

Natural and accelerated weathering performance of vacuum-hydrothermally treated moso bamboo

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ABSTRACT

Bamboo is a renewable lignocellulosic material, but its hygroscopicity and limited weathering resistance restrict long-term outdoor applications. Vacuum-hydrothermal (VH) treatment offers a chemical-free strategy for improving dimensional stability; however, the influence of treatment severity on subsequent weathering performance remains insufficiently understood. In this study, moso bamboo was treated at 160–240 °C for 4–16 h, and selected specimens treated at 160 and 180 °C for 4 or 8 h were further subjected to 720 days of natural weathering and 500 h of xenon-arc accelerated weathering. Results showed that VH treatment markedly reduced moisture-related properties, with equilibrium moisture content decreasing from 11.5 % in untreated bamboo to 5.6 % at 200 °C for 16 h and thickness swelling decreasing from 7.0 % to 3.9 % even under the mild 160 °C for 4 h treatment. However, increasing treatment severity caused greater mass loss and mechanical deterioration. After 720 days of natural weathering, untreated specimens exhibited the highest mass loss (14.7 %), whereas VH-treated specimens showed lower values of 11.4–12.2 %. Nevertheless, the modulus of elasticity of all groups decreased substantially, with the VH-treated groups retaining 42.3–59.9 % of their initial values. In contrast, accelerated weathering produced more variable responses, and the 180 °C for 8 h group exhibited the highest mass loss (8.9 %), indicating increased susceptibility under relatively severe VH treatment. Surface SEM observations revealed progressive cracking, while cross-sectional SEM showed morphological alterations beneath the exposed surface. ATR-FTIR analysis revealed relative changes in hemicellulose- and lignin-associated bands and in the carbonyl region, indicating progressive surface chemical changes with distinct temporal responses under natural and accelerated weathering. These results demonstrate that VH treatment severity governs a trade-off between initial improvement in moisture-related properties and subsequent susceptibility to weathering-induced deterioration. Among the selected conditions, treatment at 160 °C for 8 h provided a comparatively favorable compromise among reduced hygroscopicity, retained mechanical integrity, and resistance to weathering-associated deterioration. This study provides a practical and quantitative basis for evaluating the natural and accelerated weathering performance of VH-treated bamboo, while highlighting the need to interpret accelerated-weathering results as complementary to, rather than directly equivalent to, outdoor exposure.

1. Introduction

In recent years, increasing attention to environmental sustainability and the efficient utilization of renewable resources has stimulated broad interest in natural lignocellulosic materials such as wood and bamboo

[1]. Although bamboo forests account for only a small proportion of the global forest area, bamboo is widely distributed across tropical and temperate regions and grows under diverse ecological conditions, from sea level to altitudes of up to 4,300 m [2,3]. Owing to its rapid growth, early maturity, high productivity, and favorable mechanical

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performance, bamboo has been regarded as a highly promising renewable biomaterial for a wide range of structural and non-structural applications [1].

The physical and mechanical properties of bamboo are closely associated with its multiscale hierarchical architecture, in which the organization of structural tissues and major cell-wall polymers collectively contributes to its material performance [4]. Recent advances in bamboo modification have further demonstrated that deliberate regulation of cell-wall chemistry and hierarchical structure can markedly alter mechanical performance, dimensional stability, and durability [5]. Among bamboo species, moso bamboo (*Phyllostachys pubescens* (Pradelle) Mazel ex J.Houz.) is one of the most important commercial resources because of its abundant availability and high utilization value. However, like other lignocellulosic materials, bamboo exhibits high hygroscopicity, which leads to dimensional instability through repeated moisture adsorption and desorption under changing environmental conditions [6,7]. This behavior results in swelling and shrinkage, thereby limiting its long-term performance in outdoor applications. In addition, the relatively high contents of starch and soluble sugars in bamboo increase its susceptibility to fungal attack and insect infestation, which further compromise its appearance, durability, and service life [1, 2]. Therefore, understanding how modification-induced changes in bamboo structure and cell-wall polymers influence material performance is essential for developing effective modification strategies and expanding the applicability and durability of bamboo products.

Thermal modification has been widely recognized as an environmentally friendly approach for improving the dimensional stability and durability of lignocellulosic materials. Previous studies have demonstrated that thermal treatment can effectively enhance the dimensional stability of wood and bamboo [8–10], while also improving durability and decay resistance [11–14]. These advantages make thermal modification a promising strategy for overcoming the inherent limitations of bamboo under outdoor service conditions. However, the practical value of thermal treatment depends on achieving an appropriate balance between improved stability and acceptable mechanical performance.

According to the heating medium employed, thermal modification technologies are generally classified into hydrothermal treatment, inert gas heat treatment, and oil heat treatment [11]. Among these methods, inert gas treatment has been extensively studied, whereas oil heat treatment provides relatively uniform heat transfer but may leave oil residues that interfere with subsequent utilization. In contrast, hydrothermal treatment uses water vapor as the heating medium, which can reduce thermal gradients, alleviate rapid-heating defects such as cracking, and lower energy consumption and processing costs [15]. Steam-assisted processing has also been explored as a practical approach for improving bamboo formability and value-added utilization in engineered products [16,17]. More specifically, recent studies have shown that saturated-steam and hydrothermal treatments can alter the chemical composition, cellular structure, and cell-wall mechanical properties of bamboo, while also influencing its physical performance and reported biological resistance [18,19]. Furthermore, pre-vacuuming the treatment chamber before heating removes air from both the chamber and the bamboo, thereby creating a low-oxygen environment and reducing the boiling point of water. This facilitates steam generation and penetration at lower temperatures and can effectively reduce cracking during treatment [20]. Accordingly, vacuum-hydrothermal (VH) treatment represents a promising thermal modification strategy for bamboo.

In recent years, bamboo has been increasingly used in engineered laminated products, outdoor materials, and construction-related applications, where weathering critically governs long-term performance. Surface discoloration, cracking, and property deterioration caused by prolonged environmental exposure can substantially reduce the service life and application value of bamboo products. Although previous studies have reported the effects of thermal treatment on the surface characteristics and chemical composition of bamboo [6,7], the relationship between VH treatment severity, surface chemical

characteristics, and subsequent long-term weathering performance remains insufficiently understood. In particular, how different VH treatment conditions influence moisture-related properties, surface deterioration, and mechanical retention during prolonged weathering has not been systematically evaluated.

Therefore, this study investigated the effects of different VH treatment conditions on moso bamboo by examining changes in physical, mechanical, and chemical properties. Selected VH-treated specimens with relatively balanced initial properties were further subjected to natural weathering and xenon-arc accelerated weathering tests to assess color change, mass loss, dimensional stability, mechanical retention, surface morphology, and ATR-FTIR characteristics during exposure. The aim of this work was to clarify how VH treatment severity influences the long-term weathering performance, surface chemical characteristics, and mechanical retention of moso bamboo, thereby identifying treatment conditions that provide a favorable balance between dimensional stability, mechanical integrity, and weathering resistance.

2. Experiments

2.1. Materials

Moso bamboo, approximately 4 years old, was obtained from a local supplier in Taiwan as a single batch of pre-cut strips. According to the supplier's preparation procedure, the strips were prepared from the middle portions of the culm internodes after removal of the outer epidermal layer and were supplied at dimensions of approximately 900 mm × 30 mm × 5 mm. Approximately six strips were obtained from each culm. Based on the total number of strips purchased, the material batch was estimated to represent approximately 100 bamboo culms. However, because the strips were supplied without culm-specific identification, the original culm identity of individual strips could not be retrospectively determined. The same batch of bamboo strips was used for both initial property characterization and subsequent weathering experiments.

2.2. VH treatment of moso bamboo

The purchased bamboo strips were sequentially allocated among the untreated and VH treatment groups. Because culm-specific identification was not available, individual strips could not be assigned according to their original culm identity. Replicate specimens used for subsequent property measurements were prepared, as far as practicable, from different bamboo strips to reduce dependence on any single strip. Accordingly, the sample sizes (n) reported in this study represent independent strip- or specimen-level replicates rather than independent biological replicates at the culm level. The prepared bamboo strips were subjected to VH treatment in a semi-industrial chamber (San Neng Ltd., Chiayi, Taiwan) with controlled temperature and pressure, as schematically illustrated in Fig. 1. Distilled water equivalent to 1/100 (v/v) of the chamber volume was added. The chamber was evacuated to below 23 kPa using a vacuum pump to establish a low-pressure environment. The temperature was then increased at a heating rate of 3 °C/min to the target temperatures of 160, 180, 200, 220, and 240 °C, followed by treatment under saturated steam conditions for 4, 8, or 16 h. After treatment, the specimens were air-dried and conditioned at 20 °C and 65 % relative humidity (RH) prior to further analyses. Because of the large number of specimens required for the entire experiment and the limited capacity of each VH treatment run, multiple VH treatment runs were conducted. All specimens originated from the same purchased batch of moso bamboo, but different specimen sets and VH treatment runs were used for the initial treatment screening and subsequent weathering experiments, as described below.

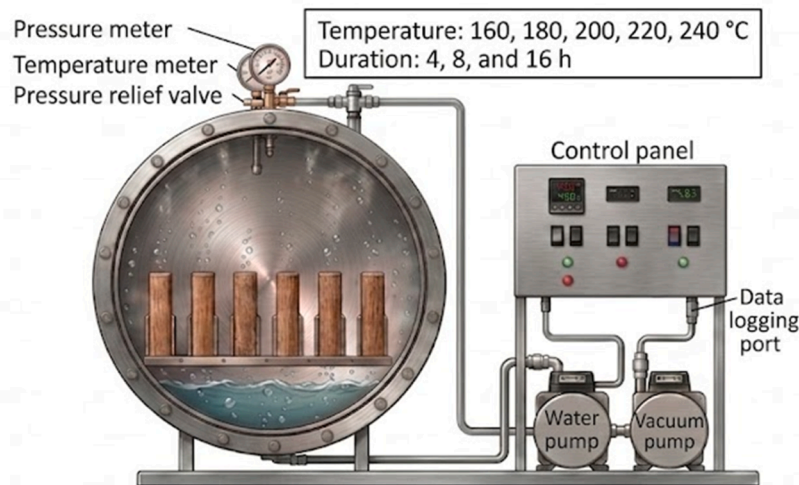


Fig. 1. Schematic illustration of the vacuum-hydrothermal (VH) treatment apparatus and treatment conditions. The schematic image was generated using ChatGPT (GPT-5.5 Thinking, OpenAI) as an explanatory visual aid for the experimental setup and was subsequently reviewed, edited, and verified by the authors for scientific accuracy.

2.3. Natural weathering and accelerated weathering tests

In this study, untreated bamboo and specimens treated under four VH conditions (160 °C-4 h, 160 °C-8 h, 180 °C-4 h, and 180 °C-8 h) were selected for both natural and accelerated weathering tests based on the overall balance between dimensional stability and mechanical performance. The specimens used for the natural weathering experiment were prepared in separate VH treatment runs from those used for the initial treatment screening and accelerated weathering experiment. Accordingly, the day-0 values reported for the natural weathering experiment represent the baseline properties of the specific specimen sets assigned to this experiment and are not necessarily identical to the initial values reported in Table 1. For the natural weathering test, the specimens were cut to dimensions of 150 mm (L) × 20 mm (W) × 5 mm (T). The inner surface of the bamboo culm was oriented southward on a test rack inclined at 45°, which was installed on the rooftop of the Department of Forestry, National Chung Hsing University, Taichung, Taiwan (24.12° N, 120.67° E). The outdoor exposure period extended from November

2023 to March 2026. The specimens were evaluated up to 720 days of exposure, and the monthly average climatic conditions during the experimental period are summarized in Fig. 2. Each treatment group consisted of 81 specimens, including 15 specimens assigned for periodic measurements of surface color, microstructure, physical properties, and surface chemical characteristics on days 0, 7, 15, 30, 60, 90, 180, 360, 540, and 720 of outdoor exposure. In addition, 55 specimens were allocated to destructive mechanical testing on days 0, 180, 360, 540, and 720, with 11 specimens tested at each time point. The remaining 11 specimens were used for dimensional stability evaluation on days 0, 30, 60, 90, 180, 360, 540, and 720.

For the accelerated weathering test, the specimens were cut to dimensions of 80 mm (L) × 20 mm (W) × 5 mm (T). The specimens assigned to the accelerated weathering experiment originated from the same VH treatment runs as those used for the initial treatment screening. However, because the capacity of the xenon-arc weathering chamber limited the number of specimens that could be exposed simultaneously, separate specimen sets were allocated for some property measurements.

