Mechanism of Ink and Pigment Detachment from Palm Leaf Manuscripts Driven by Hygroexpansion

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Palm-leaf manuscripts use palm leaves as their medium. The inherent poor dimensional stability of this biological material is the main reason for ink and pigment detachment. As a valuable cultural heritage, the detachment of ink or pigment on the surface of palm leaf manuscripts under humid-dry cycling poses a critical challenge in the field of conservation. This study simulated traditional palm leaf manuscript preparation and employed accelerated humid-dry cycling to elucidate ink/pigment detachment mechanisms. The substrate—composed of cellulose, lignin, hemicellulose, waxes, and pectin—exhibits anisotropic deformation during cycling: thickness/tangential expansion significantly exceeds longitudinal direction. Chromatic analysis showed minor ΔE^* increases in substrate, ink, and pigment, confirming stable chemistry. Color changes primarily resulted from interfacial microcracks and light scattering due to physical deformation. Detachment area escalated with cycles, driven by substrate-ink/pigment expansion mismatch. This induces interfacial tensile-compressive stress cycling, causing mechanical fatigue, adhesion loss, and eventual powdering/flaking. Key conservation strategies include stabilizing environmental humidity and developing flexible protective coatings to buffer interfacial stress. This provides theoretical foundations for scientific preservation of palm leaf manuscripts and conservation material design.

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INTRODUCTION

Palm leaf manuscripts, as vital documentary substrates before the widespread use of paper in South Asia, Southeast Asia, and certain regions of China such as Tibet and Yunnan, encapsulate abundant historical, cultural, and religious information, and are regarded as invaluable cultural heritage (Vijaya Lakshmi *et al.* 2018; Zhang *et al.* 2021). The ink and pigments on palm-leaf manuscripts constitute an ancient writing and painting system, composed of colorants such as carbon black and inorganic mineral pigments combined with an animal glue binding medium. This material system was hand-prepared according to traditional techniques and applied onto the palm-leaf substrate. However, after centuries of preservation, these manuscripts face severe challenges from environmental factors, leading to progressive degradation of their physical structure and the stability of their inks and pigments (Zhang *et al.* 2022; Chu *et al.* 2024; Lian *et al.* 2024). Among these, cyclic fluctuations in temperature and humidity are widely considered to be key contributors to material deterioration, particularly the detachment of ink or pigments.

The palm leaf, characterized by a surface layer of silica, possesses inherently low water absorption and a smooth texture. This hydrophobic nature limits ink penetration and anchorage, resulting in weak ink or pigment adhesion. Zhang *et al.* (2025) revealed that exposure to extremely dry or humid conditions adversely affects palm leaf manuscripts. Under dry conditions, they exhibit warping, cracking, reduced mechanical strength, decreased hygroscopicity, and chemical degradation. In humid environments, fungal growth compromises the structural integrity of the manuscripts. These alterations lead to changes in color, gloss, hygroscopic behavior, cellulose crystallinity, and thermal stability, thereby diminishing mechanical properties. Therefore, it is hypothesized that there may be a connection between the detachment of ink and pigments and the dimensional changes of the substrate (Zhang *et al.* 2025).

The precise mechanism by which humid-dry aging induces the physical detachment of inks or pigments remains unclear. While substrate degradation under aging has been partially characterized, systematic experimental investigations into ink-substrate interfacial failure mechanisms under cyclic stress remain scarce. Understanding this process is essential for developing targeted preventive and restoration strategies.

This study addressed the critical knowledge gap by investigating ink/pigment detachment behavior and underlying mechanisms during humid-dry aging. At present, in the Xishuangbanna Dai Autonomous Prefecture of Yunnan Province, China, there are still inheritors proficient in the traditional techniques of palm-leaf manuscript craftsmanship who impart knowledge to the public. This serves as the technical foundation for current preparation of simulated palm-leaf manuscript samples. An accelerated aging test, involving controlled humid-dry cycles, was conducted on samples of palm leaf manuscripts produced by traditional simulation methods. This work comprehensively examined morphological evolution and pigment detachment progression across aging stages, chemical alterations in structural com-ponents (lignin, cellulose, hemicellulose) and extractives, alongside associated shifts in color fidelity, surface gloss, and dimensional stability. Through integrated extended depth-of-field microscopy, chemical profiling, and chromatic/morphological characterization, this work elucidates microscopic interfacial failure mechanisms. The findings provide theoretical foundations and experimental benchmarks essential for advancing scientific preservation protocols, developing preventive protection strategies for fragile media, and guiding next-generation restoration material design.

