

Morphological Remodeling and Performance of Cured Tannin-Sucrose Adhesive Layer: Enhancement by Catalyst

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New adhesives were developed using sustainable bayberry tannin and sucrose as raw materials. Through introducing three catalysts—citric acid, p-toluenesulfonic acid, and phytic acid—a comprehensive analysis of their differential impact mechanisms on catalyzing sucrose conversion, promoting cross-linking reactions, and shaping the microstructure of the adhesive was conducted. The results showed that under the phytic acid catalytic system, the yield of 5-hydroxymethylfurfural (5-HMF) reached 17.5 µg/mL, which was higher than that of p-toluenesulfonic acid (14.1 µg/mL) and citric acid (12.9 µg/mL). The introduction of catalysts led to a stepwise improvement in the mechanical properties of the adhesive. The adhesive catalyzed by phytic acid exhibited excellent bonding strength and water resistance, reflecting its advantage in promoting deep cross-linking between 5-HMF and tannin. Scanning electron microscopy results intuitively demonstrated the reshaping of the adhesive layer morphology by the catalysts, evolving from the loose and porous structure of the blank group to a dense, wrinkled morphology after the action of the catalysts. The results of thermogravimetric analysis further quantified the enhancement effect of the catalysts on the thermal stability of the network structure, with the three-dimensional network structure built by the phytic acid system exhibiting superior thermal protection capabilities.

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INTRODUCTION

In the contemporary wood processing industry, wood adhesives, as a key bonding material, directly determine the quality and durability of wood products. With increasing global emphasis on environmental protection and sustainable development, the traditional wood adhesive industry is facing severe challenges. Adhesives based on formaldehyde, isocyanates, and other chemical raw materials release a large amount of harmful gases during production and use. For example, formaldehyde is a well-known indoor air pollutant, and long-term exposure can lead to respiratory diseases and even cancer, posing a serious threat to human health (Wang *et al.* 2023; Staciwa *et al.* 2024; Zhang *et al.* 2024a; Deng *et al.* 2025). In addition, these raw materials are mostly derived from non-renewable resources. As these resources become increasingly scarce, their production and usage costs continue to rise, severely restricting the sustainable development of the wood processing industry (Liu *et al.* 2019; Mirski *et al.* 2020; Zheng *et al.* 2022a).

Against this backdrop, the development of green, environmentally friendly, and efficient wood adhesives has become a research hotspot in the field of materials science. Biomass-based adhesives, with their renewable, biodegradable, and environmentally friendly characteristics, have gradually emerged and attracted widespread attention from researchers worldwide (Zhang *et al.* 2018, 2020; Qu *et al.* 2023; Dai *et al.* 2024; Wu *et al.* 2024; Liu *et al.* 2025; Zheng *et al.* 2025). Numerous studies have shown that plant tannins and polysaccharides are ideal raw materials for the preparation of biomass-based wood adhesives. Plant tannins are rich in active functional groups, such as phenolic hydroxyl and catechol groups, which endow them with strong reactivity and the ability to cross-link with various substances (Arias *et al.* 2021; Gu *et al.* 2023; Li *et al.* 2023; Oktay *et al.* 2024). Polysaccharides, on the other hand, are characterized by their wide availability, low cost, and good film-forming properties. The synergistic action of these two components is expected to produce high-performance wood adhesives (Zheng *et al.* 2022b; Zhang *et al.* 2025).

Bayberry tannin, as a highly promising plant tannin resource, has the advantages of wide availability, low cost, and renewability. Its main components are catechin compounds, which contain abundant phenolic hydroxyl groups that can cross-link with various substances to form stable network structures (Arias *et al.* 2021; Gu *et al.* 2023; Xiao *et al.* 2023; Wu *et al.* 2025). Sucrose, a common polysaccharide, also has advantages of wide availability and low cost, as well as good film-forming and biocompatible properties. However, sucrose itself has poor bonding properties and needs to be chemically modified to enhance its reactivity to meet the performance requirements of wood adhesives. In the chemical modification of sucrose, catalysts play a crucial role. Studies have shown that catalysts can significantly accelerate the dehydration reaction of hydroxyl groups in sucrose molecules to generate 5-hydroxymethylfurfural (5-HMF). This intermediate product has high reactivity and can cross-link with tannins to form a stable network structure, thereby improving the bonding performance of the adhesive (Zhao and Umemura 2015, 2015; Zhao *et al.* 2015, 2020; Yang *et al.* 2023).

