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Combined Wood Modification Using Bark Extractive Impregnation and Ammonia Fuming

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Abstract

The present study investigated, for the first time, the synergistic effects of combining bark extractive impregnation with ammonia fuming, revealing promising enhancements in both the aesthetic and functional attributes of wood. This combined approach uses the bioactive and protective properties of natural extractives while also making ammonia fuming effective for wood species with low extractive content. Bark extracts of pedunculate oak (*Quercus robur* L.) and European beech (*Fagus sylvatica* L.) were investigated on wood specimens of European beech (with and without red heartwood), hornbeam (*Carpinus betulus* L.), and gray poplar (*Populus* × *canescens*). Oak bark extract contained substantially higher total extractives and polyphenols than beech bark extract. Treatments caused only minor changes in density and equilibrium moisture content (generally within ±8%), while some shrinkage parameters changed more noticeably. The largest improvement occurred in beech false heartwood, where volumetric shrinkage decreased by up to 16%. Both treatments darkened the wood surface, with oak bark extract producing significantly larger color changes; ammonia fuming further intensified these effects, especially in poplar. Durability improved markedly: mass loss caused by *Coniophora puteana* (Schumach.) P. Karst. and *Trametes versicolor* (L.) Lloyd decreased from 27%–38% in untreated samples to 0.58%–7.73% after treatment, improving durability classification from Durability Class 4 to classes 1–2. Bark extract treatments slightly reduced hardness but generally maintained or slightly increased compression strength.

Keywords: ammonia fuming; bark extractives; wood impregnation; wood modification; wood durability; physical properties; color change; dimensional stability

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1. Introduction

Wood, a renewable and versatile material, is widely used in construction, furniture, and other industries. Yet, its inherent limitations—such as susceptibility to moisture, decay, and dimensional instability—require modification to enhance its performance [1–5]. Among the various wood modification techniques, chemical treatments have gained prominence for their ability to improve durability, dimensional stability, and mechanical properties [1,3,6,7]. Chemical modification (e.g., acetylation, furfurylation) and thermal modification remains a primary strategy for enhancing wood properties, with implementing technologies being widely studied and commercialized [1–3,8]. The integration of nanotechnology and green chemistry principles is also expanding the possibilities for wood functionalization [3,5].

Bark is defined as the tissues external to and surrounding the vascular cambium, typically accounting for 9% to 15% of a log's volume or up to 21% of its dry weight [9]. Chemically, bark differs from wood by containing significantly higher levels of ash and extractives, such as suberin and polyphenols like tannins and flavonoids [9–11]. It is often treated as a low-value byproduct or waste material, although it represents a rich source of bioactive compounds.

Impregnation with natural bark extractives uses the rich chemical composition of bark, which can impart bioactivity and resistance to biological degradation [12–14]. Bark extractives are rich in polyphenolic compounds and other bioactive molecules, which can be harnessed to improve wood's resistance to decay, moisture, and biological attack [12–14]. Studies have shown that bark extractives can significantly increase antioxidant activity and may contribute to wood color and UV-absorbing properties [7,11,15] and enhance durability when used in wood impregnation [13,14].

Yingprasert et al. concluded that *Acacia mangium* Willd. bark extract vacuum impregnated into rubberwood significantly enhanced multiple properties. For dimensional stability, the treatment achieved an optimal anti-swelling efficiency (ASE) of 89.02% in the radial direction and 86.79% in the tangential direction using a 10% concentration. Regarding fungal decay resistance, the mass loss from white-rot fungus (*Trametes versicolor* L.) was reduced from 28.26% to 9.23%, and mass loss from brown-rot fungus (*Gloeophyllum striatum* Murill) dropped from 26.36% to 5.18% when a 20% concentration was used. Additionally, termite resistance improved, with the 10% concentration reducing the mass loss caused by *Coptotermes gestroi* (Wasmann) to 20.50% compared with 27.24% in untreated rubberwood [16].

Passialis et al. found that Aleppo pine (*Pinus halepensis* Mill.) bark extract, when used as a hydrophobic substance in water-repellent formulations, effectively improved the water repellency of beech wood (*Fagus sylvatica* L.) and Scots pine wood (*Pinus sylvestris* L.). Impregnated wood took 5 to 83 times longer to reach half maximum swelling compared with untreated controls. They also noted that extractives from broadleaved oak (*Quercus conferta* Kit.) were found to be effective water barriers to a varying degree [17].

Karim and Mazlan concluded that Gabon hazel (*Coula edulis* Baill.) bark extracts enhanced the fungal decay resistance of wood products. Their research demonstrated that at a concentration of 1000 mg/L, the extract completely suppressed the growth of brown-rot fungi such as *Coniophora puteana* (Schumach.) P. Karst. and *Rhodonia placenta* (Fr.) Cooke [18].

In the research of Feng et al., bark pyrolysis oils were used for the impregnation of wood blocks, improving the decay resistance of the material without negatively impacting the mechanical properties of the wood [9].

Alfredsen et al. created methanol bark extracts from sessile oak (*Quercus petraea* Lieb.) to screen its antifungal activity against various fungi species. The result showed that sessile oak bark extract produced over 25% antifungal activity against brown and white rot in laboratory agar plate bioassays [19].

Mori et al. screened acetone extracts from the barks of deciduous trees, including beech species (*Fagus crenata* Blume) and oak species (*Quercus mongolica* var. *grosseserrata* and *Quercus dentata* Thunb.). The result showed that these specific species exhibited only weak activity against wood decay fungi, categorized in the 0%–25% antifungal activity range [20].

Ammonia fuming, on the other hand, is a traditional method known for altering wood color and inducing chemical changes that can affect both aesthetics and material properties [21,22]. Ammonia fuming is traditionally used to darken wood and modify its surface chemistry. The process involves exposing wood to ammonia vapor, which reacts

with wood components—particularly carbonyl, aromatic, and alcohol groups—resulting in color changes and potential alterations in mechanical properties [21,22]. Besides the color effects, ammonia treatment can also increase density and affect the modulus of rupture and elasticity [21].

However, ammonia treatment is only applied to wood species which are naturally rich in polyphenolic extractives (e.g., European oak species (*Quercus robur* L. and *Quercus petreaea* L.), black locust (*Robinia pseudoacacia* L.), etc.). The advantages of ammonia treatment—like color enhancement—are more difficult to achieve in wood species with low extractive content.