EXPERIMENTAL

Materials

The palm leaf material used in this study was sourced from Xishuangbanna Dai Autonomous Prefecture, Yunnan Province, China. The preparation of palm leaf manuscripts in this region follows traditional craftsmanship, with key procedures including leaf collection, trimming, steaming, polishing, flattening, air-drying, inscribing, inking, and binding. The palm leaf material was then cut into samples measuring 40 mm × 20 mm. To simulate the writing characteristics of actual palm leaf manuscripts, the samples were stained using inks prepared from pot soot, cinnabar, and plant oil. As a result, simulated palm leaf manuscripts bearing red and black inscriptions were obtained. In addition, ancient palm-leaf manuscripts were collected from the Xishuangbanna Dai Autonomous Prefecture and used for material analysis.

Material Characterization of Palm Leaf Manuscripts

Fourier transform infrared spectroscopy (FTIR)

ATR-FTIR spectra were collected using a ThermoFisher iS5 instrument (USA), with a spectral resolution of 4000 to 400 cm⁻¹ and an average of 64 scans per sample. The ATR crystal was diamond, with a 45° incident angle, a refractive index of 1.0 for the sample, and 1.62 for the crystal.

Gas chromatography—mass spectrometry (GC-MS)

GC-MS analysis was performed using a Shimadzu GCMS-TQ8050 system equipped with a Waters HP-5MS UI capillary column (30 m, 0.25 mm ID, 0.25 μ m film thickness), and a Shimadzu HS-20 headspace sampler. GC conditions included split injection (split ratio 10:1), an injection port temperature of 35 °C, and constant linear velocity mode. The temperature program started at 40 °C (held for 2 min), then increased at 15 °C/min to 280 °C (held for 10 min). The MS was operated in electron ionization mode with an ion source temperature of 200 °C, using full scan mode from 40 to 500 m/z.

Aging Tests

Humid-dry cyclic aging test

An accelerated aging simulation was performed on palm leaf manuscript samples using an alternating humid-dry cycle method. Samples were placed in a constant temperature and humidity chamber and alternately exposed to two extreme environmental conditions: high temperature/high humidity (65 °C, 80% RH) and low temperature/low humidity (25 °C, 30% RH). Each exposure lasted 48 h before immediately switching to the alternate environment. The humid-dry cycle was repeated until a total of 27 cycles was completed.

3D optical microscopy

A VHX-6000 3D microscope (KEYENCE, Japan) was used to observe the surface morphology of the manuscript samples, including substrate, ink, and pigment regions, at high resolution. Based on the acquired micrographs, ImageJ software was used to quantitatively evaluate the detachment area ratio of inks and pigments to assess the degree of surface material loss during the aging process.

Chemical composition analysis

Aged and unaged palm leaf samples were ground into 18- to 60-mesh powder. Chemical analysis was performed following the method reported by Chu *et al.* (2025), and each group consisted of three parallel samples. Benzene–ethanol extractives were measured using a 2:1 (v/v) benzene–ethanol solution. After extraction, Klason lignin (acidinsoluble lignin) was determined by sulfuric acid hydrolysis. Holocellulose content was measured according to Chinese National Standard GB/T 2677.10 (1995). Specifically, holocellulose was isolated from benzene–ethanol extracted samples using glacial acetic acid and sodium hypochlorite. The isolated holocellulose was subsequently used for α -cellulose content determination *via* the sodium hydroxide method. Hemicellulose content was calculated as the difference between holocellulose and α -cellulose contents.