This study focused on the field of wood adhesives, using bayberry tannin and sucrose as the main raw materials and selecting citric acid, p-toluenesulfonic acid, and phytic acid as catalysts to prepare bayberry tannin-sucrose wood adhesives. The aim was to investigate the effects of different catalysts on the properties of the adhesives. This research is intended to provide a solid theoretical basis for the development of high-performance, environmentally friendly biomass-based wood adhesives and to promote the widespread application of biomass-based wood adhesives in the wood processing industry and other related fields.

EXPERIMENTAL

Materials

Bayberry (*Myrica rubra*) tannin, industrial grade (particle size approximately 150 μm), obtained from Wuming Tannin Factory, Guangxi, China was used. The sucrose, purity 99.0%, analytical grade, was purchased from Chengdu Jinshan Chemical Reagent Co., Ltd. Citric acid (CA), p-toluenesulfonic acid (PTA), and phytic acid (PA) analytical grade, were from Shanghai Macklin Biochemical Co., Ltd. Poplar veneer (*Populus* spp.) with a moisture content 8 to 10%, dimensions 400 mm $\times$ 400 mm, thickness 1.5 mm, was purchased from Suqian, Jiangsu, China.

Preparation of Tannin-Sucrose Adhesive and Bonding Performance Testing

Under ambient temperature conditions, 33.3 g of distilled water was added to a round-bottom three-neck flask equipped with a mechanical stirrer, thermometer, and condenser. The mixture was then slowly heated to 85 °C. Once the temperature stabilized, 20 g of sucrose was added to the flask and continuously stirred until the sucrose was completely dissolved. Subsequently, 30 g of tannin was added to the flask in batches, with continuous stirring to ensure thorough mixing with the sugar solution. Immediately after, 0.5% sodium dodecylbenzenesulfonate as a dispersant was added to the mixture, and stirring was maintained for 5 min to ensure uniform dispersion. Laboratory-made three-layer poplar plywood was prepared. A certain amount of adhesive was weighed and mixed with 16% of different catalysts (including citric acid, p-toluenesulfonic acid, and phytic acid) using a stirring device until the catalysts were uniformly dispersed in the adhesive and the obtained adhesives were respectively labeled as TCA, TPTA, and TPA. The adhesive was applied to the surface of the poplar veneer at a rate of 160 g/m² on one side. After assembly, the veneers were cold-pressed for 10 min, followed by hot pressing. The hot-pressing process was as follows: hot pressing temperature of 215 °C, unit hot pressing pressure of 1.5 MPa, and hot-pressing time of 1 min/mm. After hot-pressing was completed, the bonding strength of the three-layer poplar plywood was tested in accordance with the national standard GB/T 17657 (2022). For the bonding strength in warm water, the specimens were immersed in water at 63 ± 3 °C for 3 h, then condition them at room temperature for 10 min before testing. And for the bonding strength in boiling water, the specimens were boiled for 3 h, then conditioned at room temperature for 10 min before testing. The reported strength was the mean value of 10 samples.

Measurement of 5-Hydroxymethylfurfural Content

Under ambient temperature conditions, 33.3 g of distilled water was weighed and placed in a round-bottom three-neck flask equipped with a mechanical stirrer, thermometer, and condenser. The mixture was then slowly heated to 85 °C and maintained at this temperature. Once the temperature stabilized, 20 g of sucrose was slowly added to the flask and stirred at a moderate rate until the sucrose was completely dissolved. Subsequently, 3.2 g of catalyst (either citric acid, p-toluenesulfonic acid, or phytic acid) was slowly added to the flask. The reaction was carried out with continuous stirring at 85 °C for 2 h. After the reaction was complete, the mixture was cooled to room temperature. The content of 5-HMF was measured using the UV spectrophotometric method, as referenced in Xiao *et al.* (2023). The reported 5-HMF content was the mean value of 10 samples.

Thermogravimetric (TG) Testing

The cured adhesive powder was subjected to TG analysis. A Netzsch TG 209 F3 thermogravimetric analyzer (Germany) was used for the testing under nitrogen protection. The heating rate was set at 10 °C/min, and the temperature range was from 30 to 600 °C.