According to Doczekalska et al., thermochemical modification using a 10% aqueous ammonia solution at 130 °C for 24 h notably enhances beech wood's structural performance, increasing the MOE by approximately 22.8% and the compression strength parallel to the grain by 39% [23].

When ammonia gas is used in the industrial densification process known as "Lignamon", Pařil et al. [24] and Rousek et al. [25] report even more dramatic gains, with the MOR increasing by 78%, the MOE by 86%, and the Janka hardness by approximately 150% compared with native beech.

Conversely, for bending applications, Šprdlík et al. [26] found that a combined treatment of water and ammonia vapor increases wood plasticity by reducing the MOE by 43.6% in its plasticized state, allowing the wood to be bent at sharper angles for furniture production.

Weigl-Kuska et al. determined that gaseous ammoniation notably increases the EMC at standard climate with hornbeam (*Carpinus betulus* L.), exhibiting one of the strongest increases in water affinity. Poplar demonstrated the most extreme response among the tested hardwood species, with its anisotropy coefficient increasing by approximately 1.0 unit compared with reference wood. Hornbeam also experienced a notable decrease in differential radial shrinkage, dropping from a median of roughly 0.25% down to 0.23% after modification [27].

In another research of Weigl-Kuska et al., they observed that 20 days of ammonia fumigation provides stabilized brightness for beech, poplar, and hornbeam when exposed to UV light, effectively preventing the natural darkening or graying typically seen in untreated samples [28].

Both bark extractive impregnation and ammonia fuming are effective methods for modifying wood properties, each with distinct mechanisms and benefits [1,3,12,21,29]. Bark extractives offer a sustainable, bio-based approach to enhancing wood durability and resistance, leveraging the natural chemical diversity of tree bark [12–14].

The combination of bark extracts' impregnation followed by ammonia treatment of wood is promising, as the beneficial effects of ammonia fuming (improved fire resistance, color changes, durability, and potentially novel functionalities) can be extended to other wood species too, which aligns with green chemistry and sustainability goals [3,5,29].

To the best of our current knowledge, the direct studies on the sequential use of bark extractive impregnation followed by ammonia fuming are limited, and most available research focuses on each method individually or in combination with other treatments (e.g., thermal, mineralization) [29,30]. The quality of evidence is generally strong for the individual methods, with multiple experimental and review studies supporting their efficacy, but more targeted research is needed to fully understand the interactions and optimize the combined approach.

The present research explored the synergistic potential of combining these two methods—impregnating wood with bark extractives followed by ammonia fuming—which aims to extend applications of less durable wood species and improve utilization of bark, which is mostly regarded as a waste material and a byproduct of wood logging and pro-

cessing. By using these methods together, the synergistic effect of the different mechanisms of action can be exploited. All this is achieved by using less utilized biomaterials, which also takes into account sustainability aspects. Bark and wood materials were selected based on their relevance and economic importance in Hungary. Accordingly, the bark extracts of European beech and pedunculate oak (*Quercus robur* L.) were investigated, and wood materials of European beech (with and without false heartwood), gray poplar sapwood (*Populus* × *canescens*), and European hornbeam were impregnated. The changes of color, density, wood–water relations, durability, and mechanical properties were compared and evaluated after bark extract impregnation and ammonia-fuming.

The aims of this research align with the growing emphasis on sustainable, green chemistry-based wood modification strategies that utilize bio-based resources and minimize environmental impact [3,5].

2. Materials and Methods

2.1. Materials

2.1.1. Bark Samples

Bark samples were collected from mature, healthy beech and pedunculate oak trees felled in December 2023 in the forests managed by TAEG Ltd. (Sopron, Hungary). Approximately 2 kg of bark was removed from the trunk of each species at heights between 1.5 and 3.0 m above the ground. The bark samples were transported to the laboratory and air-dried at room temperature in the dark for one week. During sampling and drying, special care was taken to preserve large bark pieces in order to minimize enzymatic oxidation and the potential loss of polyphenolic compounds [31,32].

2.1.2. Bark Extracts

The dried bark material was subsequently ground using a hammer mill equipped with a 0.5 cm sieve (Retsch SK3, Tersch GmbH, Haan, Germany). Extraction was performed by mixing 240 g of ground bark with 8 L of boiling distilled water, followed by maceration for 24 h. The resulting mixture was then filtered through filter paper, yielding a total of 6 L of bark extract, which was used for the impregnation treatments.

2.1.3. Wood Samples

Wood samples were taken from trees of beech, beech with false heartwood, hornbeam, and gray poplar sapwood felled in December 2023 in the forests managed by TAEG Ltd. (Sopron, Hungary).

2.2. Methods

2.2.1. Characterization of the Extracts

The chemical composition of the prepared extracts was characterized by determining the total extractive content (TEX) and the total polyphenol content (TPC) using the Folin–Ciocâlteu method.

For TEX determination, 5 mL of each extract was evaporated to dryness at room temperature for 72 h, and the remaining solids were weighed. The TEX content was given in grams of extractives per liter of extract solution (g/L). Measurements were performed in triplicate.

TPC determination was determined by applying the Folin–Ciocâlteu assay [33], with gallic acid used as the calibration standard. Briefly, the extract solution was mixed with 2.5 mL of tenfold-diluted Folin–Ciocâlteu reagent (VWR Chemicals, Debrecen, Hungary). After 1 min, 2.0 mL of 0.7 M Na_2CO_3 (VWR Chemicals, Debrecen, Hungary) solution was added, and the reaction mixture was heated in a water bath at 50 °C for 5 min. The reaction was terminated by cooling the mixture to room temperature in a cold-water bath. Solution

absorbance was measured at 760 nm using a Hitachi U-1500 type spectrophotometer (Hitachi Ltd., Tokyo, Japan). Results are expressed as grams of gallic acid (VWR Chemicals, Debrecen, Hungary) equivalents per liter of extract solution (g GAE/L). All determinations were carried out in triplicate.