Color change analysis

A bench-top colorimeter (CR-400, Konica Minolta, Japan) was used to measure surface colorimetric parameters: lightness (L^*), red–green axis (a^*), and yellow–blue axis

(b^*). To evaluate color differences among samples, total color difference (ΔE^*) was calculated using the following formula,

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \tag{1}$$

where ΔL^* , Δa^* , and Δb^* represent changes in lightness, red-green, and yellow-blue values before and after treatment, respectively.

Dimensional stability test

To explore the effect of moisture on size change, additional specimens were immersed fully in distilled water. The immersion lasted for 72 h (3 days) to reach full saturation. After soaking, dimensional changes were recorded to evaluate the impact of water on sample size.

RESULTS AND DISCUSSION

Material Analysis of Palm Leaf Manuscripts

The infrared (IR) spectral analysis (Fig. 1) revealed several characteristic absorbance peaks. A broad band at 3290 cm⁻¹ corresponds to O–H stretching vibrations, while strong peaks at 2918 cm⁻¹ and 2850 cm⁻¹ are attributed to asymmetric and symmetric C–H stretching of –CH₃ and –CH₂ groups, respectively (Kayabaş and Yildirim 2021; Mahdi *et al.* 2023). The absorbance peak near 1740 cm⁻¹ may originate from ester carbonyl groups or acetyl groups in hemicellulose. The 1640 cm⁻¹ band could be associated with the stretching vibration of amide carbonyls or aromatic C=C skeletal vibrations (Gorgulu *et al.* 2007; Ivanova and Singh 2003). Peaks at 1460 cm⁻¹ and 1370 cm⁻¹ are assigned to bending vibrations of –CH₃ and –CH₂, respectively, which are typical for lignin. Additionally, the peak at 1733 cm⁻¹ suggests the presence of pectin, which generally exhibits C=O stretching in the 1740 to 1760 cm⁻¹ range.

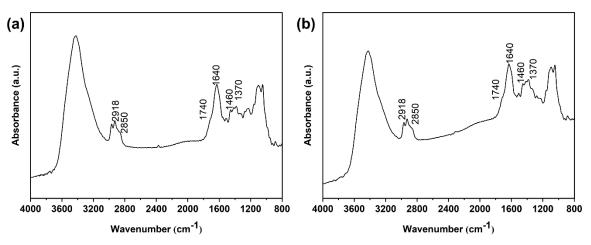


Fig. 1. Fourier transform infrared spectrum of the palm leaf manuscripts substrate

Pectin is a complex polysaccharide composed of 1,4-linked α-D-galacturonic acid and is a key component of the middle lamella in terrestrial plant cell walls, facilitating cell adhesion. Because the palm leaves used as manuscript substrates originate from *Arecaceae* species, they typically contain surface waxes. The infrared features of palm wax include O–H stretching at 3300 cm⁻¹, –CH₃ and –CH₂ peaks at 2960 and 2850 cm⁻¹, sharp and intense C=O peaks within 1650 to 1900 cm⁻¹, and C–O stretching bands in the 1050 to 1250 cm⁻¹ range. It is important to note that the symmetric C-H bonds in long-chain aliphatic compounds exhibit inherently weak infrared absorbance. Therefore, the absence of strong signals does not preclude the presence of waxes, and their composition was further unequivocally confirmed by the more sensitive GC-MS analysis. Collectively, the FTIR analysis suggests that the material of palm leaf manuscripts consists primarily of cellulose, hemicellulose, and lignin, with additional components such as plant waxes, pectin, and water-soluble compounds.