Table 1

The properties of mass loss (ML), density, equilibrium moisture content (EMC), moisture excluding efficiency (MEE), thickness swelling (TS), anti-swelling efficiency (ASE), modulus of elasticity (MOE), and modulus of rupture (MOR) of moso bamboo before (untreated) and after VH treatment.

Sample	ML (%)	Density (kg/m ³)	EMC (%)	MEE (%)	TS (%)	ASE (%)	MOE (GPa)	MOR (MPa)
Untreated	-	771.0 ± 48.9 ^a	11.5 ± 0.4 ^a	-	7.0 ± 1.2 ^a	-	11.1 ± 0.4 ^a	165.4 ± 6.1 ^a
160 °C-4h	1.7 ± 0.3 ^f	705.2 ± 88.6 ^{ab} *	8.4 ± 0.3 ^b	26.8 ± 2.6 ^g	3.9 ± 0.5 ^b	44.0 ± 6.9 ^e	9.5 ± 0.7 ^{ab}	130.9 ± 14.3 ^{ab}
160 °C-8h	2.1 ± 0.5 ^f	719.5 ± 98.8 ^{ab} *	7.6 ± 0.3 ^c	33.9 ± 2.6 ^f	3.6 ± 0.6 ^b	49.2 ± 8.9 ^{de}	10.4 ± 0.5 ^a	127.5 ± 8.6 ^{bc}
160 °C-16h	2.3 ± 0.5 ^f	676.2 ± 74.6 ^{ab} ***	7.3 ± 0.2 ^{cd}	37.0 ± 1.5 ^{ef}	3.7 ± 0.5 ^b	47.2 ± 7.6 ^{de}	9.8 ± 0.9 ^{ab}	113.4 ± 11.3 ^{bcd}
180 °C-4h	2.0 ± 0.4 ^f	647.3 ± 101.1 ^{ab} ***	7.6 ± 0.2 ^c	33.9 ± 1.4 ^f	3.2 ± 0.7 ^b	54.9 ± 9.3 ^{de}	9.0 ± 1.1 ^{ab}	113.2 ± 11.3 ^{bcd}
180 °C-8h	3.6 ± 0.7 ^{ef}	639.7 ± 108.0 ^{ab} ***	6.8 ± 0.3 ^{de}	40.8 ± 2.3 ^{de}	3.0 ± 0.6 ^{bc}	58.0 ± 8.0 ^{cde}	9.3 ± 1.5 ^{ab}	106.2 ± 15.5 ^{bcd}
180 °C-16h	5.2 ± 1.3 ^e	658.4 ± 90.7 ^{ab} ***	5.8 ± 0.2 ^{gh}	49.5 ± 1.8 ^{ab}	2.3 ± 0.6 ^{bcd}	66.8 ± 8.1 ^{abcd}	11.0 ± 1.2 ^a	124.9 ± 12.6 ^{bc}
200 °C-4h	4.6 ± 1.5 ^e	694.7 ± 117.0 ^{ab} *	6.0 ± 0.2 ^{fgh}	47.9 ± 2.1 ^{abc}	2.5 ± 0.6 ^{bcd}	64.7 ± 8.3 ^{bcd}	10.2 ± 0.9 ^a	93.7 ± 12.0 ^{cdef}
200 °C-8h	10.8 ± 2.2 ^d	650.5 ± 43.0 ^{ab} ***	5.7 ± 0.4 ^{gh}	50.4 ± 3.6 ^{ab}	1.0 ± 0.6 ^d	85.9 ± 8.9 ^{ab}	9.7 ± 0.7 ^{ab}	109.0 ± 14.3 ^{bcd}
200 °C-16h	12.6 ± 1.4 ^d	627.3 ± 108.5 ^{ab} ***	5.6 ± 0.2 ^h	51.0 ± 1.8 ^a	1.5 ± 1.3 ^{cd}	79.0 ± 18.8 ^{abc}	9.8 ± 1.1 ^{ab}	108.4 ± 19.9 ^{bcd}
220 °C-4h	10.5 ± 2.2 ^d	680.3 ± 79.2 ^{ab} ***	6.2 ± 0.3 ^{fgh}	46.1 ± 2.8 ^{bc}	1.2 ± 0.6 ^d	82.2 ± 9.1 ^{ab}	10.0 ± 0.8 ^{ab}	102.0 ± 25.0 ^{bcd}
220 °C-8h	16.6 ± 1.2 ^c	644.6 ± 54.5 ^{ab} ***	6.4 ± 0.3 ^{ef}	44.6 ± 2.8 ^{cd}	1.3 ± 0.4 ^d	81.4 ± 5.7 ^{ab}	9.0 ± 0.8 ^{ab}	73.8 ± 19.9 ^{efg}
220 °C-16h	19.3 ± 0.6 ^b	596.3 ± 72.0 ^b ***	6.0 ± 0.3 ^{fgh}	48.2 ± 2.6 ^{abc}	1.4 ± 0.7 ^{cd}	80.1 ± 10.5 ^{ab}	7.9 ± 1.0 ^b	83.4 ± 11.3 ^{defg}
240 °C-4h	16.2 ± 0.9 ^c	672.2 ± 70.4 ^{ab} ***	6.0 ± 0.3 ^{fgh}	48.1 ± 2.6 ^{abc}	1.1 ± 0.6 ^d	85.0 ± 8.9 ^{ab}	10.2 ± 0.9 ^a	75.0 ± 22.5 ^{efg}
240 °C-8h	21.3 ± 0.7 ^b	671.4 ± 70.1 ^{ab} ***	6.2 ± 0.2 ^{fg}	46.4 ± 1.8 ^{bc}	0.9 ± 0.6 ^d	87.2 ± 8.5 ^a	9.2 ± 0.5 ^{ab}	58.5 ± 6.2 ^{fg}
240 °C-16h	23.8 ± 1.3 ^a	653.5 ± 100.5 ^{ab} ***	6.1 ± 0.3 ^{fgh}	47.4 ± 2.6 ^{abc}	1.4 ± 0.9 ^{cd}	79.9 ± 13.4 ^{abc}	7.9 ± 0.6 ^b	55.0 ± 6.6 ^g

Values for the various properties are mean ± SD (ML, Density, EMC, and MEE, $n = 15$; TS and ASE, $n = 10$; MOE and MOR, $n = 9$). Values within a column with different letters indicate significant differences ($p < 0.05$).

*: $p < 0.05$.

***: $p < 0.001$ compared with untreated samples.

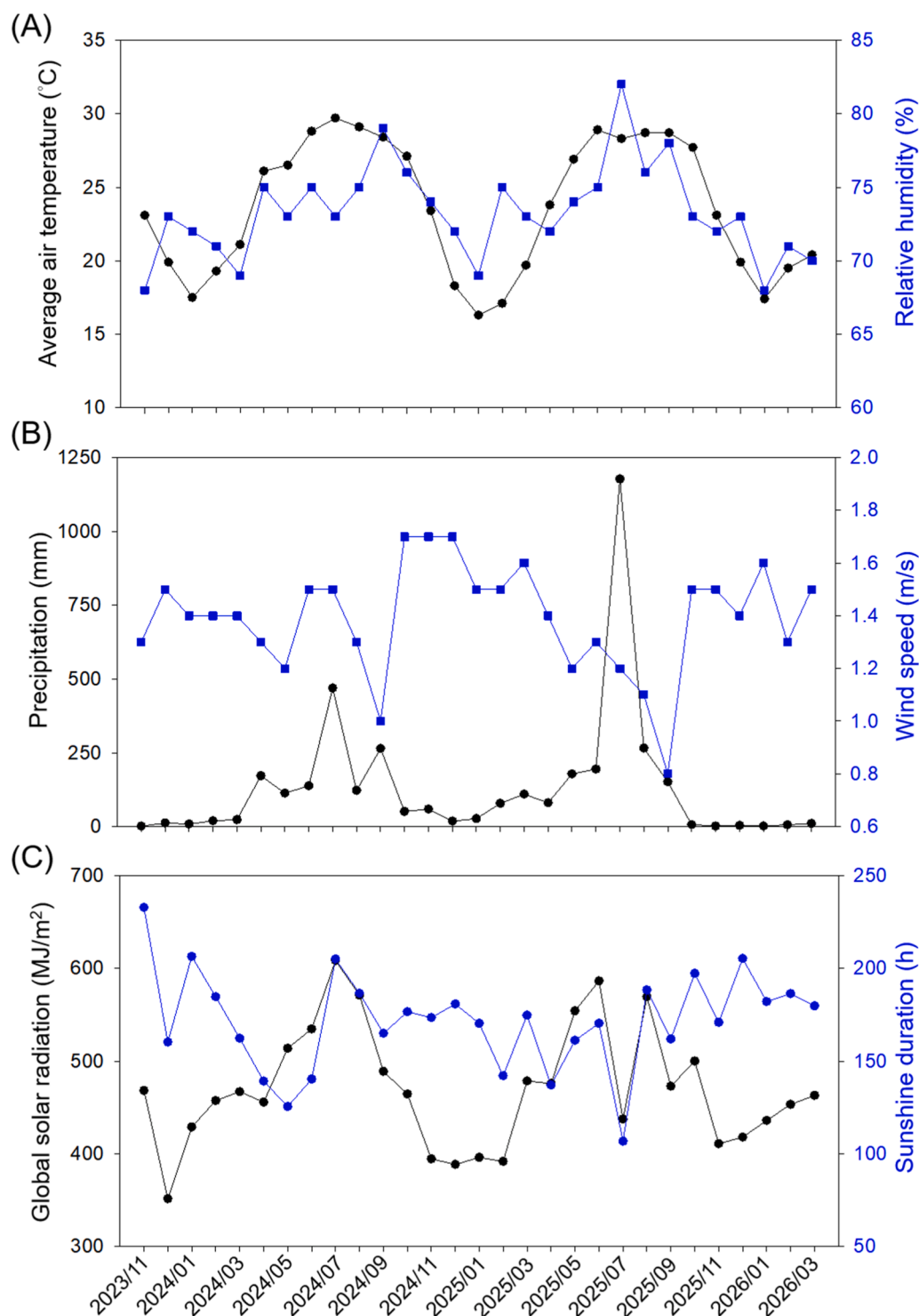


Fig. 2. Monthly climatic conditions at the natural weathering site in Taichung, Taiwan, from November 2023 to March 2026: (A) average air temperature and relative humidity; (B) precipitation and wind speed; and (C) global solar radiation and sunshine duration.

Baseline mechanical properties, EMC, and MEE were therefore determined using the specimens specifically assigned to the accelerated weathering experiment before exposure, whereas the specimens used for TS and ASE evaluation were retained from the initial treatment-screening experiment. The accelerated weathering test was conducted in accordance with Cycle 1 of ASTM G155-13 [21]. The specimens were exposed in a xenon-arc weathering chamber (Q-SUN Xenon test chamber model Xe-3-HDS, Q-LAB, Westlake, OH, USA) at an irradiance of 0.35 W/m² at 340 nm. Each cycle consisted of 102 min of light exposure

followed by 18 min of simultaneous light exposure and water spray. The black panel temperature was maintained at 63 °C, and the total exposure duration was 500 h. Each treatment group consisted of 55 specimens, including 15 specimens assigned for periodic analyses of surface color, microstructure, and surface chemical analyses at 0, 2, 4, 8, 16, 32, 64, 100, 200, 300, 400, and 500 h. In addition, 25 specimens were assigned to destructive mechanical testing at 0, 100, 200, 300, and 500 h, with five specimens tested at each time point. The remaining 15 specimens were used for dimensional stability evaluation at 0, 32, 64, 100, 200,

300, and 500 h.

For periodic measurements, the same specimens were repeatedly evaluated at the scheduled exposure intervals, whereas independent specimens were used for destructive bending tests at each time point. Specimens assigned to dimensional-stability evaluation were used exclusively for water-immersion measurements to avoid interference with mechanical testing and surface chemical analyses.

2.4. Property analysis

2.4.1. Surface images and color measurement

Surface images were recorded using a multifunctional scanner (M1120 MFP, HP, Beijing, China). Color measurements were performed using a UV/Vis spectrophotometer (Lambda 850+, PerkinElmer, Waltham, MA, USA) according to the CIE $L^*a^*b^*$ color system. The color parameters included lightness (L^*), red–green coordinate (a^*), and yellow–blue coordinate (b^*). The total color difference (ΔE^*) was calculated according to the following Eq. (1):

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (1)$$

2.4.2. Surface and cross-sectional structural observations

The surface and cross-sectional morphologies of untreated and VH-treated moso bamboo specimens before and after weathering were examined using scanning electron microscopy (SEM) (TM3000, Hitachi, Tokyo, Japan). For surface observations, the exposed surfaces of unweathered specimens and specimens subjected to natural or xenon-arc accelerated weathering were examined directly. For cross-sectional observations, specimens after natural or xenon-arc accelerated weathering were transversely cut at the midpoint of their longitudinal length, and the freshly exposed cross sections were examined to evaluate tissue deterioration from the weather-exposed surface toward the interior along the thickness direction. Prior to SEM observation, each specimen was mounted on an aluminum stub using carbon adhesive tape, and the images were obtained at an accelerating voltage of 15 kV. For surface observations, five independent specimens were examined for each condition ($n = 5$), with three locations observed on each specimen. For cross-sectional observations, five independent specimens were examined for each condition ($n = 5$). Representative micrographs were selected for presentation.