Scanning Electron Microscopy (SEM) Testing

The cross-section of the cured adhesive layer was sputter-coated with gold and then observed using a Zeiss GeminiSEM 300 scanning electron microscope (Germany).

RESULTS AND DISCUSSION

Analysis of 5-HMF Content

The key mechanism for the excellent bonding performance of the bayberry tannin-sucrose adhesive lies in the conversion of sucrose to 5-HMF, followed by the cross-linking reaction between 5-HMF and tannin to form a stable network structure (Xiao *et al.* 2022, 2023). In this critical step of converting sucrose to 5-HMF, the choice of catalyst plays a crucial role. As shown in Fig. 1, there were differences in the catalytic effects of citric acid, p-toluenesulfonic acid, and phytic acid on the generation of 5-HMF from sucrose. Phytic acid exhibited the best catalytic performance, with a 5-HMF content of 17.5 µg/mL in the product; p-toluenesulfonic acid was the second best, with a 5-HMF content of 14.1 µg/mL; and citric acid had the lowest 5-HMF content at 12.9 µg/mL. This result clearly indicates that different catalysts have varying efficiencies in promoting the conversion of sucrose to 5-HMF. The differences in performances can be tentatively attributed to their different acid strengths and interactions with the reactants.

Citric acid is a weakly acidic catalyst that can release a certain amount of hydrogen ions. When the hydroxyl groups of sucrose molecules combine with H^+ , the activity of the hydroxyl groups is enhanced, making it easier for them to lose water molecules and initiate the reaction pathway for the conversion of sucrose to 5-HMF (Xiao *et al.* 2022). However, due to its relatively weak acidity, citric acid provides a limited number of H^+ ions, which to some extent restricts the dehydration reaction rate and extent of sucrose, resulting in a relatively low yield of 5-HMF. Moreover, citric acid molecules have a specific spatial structure with a relatively large volume. This may cause steric hindrance when interacting with sucrose molecules. Such steric hindrance can impede the full contact between sucrose molecules and the catalyst, preventing some sucrose molecules from effectively participating in the catalytic reaction and thereby affecting the overall catalytic efficiency.

p-Toluenesulfonic acid is a strong acidic catalyst that can release a large amount of H^+ . These H^+ ions interact with sucrose molecules, making the hydroxyl groups in sucrose more prone to dehydration and thus accelerating the reaction rate for the conversion of sucrose to 5-HMF (Sakai *et al.* 2023). The high concentration of H^+ can more effectively reduce the activation energy of the reaction, enabling more sucrose molecules to overcome the reaction energy barrier and thereby increasing the efficiency of 5-HMF formation. In addition to providing acidic centers, the sulfonic acid groups in p-toluenesulfonic acid molecules may also interact with intermediate products formed during the reaction. This interaction can stabilize the structure of the intermediates, keeping them in a state that is more readily converted further, thereby improving the selectivity and efficiency of the reaction.

Phytic acid has a unique molecular structure containing multiple hydroxyl and phosphate groups. The hydroxyl groups can form hydrogen bonds with sucrose molecules. This hydrogen bonding enhances the interaction between sucrose and the catalyst, making the adsorption of sucrose molecules on the catalyst surface more secure and favorable for the reaction to proceed. Meanwhile, the phosphate groups provide acidic centers that promote the dehydration reaction of hydroxyl groups in sucrose molecules. The synergistic action of multiple hydroxyl and phosphate groups makes it easier for sucrose molecules to undergo dehydration reactions under phytic acid catalysis, thereby improving the efficiency of 5-HMF formation (Zheng *et al.* 2022b). Moreover, during the catalysis of sucrose conversion to 5-HMF, phytic acid may interact with sucrose molecules in complex ways to form highly reactive intermediates. These active intermediates are more likely to

be converted to 5-HMF than sucrose molecules themselves.

