KH550-Modified PVOH Sizing Agent for Tibetan Paper: Performance Enhancement and Antibacterial Function of Bio-Based Systems

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A composite sizing system was developed based on polyvinyl alcohol (PVOH) and the silane coupling agent KH550, with the innovative incorporation of root extract from *Stellera chamaejasme* L.—a characteristic raw material for traditional Tibetan paper. A novel bio-based surface sizing agent with integrated strength enhancement, water resistance, and natural antibacterial properties was successfully prepared. Investigation of KH550 dosage revealed that it acts as a "chemical bridge" between PVOH and paper fibers *via* a cross-linking reaction, in which it condenses with hydroxyl groups on cellulose and PVOH to form covalent Si–O–C bonds. This enhanced interfacial bonding strength and film density, whereas excessive addition led to molecular aggregation. The optimal paper properties were obtained at a KH550 dosage of 1 g, with a sizing weight of 34.1%, tensile index of 50.1 N·m/g, sizing degree of 33 s (43.5% higher than that with PVOH alone), tear index of 219.5 mN·m²/g (163% higher than that of the unsized control), and water contact angle of 106°. Performance indicators were significantly improved compared with the control group without KH550. Notably, the *Stellera chamaejasme* root extract derived from traditional Tibetan papermaking raw materials effectively endows the paper coating with excellent antibacterial properties.

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Keywords: Bio-based sizing agent; Stellera chamaejasme L; Antibacterial components; KH550 modified PVOH; Tibetan paper; Surface sizing

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INTRODUCTION

Tibetan paper, a cultural heritage of the Tibetan Plateau, encapsulates millennia of historical memory (Han *et al.* 2021). Its origins date back to the mid-7th century CE, when local artisans produced this distinctive handmade paper using indigenous materials, including the root of *Stellera chamaejasme* L. The plant is biologically toxic, particularly its pollen and roots (Wu *et al.* 2022). Paper made from this plant retains bioactive compounds that confer long-lasting resistance to insects and microbial degradation (Xiao-Qin *et al.* 2018), making it essential for manuscript preservation. Modern studies have identified flavonoids and coumarins in the roots as the key compounds responsible for its

antimicrobial effects (Song *et al.* 2023). In addition to its durability, Tibetan paper exhibits excellent tensile strength, flexibility, and aesthetic qualities. These attributes render it indispensable in restoring ancient scriptures and creating high-end calligraphy and paintings (Fig. 1). The archival performance of paper, particularly the long-term stability of its chemical components under storage conditions, is therefore of critical importance for such applications (Hubbe *et al.* 2023).

Tibetan paper is prone to absorb moisture from the air during application, resulting in deformation and warping. In artistic creation, ink and pigments are easy to bleed and diffuse on its surface, which affects the visual quality of the finished works. Even minor changes in environmental humidity will cause stress changes in the paper, which severely limits its modern application and promotion (Dutt *et al.* 2012).

Fig. 1. Traditional Tibetan paper and its raw material *Stellera chamaejasme* L. root

In modern papermaking, surface sizing is the primary method to address issues such as low strength, poor water resistance, and surface dusting (Wang *et al.* 2022; Yang *et al.* 2022). This process involves applying a sizing agent with functional components onto the paper surface under controlled conditions (Li *et al.* 2011). After drying, the agent penetrates fiber gaps and encapsulates the fibers, enhancing inter-fiber bonding and overall paper performance. Currently, surface sizing agents are diverse and mainly fall into two categories: natural polymers and synthetic polymers.

The core reason for the application of silane coupling agent KH550(γ-aminopropyltriethoxysilane) in surface sizing system lies in its molecular structure, which contains both amino functional groups and siloxane groups. It can form stable chemical bonds with the hydroxyl groups on the surface of paper fibers, and meanwhile maintain good compatibility with the organic components in the sizing agent system. This effectively enhances the bonding strength between the sizing agent and fibers, further improves the surface strength, water resistance and overall mechanical properties of the paper, and optimizes the stability and film-forming effect of the sizing system, so as to meet the production demand of high-quality paper in modern papermaking.