2.4.3. Physical properties and dimensional stability

For specimens retrieved from natural weathering, visible dust and other loosely attached foreign deposits were gently removed from the specimen surfaces using a soft brush without intentionally abrading the weathered bamboo surface. The cleaned specimens were subsequently oven-dried to constant mass before mass determination and subsequent property measurements. The oven-dry mass of the specimens was determined before and after VH treatment, as well as at different exposure intervals during the weathering tests, and the mass loss (ML) was subsequently calculated. The equilibrium moisture content (EMC) of the specimens was measured at 20 °C and 65 % RH. Moisture excluding efficiency (MEE) was then calculated according to a previous study [22]. To evaluate dimensional stability, the specimens were immersed in distilled water at 20 ± 5 °C for 1 week. The thickness was measured before and after immersion, and the thickness swelling (TS) was calculated. Anti-swelling efficiency (ASE) was further determined according to the method proposed by Prakash and Mahadevan [23].

2.4.4. Bending properties

The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined according to CNS 454 [24]. For the initial characterization of untreated and VH-treated bamboo (Table 1), specimens were conditioned at 20 °C and 65 % RH for 2 weeks until constant mass was reached before mechanical testing. For the natural and accelerated

weathering experiments, specimens collected at each designated exposure interval were oven-dried at 65 °C for 2 days until constant mass was reached before testing. The same oven-drying procedure was also applied to the unweathered specimens at day 0 and 0 h. After conditioning or oven drying, the dimensions of each specimen were measured. A three-point bending test was then performed using a support span equal to 14 times the specimen thickness, corresponding to a span-to-depth ratio of 14:1, at a loading rate of 5 mm/min. During the test, the maximum load and the corresponding load–deformation data were recorded. The MOR and MOE values, as well as their retention ratios during weathering, were calculated using the following Eqs. (2–5):

$$\text{MOR (MPa)} = 3PL/2bh^2 \quad (2)$$

$$\text{MOE (MPa)} = \Delta PL^3/4\Delta Ybh^3 \quad (3)$$

$$\text{MOR retention ratio (\%)} = (\text{MOR}_t / \text{MOR}_0) \times 100 \quad (4)$$

$$\text{MOE retention ratio (\%)} = (\text{MOE}_t / \text{MOE}_0) \times 100 \quad (5)$$

where P is the maximum load (N), L is the span (mm), ΔP is the difference between the upper and lower load limits within the proportional limit (N), ΔY is the bending deformation at the center of the span corresponding to ΔP (mm), b is the specimen width (mm), and h is the specimen thickness (mm). MOR_0 and MOE_0 represent the mean MOR and MOE values of the independent unweathered baseline specimens, respectively, whereas MOR_t and MOE_t represent those measured after weathering.

2.4.5. ATR-FTIR analysis

Relative surface chemical changes were analyzed using a Fourier transform infrared (FTIR) spectrometer (Spectrum 100, PerkinElmer, Beaconsfield, UK) equipped with a deuterated triglycine sulfate (DTGS) detector and an attenuated total reflectance (ATR) accessory (MIRacle, Pike Technologies, Madison, WI, USA). Spectra were collected over the range of 4000–650 cm^{-1} at a resolution of 4 cm^{-1} using 32 scans per sample. Fifteen specimen-level replicates were analyzed for each treatment at every scheduled weathering interval ($n = 15$). Although FTIR spectra were collected at all scheduled exposure times, only selected representative time points were presented in Fig. 10 to facilitate graphical comparison of the temporal spectral changes. For natural weathering, spectra at 0, 90, 180, and 720 days were selected for presentation, whereas for xenon-arc accelerated weathering, spectra at 0, 64, 100, and 500 h were selected. At each selected treatment–time combination, a representative spectrum was chosen from the 15 replicate spectra for graphical presentation. All FTIR spectra were baseline-corrected and normalized using the absorption band at 898 cm^{-1} as an internal reference peak. Because all spectra were normalized to the cellulose-associated band at 898 cm^{-1} , changes in the relative intensities of lignin- and hemicellulose-associated bands were used as semi-quantitative indicators of relative surface chemical changes during VH treatment and subsequent weathering.

To further evaluate changes in the carbonyl region during weathering, the FTIR spectrum of each individual specimen was processed separately. Each spectrum was first normalized by multiplicative scaling such that the absorbance at 898 cm^{-1} was set to 0.5. The exported spectra had a sampling interval of 1 cm^{-1} , and the integrated absorbance areas over 1729–1720 and 1740–1731 cm^{-1} were calculated separately for each specimen using trapezoidal numerical integration. The ratio $A_{1729-1720 \text{ cm}^{-1}}/A_{1740-1731 \text{ cm}^{-1}}$ was then used to characterize relative changes in carbonyl-associated absorption. For the quantitative comparison reported in Table 2 and the subsequent mixed-effects analysis, the carbonyl-area ratios were evaluated at the same representative time points selected for Fig. 10. Thus, the analysis included 0, 90, 180, and 720 days for natural weathering and 0, 64, 100, and 500 h for xenon-arc

Table 2

Changes in ATR-FTIR carbonyl-band area ratios ($A_{1729-1720} \text{ cm}^{-1}/A_{1740-1731} \text{ cm}^{-1}$) of untreated and VH-treated moso bamboo at selected time points during natural outdoor and xenon-arc accelerated weathering.

Weathering regime	Weathering time	Untreated	160 °C-4 h	160 °C-8 h	180 °C-4 h	180 °C-8 h
Natural outdoor weathering	0 d	1.02 ± 0.02 ^{Bb}	1.00 ± 0.03 ^{Cb}	1.00 ± 0.04 ^{Bb}	1.02 ± 0.04 ^{Bab}	1.06 ± 0.05 ^{ABa}
	90 d	1.08 ± 0.03 ^{Aab}	1.04 ± 0.05 ^{Bb}	1.06 ± 0.02 ^{Aab}	1.08 ± 0.06 ^{Aa}	1.07 ± 0.02 ^{Aab}
	180 d	1.10 ± 0.10 ^{Aa}	1.05 ± 0.04 ^{ABa}	1.06 ± 0.04 ^{Aa}	1.04 ± 0.04 ^{ABa}	1.04 ± 0.04 ^{ABa}
	720 d	1.10 ± 0.04 ^{Aa}	1.09 ± 0.03 ^{Aa}	1.07 ± 0.02 ^{Aa}	1.09 ± 0.02 ^{Aa}	1.03 ± 0.04 ^{Bb}
Xenon-arc accelerated weathering	0 h	1.04 ± 0.02 ^{BCab}	1.04 ± 0.04 ^{BCab}	1.06 ± 0.03 ^{Aa}	1.03 ± 0.02 ^{Bb}	1.06 ± 0.02 ^{Aa}
	64 h	1.07 ± 0.04 ^{ABa}	1.06 ± 0.04 ^{Aa}	1.07 ± 0.02 ^{Aa}	1.06 ± 0.03 ^{Aa}	1.07 ± 0.04 ^{Aa}
	100 h	1.07 ± 0.02 ^{Aa}	1.06 ± 0.04 ^{ABa}	1.05 ± 0.02 ^{ABa}	1.03 ± 0.02 ^{Bb}	1.06 ± 0.02 ^{Aa}
	500 h	1.03 ± 0.02 ^{Ca}	1.03 ± 0.02 ^{Ca}	0.99 ± 0.02 ^{Bb}	0.99 ± 0.02 ^{Cb}	1.03 ± 0.01 ^{Ba}

Values are presented as mean ± SD ($n = 15$). Ratios were calculated separately for each specimen. Within each weathering regime, different superscript lowercase letters indicate significant differences among treatments within the same weathering time, whereas different superscript uppercase letters indicate significant differences among weathering times within the same treatment. Means sharing at least one letter are not significantly different. Statistical comparisons were based on estimated marginal means from linear mixed-effects models with Holm adjustment for multiple comparisons ($p < 0.05$). The 1730 cm^{-1} boundary was excluded from both integration regions to minimize spectral overlap.

accelerated weathering. The two integrated areas used to calculate the ratio were obtained from the same multiplicatively scaled spectrum; therefore, the common normalization factor cancels mathematically in the area ratio. The normalization at 898 cm^{-1} was retained for spectral display and comparison of relative band profiles, whereas the calculation of the carbonyl-area ratio itself does not require the assumption that the 898 cm^{-1} band remains absolutely invariant after weathering. Because ATR-FTIR provides semi-quantitative information primarily on surface functional groups, changes in this ratio and in the characteristic absorption bands were interpreted as indicators of relative changes in cell-wall polymer-associated structures rather than as measures of the absolute concentrations of specific degradation products or as direct quantitative evidence of specific degradation pathways.

2.5. Statistical analysis

Statistical analyses were performed using SPSS software (IBM SPSS Statistics, IBM Corp., Armonk, NY, USA) for comparisons involving independent specimen groups. One-way analysis of variance (ANOVA) was used to evaluate differences among VH treatment groups in the initial treatment-characterization experiment and for destructive measurements in which independent specimens were tested at each sampling time, followed by Scheffé's or Games–Howell post hoc tests where appropriate. Comparisons between two independent groups were conducted using Student's *t*-test. Differences were considered statistically significant at $p < 0.05$. For non-destructive weathering measurements, including surface color, mass loss, EMC, MEE, TS, and ASE, the same specimens were repeatedly evaluated over the scheduled exposure intervals. These data were used primarily to describe the temporal evolution of physical and surface properties during natural and xenon-arc accelerated weathering and were therefore presented descriptively without inferential comparisons among repeated observations. Accordingly, the one-way ANOVA described above was not applied to these longitudinal repeated-measurement data.

For the FTIR carbonyl-area ratio, inferential comparisons were performed using the selected exposure times reported in Table 2 and shown in Fig. 10. Specifically, the analysis included 0, 90, 180, and 720 days for natural weathering and 0, 64, 100, and 500 h for xenon-arc accelerated weathering. At each selected treatment–time combination, the area ratios calculated separately from the 15 specimen-level replicate spectra were analyzed using linear mixed-effects models to account for repeated measurements of the same specimens over time. Treatment, weathering time, and their interaction were included as fixed effects, while specimen was included as a random intercept. Because residual variability differed among weathering times, heterogeneous residual variances among weathering times were allowed in the model. Post-hoc comparisons of estimated marginal means were performed with Holm adjustment for multiple comparisons. Natural and xenon-arc accelerated

weathering datasets were analyzed separately.

3. Results and discussion

3.1. Property changes of moso bamboo under VH treatment

The results showed that mass loss (ML) increased significantly with increasing treatment temperature and duration. When the treatment conditions were below 200 °C-8 h, the ML values of VH-treated specimens remained below 10 %. Beyond this threshold, ML increased markedly. Because ML was calculated from oven-dry mass, the initial mass loss is more appropriately attributed to the release of extractives, volatile compounds, and low-molecular-weight degradation products rather than to the evaporation of bound water [25,26]. As treatment temperature and duration increased, hemicellulose degradation became more pronounced, and deacetylation reactions generated acetic acid, which could further catalyze the degradation of polysaccharides and lignin [11,27]. When the treatment severity reached 220 °C-8 h, ML rose sharply to 16.6 %, suggesting more extensive thermal alteration of lignocellulosic components under high-temperature and prolonged treatment conditions, which likely contributed to the substantial increase in ML.

As shown in Table 1, the untreated moso bamboo exhibited an air-dry density of 771.0 kg/m^3 . For the VH-treated specimens, the air-dry density ranged from 596.3 to 719.5 kg/m^3 , with the lowest value observed after the 220 °C-16 h treatment. Although changes in density were less pronounced than those in ML, the overall decrease in density with increasing treatment severity was consistent with the progressive removal of extractives and thermal alteration of lignocellulosic components. The variation in density among treatment groups may also partly reflect the inherent anatomical heterogeneity of bamboo strips within the commercial material batch, in addition to treatment-induced mass loss. Therefore, density changes should be interpreted together with ML and mechanical properties, rather than as an independent indicator of treatment effectiveness.