In summary, citric acid, p-toluenesulfonic acid, and phytic acid were found to exhibit different catalytic efficiencies in generating 5-HMF from sucrose, in the order of phytic acid > p-toluenesulfonic acid > citric acid. This difference was attributed to the different acid strengths, molecular structures, and interaction modes of the catalysts with the reactants. Phytic acid, with its multiple hydroxyl and phosphate groups working synergistically, showed the highest catalytic efficiency in promoting the dehydration of sucrose to form 5-HMF; p-toluenesulfonic acid achieved the second-best catalytic effect by relying on its strong acidity and intermediate product stabilization; and citric acid, limited by its relatively weak acidity and steric hindrance factors, had the lowest catalytic efficiency.

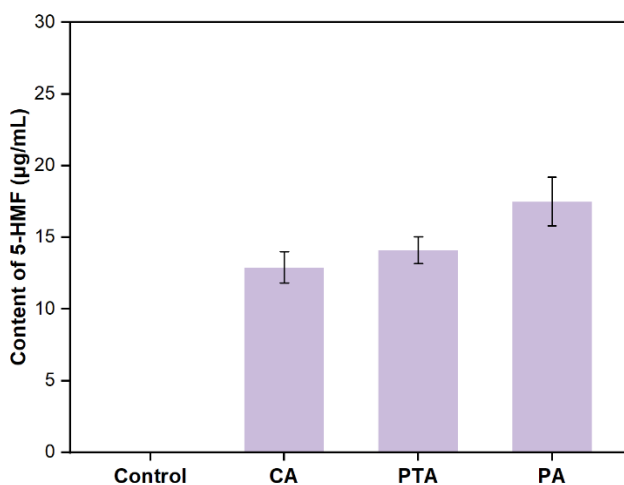


Fig. 1. Influence of different catalysts on the content of 5-HMF generated from sucrose

Bonding Performance

As shown in Fig. 2, the introduction of catalysts had a complex impact on the bonding performance of the adhesive, which is underpinned by rich principles of chemical reactions and the evolution of material structures.

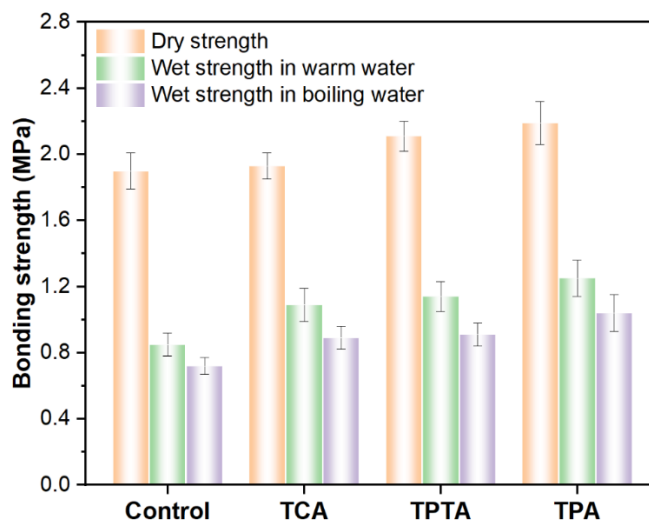


Fig. 2. The influence of different catalysts on the tannin-sucrose bonding performance

When no catalyst was added, the dry strength of the adhesive was 1.90 MPa, while the strength in warm water and boiling water was 0.85 and 0.72 MPa, respectively, indicating an overall low strength level. This is because, in the absence of a catalyst, the conversion of sucrose to 5-HMF is hindered, and the conversion efficiency cannot be effectively improved. The sluggishness of this conversion reaction directly results in insufficient cross-linking reactions between sucrose and tannin, leading to a loose and unstable network structure of the adhesive. Consequently, the bonding strength exhibited by the adhesive under different environmental conditions was relatively weak.

When citric acid was introduced as a catalyst, the performance of the adhesive improved to some extent. The dry strength increased slightly to 1.93 MPa, and the strength in warm water and boiling water reached 1.09 and 0.89 MPa, respectively. As a weakly acidic catalyst, citric acid can release a certain amount of H^+ in the reaction system, which effectively enhances the activity of the hydroxyl groups in sucrose molecules, thereby initiating and promoting the conversion of sucrose to 5-HMF. However, due to its relatively weak acidity, citric acid can only provide a limited amount of H^+ , which restricts the yield of 5-HMF and, consequently, the depth and extent of the cross-linking reactions between sucrose and tannin.