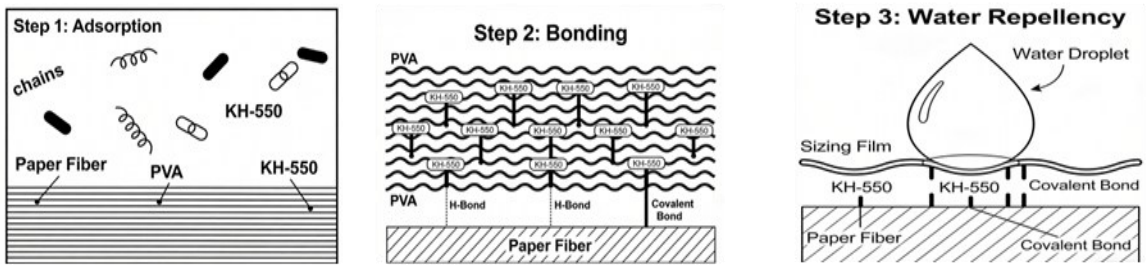

Fig. 2. Schematic diagram of sizing agent mechanism

The primary reason for the utilization of silane coupling agent KH550 (Chi *et al.* 2024; Liu *et al.* 2025), in a surface sizing system is attributable to its molecular composition, which encompassed both amino functional groups and siloxane groups (Yang *et al.* 2023). It has been demonstrated to form stable chemical bonds with the hydroxyl groups present on the surface of paper fibers (Liu *et al.* 2011), while concurrently maintaining optimal compatibility with the organic components that comprise the sizing agent system (Pan *et al.* 2020). This approach has been shown to enhance the bonding strength between the sizing agent and fibers (Sun *et al.* 2022), improve the surface strength, water resistance, and overall mechanical properties of the paper, and optimize the stability and film-forming effect of the sizing system. The result is a paper that meets the production demand for high-quality paper in modern papermaking (Hagiopol and Johnston 2011).

Currently, PVOH and KH550 graft-modified sizing agents are promising for surface sizing (Gu *et al*. 2026), due to their excellent film-forming properties and environmental sustainability (Wang *et al.* 2006). However, their limited functionality restricts their ability to meet the growing demand for long-term antibacterial and hygienic safety in high-end paper products, highlighting the need to enhance their performance and impart new functionalities (Pan *et al.* 2024). Systematic research on surface sizing agents for Tibetan paper not only advances traditional papermaking techniques but also supports cultural heritage preservation and sustainable industry development.

This study aims to develop a novel bio-based surface sizing agent with superior sizing performance and durable antibacterial functionality. The innovation lies in using a KH550/PVOH modified matrix to enhance interfacial bonding and water resistance, providing a stable substrate for functional components. The incorporation of *Stellera chamaejasme* L root extract introduces natural antibacterial properties.

The study proceeded as follows: (1) synthesis and characterization of a KH550 graft-modified PVOH matrix; (2) optimization of antibacterial component extraction from *Stellera chamaejasme* L roots and compounding with the PVOH matrix; (3) systematic evaluation of film-forming behavior, sizing performance, physical properties, and antibacterial efficacy. This work develops a high-performance functional sizing agent and offers a reference strategy for targeted functionalization of bio-based polymer materials.

EXPERIMENTAL

Raw Materials and Reagents

The analytical-grade reagents used in this study included glycerol, sodium hydroxide (Xilong Scientific), polyvinyl alcohol (no pre-silanized) (PVOH-1799, Aladdin), γ-aminopropyltriethoxysilane (KH550), acetic acid, and potassium bromide (SP grade, Kelong Chemical). *Staphylococcus aureus* was obtained from Hunan Fenghui Biotechnology. *Stellera chamaejasme* L. roots were collected from local herdsmen in the Tibetan region and authenticated prior to use.

Preparation of the Modified Sizing Agent

First, 10 g PVOH with varying alcoholysis degrees was dissolved in distilled water at 90 °C for 3 h under stirring. After cooling, KH550 (0, 0.5, 1, 1.5, or 2g) was added dropwise, the pH was adjusted to 6 to 7 with 10 wt.% acetic acid, and the mixture was reacted at 60 °C for 2 h to obtain the modified PVOH (KPVOH) dispersible agent. This agent was blended with 10% modified PVOH solution and glycerol at a 3:4:1 mass ratio,

diluted with deionized water to adjust solid content, and kept at 70 °C.

Paper sheets (30 cm × 30 cm) were impregnated with the sizing agent, roller-coated at 4.0 mm·s^{-1}, and dried at 105 °C for 10 min. All specimens were conditioned for 24 h before testing.

Structural and Performance Testing of Sized Tibetan Paper

Sizing weight determination

Sizing weight is a key parameter for assessing sizing effectiveness and cost efficiency. Accurate measurement ensures objective evaluation of sizing uniformity and material consumption.

Before testing, specimens were conditioned under 23 °C, 50 RH. Ten circular samples (100 cm² each) were cut from uncoated and sized paper and weighed using a precision balance. Average basis weights before (Q_b) and after sizing (Q_a) were calculated. Sizing weight was determined using Eq. 1 and expressed as a percentage,

$$P = \frac{Q_a - Q_b}{Q_b} \times 100\% \quad (1)$$

where P is the loading level of the sizing agent (%), Q_a is the paper basis weight after sizing (g/m²), and Q_b is the paper basis weight before sizing (g/m²).

Tensile Index Test

Tensile index is a key parameter for evaluating paper strength and durability, reflecting fiber bonding strength and resistance to tensile stress during processing and use.