The effects of different VH treatment conditions on the dimensional stability of bamboo were further evaluated on the basis of EMC, MEE, TS, and ASE (Table 1). With respect to EMC and MEE, the untreated bamboo exhibited an EMC of 11.5 %. Following VH treatment, EMC decreased with increasing temperature and duration when the treatment temperature was below 220 °C. Above this point, however, the trend reversed and EMC began to increase. Among all treatment groups, the 200 °C-16 h condition resulted in the lowest EMC value (5.6 %) and the highest MEE (51 %). Notably, even the relatively mild 160 °C-4 h treatment produced a significant reduction in EMC (8.4 %) compared with the untreated specimens, with a corresponding MEE of 27 %, indicating that mild VH treatment was sufficient to effectively reduce the hygroscopicity of moso bamboo.

Regarding TS and ASE, the untreated bamboo exhibited a TS value of 7.0 %. After VH treatment, the TS values of all treated specimens decreased with increasing treatment severity. At 160 °C-4 h, the TS value was already reduced to 3.9 %, which differed significantly from that of the untreated group, indicating that a relatively mild VH treatment was sufficient to improve the dimensional stability of moso bamboo. Furthermore, under treatment conditions more severe than 200 °C-8 h, no further significant reduction in TS was observed, with values ranging from 0.9 to 1.5 %. Similarly, ASE increased with increasing treatment temperature and duration, except for the 240 °C-16 h treatment. The highest ASE value (87.2 %) was observed at 240 °C-8 h. However, at treatment severities of 200 °C-8 h and above, no significant differences in ASE were detected among the treated groups, and all ASE values exceeded 79.0 %. These findings indicate that a VH treatment of 200 °C for 8 h was sufficient to achieve substantial improvement in the dimensional stability of moso bamboo.

Overall, EMC and TS decreased significantly with increasing treatment temperature and duration, whereas MEE and ASE showed corresponding upward trends. These changes can be attributed to chemical

transformations occurring during VH treatment. The degradation of polysaccharides generates more hydrophobic compounds such as furfural, thereby reducing hygroscopicity [11,27]. In addition, the dehydration of hemicelluloses decreases the number of accessible hydroxyl groups, while lignin cross-linking reactions further reduce hydroxyl functionality, collectively resulting in reduced hydrophilicity [11,19,27].

Because acetic acid generated during hemicellulose thermal degradation may negatively affect bending strength [11,28], the mechanical properties of the specimens were also evaluated (Table 1). The untreated bamboo exhibited an MOE of 11.1 GPa. No significant reductions in MOE were observed for most VH-treated groups, with values ranging from 9.0 to 11.0 GPa, except for the 220 °C-16 h and 240 °C-16 h groups, both of which exhibited an MOE of 7.9 GPa. The stability of MOE under mild treatment conditions may reflect a balance between competing effects. On the one hand, partial hemicellulose degradation and deacetylation can reduce stress transfer between cell-wall components. On the other hand, reduced moisture accessibility, lignin condensation or rearrangement, and an increased relative contribution of cellulose-rich

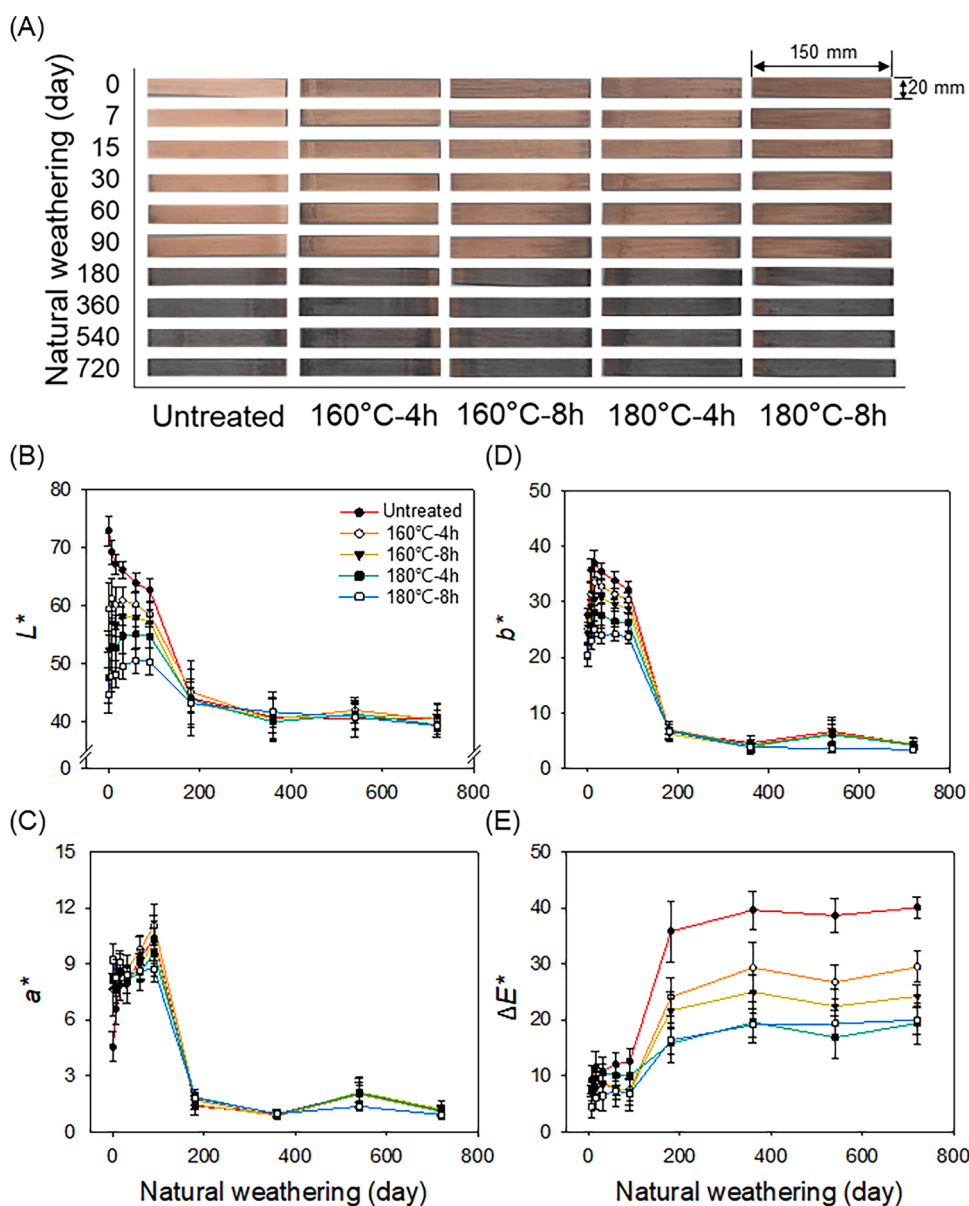


Fig. 3. Surface images (A) and L^* (B), a^* (C), b^* (D), and ΔE^* (E) values of untreated and VH-treated moso bamboo during the natural weathering test. Values are mean $\pm$ SD ($n = 15$).

structures may partly maintain stiffness under mild VH conditions [29]. As a result, no significant reductions in MOE were observed for most mildly treated specimens. However, when the treatment severity increased to 220 °C for 16 h or 240 °C for 16 h, extensive hemicellulose degradation and acid-catalyzed depolymerization became more pronounced, resulting in lower MOE values. Accordingly, mild to moderate VH treatment conditions improved dimensional stability without substantially compromising mechanical performance, whereas excessive treatment was associated with greater mass loss and deterioration of mechanical properties. Therefore, the 160 °C and 180 °C treatments

with durations of 4 and 8 h were selected for subsequent weathering tests to further evaluate long-term durability and practical applicability.

3.2. Weathering performance of VH-treated moso bamboo

3.2.1. Color changes

VH-treated bamboo exhibited progressively darker coloration with increasing treatment severity (Fig. 3A and 4A), which can be attributed to the formation of chromophoric structures associated with unsaturated bonds and aromatic compounds [30]. During the 720-day natural

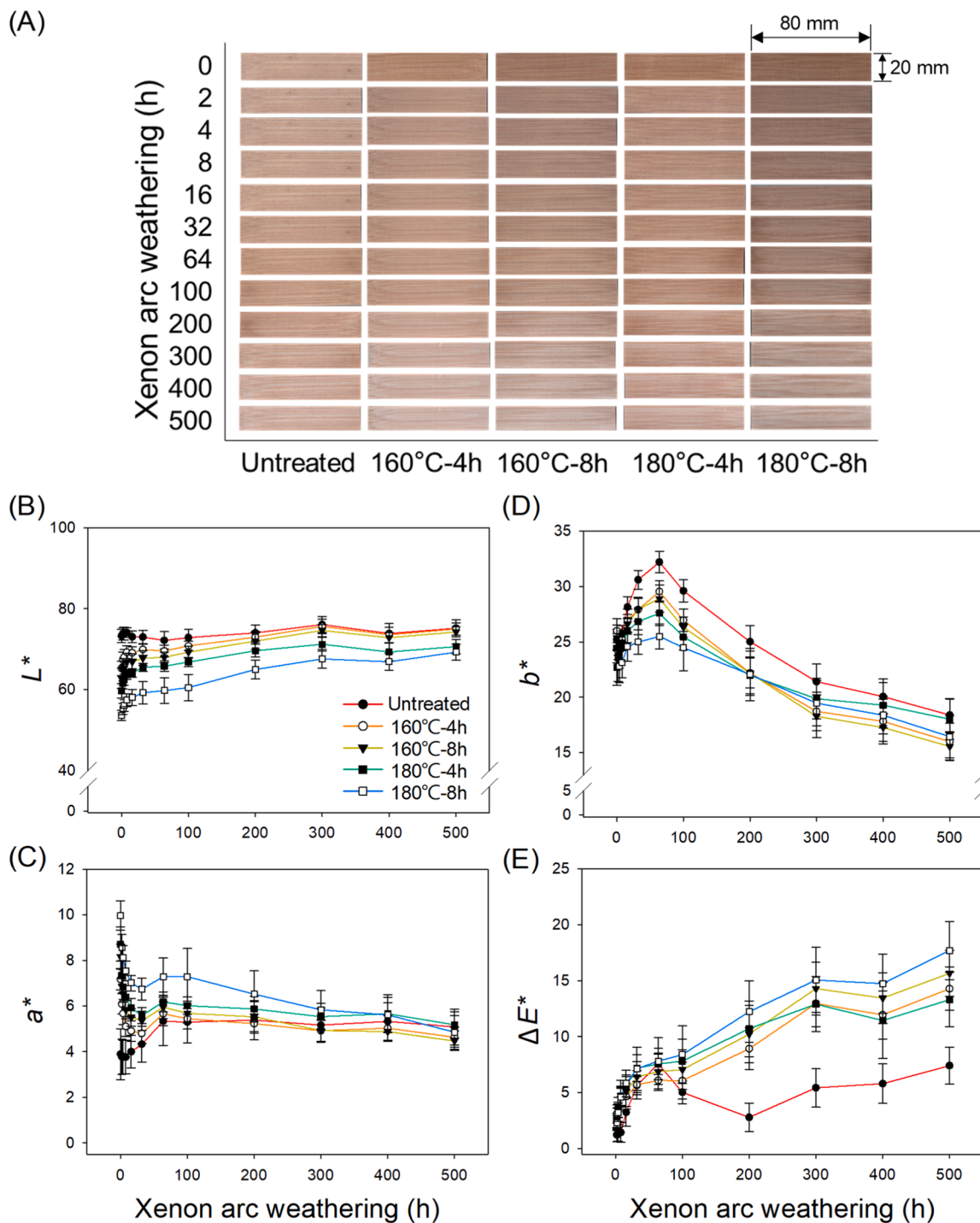


Fig. 4. Surface images (A) and L^* (B), a^* (C), b^* (D), and ΔE^* (E) values of untreated and VH-treated moso bamboo during the xenon-arc accelerated weathering test. Values are mean $\pm$ SD ($n = 15$).

weathering test, the surface color of all specimens gradually darkened with increasing exposure time (Fig. 3A). Color measurements (Fig. 3B–E) showed that, after 720 days of exposure, the untreated specimens exhibited decreases in L^* from 72.8 to 40.5, in a^* from 4.5 to 1.1, and in b^* from 27.6 to 4.4. For the VH-treated groups, L^* decreased from 44.7–59.4 to 39.3–40.9, a^* from 7.7–9.2 to 0.9–1.2, and b^* from 20.2–25.2 to 3.4–4.5. Overall, all groups showed a reduction in lightness accompanied by a shift in hue toward the blue–green direction, and their ΔE^* values increased with prolonged exposure time. The untreated specimens exhibited the largest total color change ($\Delta E^* = 40.0$), whereas the 180 °C–4 h treatment exhibited the smallest change ($\Delta E^* = 19.4$).