To confirm these results and resolve overlapping peaks of cellulose, hemicellulose, lignin, pectin, and waxes, gas chromatography-mass spectrometry (GC-MS) was employed on two representative leaf samples. The chromatographic profiles of samples S1 and S2 (Fig. 2a and b) were analyzed, with identified compounds listed in Tables 1 and 2. GC–MS analysis revealed the presence of monoterpenes and long-chain saturated alkanes. Compounds such as methylcyclopentyl acetate and 2,2,6,7-tetramethyl-10-oxabicyclo-[4.3.0.1(1,7)]dec-5-one were identified in S1. These monoterpenes are commonly found in secretory structures (e.g., resin ducts or glandular tissues) of higher plants and exhibit biological activities including microbial resistance and pollinator attraction. Long-chain saturated hydrocarbons, which constitute primary components of plant waxes, were also detected. Plant waxes typically comprise hydrocarbons (predominantly n-alkanes, branched alkanes, and cycloalkanes), aromatic compounds (e.g., substituted benzenes), and aliphatic compounds (e.g., fatty acids, alcohols), with saturated alkanes representing the dominant group. The above GC-MS results indicate that in addition to cellulose, hemicellulose, and lignin, the palm leaf manuscript substrate also contains plant waxes and other components.

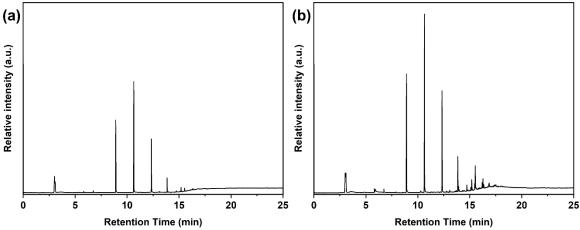


Fig. 2. The gas chromatography–mass spectrometry of palm leaf manuscripts. (a) S1; (b) S2

Peaks	Retention time (min)	Chemical substances
1	7.890	3-Methylcyclopebtyl acetate
2	12.755	2,2,6,7-Tetramethyl-10-oxatricyclo[4.3.0.1(1,7)]decan-5-one
3	13.080	Tetradecane
4	13.700	Triethylidene mannitol
5	15.100	Octadecane
6	15.525	Pentadecane,2,6,10,14-tetramethyl-
7	16.190	Heneicosane
8	16.270	Eicosane
9	16.830	Dotriacontane
10	16.87	Heneicosane

Table 1. The Results of GC-MS of S1

Table 2. The Results of GC-MS of S2

Peaks	Retention time (min)	Chemical substances
1	14.725	Heneicosane
2	15.100	Pentadecane, 2,6,10,14-tetramethyl-
3	15.525	Pentadecane, 2,6,10,14-tetramethyl-

Humid-Dry Cyclic Aging Analysis

Figure 3 illustrates the macroscopic changes in the palm leaf manuscript substrate, ink, and pigment during the humid-dry aging cycles. In the initial state, the substrate surface was relatively intact, with sharp and uniformly dark black ink lines and bright, evenly distributed red pigment.

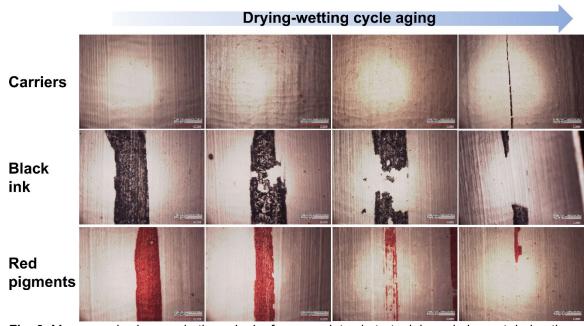


Fig. 3. Macroscopic changes in the palm leaf manuscript substrate, ink, and pigment during the wet-dry aging cycles

As the humid-dry cycles proceeded, microcracks began to appear on the substrate surface, likely due to internal stress variations caused by alternating moisture conditions. For the black ink, slight edge blurring appeared during the early stages of the cycling,

possibly due to moisture-induced migration or diffusion of ink components. With increased cycles, ink detachment became increasingly evident, with thinning and large-scale flaking in some areas, indicating reduced adhesion be-tween the ink and the substrate. The red pigment showed slight fading in the early stages, which may be related to dissolution or dilution caused by moisture. As cycling progressed, pigment loss became more pronounced, with noticeable flaking and mott-ling, likely due to weakened bonding between the pigment and substrate under cyclic moisture conditions.