Measurements were conducted using a horizontal computer-controlled tensile tester (YT-WL). Specimens (250 mm × 15 mm) were tested with an 180 mm gauge length at 20 mm/min. At least ten valid specimens were tested per group. The maximum tensile force at break was recorded, and the average value ($\overline{F}$) was used to calculate the tensile index according to Eq. 2,

$$Y = \frac{\overline{F}}{(L_w \times g) \times 10^{-3}} \quad (2)$$

where Y is the tensile index (N·m/g), $\overline{F}$ is the average tensile force (N), L_w is the width of the specimen (mm), and g is the basis weight of the specimen (g/m²).

Tear Index Test

Tear index quantifies paper's resistance to tear propagation, reflecting toughness and service performance, particularly for cultural and packaging papers. Tests were performed using a computer-controlled tear tester (YT-SLD1000) following the Elmendorf method (Perdoch *et al.* 2022). Specimens (63 mm × 50 mm) were taken from both machine and cross directions, with at least eight per direction. After calibration, each specimen was clamped, a 20 mm incision was made, and the pendulum was released. The tear force (F) was recorded, and the tear index was calculated using Eq. 3 based on F and specimen basis weight (G),

$$X = \frac{F}{G} \quad (3)$$

where X is the tear index (mN·m²/g), F is the tear strength (mN), and G is the basis weight of the specimen (g/m²).

Brightness Test

Brightness is a key optical property that determines paper's visual quality, print contrast, and color fidelity. Measurements were conducted using a brightness colorimeter (YT-48A) following the 45/0 directional reflectance method. After warm-up and calibration with a black cylinder and standard white plate, the R457 blue-light reflectance (ISO brightness) and Lightness values (L^*) were recorded. Each sample was measured at five different positions, and the average value was reported.

Sizing Degree Test

Sizing degree reflects paper's resistance to water penetration, which is essential for evaluating its suitability in liquid-resistant applications such as writing and packaging. A preconditioned 50 mm × 50 mm specimen was folded into a V-shaped trough (60° base angle). A 1.0% $FeCl_3$ solution was pipetted along the top edge, and the trough was immediately placed above a 2.0% NH_4CNS solution. Timing began upon placement and stopped when the first red spot appeared due to solution permeation. The recorded time (s) was taken as the sizing degree.

Water Contact Angle Test

Water contact angle quantitatively characterizes the surface wettability, free energy, and sizing uniformity of paper, directly reflecting the modification effect of sizing agents on fiber surfaces and linking surface chemistry with macroscopic liquid resistance. Measurements were performed using an optical contact angle instrument (Sheng Ding SDG-350) *via* the static drop method. A flattened specimen was fixed on the stage, and ~2 μL of ultrapure water was dispensed vertically from ~2 mm onto the surface. The droplet image was captured, and the static contact angle at 2 s after deposition was determined.

Scanning Electron Microscopy (SEM)

SEM was performed using a Hitachi Su3500 microscope. Specimens were cut into approximately 5 mm × 5 mm pieces, mounted on the stage with conductive adhesive, and sputter-coated with a 10 nm gold layer. Imaging was conducted at an acceleration voltage of 5.0 to 15.0 kV and appropriate working distance, with surface and cross-sectional regions observed at multiple magnifications.

Fourier Transform Infrared (FT-IR) Spectroscopy

FT-IR spectra were collected by a Nicolet FT-IR spectrometer, and acquired over 4000 to 800 cm^{-1}, with 32 scans per spectrum to ensure high signal-to-noise ratio. A background spectrum was recorded prior to each measurement. Each sample was measured at least three times at different positions.

Preparation of *Stellera chamaejasme* L Antibacterial Components and Antibacterial Assay

The milled roots of *Stellera chamaejasme* L. (approx. 8 g) were extracted with ethanol at a solid-to-liquid ratio of 1:30 (w/v) under magnetic stirring at r.t for 24 h. The solvent was renewed after 8 h to ensure exhaustive extraction. The combined extracts were filtered and concentrated under reduced pressure using a rotary evaporator. The resulting concentrate was stored in amber glass vials at 4 °C until further use.

The ethanolic extract of *Stellera chamaejasme* L roots was applied to standard A4 paper at a coating weight of 3 g/m². Circular discs (d≈5 mm) were punched from the

coated paper, sterilized, and placed onto agar plates pre-inoculated with a standardized bacterial suspension. Untreated paper discs served as controls. After incubation at 37 °C for 18 to 24 h, antimicrobial activity was evaluated by measuring the diameter of the inhibition zone surrounding each disc.