In the accelerated weathering test, the surfaces of all specimens became noticeably grayish-white after 500 h of exposure (Fig. 4A). Color measurements (Fig. 4B–E) indicated that, for the untreated specimens, L^* increased from 73.3 to 75.2, a^* increased from 3.9 to 5.1, and b^* decreased from 25.2 to 18.4. For the VH-treated groups, L^* increased from 53.6–65.2 to 69.2–75.0, whereas a^* decreased from 7.2–10.0 to 4.5–5.2, and b^* decreased from 22.7–26.0 to 15.6–18.0. All groups exhibited an increase in lightness and a shift toward the blue–green direction, and their ΔE^* values generally increased with exposure time and tended to increase with greater VH treatment severity. Among the groups, the 180 °C–8 h treatment exhibited the largest color change ($\Delta E^* = 17.7$), whereas the untreated group showed the smallest change ($\Delta E^* = 7.4$).

The observed color changes during weathering are consistent with photo-oxidative changes in lignin-associated structures, as suggested by the changes in the lignin-associated FTIR bands discussed later. Lignin strongly absorbs radiation in the range of 280–400 nm; upon absorbing light energy, free radicals are generated, which subsequently react with

oxygen to form chromophoric groups containing carbonyl and carboxyl functionalities, resulting in surface darkening [31]. In addition, leaching during weathering may gradually remove leachable degradation products [32–34], thereby exposing the whitish cellulose-rich surface and ultimately causing lightening. The accelerated weathering results obtained in this study (Fig. 4) were consistent with this trend and agreed with the observations reported by Kim et al. [35] for moso bamboo exposed outdoors for one year. In contrast, under natural outdoor weathering, the combined influence of both abiotic and biotic environmental factors led to an overall darker surface appearance.

3.2.2. Surface structural changes

The surface morphologies of untreated and VH-treated moso bamboo after weathering are shown in Fig. 5. In both natural and accelerated weathering tests, distinct surface cracks and checks developed in all specimens (Fig. 5B–F and 5H–L). These morphological changes are consistent with the progressive deterioration commonly associated with photochemical degradation of lignocellulosic materials. In particular, the middle lamellae, which are relatively rich in lignin and contribute to intercellular adhesion, are susceptible to photodegradation. Previous long-term weathering observations of bamboo-based materials have also shown micro-check development in the compound middle lamellae and cell-corner regions, where the degree of lignification is relatively high [34]. Deterioration of these lignin-rich intercellular regions may weaken bonding between adjacent cells, thereby facilitating cell separation and the initiation and propagation of cracks [34,36]. The changes in lignin-associated chemical structures observed by ATR-FTIR, as discussed in Section 3.2.6, are consistent with this interpretation.

Cross-sectional SEM observations were further performed to qualitatively examine morphological changes from the weather-exposed

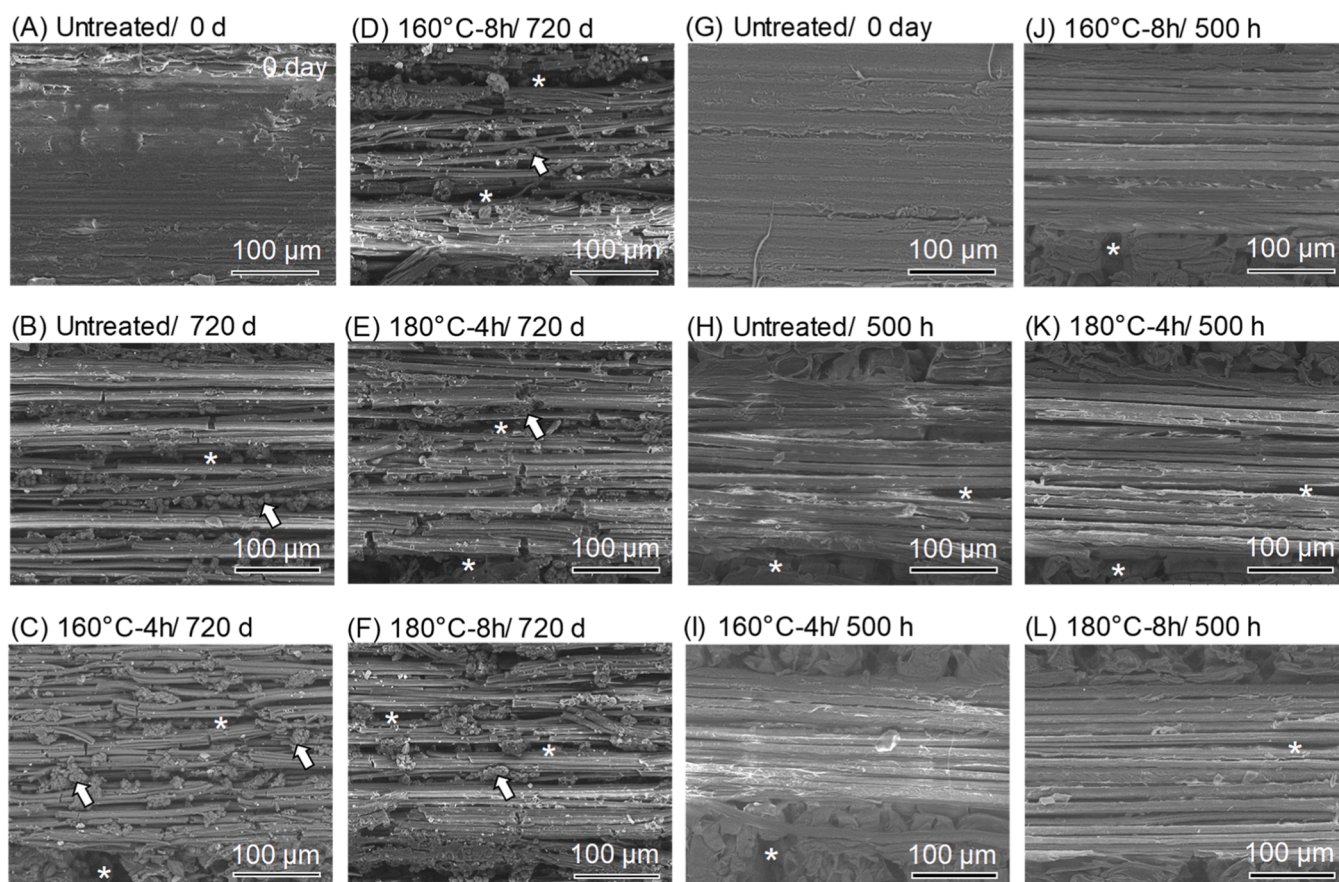


Fig. 5. SEM images of untreated and VH-treated moso bamboo surfaces before weathering (A, G) and after the natural weathering test (B–F) or the xenon-arc accelerated weathering test (H–L). Asterisks (*) indicate cracks, and arrows indicate spore-like surface deposits observed after natural weathering.

surface toward the interior along the thickness direction (Fig. 6). The unweathered untreated specimens exhibited relatively intact cellular structures with well-defined cell-wall boundaries (Fig. 6A and 6G). After 720 days of natural weathering, varying degrees of cell-wall deformation, local disruption, and irregular cellular structures were observed in the cross-sectional regions beneath the exposed surfaces of both untreated and VH-treated specimens (Fig. 6B–F). Similar morphological alterations were also evident after 500 h of accelerated weathering (Fig. 6H–L), including deformation and disruption of cellular structures adjacent to the exposed surface. These morphological features are consistent with progressive weakening and disruption of intercellular and cell-wall structures during weathering and may be associated with the development and propagation of microcracks beneath the exposed surface. These cross-sectional observations provide qualitative morphological evidence that weathering-associated structural alterations were detectable from the weather-exposed surface toward the interior. However, because SEM provides morphological rather than depth-resolved chemical information, these observations cannot determine the penetration depth or chemical nature of the weathering-induced alterations. Accordingly, the cross-sectional SEM observations should be interpreted as qualitative morphological evidence and do not establish a direct causal relationship between surface deterioration and the observed changes in bulk mechanical properties.

In the natural weathering test, which was conducted under outdoor conditions, visible deposition of dust and possible biological deposits was observed on the bamboo surface, together with more severe surface deterioration (Fig. 5B–F). Hydrothermal treatment has previously been reported to improve the mildew resistance of moso bamboo [19].

Although biological resistance was not independently evaluated in the present study, this finding provides relevant context for interpreting the possible biological deposition observed during natural weathering. In contrast, specimens subjected to accelerated weathering showed no apparent biological deposition and exhibited less severe surface degradation (Fig. 5H–L). These observations highlight the different contributions of biotic and abiotic factors under natural and accelerated weathering conditions. These findings also indicate that natural and accelerated weathering should be interpreted as complementary rather than interchangeable ageing environments. Natural exposure involved the combined effects of photochemical oxidation, moisture cycling, leaching, dust deposition, and possible biological activity, whereas xenon-arc exposure mainly emphasized abiotic photo-oxidative degradation coupled with repeated water spray.

3.2.3. Mass loss changes

The ML values of untreated and VH-treated bamboo during weathering are shown in Fig. 7. In the 720-day natural weathering test (Fig. 7A), the untreated specimens exhibited the highest ML (14.7 %), whereas the treated groups ranged from 11.4 % to 12.2 %. In the 500-h accelerated weathering test (Fig. 7B), the 180 °C-8 h group exhibited the highest ML (8.9 %), whereas the other treatment groups ranged from 4.6 % to 6.1 %. The variation in ML among groups was associated with the different deterioration processes occurring during weathering. Surface cracks and checks formed during exposure may have facilitated light penetration and moisture ingress into the bamboo interior, thereby accelerating chemical deterioration and the leaching of degradation products. In the accelerated weathering test, the absence of visible

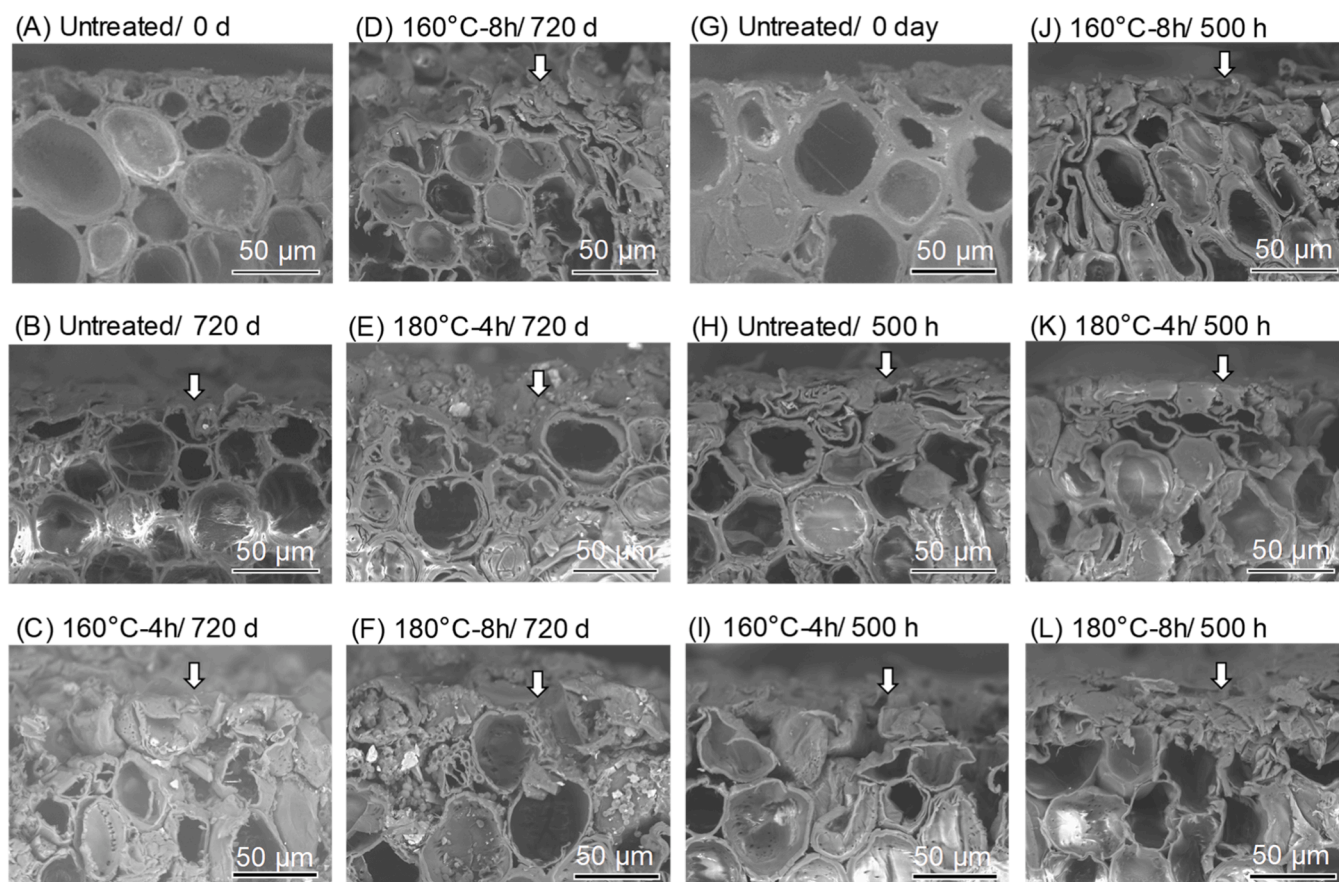


Fig. 6. Cross-sectional SEM images showing the thickness-direction morphology of untreated and VH-treated moso bamboo specimens before weathering (A, G) and after natural weathering (B–F) or xenon-arc accelerated weathering (H–L). The specimens were transversely cut at the midpoint of their longitudinal length, and the freshly exposed cross sections were observed from the weather-exposed surface toward the interior along the thickness direction. Arrows indicate the weather-exposed surface, which is positioned at the top of each image.