In comparison, when p-toluenesulfonic acid was used as a catalyst, the bonding performance of the adhesive achieved more improvement. The dry strength increased to 2.11 MPa, and the strength in warm water and boiling water reached 1.14 and 0.91 MPa, respectively. As a strong acidic catalyst, p-toluenesulfonic acid can release a large amount of H^+ in the reaction system, which greatly accelerates the conversion of sucrose to 5-HMF and improves the yield of 5-HMF. With the increased content of 5-HMF, the cross-linking reactions between 5-HMF and tannin can proceed more fully, resulting in a more compact and stable network structure of the adhesive and effectively enhancing the strength of the adhesive. In addition, the sulfonic acid groups in p-toluenesulfonic acid molecules have unique interactions with the intermediate products in the reaction. These interactions not only stabilize the structure of the intermediate products, preventing them from decomposing or undergoing side reactions during the reaction process, but also improve the selectivity and efficiency of the entire reaction, further optimizing the performance of the adhesive.

When phytic acid was selected as the catalyst, the bonding performance of the adhesive reached its optimal state. The dry strength was as high as 2.19 MPa, and the strength in warm water and boiling water reached 1.25 and 1.04 MPa, respectively. Phytic acid has a unique molecular structure containing multiple hydroxyl and phosphate groups. On the one hand, the hydroxyl groups can form hydrogen bonds with sucrose molecules, enhancing the interaction between sucrose molecules and making sucrose more securely adsorbed in the reaction system, providing a more stable substrate environment for subsequent chemical reactions. On the other hand, the phosphate groups act as acidic centers, providing efficient catalysis for the dehydration reaction of sucrose, further promoting the conversion of sucrose to 5-HMF. The synergistic action of these two types of groups not only improves the yield of 5-HMF but also likely forms a highly reactive intermediate between sucrose and phytic acid. Compared with ordinary reaction intermediates, this intermediate is more readily converted to 5-HMF, allowing the cross-linking reactions between 5-HMF and tannin to proceed more fully and ultimately forming a more robust and dense adhesive network structure.

Thermal Stability Analysis

Figures 3 and 4 present the thermogravimetric analysis results of the solidified products of the bayberry tannin-sucrose adhesive system. In the absence of a catalyst, the solidified product exhibited a residual carbon content of 51.9% and a maximum thermal decomposition temperature of 294 °C. This indicates that without a catalyst, the network structure of the adhesive was relatively unstable and prone to decomposition during the thermal degradation process. This finding is consistent with the previously mentioned low bonding strength results, strongly confirming that the instability of the network structure is the key factor causing the adhesive's deficiencies in thermal resistance and mechanical properties.

When citric acid was introduced as a catalyst, the residual carbon content of the solidified product increased to 54.1%, while the maximum thermal decomposition temperature slightly decreased to 291 °C. Although the increase in residual carbon content reflects a certain enhancement in the stability of the network structure, the decrease in the maximum thermal decomposition temperature suggests that some potential changes may have occurred during the thermal degradation process. This may be related to the relatively limited amount of H⁺ provided by citric acid, a weakly acidic catalyst. Despite its ability to promote some cross-linking reactions, this limited effect did not significantly improve the thermal stability of the network structure.

When p-toluenesulfonic acid was used as a catalyst, the residual carbon content of the solidified product further increased to 56.0%, and the maximum thermal decomposition temperature also rose to 303 °C. This indicates that p-toluenesulfonic acid, as a strong acidic catalyst, not only effectively promotes the conversion of sucrose to 5-HMF but also enhances the stability of the adhesive's network structure, allowing it to maintain a more intact structural state at higher temperatures. This improvement in thermal stability corroborates the previously mentioned substantial increase in bonding strength, revealing that p-toluenesulfonic acid plays an important role in driving cross-linking reactions and optimizing the network structure.

When phytic acid was used as a catalyst, the residual carbon content of the solidified product reached the highest value of 56.2%, with the maximum thermal decomposition temperature also stabilized at 303 °C. The multiple hydroxyl and phosphate groups in phytic acid molecules work synergistically, not only greatly improving the yield of 5-HMF but also potentially interacting with sucrose to form highly reactive intermediates, thereby deeply optimizing the network structure. This optimized network structure demonstrates higher residual carbon content and excellent thermal stability during the thermal degradation process, which is highly consistent with the highest bonding strength achieved by the adhesive when phytic acid is used as a catalyst (Zhang *et al.* 2025).