2.2.2. Impregnation of Wood Specimens

Impregnation of all samples was carried out in accordance with EN 113-1:2020 [34]. Here, dry test specimens with a known initial mass (m_0) were impregnated with the beech bark extract or the oak bark extract. The procedure consisted of applying a vacuum of 0.7 kPa for 15 min, followed by the introduction of the extractives into the vessel and maintaining the specimens immersed at atmospheric pressure for 2 h. After impregnation, the specimens were immediately weighed to determine the mass after treatment (m_1). Retention values (kg/m^3) were calculated based on the mass data. Following impregnation, the samples were conditioned for 4 weeks at 20 °C and 65% relative humidity.

2.2.3. Ammonia Fuming of Samples

Fuming was performed in 30 cm diameter glass desiccator chambers, each with a total volume of 16 L. In total, three desiccator units were used for fuming. A total of 35 mL of concentrated (30% m/m) ammonium hydroxide solution (VWR Chemicals, Debrecen, Hungary) was measured into Petri dishes and placed at the bottom of the desiccators. Above the solutions, wood specimens were arranged at equal distances on a perforated ceramic holder. The specimens were arranged so that all three desiccators contained the same number of wood samples. Special care was taken to ensure that the liquid ammonium hydroxide solution did not come into direct contact with the wood specimens. The desiccators were covered with their glass lids, and fuming was carried out for 24 h. After the treatment, the lids were removed, and the samples were taken out of the chambers and conditioned at laboratory conditions for one day. For reference (control) measurements, respective non-impregnated wood specimens were also placed in the desiccators and fumed.

Figure 1 depicts the arrangement of the wood specimens in the fuming chamber and the color change during the fuming process.

Figure 1. Arrangement of the wood specimens in the fuming chamber (the photo was taken here without a lid). The initial color of the wood samples (on the left) changed after fuming (on the right). Note the Petri dish at the bottom of the chamber holding the treatment medium (30% NH_4OH solution).

2.2.4. Color Measurement

Surface color evaluation was carried out before and after impregnation and fuming with a Konica Minolta CM-2600d spectrophotometer (Konica Minolta Inc., Tokyo, Japan) based on the CIELAB color space. In the CIELAB color space, L* represents lightness (0 = black, 100 = white), a* indicates the position on the green (−) to red (+) axis, and b* corresponds to the blue (−) to yellow (+) axis. The overall color difference (ΔE*) is a combined parameter calculated from changes in the L*, a*, and b* coordinates. Readings were obtained under D65 standard illumination conditions using a 10° observer angle and an aperture size of 8 mm. For each sample type, ten individual specimens were measured.

2.2.5. Durability Test

Biological durability was assessed in accordance with EN 113-2:2020 [34] using specimens sized 15 × 25 × 50 mm (radial × tangential × longitudinal). The brown-rot fungus *Coniophora puteana* and white-rot fungus *Trametes versicolor* were employed as test organisms. Five specimens per treatment and five untreated controls were exposed for a total test period of 16 weeks.

2.2.6. Conditioning

The test samples for hygroscopic properties, hardness, and compression strength tests were conditioned at 20 °C room temperature and 65% relative humidity in a Memmert HPP260 climate chamber (Memmert GmbH, Schwabach, Germany) until reaching constant mass.

2.2.7. Measurment of the Hygroscopic Properties

For the determination of hygroscopic properties, 10 specimens with dimensions of 20 mm × 20 mm × 30 mm (radial × tangential × longitudinal) were prepared from each wood species and treatment type. After conditioning, their dimensions in the radial, tangential, and longitudinal directions were measured using a Mitutoyo 543730B digimatic indicator (Mitutoyo Ltd., Kawasaki, Japan), and their mass was determined using a KERN ABJ220-4M laboratory scale (Kern & Sohn, Balingen, Germany). The specimens were subsequently dried at 105 °C in a conventional oven until constant mass was reached and then remeasured using the same procedures. The equilibrium moisture content ($EMC_{20/65}$) was determined in accordance with ISO 13061-1:2014 [35], and the conditioned ($\rho_{20/65}$) and oven-dry (ρ_0) density were calculated according to ISO 13061-2:2014 [36].

Linear shrinkage in the radial (S_{rad}), tangential (S_{tan}), and longitudinal (S_{lon}) directions were calculated by dividing the dimensional change by the conditioned dimension. Volumetric shrinkage (S_V) was determined by dividing the change in volume by the conditioned volume. The shrinkage coefficient (SC) was calculated between the oven-dry and conditioned state by dividing S_V with $EMC_{20/65}$. Anti-swelling efficiency (ASE) was determined by dividing the SC_V difference between the treated and control state by the SC_V of control.

2.2.8. Brinell Hardness Test

Brinell hardness measurements were carried out on the cross-section and the tangential surfaces using an Instron 4208 universal testing machine (Norwood, MA, USA), with 500N force, following the procedure described in EN 1534:2020 [37]. Test specimens had dimensions of 20 mm × 50 mm × 50 mm (radial × tangential × longitudinal). For each treatment group, ten samples were tested and compared with ten untreated counterparts.

2.2.9. Compression Strength Test

Compression strength in the longitudinal direction was determined using an Instron 4208 universal testing machine (Norwood, MA, USA) in accordance with ISO 13061-5:2020 [38].

Specimens measuring 20 mm × 20 mm × 30 mm (radial × tangential × longitudinal) were used. A total of 10 samples were evaluated for each type.

2.2.10. Statistical Evaluation

The datasets were first examined for compliance with normal distribution assumptions. The influence of the applied treatments on the measured material characteristics was then evaluated using variance analysis followed by Fisher's least significant difference post hoc test at a significance level of $p < 0.05$. All statistical analyses were performed using Statistica 10.0 (TIBCO Software Inc., Palo Alto, CA, USA) software.

3. Results

3.1. Composition of the Extracts

Table 1 presents the results of the chemical analysis of the extracts. According to the results, the TPC and TEX of oak bark are significantly higher compared with those of beech, which is consistent with the authors' earlier findings: the TPC of aqueous beech bark extracts ranged between 29.3 and 34.2 mg QE/g d.w. [39,40], while the corresponding values for oak were 46.2–71.6 mg QE/g d.w. [41]. It should be emphasized, however, that these earlier results refer to the dry mass of the bark rather than the extract solution, and a different standard was used (quercetin instead of gallic acid). Nevertheless, the present results clearly justify that oak bark extracts contain higher amounts of extractives—particularly polyphenols—compared with beech.