RESULTS AND DISCUSSION

FT-IR Spectroscopy Analysis

FTIR was employed to characterize PVOH, KH550, and the modified product (KPVOH-2), as shown in Fig. 3. In the 3400 to 3500 cm^{-1} region, PVOH exhibited a broad characteristic peak at 3441 cm^{-1}, which was attributed to -OH stretching vibrations. For KPVOH-2, this peak shifted to 3429 cm^{-1} with noticeable broadening, suggesting partial disruption of the hydrogen-bonding network in PVOH and involvement of hydroxyl groups in condensation with KH550. In the 1500 to 1650 cm^{-1} region, KPVOH-2 displayed a new absorbance peak at 1621 cm^{-1}, corresponding to the N–H bending vibration of KH550, directly confirming successful grafting of KH550 onto PVOH chains. In the 1000 to 1250 cm^{-1} region, the C-O-C stretching vibration of PVOH shifted from 1098 to 1100 cm^{-1} in KPVOH, accompanied by a new enhanced peak at 1241.7 cm^{-1}. These were assigned to Si-O-C (PVOH-O-Si) and Si-O-Si stretching vibrations, further verifying covalent bond formation *via* condensation between PVOH hydroxyl groups and KH550 siloxane groups. These results collectively confirm the successful reaction between KH550 and PVOH, consistent with the experimental design.

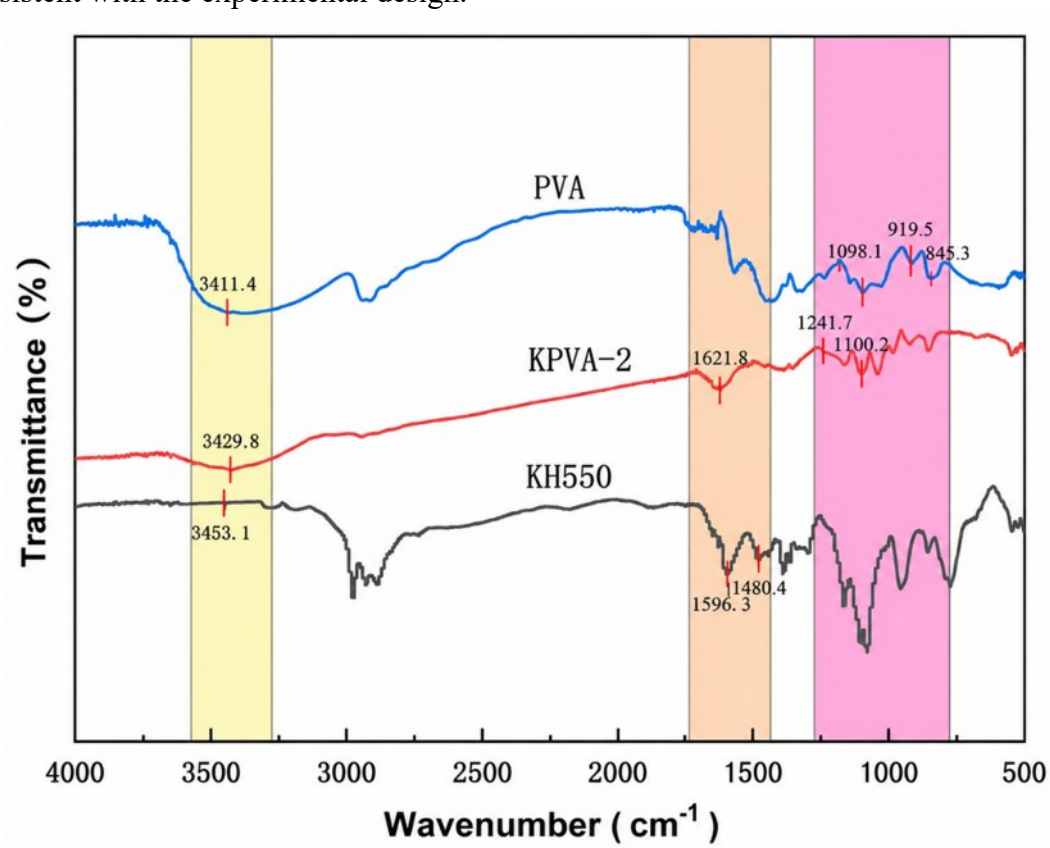

Fig. 3. FT-IR spectra of the PVOH, KH550, and KPVOH-2

Sizing Efficiency of Modified PVOH Sizing Agent

Figure 4 illustrates the effect of KH550-modified PVOH on the sizing weight and sizing degree of paper. Both exhibited an initial increase followed by a decrease with increasing KH550 dosage. As KH550 dosage increased to 1 g, sizing weight rose to 34.1% and sizing degree to 33 s (a 43.5% increase over pure PVOH). This enhancement is attributed to cross-linking between KH550 and PVOH, which improves sizing retention and promotes formation of a denser, more uniform film. However, at KH550 dosages above 1 g, both properties declined. At 1.5 g and 2 g, sizing degree dropped to 30 s and 29 s, respectively. Excessive KH550 disrupts the dispersion stability of the PVOH sizing solution, inducing molecular agglomeration. This reduces adhesion of the sizing agent on fibers, decreasing sizing weight, and introduces defects during film formation, compromising barrier function and reducing sizing degree. In short, appropriate KH550 addition synergistically enhances both sizing weight and sizing degree via cross-linking.