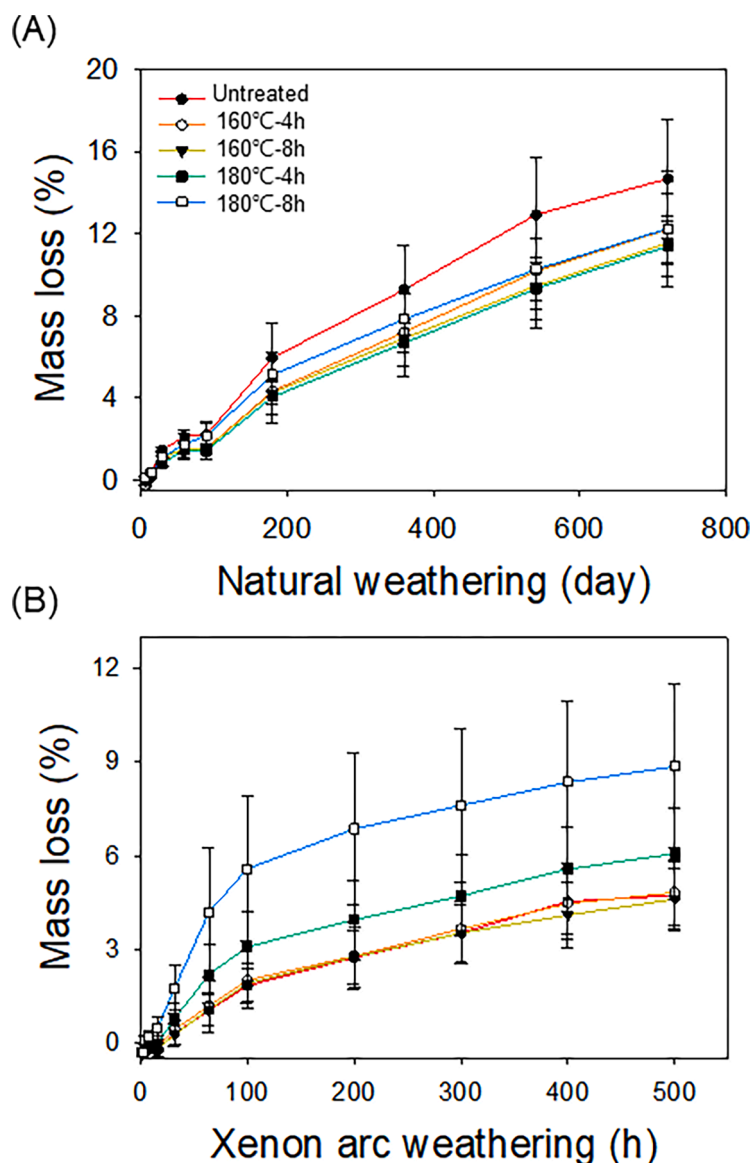


Fig. 7. Mass loss of untreated and VH-treated moso bamboo during the natural weathering test (A) and the xenon-arc accelerated weathering test (B). Values are mean $\pm$ SD ($n = 15$).

biological deposition enabled a more direct evaluation of physico-chemical degradation caused by UV irradiation and water spray. The results suggest that higher-severity VH treatment, particularly 180 °C-8 h, may slightly reduce resistance to accelerated weathering, possibly because the greater treatment-induced alteration associated with the more severe VH condition rendered the material more susceptible to mass loss under intense UV irradiation and water spray. In contrast, under outdoor exposure (Fig. 7A), possible biotic factors, together with rainfall, dust deposition, and moisture cycling, may also have contributed to surface deterioration. Hydrothermal treatment has previously been reported to improve the mildew resistance of moso bamboo [19], which may be relevant to the comparatively lower mass loss observed in the VH-treated specimens during natural weathering. However, because biological resistance was not independently evaluated in the present study, the lower mass loss cannot be directly attributed to an anti-mildew effect. Overall, the observed mass-loss patterns varied with VH treatment severity and exposure conditions, with relatively mild VH conditions generally showing lower mass loss than the more severe treatment under accelerated weathering.

3.2.4. Dimensional stability changes

The effects of VH treatment on the dimensional stability of moso bamboo during weathering were further evaluated through changes in TS, ASE, EMC, and MEE (Fig. 8). With respect to TS and ASE, the natural weathering experiment used a separately VH-treated specimen set, as described in Section 2.3. In the natural weathering test (Fig. 8A–B), the TS value of the untreated specimens decreased from 1.3 % at day 0 to 0.6 % at day 720. The VH-treated groups also generally showed a decreasing trend and reached 0.62–0.73% at day 720. Correspondingly, ASE decreased markedly over the exposure period, from initial values ranging from –13.3 % to 18.1 % to final values ranging from –25.4 % to –6.7 % at day 720. It should be noted that TS reflects the macroscopic dimensional response of the specimen in the thickness direction and does not directly represent water uptake or water accessibility. Therefore, the formation of cracks during weathering may facilitate water penetration without necessarily resulting in greater thickness swelling. Previous studies have reported that natural weathering of bamboo can result in the development of surface checks and erosion extending into the near-surface region, accompanied by preferential alterations of lignin and hemicelluloses [35]. Consistent with these previous

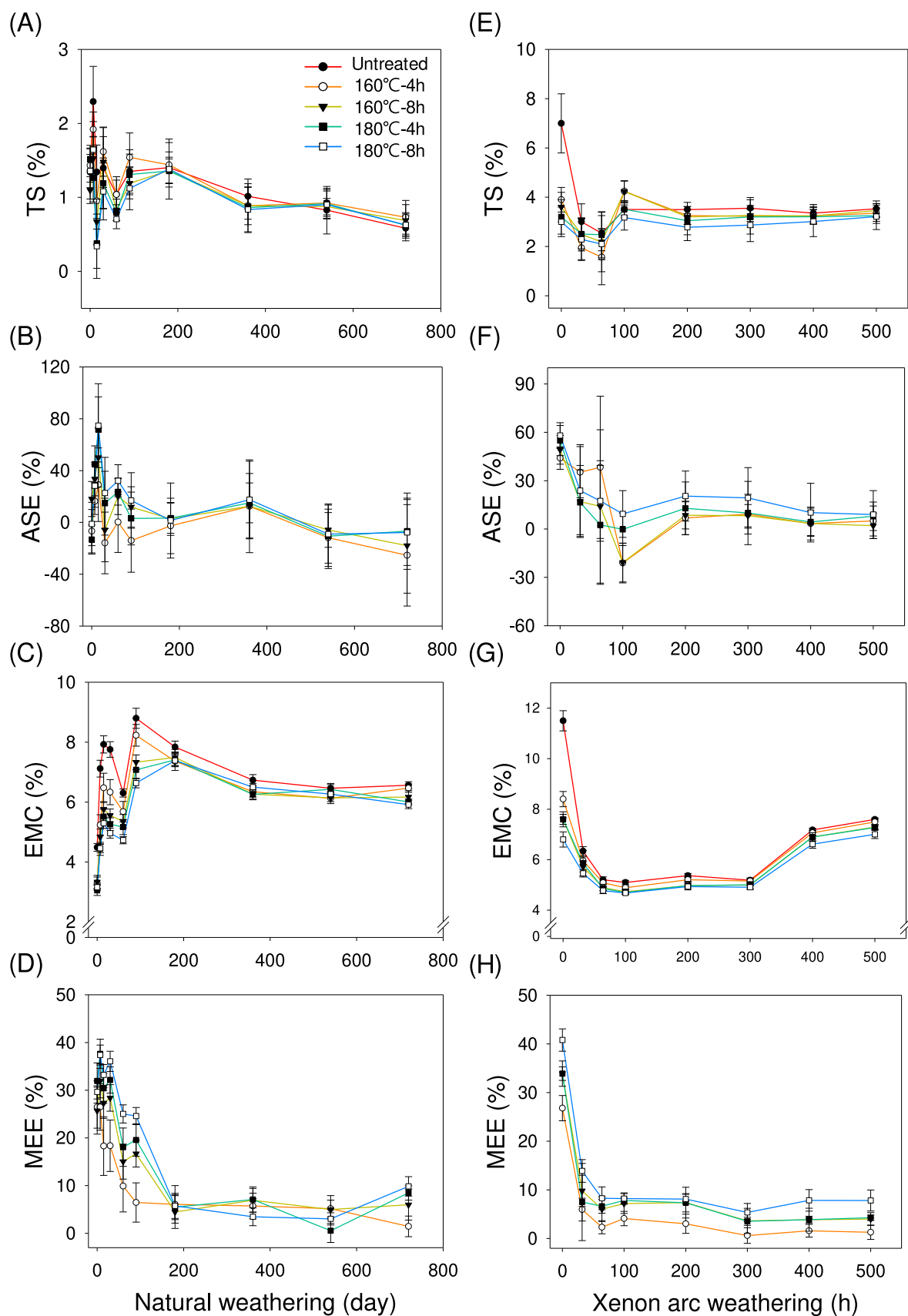


Fig. 8. Thickness swelling (TS), anti-swelling efficiency (ASE), equilibrium moisture content (EMC), and moisture excluding efficiency (MEE) of untreated and VH-treated moso bamboo during the natural weathering test (A–D) and the xenon-arc accelerated weathering test (E–H). Values are mean $\pm$ SD ($n = 11$ for TS, ASE, EMC, and MEE in the natural weathering test; $n = 10$ for TS and ASE, and $n = 5$ for EMC and MEE in the xenon-arc accelerated weathering test).

observations, the present surface and cross-sectional SEM images showed progressive cracking, cell-wall deformation, local disruption, and irregular cellular structures in the weathered specimens (Figs. 5 and 6). Such weathering-associated structural deterioration may reduce the continuity of the cellular network and the amount of intact tissue contributing to coordinated thickness-direction swelling. In addition, water entering newly formed cracks, cell lumina, or other void spaces may increase water accessibility without producing a proportional increase in macroscopic thickness swelling. These effects may partly explain the decrease in TS observed after prolonged weathering. In addition, leaching and the partial loss or alteration of hygroscopic cell-wall components during weathering may reduce the amount of swellable material, while structural collapse or changes in specimen geometry may further limit coordinated thickness-direction expansion. Accordingly, the reduced TS should not be interpreted as improved resistance to moisture ingress, but rather as an altered dimensional response of the weathered bamboo tissue. The negative ASE values at the later stage of natural weathering therefore indicate that the initial

dimensional-stabilizing advantage of VH treatment was no longer maintained relative to the weathered untreated specimens, rather than directly indicating greater or lower water accessibility. In the accelerated weathering test (Fig. 8E–F), the TS value of the untreated specimens decreased from 7.0 % at 0 h to 3.5 % at 500 h, whereas those of the VH-treated groups decreased from 3.0–3.9 % to 3.2–3.5 %. Correspondingly, ASE sharply declined from 44.0–58.0 % to only 2.0–8.9 %, indicating a gradual loss of dimensional stability under intense UV irradiation and repeated water spray.