Figure 4 shows the trends and error ranges of L^* , a^* , b^* , and ΔE^* values for the aged palm leaf manuscript substrate. Figure 4a indicates a gradual increase in L^* during early aging, a sharp rise between cycles 5 and 7, followed by stabilization (Li *et al.* 2024). Figure 4b shows that a^* decreased initially but rose significantly after the third cycle, indicating enhanced redness. Figure 4c displays the b^* values across cycles. The observed minor fluctuations, particularly the slight rise after cycle 4 and the subsequent decline after cycle 20, are of a magnitude that could be attributable to measurement variability, such as minor instrument calibration drift or heterogeneity in the leaf surface yellowness. Figure 4d reveals continuous growth in ΔE^* , especially during cycles 1 to 12, which stabilized afterward, reflecting combined chemical and physical changes.

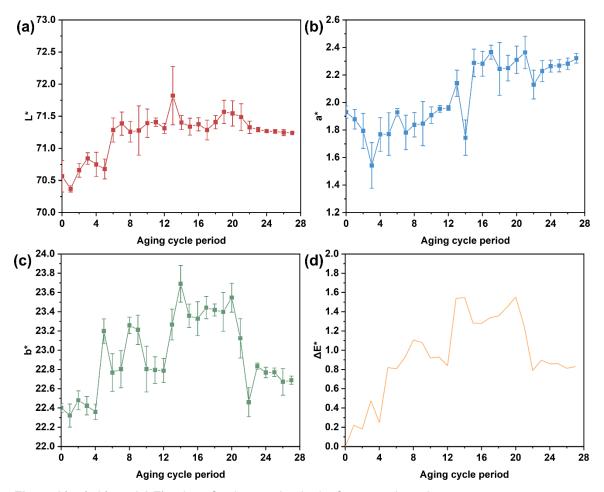


Fig. 4. L^* , a^* , b^* , and ΔE^* values for the aged palm leaf manuscript substrates

Figure 5 presents the changes in colorimetric indices of the black ink. Its initial L^* value fluctuated slightly between 29.0 and 30.0, gradually increased with cycles, and

showed a significant inflection point. The a^* value showed minor fluctuations, while b^* exhibited an overall upward trend. ΔE^* increased with aging, indicating reduced color stability and growing deviation from the original state.

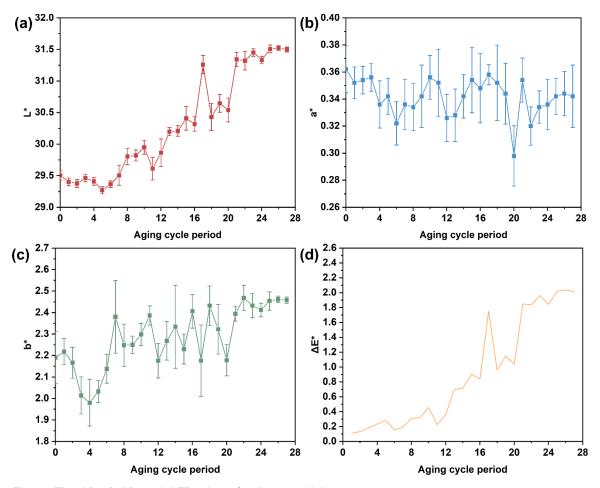


Fig. 5. The L^* , a^* , b^* , and ΔE^* values for the aged ink

Figure 6 shows the chromatic changes of the red pigment. The L^* value slowly increased initially but rose sharply to 46.5 to 47.0 after cycles 20 to 28, possibly due to enhanced reflectance or increased surface roughness. The a^* value increased steadily after the fifth cycle, suggesting a redder hue caused by oxidation or compositional shifts. The b^* value remained stable in the early stages and then rose slowly, indicating a yellowing trend likely caused by degradation or chemical reactions. ΔE^* also continuously increased over the aging cycles.

In summary, although aging cycles had some influence on the colorimetric indices of the palm leaf manuscript body, ink, and pigment, the changes were not particularly significant, indicating that their chemical compositions were relatively unaffected.