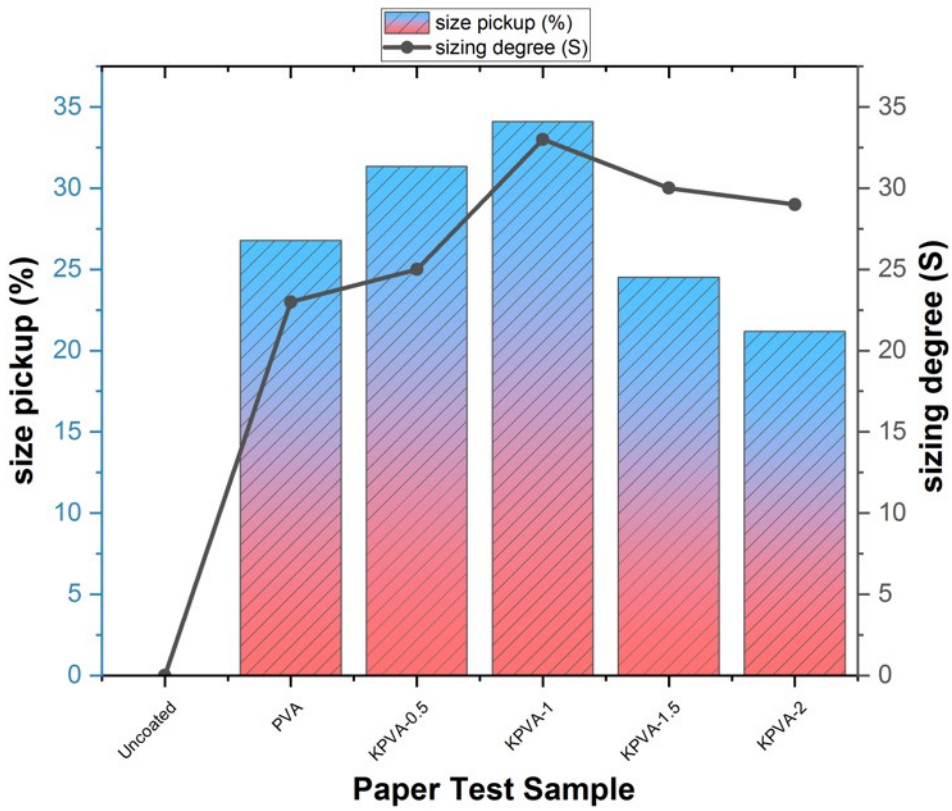

Fig. 4. Sizing degree and sizing weight of paper treated with different KH550 dosages

Optical Properties of Sized Paper

The ISO brightness and lightness (L^*) of paper after different sizing treatments are illustrated in Fig. 5.

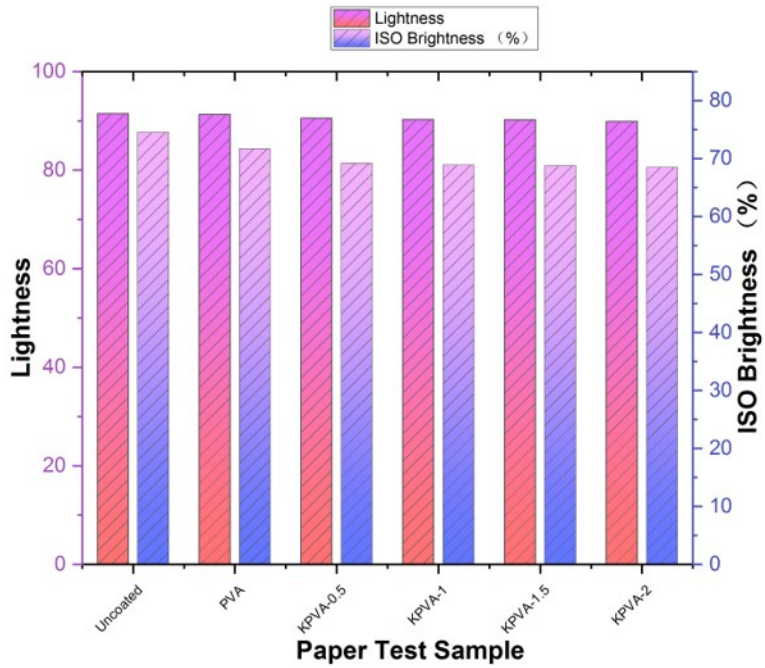

Fig. 5. Whiteness and ISO brightness of paper treated with different KH550 dosages

Both parameters decreased following sizing. The uncoated sample exhibited optical properties, with an ISO brightness of 74.5% and lightness (*L* value) of 91.5. After PVOH sizing alone, both parameters showed only minor reductions: ISO brightness decreased to 71.7%, while lightness remained nearly unchanged at 91.3. With the incorporation of KH550, the ISO brightness further decreased to approximately 70%. Interestingly, the decrease in brightness became less pronounced above a KH550 dosage of 1 g. Similarly, the ISO brightness dropped by only about 2.5 percentage points from the uncoated value. These marginal changes indicate that the sizing treatments, including the incorporation of KH550, have a limited impact on the overall whiteness of the paper.