Table 1. The total extractive content and total polyphenolic content of the beech and oak bark extracts used for the impregnation treatments. Average values are presented with the standard deviation in brackets. Different letters indicate statistically significant differences ($p < 0.05$), specifically between the two bark extracts.

	Beech Bark Extract	Oak Bark Extract
Total extractive content (g/L)	5.13 (0.42) a	18.67 (0.50) b
Total polyphenol content (g GAE/L)	0.97 (0.15) a	10.1 (0.10) b

3.2. Hygroscopic Properties

Table 2 shows the results of density, EMC, shrinkage, shrinkage coefficients, and ASE. Across all tested wood species, the treatments with oak bark extract (OB) and beech bark extract (BB) resulted in relatively small changes in density and equilibrium moisture content compared with the untreated controls (CO), while larger relative changes were observed in several shrinkage parameters.

Changes in density were generally small, mostly within approximately ±8%. The largest relative increase occurred in poplar after BB treatment, where $\rho_{20/65}$ increased from 0.41 to 0.44 g cm^{-3} (+7.3%), and ρ_0 increased from 0.39 to 0.42 g cm^{-3} (+7.7%). The largest decreases in density were observed in beech false heartwood, where $\rho_{20/65}$ decreased from 0.74 to 0.71 g cm^{-3} (−4.1%) and ρ_0 decreased from 0.71 to 0.67 g cm^{-3} (−5.6%) after BB treatment. Among the tested species, the density of hornbeam was not significantly affected by the treatments.

EMC showed similarly small variations. The largest decrease occurred in beech false heartwood, where EMC decreased from 12.32% in the control to 11.35% after BB treatment (−7.9%) and to 11.42% after OB treatment (−7.3%). The largest increase was recorded in hornbeam after OB treatment, where EMC increased from 11.01% to 11.39%

(+3.5%). Among the tested species, the EMC of poplar was not significantly affected by the treatments.

Table 2. Results of hygroscopic property testing: untreated (CO), treated with oak bark extract (OB), with beech bark extract (BB), density at 20 °C 65% ($\rho_{20/65}$), oven-dry density (ρ_0), equilibrium moisture content ($EMC_{20/65}$), linear shrinkage in radial (S_{rad}), tangential (S_{tan}) and longitudinal direction (S_{lon}), volumetric shrinkage (S_V), shrinkage coefficient in radial (SC_{rad}), tangential (SC_{tan}) and longitudinal direction (SC_{lon}), volumetric shrinkage coefficient (SC_V), and anti-swelling efficiency (ASE_V). Average values are presented with standard deviation in brackets. Different letters indicate statistically significant differences ($p < 0.05$), specifically between the untreated and extract-treated samples of a given species.

Wood Species	Treatment	$\rho_{20/65}$ (g/cm^3)	ρ_0 (g/cm^3)	$EMC_{20/65}$ (%)	S_{rad} (%)	S_{tan} (%)	S_{lon} (%)	S_V (%)	SC_{rad}	SC_{tan}	SC_{lon}	SC_V	ASE_V (%)
Beech	CO	0.75 (0.01) b	0.72 (0.01) b	10.94 (0.03) a	2.29 (0.18) a	3.89 (0.16) a	0.10 (0.05) a	6.19 (0.27) ab	0.21 (0.02) a	0.36 (0.01) ab	0.01 (0.00) a	0.57 (0.03) b	
	OB	0.73 (0.01) a	0.70 (0.01) a	11.17 (0.07) b	2.14 (0.29) a	3.87 (0.30) a	0.11 (0.08) a	6.04 (0.24) a	0.19 (0.03) a	0.35 (0.03) a	0.01 (0.01) a	0.54 (0.02) a	2.47
	BB	0.73 (0.00) a	0.70 (0.01) a	11.18 (0.03) b	2.24 (0.15) a	4.14 (0.14) b	0.13 (0.09) a	6.41 (0.23) c	0.20 (0.01) a	0.37 (0.01) b	0.01 (0.01) a	0.57 (0.02) b	−3.56
Beech (false heartwood)	CO	0.74 (0.02) b	0.71 (0.02) b	12.32 (0.08) c	2.67 (0.33) b	4.61 (0.20) c	0.12 (0.09) a	7.26 (0.49) c	0.22 (0.03) a	0.37 (0.02) b	0.01 (0.01) a	0.59 (0.04) b	
	OB	0.71 (0.02) a	0.68 (0.02) a	11.42 (0.05) b	2.34 (0.13) ab	4.09 (0.18) b	0.20 (0.04) a	6.53 (0.25) b	0.21 (0.01) a	0.36 (0.02) b	0.02 (0.00) a	0.57 (0.02) b	10.10
	BB	0.71 (0.02) a	0.67 (0.02) a	11.35 (0.05) a	2.22 (0.27) a	3.72 (0.38) a	0.22 (0.15) a	6.07 (0.33) a	0.20 (0.02) a	0.33 (0.03) a	0.02 (0.01) a	0.53 (0.03) a	16.39
Hornbeam	CO	0.76 (0.01) a	0.73 (0.01) a	11.01 (0.18) a	2.21 (0.12) ab	3.70 (0.14) a	0.19 (0.04) a	6.01 (0.19) a	0.20 (0.01) ab	0.34 (0.01) a	0.02 (0.00) a	0.55 (0.02) a	
	OB	0.76 (0.02) a	0.72 (0.02) a	11.39 (0.16) b	2.47 (0.27) b	3.60 (0.23) a	0.15 (0.06) a	6.13 (0.21) a	0.22 (0.02) b	0.32 (0.02) a	0.01 (0.01) a	0.54 (0.02) a	−2.02
	BB	0.76 (0.01) a	0.73 (0.01) a	11.18 (0.18) a	1.74 (0.79) a	4.38 (0.58) b	0.18 (0.05) a	6.22 (0.35) a	0.16 (0.07) a	0.39 (0.05) b	0.02 (0.00) a	0.56 (0.03) a	−3.52
Poplar	CO	0.41 (0.01) a	0.39 (0.01) a	10.93 (0.42) a	1.37 (0.31) a	2.84 (0.27) a	0.18 (0.14) a	4.34 (0.40) a	0.12 (0.02) a	0.26 (0.03) a	0.02 (0.01) a	0.40 (0.03) a	
	OB	0.43 (0.01) b	0.40 (0.01) b	10.91 (0.14) a	1.38 (0.24) a	2.81 (0.14) a	0.14 (0.07) a	4.29 (0.27) a	0.13 (0.02) a	0.26 (0.01) a	0.01 (0.01) a	0.39 (0.03) a	1.26
	BB	0.44 (0.01) b	0.42 (0.01) c	10.82 (0.17) a	1.43 (0.13) a	2.92 (0.15) a	0.21 (0.13) a	4.51 (0.09) a	0.13 (0.01) a	0.27 (0.01) a	0.02 (0.01) a	0.42 (0.01) a	−3.81

