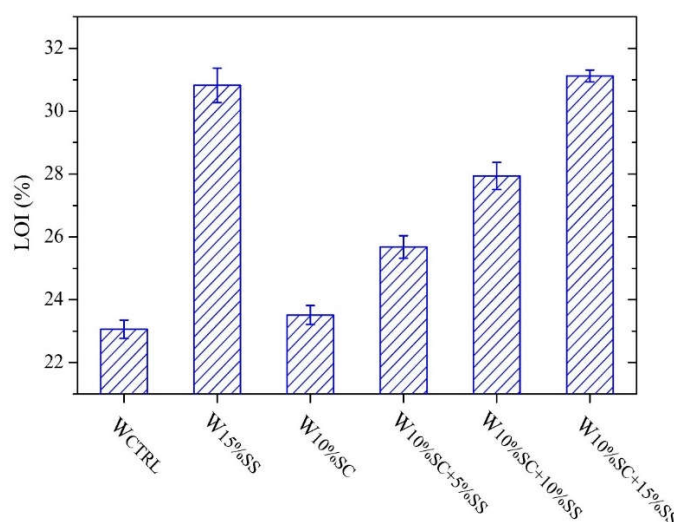


Figure 6. Limiting oxygen index (LOI) of Acacia hybrid untreated and treated with 15% SS or 10% SC and varying concentrations of SS during the combustion process

3.7. Micrographic analysis

A series of SEM images of cross sections of wood (Fig. 7) give a direct visualization of modifier distribution in wood matrix and microstructure changes of wood cell walls. The SEM image of untreated wood shows hollow cell lumina (Fig. 7a). After 15% SS impregnation, part of the lumen is entirely or partially filled with SS (Fig. 7b). Treatment with 10% SC (Fig. 7c) or combinative treatments with SS and SC, lumens of both vessels and fibers were filled

with polymerized SC or SC/SS (Fig. 7d). The SC/SS deposited in the cell lumina, forming the tubular-shaped or cylindrical-shaped inclusions as a hybrid body after polymerization (Fig. 7d), which may thicken the cell wall, which may mechanically strengthen the cell walls. Compared with $W_{10\%SC}$, $W_{10\%SC+SS}$ exhibited a larger overall filling ratio and uniform distribution, which was also confirmed by WPG results.

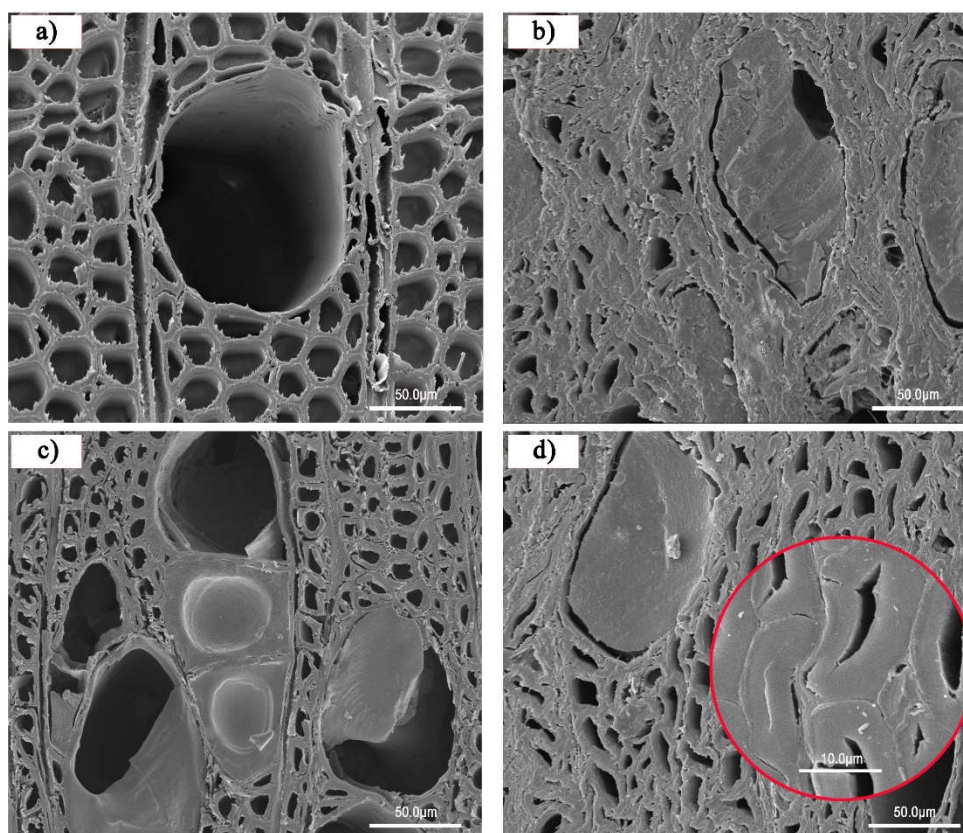


Figure 7. SEM micrographs of the cross-section of Acacia hybrid untreated (a) and treated with 15% SS (b), 10% SC (c), and 10% SC+15% SS (d), respectively

Notably, the 15% SS and SC/SS-treated wood sample displays significant structural changes with the deformation of the vessels and cell walls. Some places showed severe deformation, where the cell lumens were almost entirely closed (Fig. 7b, d). The cause of this phenomenon may be due to the partial dissolution of hemicellulose and lignin from the cell wall in the alkaline solution during

treatment [23]. This result is similar to the previous study of Kollmann and Sachs [24] and Nguyen et al. [10], who found similar characteristics in poplar wood when treated with an alkaline solution. Within the cell wall, lignin acts as a matrix that binds cellulose fibers together, while hemicelluloses provide structural strength [25, 26]. After Acacia hybrid treatment with SS or SC/SS solution with a high

PH value (Table 1), hemicellulose and lignin were partially removed, which lowered the mechanical properties of the cell wall, causing the cellulose microfibrils to collapse, and the porosity of the wood surface to increase. As a result, the cell wall structure also became looser, easy to collapse and deform, but it does so without fracturing them. This critical attribute combines the increasing WPG, resulting in the significantly increased density of treated wood. Significant increases in MOE are realized and are roughly proportional to the increase in density [27]. MOR and hardness are also increased and may improve other properties further.

4. CONCLUSION

In this study, the Acacia hybrid wood was treated with SC/SS to improve the mechanical, flame-retardant properties and the fixation of SS. The results showed that the MOR and MOE of wood bending and surface hardness of the treated wood were enhanced due to the incorporation of the polymeric SC inclusions and the needle-like silica rods in the cell lumina. SS was also effectively fixed in the Acacia hybrid wood. In addition, the flame retardancy of the treated Acacia hybrid wood was also investigated. The TGA and LOI test results showed that treatment with SC/SS resulted in the pyrolysis of wood at lower temperatures and difficulty to flame, as evidenced by the increased LOI levels in response to the incorporation of SC/SS. The results of this study demonstrate the feasibility of the treatment wood with a combination of SC/SS; however, the BE and ASE decreased due to cell wall degradation in the alkaline environment. Treated wood is suitable for use in indoor domains, such as flooring and furniture.

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