Mechanical Properties of Sized Paper

Tensile strength is a key indicator of paper durability, reflecting inter-fiber bonding enhanced by PVOH sizing. As shown in Fig. 6, the tensile index initially increased with KH550 dosage, peaking at 50.12 N·m/g at 1 g—a 17.5% improvement over PVOH alone and 20.9% over the uncoated control. This enhancement is attributed to KH550's coupling effect, which transforms the PVOH-fiber interface from physical adhesion to combined physical-chemical bonding *via* cross-linking, improving interfacial strength and stress distribution. Beyond 1 g, the tensile index slightly declined, it is due to PVOH agglomeration, though residual cross-linking sustained relatively high performance.

Tear index, reflecting resistance to tearing, increased substantially after sizing. The uncoated sample exhibited 83.5 mN·m²/g. PVOH sizing alone raised it to 161 mN·m²/g (a 92.3% increase). KH550 further enhanced tear index, reaching a peak of 220 mN·m²/g at 1 g, which was 36.7% above PVOH alone and 163.3% above the uncoated control. At dosages above 1 g, tear index gradually decreased, consistent with tensile trends and attributable to PVOH agglomeration. This decrease is consistent with observations in a related study (Guo *et al*. 2021) where excessive KH550 also led to reduced tear performance.

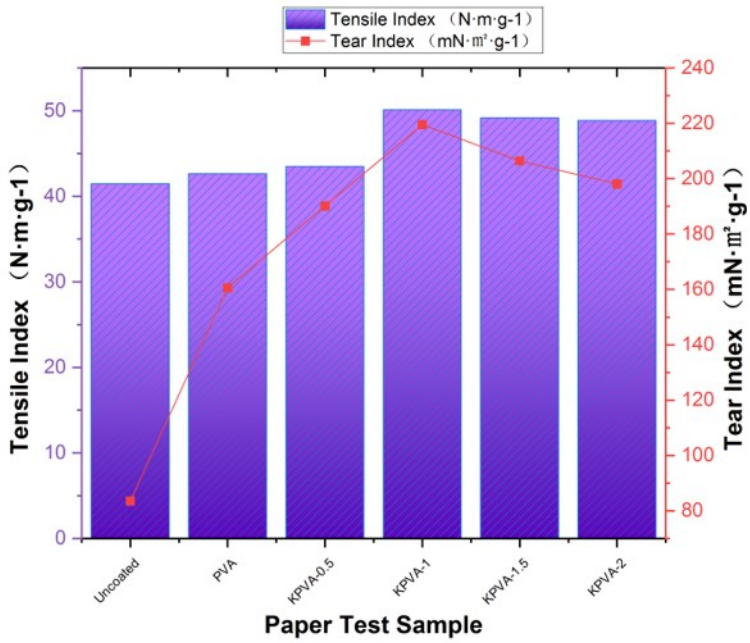

Fig. 6. Tear index and tensile index of paper treated with different KH550 dosages

Surface Wettability of Sized Paper

The water contact angle directly reflects the film-forming effect of the sizing agent and the liquid repellency of the paper (Fig. 7). The uncoated sample exhibited a contact angle of only 32.0°, indicating strong surface hydrophilicity, contrast to the strong hygroscopic property of Tibetan paper. As KH550 dosage increased, the contact angle grew and then gradually declined. At optimal dosage, KH550 cross-linked with PVOH and fibers, enhancing film density and continuity while reducing hydrophilic group exposure (Kupalang *et al.* 2024). The hydrophobic organosilane segments further formed a hydrophobic layer on the paper surface, collectively increasing contact angle. Excess KH550 induced molecular aggregation, disrupting film uniformity and re-exposing hydrophilic groups, while compromising the integrity of the hydrophobic layer, leading to reduced contact angles.