In summary, the introduction of different catalysts was found to have an impact on the thermal stability and structural stability of the solidified products of the bayberry tannin-sucrose adhesive. This finding is consistent with the previous analysis of bonding performance, further demonstrating the important role of catalysts in improving the properties of adhesives.

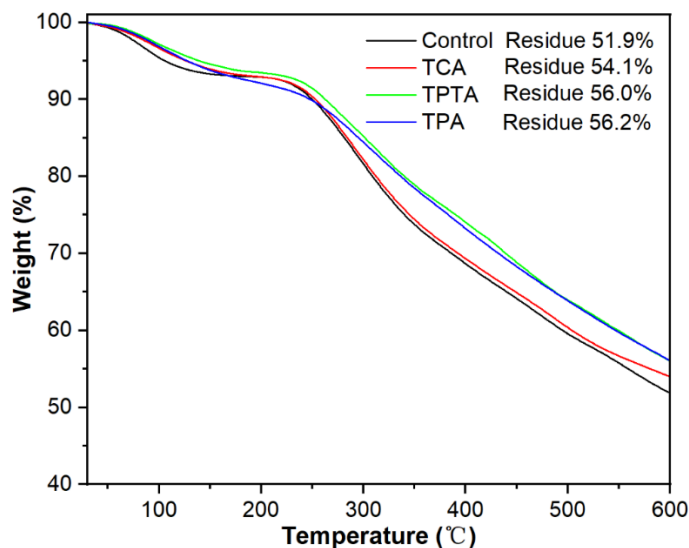


Fig. 3. TG analysis results of tannin-sucrose adhesive

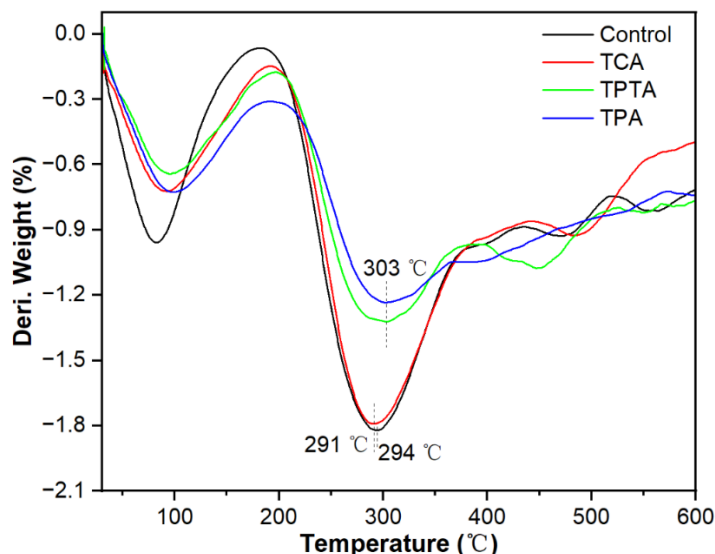


Fig. 4. TG analysis results of tannin-sucrose adhesive

SEM Analysis

As shown in Fig. 5, in the absence of a catalyst, the cross-section of the solidified product of the bayberry tannin-sucrose adhesive exhibited a loose and porous microstructure, accompanied by cracking phenomena. This unique microstructural characteristic corroborates the results of the thermogravimetric analysis, jointly revealing that in the absence of a catalyst, the network structure formed by the adhesive was relatively loose and unstable. The cross-linking and intermolecular interactions within the adhesive may have been insufficient to support a dense and tough structural system, leading to the observed porous and easily cracked macroscopic appearance.

When citric acid was introduced as a catalyst in the reaction system, the microstructure of the cross-section of the solidified product underwent changes, with a notable increase in density and no apparent cracking. This experimental result indicates that the addition of citric acid effectively promoted the formation and densification of the adhesive's network structure. The adhesive became more complete and stable at the

microstructural level, with its components connecting and interacting more tightly, forming a more robust network system. Moreover, this change in microstructure is highly consistent with the increased residual carbon content observed in the thermogravimetric analysis. It strongly demonstrates that the introduction of citric acid as a catalyst plays a crucial role in enhancing the stability of the network structure, thereby improving the adhesive's thermal stability and mechanical properties. This enables the adhesive to exhibit stronger resistance and adaptability to external heat sources and mechanical stresses.