Regarding EMC and MEE, natural weathering (Fig. 8C–D) led to an increase in EMC in both untreated and VH-treated groups, from 4.5 % to 6.6 % and from 3.1–3.3 % to 5.9–6.5 %, respectively. These values showed a convergence in hygroscopicity among the groups during long-term outdoor exposure. Consequently, the MEE values of the treated specimens decreased from 25.7–32.0 % to 1.5–9.8 % by day 720, consistent with a gradual reduction in the initial hygroscopicity-related advantage imparted by VH treatment. Similarly, in the accelerated weathering test (Fig. 8G–H), EMC decreased from 11.5 % to 7.6 % in the

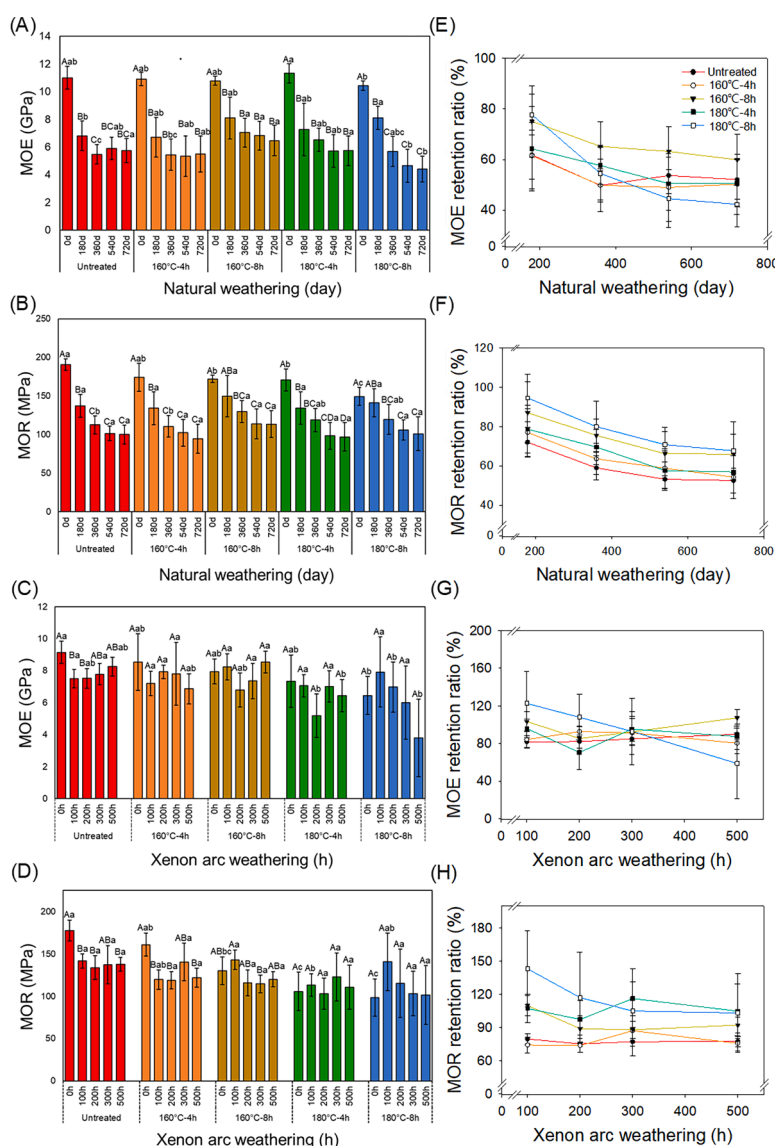


Fig. 9. Modulus of elasticity (MOE) (A, C), modulus of rupture (MOR) (B, D), MOE retention ratio (E, G), and MOR retention ratio (F, H) of untreated and VH-treated moso bamboo during the natural weathering test (A, B, E, F) and the xenon-arc accelerated weathering test (C, D, G, H). Values are mean $\pm$ SD ($n = 11$ for the natural weathering test and $n = 5$ for the xenon-arc accelerated weathering test). Bars with different uppercase letters indicate significant differences among weathering durations within the same treatment group ($p < 0.05$), whereas bars with different lowercase letters indicate significant differences among treatment groups at the same weathering duration ($p < 0.05$).

untreated group, while the VH-treated groups converged to 7.0–7.5 %. The corresponding decline in MEE from 26.8–40.8 % to 1.3–7.8 % was likewise consistent with a gradual reduction in the moisture-resistance advantage associated with VH treatment.

Overall, the present results, together with previous studies [8–10], support that thermal modification can effectively improve the dimensional stability of lignocellulosic materials. Although the dimensional-stability advantage of VH-treated bamboo gradually diminished with prolonged weathering, the observed EMC, MEE, TS, and ASE trends suggest that the initial dimensional-stabilizing effects of mild to moderate VH treatment were progressively reduced during weathering.

3.2.5. Changes in mechanical properties

Mechanical property evaluations at different exposure intervals provided further insight into the effects of weathering on the structural performance of bamboo (Fig. 9). Because the mechanical specimens used in the weathering experiments and those used for the initial treatment characterization in Table 1 differed in moisture conditioning before mechanical testing, the initial MOE and MOR values obtained from the weathering datasets should not be directly equated with those reported in Table 1. Before natural weathering, no significant differences in MOE were observed between the untreated specimens and those subjected to VH treatment, with values ranging from 10.4 to 11.3 GPa (Fig. 9A). After 720 days of outdoor exposure, the MOE of all groups decreased markedly. The untreated specimens declined to 5.7 GPa (52.1 % retention), whereas the VH-treated groups decreased to 4.4–6.5 GPa (42.3–59.9 % retention) (Fig. 9A and 9E). A similar tendency was observed for MOR. Before natural weathering, the untreated bamboo exhibited an MOR of 190.6 MPa, whereas the VH-treated groups ranged from 149.3 to 174.1 MPa (Fig. 9B). After 720 days of natural weathering, the MOR of the untreated group decreased to 100.2 MPa (52.6 % retention), whereas the VH-treated groups decreased to 94.5–113.5 MPa (54.3–67.7 % retention) (Fig. 9B and 9F). Notably, the group treated under the most severe selected condition, 180 °C for 8 h, exhibited the lowest MOE value (4.4 GPa) and the lowest MOE retention ratio (42.3 %) after 720 days of natural weathering. This finding suggests a possible over-modification effect under relatively severe VH treatment conditions.

In the accelerated weathering test, the MOE of untreated specimens remained at 8.3 GPa after 500 h, corresponding to a retention ratio of 90.2 % (Fig. 9C and 9G). In contrast, the VH-treated groups showed greater variability, with MOE values decreasing to 3.8–6.9 GPa and corresponding retention ratios of 58.9–107.4 %. Similarly, the MOR of the untreated group decreased to 137.8 MPa, corresponding to a retention ratio of 77.6 %, whereas those of the VH-treated groups ranged from 101.4 to 121.9 MPa, with retention ratios of 75.8–104.9 % (Fig. 9D and 9H). The retention ratios exceeding 100 % in some VH-treated groups should be interpreted cautiously and may reflect specimen variability and heterogeneous bamboo anatomy rather than true mechanical reinforcement. These results indicate that the effects of VH treatment on mechanical performance during accelerated weathering were more variable than those observed under natural weathering.

Overall, the decline in mechanical properties during weathering occurred concurrently with progressive surface cracking, morphological alterations detectable beneath the exposed surface (Figs. 5 and 6), and changes in lignin-associated FTIR bands. These observations provide complementary information on the physical, morphological, and surface chemical changes occurring during weathering. However, ATR-FTIR primarily provides semi-quantitative information on surface chemical characteristics, whereas MOE and MOR represent bulk mechanical responses. Similarly, the cross-sectional SEM observations provide qualitative morphological information but do not quantitatively resolve the depth or chemical nature of the observed alterations. Therefore, the concurrent changes observed by ATR-FTIR and SEM and in mechanical properties should not be interpreted as evidence of a direct causal

relationship between surface chemical or morphological alterations and bulk mechanical deterioration. Depth-resolved chemical and structural analyses would be required to determine the penetration depth of weathering-induced alterations and to clarify their potential relationship with changes in bulk mechanical properties. Nevertheless, among the selected VH conditions, the 160 °C for 8 h treatment showed comparatively favorable initial mechanical performance and mechanical retention after weathering. This observation suggests that mild to moderate VH treatment may provide a more suitable compromise between initial property modification and long-term mechanical stability than more severe treatment conditions.

3.2.6. FTIR spectral changes

The FTIR spectra of moso bamboo samples are shown in Fig. 10. The main characteristic absorption bands were observed in the regions of 3800–2750 cm^{-1} and 1800–800 cm^{-1} . The band at 3340 cm^{-1} was assigned to hydroxyl groups (–OH), while that at 2910 cm^{-1} corresponded to the asymmetric stretching vibration of methylene groups (C–H). The absorption band within the 1740–1731 cm^{-1} range was attributed mainly to the stretching vibration of carbonyl groups (C=O) in the native acetyl ester groups of hemicelluloses. A shoulder or broadened band around 1729–1720 cm^{-1} was associated with lower-wavenumber carbonyl-containing structures, including newly formed or weathering-induced aldehydes and ketones. Furthermore, absorption in the 1720–1700 cm^{-1} region was related to C=O stretching of accumulated free organic acid photoproducts or degradation intermediates [20,31,34,35].

Thermal degradation of hemicelluloses is generally initiated by deacetylation, during which released acetic acid may further catalyze polysaccharide depolymerization and promote the formation of low-molecular-weight degradation products, such as furfural, 5-hydroxymethylfurfural, organic acids, and other volatile compounds [6,11]. These chemical changes were consistent with the evolution of the carbonyl region in the FTIR spectra. Specifically, the relative intensity at approximately 1735 cm^{-1} gradually decreased, indicating cleavage or reduction of native acetyl ester groups, whereas slight broadening in the 1729–1720 cm^{-1} region was observed in VH-treated samples (Fig. 10A and 10E). To further describe these structural transformations and track changes in the carbonyl region, a semi-quantitative analysis was conducted by calculating the integrated area ratio between the lower-wavenumber carbonyl region and the native ester carbonyl region, $A_{1729-1720 \text{ cm}^{-1}}/A_{1740-1731 \text{ cm}^{-1}}$ (Table 2). The signal at 1730 cm^{-1} was treated as a transition boundary between these two regions and was not included in either integrated area to avoid overlap. The area ratios were calculated separately for each specimen ($n = 15$) and are presented as mean $\pm$ SD in Table 2. Linear mixed-effects analysis revealed significant treatment $\times$ weathering-time interactions under both natural weathering ($F(12, 210) = 6.661, p < 0.0001$) and xenon-arc accelerated weathering ($F(12, 210) = 4.754, p < 0.0001$), indicating that temporal changes in the carbonyl-area ratio differed among treatment groups. Prior to weathering, the area ratio ranged from 1.00 ± 0.03 to 1.06 ± 0.05 in the natural-weathering series and from 1.03 ± 0.02 to 1.06 ± 0.03 in the accelerated-weathering series. The differences observed among VH treatment conditions before weathering suggest that VH treatment modified the relative distribution of carbonyl-associated absorption, although the magnitude and direction of these differences varied among treatment conditions and specimen sets. These spectral changes are consistent with the deacetylation and chemical alteration of hemicellulose reported during thermal treatment of lignocellulosic materials [6,11]. However, considering the semi-quantitative nature of ATR-FTIR and the use of normalized spectra, $A_{1729-1720 \text{ cm}^{-1}}/A_{1740-1731 \text{ cm}^{-1}}$ should be interpreted only as an indicator of relative changes in the carbonyl region, rather than as a measure of the absolute concentration of specific degradation products or direct quantitative proof of a specific degradation pathway.