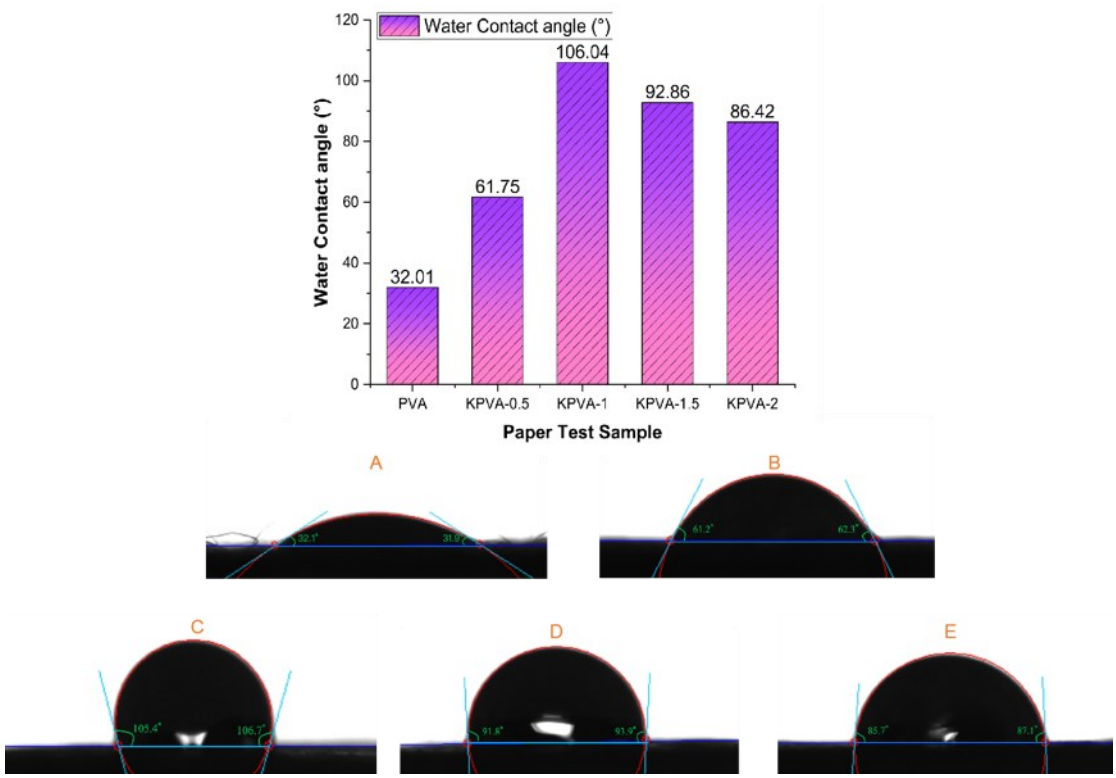

Fig. 7. Water contact angle of paper surface treated with different KH550 dosages

Characterization of the Microscopic Topography of Paper Surfaces

Scanning electron microscopy was employed to examine the surface morphology of uncoated paper and paper coated with varying KH550 dosages. The uncoated sample exhibited a loose fiber network with numerous pores and no adhesive film, consistent with its poor performance. The KPVOH-0.5 group showed a thin, discontinuous film, indicating insufficient cross-linking and limited property enhancement. In contrast, the KPVOH-1 group displayed a continuous, dense film with tightly bonded fibers, corresponding to optimal macroscopic performance. At higher dosages, agglomeration caused film defects and increased porosity, leading to performance decline. These observations confirm that KH550 at an optimal level improves interfacial bonding and film continuity, corroborating mechanical test results.

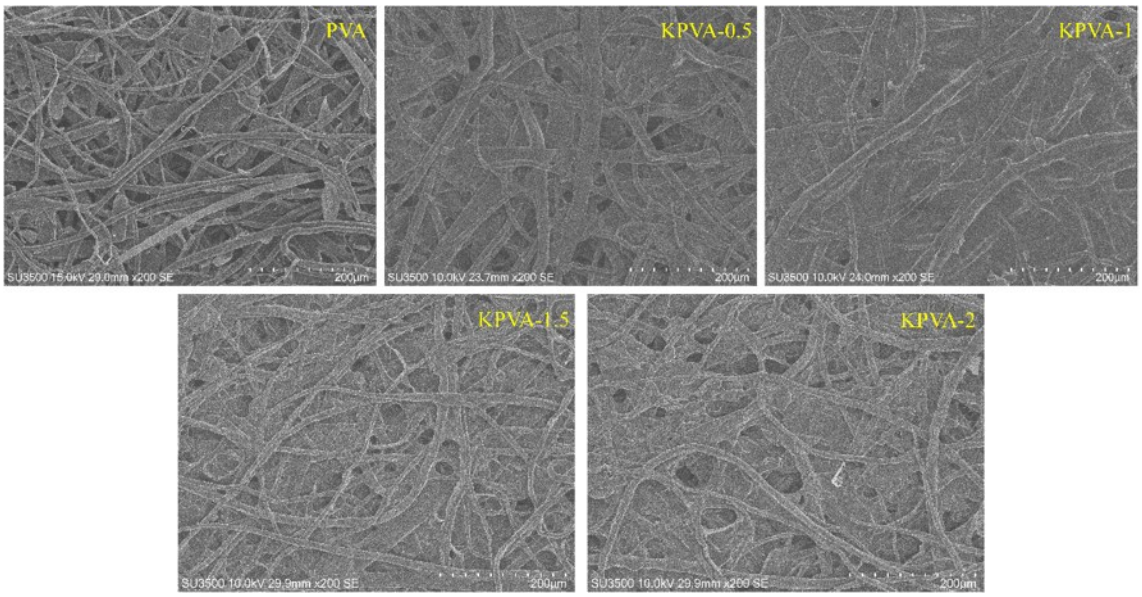

Fig. 8. Microscopic image of the paper surface

Antibacterial Performance of the Bio-based Sizing Agent

The inhibition zone method is a classic qualitative assessment technique for microbial antibacterial activity. The inhibition zone test results (Fig. 9) showed that untreated A4 paper exhibited no antibacterial activity, whereas uncoated Tibetan paper produced an inhibition zone of 7.1 mm.