The notable detachment of ink and pigment is likely attributable to physical factors, such as reduced adhesion or internal stress-induced structural failure. Wet chemical methods were employed to analyze the chemical components of the palm leaf manuscripts (Fig. 7). With increasing humid-dry cycling, changes in the main chemical components remained relatively small. Cellulose content slightly increased from 37.2% to 38.2%,

indicating its relative stability. Hemicellulose content showed a minor decrease from 23.6% to 21.9%, possibly due to its susceptibility to hydrolysis or degradation. Lignin content remained stable at 30.6% to 31.2%, demonstrating strong resistance to degradation. Extractives fluctuated within a narrow range of 8.6% to 8.8%. This overall chemical stability contrasts with the pronounced physical degradation observed in the substrate, ink, and pigment.

The microstructural damage, such as cracking and flaking, is more likely caused by internal stress changes under dry—wet cycling than by chemical deterioration. Nonetheless, such physical damage is critical to the preservation state of these artifacts and cannot be ignored.

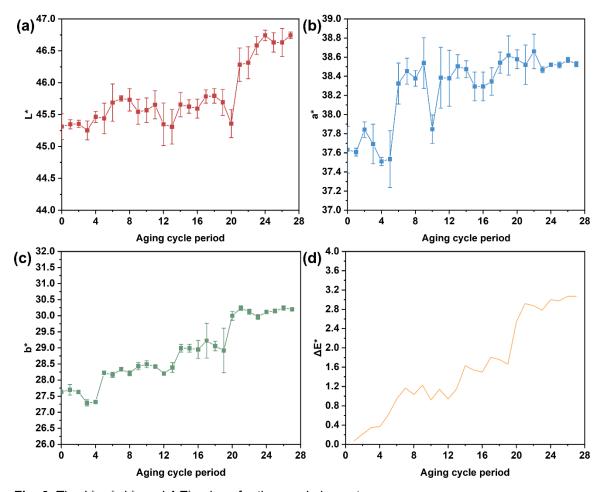


Fig. 6. The L^* , a^* , b^* , and ΔE^* values for the aged pigment

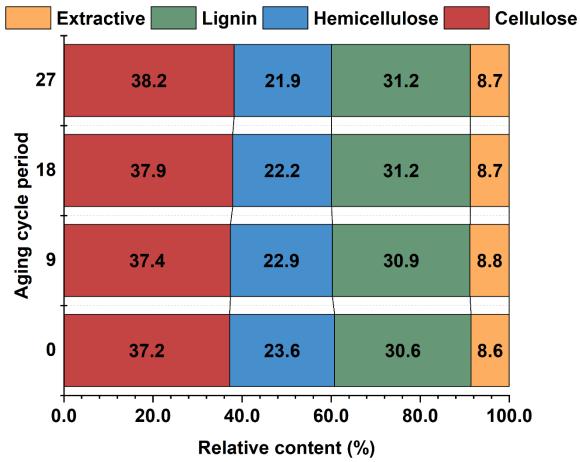


Fig. 7. The chemical components of the palm leaf manuscripts

Mechanism of Ink and Pigment Detachment in Palm Leaf Manuscripts under Cyclic Humid-Dry Aging

To investigate the effects of physical changes on ink and pigment detachment, palm leaf manuscripts were subjected to drying—saturation cycling (Fig. 8). Under these conditions, the manuscript exhibited significant deformation in the thickness and tangential directions due to the fiber structure's moisture response (Zhang et al. 2024; Zhu et al. 2024). When saturated, water molecules penetrated between cellulose microfibrils, weakening hydrogen bonds and causing expansion (Paajanen et al. 2022; Zitting et al. 2021). In contrast, longitudinal dimensional change was limited, owing to the aligned arrangement of microfibrils, which offered structural stability. This orientation, along with tight fiber packing, inhibited moisture diffusion longitudinally. During humid-dry cycles, the manuscript's dimensional instability contrasted with the relatively stable ink and pigment, causing internal stress at the interface (Chen et al. 2024; Zhang et al. 2021). Expansion of the manuscript body during wetting and its contraction during drying, combined with minimal deformation in the ink and pigment, generated alternating tensile and compressive stresses. These stresses progressively weakened adhesion, and their accumulation over multiple cycles led to ink and pigment detachment.