Shrinkage values showed larger relative changes than density and EMC. The most pronounced reductions were observed in beech false heartwood. Radial shrinkage decreased from 2.67% in the control to 2.22% after BB treatment and to 2.34% after OB treatment. Tangential shrinkage decreased from 4.61% to 3.72% with BB and to 4.09% with OB. Correspondingly, volumetric shrinkage decreased from 7.26% in the control to 6.07% after BB treatment (−16.4%) and to 6.53% after OB treatment (−10.1%).

In contrast, several parameters increased after treatment in other species. For example, tangential shrinkage in hornbeam increased from 3.70% in the control to 4.38% after BB treatment. In beech wood, tangential shrinkage increased from 3.89% to 4.14% with BB treatment.

In beech false heartwood, longitudinal shrinkage increased from 0.12% in the control to 0.20% with OB and to 0.22% with BB.

Among the tested species, the treatments did not significantly affect the linear shrinkage and volumetric shrinkage of poplar, nor the volumetric shrinkage of hornbeam.

Shrinkage coefficients generally followed similar trends as the shrinkage values. One of the largest relative changes occurred in hornbeam after BB treatment, where SC_{rad} decreased from 0.20 to 0.16. In beech false heartwood, SC_{tan} decreased from 0.37 in the control to 0.33 with BB treatment, and SC_V decreased from 0.59 to 0.53.

Among the tested species, the treatments did not significantly affect the linear shrinkage coefficient and volumetric shrinkage coefficient of poplar, nor the volumetric shrinkage coefficient of hornbeam.

The highest value for ASE was recorded for beech false heartwood treated with BB (16.39%), followed by OB treatment of the same material (10.10%). In contrast, negative ASE values were recorded in several cases, including poplar with BB (−3.81%) and beech with BB (−3.56%).

3.3. Color Measurements

The color measurements presented in Tables 3 and 4 show the changes in the CIELAB color parameters (ΔL, Δa, Δb) and the total color difference (ΔE) after impregnation with bark extracts and subsequent fuming. In all wood species and treatments, the dominant change was a decrease in lightness (ΔL), indicating darkening of the wood surface, accompanied by varying increases in redness (Δa) and yellowness (Δb). This can also be observed in Figures 1 and 2.

Table 3. Results of color measurements after oak bark extract impregnation and fuming. The values indicate the average color change after impregnation and fuming compared with the original color. L—lightness; a—redness; b—yellowness; E—color difference. The standard deviation is given in brackets.

	Color Change Compared with the Original, Untreated Color							
	After Oak Bark Extract Impregnation				**Color Change After Fuming**			
	ΔL	**Δa**	**Δb**	**ΔE**	**ΔL**	**Δa**	**Δb**	**ΔE**
Beech	−11.52 (2.98)	2.72 (0.46)	6.01 (0.69)	13.33 (2.79)	−20.79 (2.92)	3.28 (0.59)	5.39 (1.03)	21.75 (2.95)
Beech (false heartwood)	−8.44 (2.24)	1.82 (0.73)	5.71 (1.07)	10.39 (2.43)	−15.34 (1.67)	2.89 (0.57)	6.00 (0.97)	16.74 (1.78)
Hornbeam	−10.13 (1.93)	4.51 (1.09)	8.25 (1.79)	13.97 (1.88)	−18.83 (2.92)	5.81 (1.19)	8.36 (2.20)	21.55 (2.76)
Poplar	−16.28 (4.75)	7.11 (1.99)	11.23 (1.79)	21.09 (5.12)	−26.37 (6.04)	8.53 (2.03)	11.29 (1.47)	29.97 (6.30)

Table 4. Results of color measurements after beech bark extract impregnation and fuming. The values indicate the color change after impregnation and fuming compared with the original color. L—lightness; a—redness; b—yellowness; E—color difference. The standard deviation is given in brackets.

	Color Change Compared to the Original Color							
	After Beech Bark Extract Impregnation				**Color Change After Fuming**			
	ΔL	**Δa**	**Δb**	**ΔE**	**ΔL**	**Δa**	**Δb**	**ΔE**
Beech	−4.79 (0.63)	−0.45 (0.58)	1.90 (0.89)	5.27 (0.68)	−9.05 (1.03)	0.47 (0.42)	2.09 (0.82)	9.33 (1.12)
Beech (false heartwood)	−3.84 (1.93)	−0.03 (0.68)	2.03 (0.96)	4.45 (2.02)	−9.11 (3.44)	1.03 (0.88)	2.29 (1.39)	9.58 (3.45)
Hornbeam	−2.74 (0.84)	1.49 (0.46)	4.84 (1.10)	5.81 (1.21)	−7.44 (1.05)	2.18 (0.68)	4.88 (1.11)	9.20 (1.36)
Poplar	−5.57 (1.92)	2.41 (1.21)	6.59 (2.32)	8.99 (3.14)	−11.11 (2.54)	4.49 (1.61)	8.19 (2.38)	14.55 (3.66)