When p-toluenesulfonic acid and phytic acid were used as catalysts, respectively, the density of the cross-section of the solidified product further increased compared to the citric acid-catalyzed system, with a more compact microstructure. Under the action of these two catalysts, distinct wrinkle structures can be clearly observed, especially in the system catalyzed by phytic acid, where the wrinkle structures are more pronounced, with more numerous and deeper wrinkles. The formation of these wrinkle structures indicate that the adhesive had the ability to dissipate energy through self-deformation during the curing process. This unique energy dissipation mechanism effectively reduced the occurrence of stress concentration. When subjected to external stress, the adhesive was able to more uniformly distribute the stress, avoiding local damage caused by stress concentration, thereby further enhancing the adhesive's mechanical properties and reliability in practical applications.

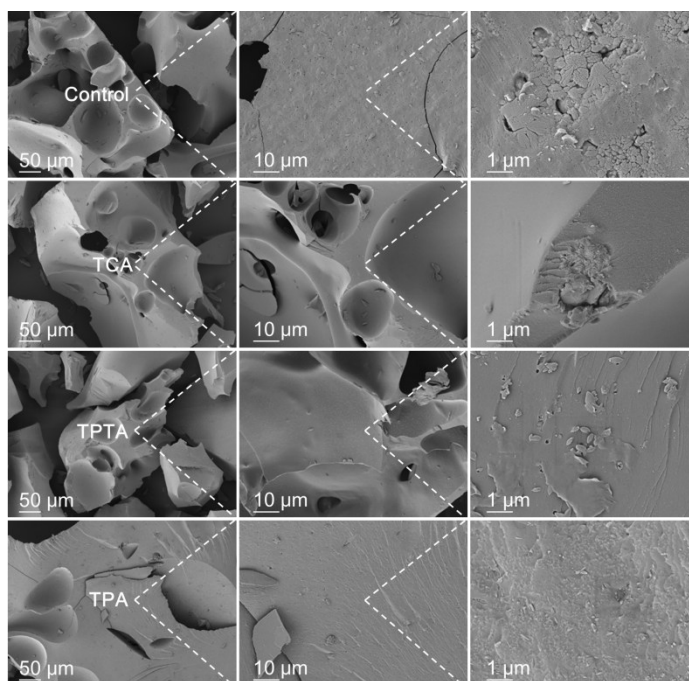


Fig. 5. SEM analysis results of tannin-sucrose adhesive

CONCLUSIONS

1. Phytic acid, with its multiple hydroxyl and phosphate groups working synergistically, efficiently catalyzed the dehydration of sucrose to form 5-HMF, achieving a 5-HMF content of 17.5 $\mu\text{g/mL}$ in the product. p-Toluenesulfonic acid, as a strong acid catalyst, accelerated the conversion of sucrose by releasing a large amount of H^+ . Citric acid, with its weaker acidity and steric hindrance effects, had the lowest catalytic efficiency.

2. The bonding performance of the bayberry tannin-sucrose adhesive was influenced by the catalysts. Without a catalyst, the adhesive had low strength, with a dry strength of only 1.90 MPa, and warm water and boiling water strengths of 0.85 and 0.72 MPa, respectively. After adding catalysts, the bonding performance of the adhesive was enhanced, with the best performance achieved when phytic acid is used as the catalyst. The dry strength reached 2.19 MPa, and the warm water and boiling water strengths were 1.25 and 1.04 MPa, respectively. This improvement was mainly attributed to the differences in 5-HMF content generated by different catalysts, which in turn affected the degree of cross-linking reactions between 5-HMF and tannin, as well as the compactness and stability of the adhesive's network structure.
3. The different catalysts reshaped the morphology of the adhesive layer, improving the thermal stability and structural stability of the solidified products of the bayberry tannin-sucrose adhesive.
4. The use of low-cost, renewable tannin and sucrose in combination with natural catalysts aligns well with green chemistry principles. Comprehensive exploration of other biomass catalysts is the direction for future research.

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