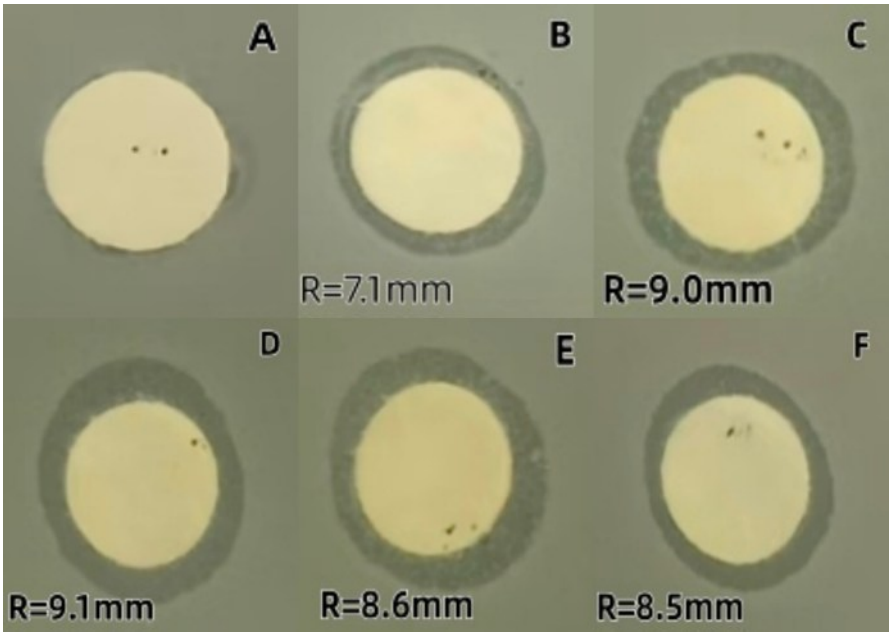

Fig. 9. Inhibition zone test results of different paper Sample: standard A4 paper (a), sample of uncoated Tibetan paper (b), Paper sample after sizing with KPVOH-0.5 (c) KPVOH-1 (d) KPVOH-1.5 (e) and KPVOH-2 (f).

In contrast, A4 papers treated with different concentrations of the extract all showed inhibition zones exceeding 7.1 mm. This is due to residual bioactive compounds from the traditional *Stellera chamaejasme* root fibers. In contrast, paper treated with the bio-based sizing agent containing *Stellera chamaejasme* L extract showed the strongest antibacterial effect, The increase results primarily from the high concentration and uniform distribution of ethanol-extracted antimicrobials in the coating (Dang *et al.* 2024, 2025), meeting the national standard for antibacterial paper.

The ethanol extract of *Stellera chamaejasme* L roots is rich in diterpenoid alkaloids, coumarins, and flavonoids, which disrupt microbial cell membranes by embedding into the phospholipid bilayer, causing leakage of intracellular components and inhibiting microbial growth. A key reason for the robust activity is the multi-target synergy of these compounds. These active compounds also bind to fiber surfaces *via* hydrogen bonds and van der Waals forces, forming a stable antibacterial layer that creates an unfavorable microenvironment for microbial survival, thereby enhancing the paper's antibacterial performance.

CONCLUSIONS

1. A bio-based surface sizing agent composed of *Stellera chamaejasme* L. root extract and KH550-modified PVOH was successfully prepared, endowing paper with an inhibition zone of 8.8 mm against S. aureus, meeting national antibacterial standards.
2. The dual mechanism involves disruption of microbial membranes by root extract components and their stable fixation onto fibers *via* hydrogen bonds and van der Waals forces, while KH550 forms a three-dimensional cross-linked network through condensation with hydroxyl groups.
3. Optimal performance was achieved at 1 g KH550, giving a sizing weight of 34.1%, tensile index of 50.1 N·m/g, tear index of 220 mN·m²/g, and water contact angle of 106°; excess KH550 caused aggregation and film defects.
4. This green functionalization strategy utilizes natural plant extracts instead of synthetic biocides, offering a sustainable approach for antimicrobial and mechanically reinforced paper with promising applications in cultural heritage conservation and specialty paper products.
5. The developed bio-based sizing agent is cost-effective due to its low KH550 dosage and renewable *Stellera chamaejasme* roots. It significantly enhanced paper strength, water resistance, and antibacterial performance, making it suitable for high-end cultural papers, antibacterial packaging, and archival conservation. Despite its promising performance, the present film system has limitations in long-term durability and industrial scalability that merit further investigation.

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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