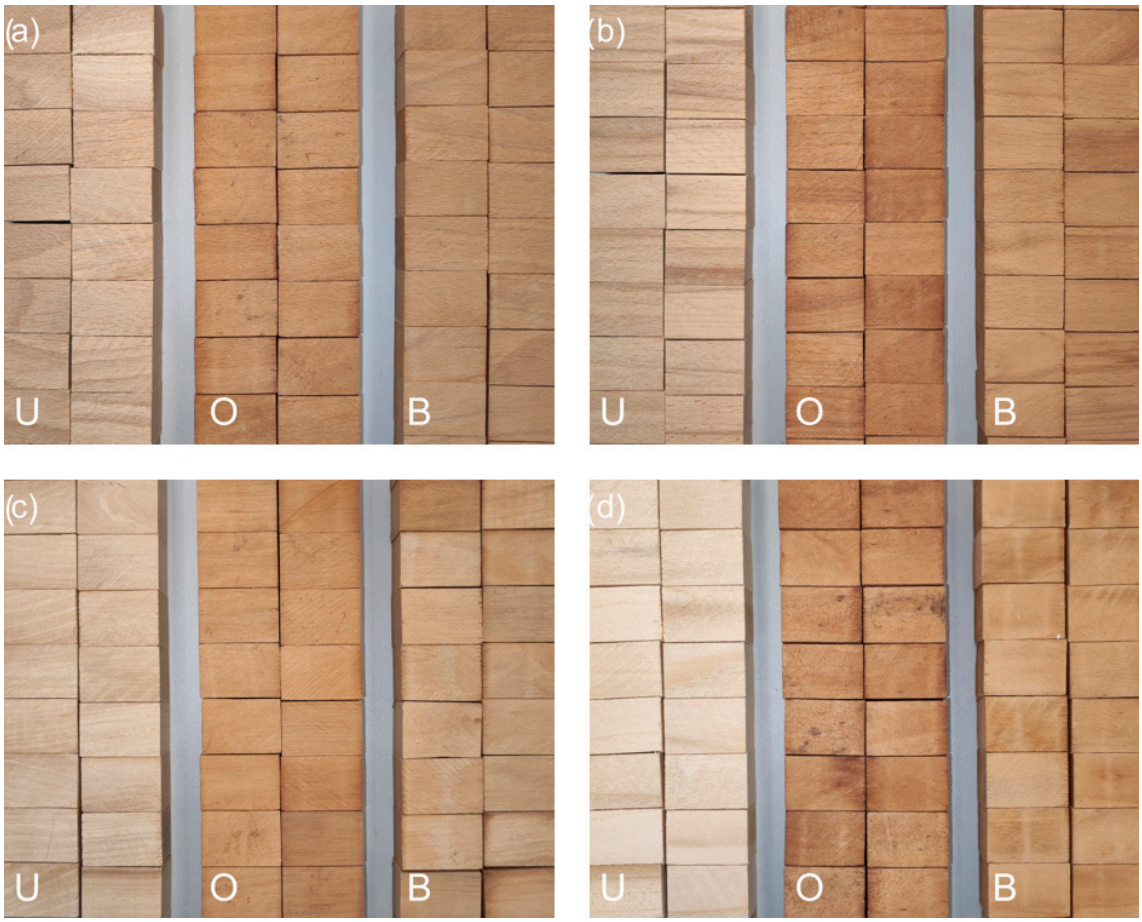

Figure 2. Photographs of samples used for color testing. The images show specimens of beech (**a**), beech with false heartwood (**b**), hornbeam (**c**), and poplar (**d**). In each image, the specimens are arranged from left to right as follows: untreated samples (U), samples impregnated with oak bark extract and subsequently fumed (O), and samples impregnated with beech bark extract and subsequently fumed (B).

After impregnation with oak bark extract, all wood species exhibited a substantial decrease in lightness. The largest decrease occurred in poplar (ΔL = −16.28), followed by beech (−11.52), hornbeam (−10.13), and beech false heartwood (−8.44). After fuming, the decrease in lightness became considerably stronger in all species. Poplar showed the largest change (ΔL = −26.37), followed by beech (−20.79), hornbeam (−18.83), and beech false heartwood (−15.34). Compared with the impregnated state, fuming increased the magnitude of lightness reduction by approximately 70%–90% depending on the species.

Redness (Δa) increased in all species after oak bark impregnation. The largest increase was observed in poplar (Δa = 7.11), followed by hornbeam (4.51), beech (2.72), and beech false heartwood (1.82). After fuming, redness increased further, reaching 8.53 in poplar and 5.81 in hornbeam, while beech and beech false heartwood reached 3.28 and 2.89, respectively.

Yellowness (Δb) also increased in all species after impregnation. Poplar showed the highest increase (11.23), followed by hornbeam (8.25), beech (6.01), and beech false heartwood (5.71). After fuming, yellowness remained similar or slightly increased depending on the species. The largest value was recorded in poplar (11.29), while hornbeam (8.36), beech (5.39), and beech false heartwood (6.00) showed smaller changes relative to the impregnated state.

After impregnation with oak bark extract, ΔE ranged from 10.39 in beech false heartwood to 21.09 in poplar. Fuming significantly increased the color difference in all species, with values reaching 29.97 in poplar, 21.75 in beech, 21.55 in hornbeam, and 16.74 in beech

false heartwood. The increase in ΔE after fuming relative to the impregnated state ranged from approximately 48% in poplar to about 66% in beech.

All color differences observed after treatment were considered significantly large, meaning that a standard observer would perceive them as distinct colors [42].

Color changes after beech bark extract impregnation were notably smaller than those observed with oak bark extract. The decrease in lightness ranged from −2.74 in hornbeam to −5.57 in poplar. After fuming, lightness decreased further, reaching −11.11 in poplar, −9.11 in beech false heartwood, −9.05 in beech, and −7.44 in hornbeam.

Changes in redness were smaller and more variable than in the oak extract treatment. After impregnation, beech and beech false heartwood showed very small or negative changes (−0.45 and −0.03), while hornbeam and poplar showed increases of 1.49 and 2.41, respectively. After fuming, redness increased in all species, reaching 4.49 in poplar, 2.18 in hornbeam, 1.03 in beech false heartwood, and 0.47 in beech.

Yellowness increased moderately in all cases. After impregnation, Δb ranged from 1.90 in beech to 6.59 in poplar. After fuming, these values increased further, with the highest value again being observed in poplar (8.19), followed by hornbeam (4.88), beech false heartwood (2.29), and beech (2.09).

The total color difference after impregnation with beech bark extract ranged from 4.45 in beech with false heartwood to 8.99 in poplar. After fuming, ΔE approximately doubled for most species, reaching 14.55 in poplar, 9.58 in beech false heartwood, 9.33 in beech, and 9.20 in hornbeam.