During natural weathering (Fig. 10A–D), the absorption intensities at

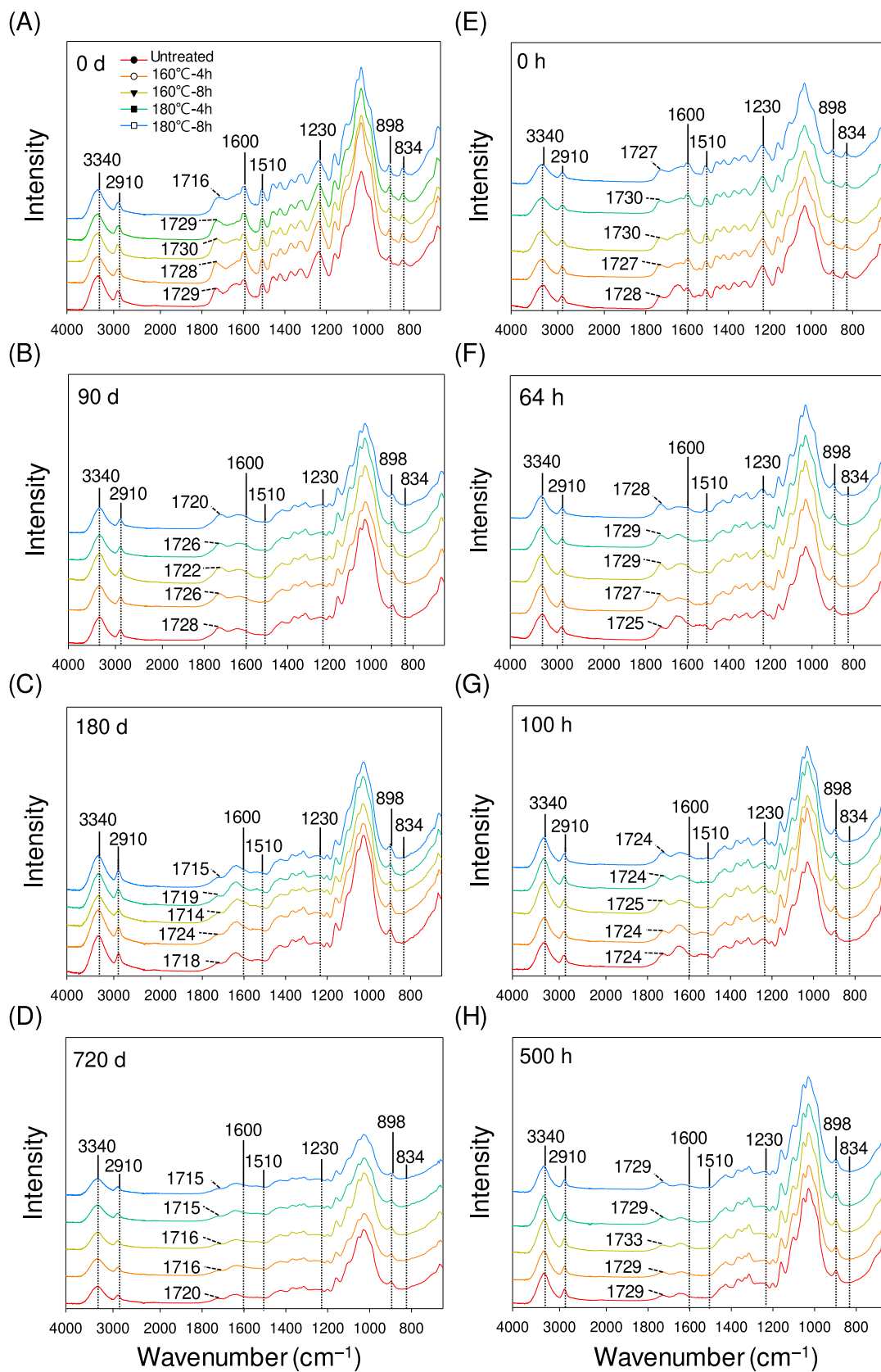


Fig. 10. FTIR spectra of untreated and VH-treated moso bamboo during the natural weathering test (A–D) and the xenon-arc accelerated weathering test (E–H).

1735, 1600, 1510, and 834 cm^{-1} gradually decreased with increasing exposure time across all groups. The decreases in the bands associated with hemicellulose and lignin are consistent with progressive changes in these polymer-associated structures under natural outdoor exposure. In particular, the decreases in the lignin-associated bands at 1600, 1510, and 834 cm^{-1} are consistent with previously reported lignin photo-oxidation and depletion from weathered lignocellulosic surfaces. In addition, a noticeable change in the carbonyl region was observed after 720 days of natural weathering. The maximum absorption in the 1740–1700 cm^{-1} region shifted from the native ester-dominated region near 1730 cm^{-1} toward a lower wavenumber, approximately 1720–1715 cm^{-1} , in the weathered specimens. This spectral shift is consistent with a reduction in native ester-associated carbonyl structures together with an increased relative contribution of lower-wavenumber carbonyl-containing structures during prolonged outdoor exposure. The carbonyl-area ratio also showed treatment-dependent temporal changes, as supported by a significant treatment $\times$ weathering-time interaction in the linear mixed-effects model ($F(12, 210) = 6.661, p < 0.0001$). The ratio generally increased with exposure time in the untreated, 160 °C-4 h, 160 °C-8 h, and 180 °C-4 h groups, whereas the 180 °C-8 h group showed a different temporal response (Table 2). After 720 days, the 180 °C-8 h group exhibited a significantly lower ratio than the other treatment groups ($p < 0.05$), while no significant differences were detected among the remaining four groups. These results indicate that the temporal evolution of carbonyl-associated absorption during natural weathering depended on the VH treatment condition.

The FTIR spectra of specimens subjected to accelerated xenon-arc weathering (Fig. 10E–H) showed a different temporal pattern in the carbonyl region. The carbonyl-area ratio exhibited treatment-dependent temporal changes, with a significant treatment $\times$ weathering-time interaction ($F(12, 210) = 4.754, p < 0.0001$). During the initial 64–100 h of exposure, changes in the 1729–1720 cm^{-1} region and in the corresponding area ratio varied among treatment groups, consistent with rapid photo-oxidative alteration of carbonyl-associated surface structures. By 500 h, the area ratios ranged from 0.99 ± 0.02 to 1.03 ± 0.02 (Table 2). At this time point, the 160 °C-8 h and 180 °C-4 h groups exhibited significantly lower ratios than the untreated, 160 °C-4 h, and 180 °C-8 h groups ($p < 0.05$). Considering the repeated water-spray cycles employed in the accelerated weathering test, the decreases observed in several treatment groups during prolonged exposure may reflect the progressive removal of water-soluble, low-molecular-weight oxidation and degradation products from the weathered surface [31,34,35]. Concurrently, the lignin-associated bands at 1600, 1510, and 834 cm^{-1} decreased markedly and became weak or nearly indistinguishable after prolonged exposure, indicating substantial changes in lignin-associated surface structures. Collectively, these spectral changes indicate that accelerated weathering produced treatment-dependent changes in carbonyl-associated absorption, with early-stage variation followed by lower ratios in several VH-treated groups after prolonged irradiation and water-spray exposure.

The different temporal trends observed under natural and accelerated weathering highlight the distinct surface chemical responses of bamboo under the two exposure environments. Under natural weathering, lower-wavenumber carbonyl-associated absorption remained relatively pronounced after prolonged exposure, which may reflect the combined effects of sunlight, rainfall, moisture cycling, surface deposition, and possible biological activity. In contrast, accelerated xenon-arc exposure produced a more rapid increase followed by a decrease in carbonyl-associated absorption under repeated irradiation and water-spray cycles. The decrease in lignin-associated bands under both weathering conditions is consistent with previous observations of weathered lignocellulosic materials [37]. Overall, the ATR-FTIR results provide semi-quantitative evidence of relative changes in hemicellulose- and lignin-associated functional groups and in the carbonyl region during VH treatment and subsequent weathering. However, because

ATR-FTIR provides semi-quantitative information on surface functional groups, these spectral changes should be interpreted as being consistent with chemical alteration of cell-wall components rather than as definitive identification of individual degradation products or specific degradation pathways.

3.3. Integrated interpretation of the weathering behavior of VH-treated bamboo

On the basis of the physical, mechanical, SEM, color, and FTIR results, the weathering behavior of VH-treated moso bamboo can be interpreted as a severity-dependent balance between initial property improvements imparted by VH treatment and susceptibility to subsequent weathering-induced deterioration. During VH treatment, the observed reductions in EMC and TS under mild to moderate treatment conditions were accompanied by changes in hemicellulose- and lignin-associated FTIR bands. These observations are consistent with previously reported thermal modification processes, including hemicellulose deacetylation and degradation as well as lignin-related condensation or rearrangement, which may reduce the accessibility of hydrophilic sites and restrict moisture uptake. However, increasing treatment severity was also accompanied by greater mass loss and deterioration of mechanical properties, suggesting that excessive modification may adversely affect the integrity of the cell-wall structure through more extensive alteration of lignocellulosic components.

During subsequent weathering, all specimens exhibited progressive discoloration, surface cracking, mass loss, and changes in hemicellulose- and lignin-associated FTIR bands. The decreases in lignin-associated absorption bands are consistent with the well-established susceptibility of lignin to photo-oxidative alteration during weathering. Because compound middle lamellae are relatively rich in lignin and play an important role in intercellular adhesion, deterioration of these regions may weaken bonding between adjacent cells. Such interfacial weakening may facilitate cell separation and the initiation and propagation of surface checks and cracks. The cross-sectional SEM observations further showed morphological alterations from the weather-exposed surface toward the interior after prolonged natural and accelerated weathering (Fig. 6), which are morphologically consistent with progressive disruption of intercellular and cell-wall structures. However, these observations should not be interpreted as direct chemical evidence of lignin degradation within the middle lamella or as quantitative determination of degradation depth. Changes in the carbonyl region of the FTIR spectra were also observed during exposure, while rainfall or repeated water-spray cycles may have contributed to the removal of water-soluble, low-molecular-weight degradation and oxidation products from the weathered surface. The different temporal changes observed under natural and accelerated weathering further indicate that these two exposure regimes represent complementary rather than directly interchangeable ageing environments. Natural weathering involved slower and more complex interactions among solar radiation, rainfall, moisture cycling, airborne deposition, and possible biological activity, whereas accelerated xenon-arc weathering emphasized controlled photo-oxidative exposure coupled with repeated water spray. Accordingly, a simple linear time-equivalence between natural outdoor weathering and xenon-arc accelerated weathering should not be assumed, because each exposure regime involves different combinations and intensities of environmental stressors. Accelerated weathering results should therefore be interpreted as complementary indicators of material response rather than direct predictors of long-term outdoor performance.

With prolonged exposure, the initial dimensional-stability advantage imparted by VH treatment gradually diminished, while mechanical performance also deteriorated to varying degrees. The development of surface cracking and subsurface morphological alterations occurred concurrently with the progressive deterioration of the weathered specimens. Relatively severe VH treatment was also associated with greater deterioration in some measured properties. However, the ATR-FTIR,

SEM, and mechanical results represent observations at different structural levels and should therefore be interpreted as complementary rather than as evidence of a direct causal relationship between surface chemical changes and bulk mechanical deterioration. Among the VH conditions examined in this study, the 160 °C-8 h treatment provided a comparatively favorable compromise among reduced hygroscopicity, retention of mechanical properties, and resistance to weathering-associated deterioration. However, this observation is based on the overall trends in the measured properties rather than on a predefined multicriteria ranking and should therefore be interpreted within the range of treatment conditions evaluated here.

4. Conclusions

This study demonstrated that the severity of vacuum-hydrothermal (VH) treatment strongly influenced the dimensional stability, mechanical performance, and subsequent weathering behavior of moso bamboo. Mild to moderate VH treatment reduced equilibrium moisture content and thickness swelling while largely retaining mechanical performance, whereas excessive treatment resulted in substantial mass loss and strength deterioration. During natural and xenon-arc accelerated weathering, all specimens exhibited discoloration, surface cracking, mass loss, changes in dimensional response, and morphological alterations detectable beneath the exposed surface. ATR-FTIR analysis indicated relative changes in hemicellulose- and lignin-associated bands and in the carbonyl region, consistent with progressive chemical alteration of the weathered bamboo surface. Overall, the results indicate a trade-off between the initial improvements in moisture-related properties imparted by VH treatment and the susceptibility to deterioration associated with excessive treatment severity. Among the conditions examined, treatment at 160 °C for 8 h provided a comparatively favorable compromise among reduced hygroscopicity, retained mechanical integrity, and resistance to weathering-associated deterioration. This comparison reflects the overall trends observed within the treatment conditions evaluated in the present study rather than a formal multicriteria ranking. Further depth-resolved chemical and structural analyses are needed to determine the chemical nature and penetration depth of weathering-induced alterations and to clarify their potential relationship with bulk mechanical deterioration. Longer-term exposure under different climatic conditions and direct evaluation of biological durability would also be valuable for assessing long-term service performance. From an application perspective, the favorable balance achieved under moderate VH conditions suggests potential for developing dimensionally stable bamboo materials for outdoor and engineered applications; however, treatment parameters should be further optimized according to the performance requirements of specific end uses.

CRedit authorship contribution statement

Jian-Wei Liu: Writing – original draft, Visualization, Supervision, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Ke-Chang Hung:** Software, Methodology, Investigation, Data curation. **Cheng-Che Tsai:** Investigation, Formal analysis, Data curation. **Wen-Shao Chang:** Supervision. **Jyh-Horng Wu:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Jyh-Horng Wu reports financial support was provided by Forestry and Nature Conservation Agency, Ministry of Agriculture, Taiwan. If there are other authors, they 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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Data availability

The data used to support the findings of this study are available from the corresponding author upon request.

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