Under cyclic humid-dry conditions, the palm leaf manuscript body exhibited poor dimensional stability, whereas the ink and pigment, which typically consist of relatively stable inorganic or organic materials, showed minimal deformation under moisture. The significant mismatch in expansion and contraction between these components generates

considerable internal stress at their interface. As the manuscript absorbs water, its volume increases—especially in the thickness and tangential directions—while the ink and pigment swell much less due to low moisture absorbency, resulting in tensile stress at the interface. Conversely, during drying, the manuscript body contracts more than the ink and pigment, creating compressive stress. These alternating tensile and compressive stresses gradually weaken interfacial adhesion. Over time, the repeated dry—wet cycling accelerates internal stress accumulation, leading to a progressive loss of adhesion and eventual detachment of ink and pigment from the manuscript surface (Fig. 9).

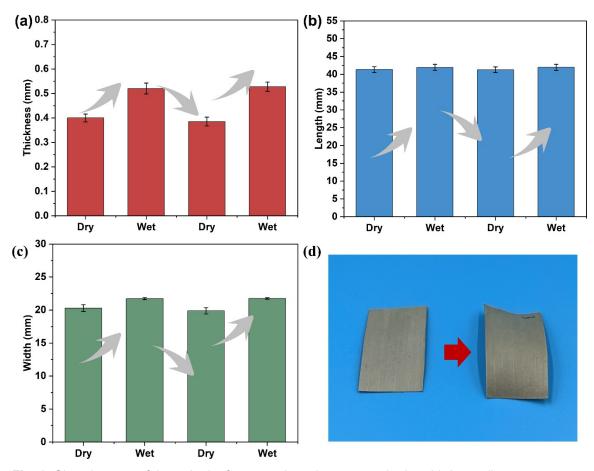


Fig. 8. Size changes of the palm leaf manuscript substrates under humid-dry cycling

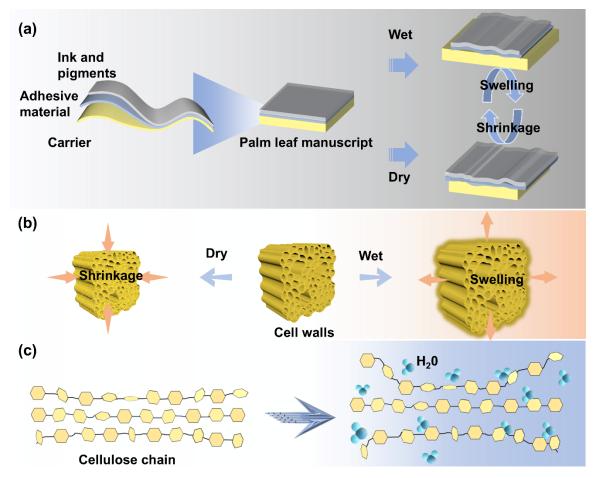


Fig. 9. Schematic diagram of the mechanism of ink and pigment detachment in palm leaf manuscripts under cyclic dry—wet aging

CONCLUSIONS

- 1. The primary mechanism of ink/pigment degradation under humid-dry aging is physical interfacial failure due to cyclic mechanical stress, not chemical degradation. Chemical analysis confirms the stability of key substrate components (cellulose, lignin), while long-term cyclic stress weakens adhesion through fatigue.
- 2. Anisotropic hygroscopic swelling of the manuscript substrate generates interfacial stress mismatches. The substrate swells significantly in thickness and tangential directions due to cellulose microfibril swelling, while remaining stable longitudinally due to fibril alignment. Ink/pigment, being dimensionally stable, experience alternating tensile and compressive stresses at the interface.
- 3. This interfacial stress mismatch leads to detachment failure. The long-term application of cyclic stresses resulting from the swelling mismatch causes mechanical fatigue at the ink/pigment-substrate interface, ultimately resulting in flaking and loss of the ink or pigment layer.

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