Comparing the two extract treatments shows that oak bark extract caused substantially larger color changes than beech bark extract. After impregnation, ΔE values for oak bark ranged from 10.39 to 21.09, whereas those for beech bark ranged from 4.45 to 8.99. After fuming, oak bark treatments produced ΔE values between 16.74 and 29.97, while beech bark treatments ranged from 9.20 to 14.55.

Among the studied species, poplar consistently showed the largest color changes in both treatments, while beech false heartwood generally showed the smallest ΔE values.

3.4. Durability Tests

The mass loss of the tested species and treatments can be seen in Table 5. The results demonstrate that untreated control specimens of all tested wood species experienced substantial mass loss when exposed to both fungal species. In the case of *Coniophora puteana*, mass loss in control samples ranged from 27.3% (poplar) to 33.7% (beech with false heartwood), while exposure to *Trametes versicolor* resulted in even higher degradation, with control values ranging between 34.4% and 37.9%. These levels indicate severe fungal attack and validate the effectiveness of the test conditions.

In contrast, treatment with bark extracts and ammonia fuming led to a pronounced reduction in mass loss for all wood species and for both fungi. Against *C. puteana*, specimens treated with oak and beech bark extracts showed mass losses between 0.58 and 2.88%, representing a reduction of more than 96% compared with the controls. Although some variations remain between the wood species, according to EN 113-1:2020 [34], this makes it Durability Class 1 (mass loss < 5%). Among the wood species, only poplar showed a significant difference in mass loss between the tested bark extracts, with beech bark extract resulting in significantly lower mass loss than oak bark extract; no significant differences were observed for the other wood species.

For *T. versicolor*, mass loss values in treated samples were higher than those observed for *C. puteana* but remained markedly lower than in the controls. Oak bark extract resulted in mass losses ranging between 4.1% and 5.5%, while beech bark extract produced values ranging from 5.4% to 7.7%. This indicates that *T. versicolor* was more tolerant and more

resistant to the treatments than *C. puteana*. Overall, oak bark extract tended to provide slightly better protection against *T. versicolor* than beech bark extract. Although some variations remain between the wood species, according to EN 113-1:2020 [34], this makes it Durability Class 1 (mass loss < 5%) and Durability Class 2 (mass loss < 10%). Among the wood species, only beech showed a significant difference in mass loss between the tested bark extracts, with oak bark extract resulting in significantly lower mass loss than beech bark extract; no significant differences were observed for the other wood species.

Table 5. Mass loss (%) of the tested wood species after the durability tests against *Coniophora puteana* and *Trametes versicolor*. Average values are presented for untreated samples (CO) and samples impregnated with oak bark (OB) or beech bark (BB) extract and fumed with ammonia. The standard deviation is given in brackets. Different letters indicate statistically significant differences ($p < 0.05$), specifically between the untreated and extract-treated samples of a given wood species and fungal species.

Mass Loss (%)	*Coniophora puteana*			*Trametes versicolor*		
	CO	OB	BB	CO	OB	BB
Hornbeam	31.60 (1.77) b	1.24 (0.18) a	1.31 (0.39) a	37.86 (2.16) b	4.84 (2.41) a	7.73 (1.96) a
Beech with false heartwood	33.74 (1.88) b	0.58 (0.09) a	1.01 (1.30) a	37.75 (3.51) b	5.46 (1.58) a	5.36 (1.67) a
Beech	31.34 (0.92) b	0.85 (0.25) a	0.93 (0.28) a	34.35 (3.31) c	4.10 (1.38) a	6.76 (0.85) b
Poplar	27.33 (1.15) c	2.88 (0.61) b	1.26 (0.29) a	36.15 (5.58) b	5.48 (1.31) a	5.63 (0.93) a

3.5. Brinell Hardness Tests

Brinell hardness values varied markedly among wood species, anatomical direction, and type of bark extract (Table 6). In all cases, hardness measured on the end grain was substantially higher than on the sides.

Table 6. Brinell hardness values of the tested wood species measured on the end grain surface and the side surface. Average values are presented for untreated samples (CO) and samples impregnated with oak bark (OB) or beech bark (BB) extract and fumed with ammonia. The standard deviation is given in brackets. Different letters indicate statistically significant differences ($p < 0.05$), specifically between the untreated and extract-treated samples of a given wood species and tested surface.

Brinell Hardness (MPa)	End Grain			Sides		
	CO	OB	BB	CO	OB	BB
Hornbeam	57.70 (14.28) a	46.34 (18.40) a	45.32 (16.31) a	31.04 (7.94) b	22.83 (4.94) a	21.40 (3.07) a
Beech with false heartwood	46.55 (10.89) a	41.24 (8.60) a	39.27 (8.04) a	23.52 (3.04) a	21.55 (3.92) a	19.97 (5.34) a
Beech	48.20 (15.52) a	51.70 (9.27) a	40.31 (9.01) a	21.82 (3.13) a	22.64 (3.20) a	21.01 (1.22) a
Poplar	29.37 (4.46) b	28.12 (4.15) ab	24.02 (4.63) a	9.45 (1.19) a	9.54 (0.82) a	10.26 (0.89) a

Among the investigated species, hornbeam exhibited the highest Brinell hardness in both orientations. The control hornbeam samples showed an end-grain hardness of 57.70 MPa, which decreased notably after treatment with oak bark extract (46.34 MPa) and

beech bark extract (45.32 MPa). A similar reduction trend was observed on the side surfaces, where hardness decreased from 31.04 MPa in the control to 22.83 MPa and 21.40 MPa after oak and beech bark extract treatments, respectively.

Beech with false heartwood showed intermediate hardness values. End-grain hardness decreased gradually from 46.55 MPa in the control to 41.24 MPa (oak bark extract) and 39.27 MPa (beech bark extract). Side hardness followed the same trend, declining from 23.52 MPa in the control to 21.55 MPa and 19.97 MPa for oak and beech bark extract treatment, respectively. In contrast, sound beech exhibited a less uniform response to the treatments. While oak bark extract slightly increased end-grain hardness (51.70 MPa) compared with the control (48.20 MPa), beech bark extract resulted in a decrease (40.31 MPa). On the side surfaces, hardness values remained relatively stable across treatments (21.82–22.64 MPa).

Poplar demonstrated the lowest Brinell hardness values among all species. End-grain hardness decreased modestly with bark extract treatments, from 29.37 MPa in the control to 28.12 MPa (oak bark extract) and 24.02 MPa (beech bark extract). Side hardness values were low and showed minimal variation: 9.45, 9.54, and 10.26 MPa for control, oak bark extract, and beech bark extract, respectively.

Among the tested species, the end-grain hardness of poplar and the side hardness of hornbeam were significantly affected by the used treatments.

Overall, the results indicate that bark extract treatments generally reduce Brinell hardness, and beech showed the highest resistance to treatment-induced changes.

3.6. Compression Strength Tests

Treatment with oak bark and beech bark extract resulted in a slight but significant increase in average values for beech (+2%–10%) and poplar wood (+3%–16%) compared with the controls (Table 7). Among the results, poplar showed the lowest standard deviation (0.93–2.13 MPa), indicating a more homogeneous structure than the other tested species.

Table 7. Compression strength values of the tested wood species. Average values are presented for untreated samples and samples impregnated with oak bark or beech bark extract and fumed with ammonia. The standard deviation is given in brackets. Different letters indicate statistically significant differences ($p < 0.05$), specifically between the untreated and extract-treated samples of a given wood species.

Compression Strength (MPa)	Untreated	Oak Bark Extract	Beech Bark Extract
Hornbeam	64.68 (4.89) ab	67.83 (2.53) b	61.36 (6.27) a
Beech	67.89 (5.11) a	69.29 (2.65) a	74.85 (2.80) b
Beech with false heartwood	66.77 (3.88) a	67.14 (3.00) a	66.96 (6.28) a
Poplar	37.83 (2.13) a	39.72 (1.77) b	43.72 (0.93) c

Hornbeam exhibited an increase after oak bark treatment (+5%), and a decrease after beech bark treatment (−5%), relative to the control. The compression strength of beech with false heartwood did not change significantly after the treatments (~67 MPa).

4. Discussion

The results demonstrate that bark extract impregnation combined with ammonia fuming can significantly influence the biological durability, color, and selected physical and mechanical properties of the investigated wood species.

The chemical analysis confirmed that oak bark extract contains substantially higher levels of total extractives and polyphenols than beech bark extract, which is reflected in the generally stronger effects observed in color modification and fungal resistance. Chemical test results correspond to the findings of [40,43]. With higher retention of bark extract, darker color and higher durability was reported by several researchers [16,19,44].

The treatments caused only minor changes in the density and hygroscopic properties. The results varied depending on anatomical characteristics and permeability. Other researchers also reported only slight changes in the density and EMC of wood treated solely with ammonia [23,27,45]. Ref. [27] reported the phenomenon ammoniation increasing the anisotropy of wood, decreasing radial shrinkage/swelling while simultaneously increasing tangential shrinkage/swelling. This phenomenon can be traced in our presented results as well.

Color measurements showed that both extracts caused darkening of the wood surface, accompanied by increases in redness and yellowness. Oak bark extract produced considerably stronger color changes than beech bark extract, and ammonia fuming significantly intensified the effect. Among the tested species, poplar showed the largest color differences. The color changes caused by the bark extract impregnations are attributed to the brown color of the impregnation solutions of bark extracts. This visually appealing color change suggests potential applications in interior design, furniture components, and decorative wood products where aesthetic enhancement is desirable.

Several papers found that the darkening of wood during ammonia fuming is caused by a chemical reaction between ammonia and tannin (native in the wood or the wood is soaked in a tannin solution). In the reaction between ammonia gas and tannins carbonyl, aromatic and alcohol groups are involved. Besides the reaction between ammonia gas and tannins, the involvement of carbonyl groups of hemicelluloses in the reaction with ammonia gas was found to be one of the reasons behind color changes [45–47].

The color of rubberwood also darkened after vacuum impregnation with *Acacia mangium* bark extract in the study of [16]. Peng et al. found that the extract of Chinese fir bark can significantly improve the photostability and surface integrity of wood during weathering [11]. In the future, oak and beech bark extracts combined with ammonia fuming could be evaluated as a means of improving the outdoor performance of low-durability beech, hornbeam, and poplar wood.

The most pronounced improvement was observed in biological durability. Mass loss caused by *Coniophora puteana* and *Trametes versicolor* decreased dramatically in treated samples compared with untreated controls, resulting in durability classes corresponding to highly durable (Durability Class 1) or durable (Durability Class 2) wood. The treatments were particularly effective against the brown-rot fungus *C. puteana* (reducing mass loss from 27.3%–33.7% to 0.6%–2.9%), while the white-rot fungus *T. versicolor* showed slightly higher tolerance (mass loss decreased from 34.4%–37.9% to 4.1%–7.7%). Oak bark extract generally provided somewhat better protection than beech bark extract, which may be attributed to its higher polyphenol content. In similar studies, the mass loss caused by *Trametes versicolor* was decreased to 9.23% using *Acacia mangium* bark extract [16], to 4.4% using *Acacia mollissima* Willd. bark extract [16], and to 3.96%–6.37% using Lignamon process [25]. *Acacia mangium* extract was even reported to decrease the mass loss caused by termites from 27.24% to 20.50%–25.18% [16]. Different explanations are attributed to the improved decay resistance of ammonia-treated wood. Simple carbohydrates are

altered by ammonia gas, which are necessary for certain fungi. There is an inhibition of fungal enzymes through substrate modification, while there is an additional formation of toxic compounds as a result of ammonia treatment [48]. The reaction of cell wall components with ammonia is supported by FTIR measurements through the reduction of peaks related to hemicelluloses [46,49]. Besides the ammonolysis of hemicelluloses, the modification of lignin contributes to the improved decay resistance through the alteration of the nutrition source of wood rotting fungi [50]. High presence of bound nitrogen at higher concentrations in wood may inhibit fungal decomposition in basidiomycetes as well [49]. Mechanical properties showed moderate changes. Brinell hardness generally decreased after treatment, particularly in hornbeam, while compression strength remained relatively stable or slightly increased in several species. Therefore, the modified wood is not more suitable for applications where high surface hardness or enhanced mechanical performance is required. Cermak and Dejmal also reported only a slight increase in the hardness of heartwood treated with ammonia fuming [45].

Beech and hornbeam can be used for flooring in the same way. However, the potential application range of the treated material cannot be expanded toward high-performance structural uses. Instead, its primary value lies in improving durability and aesthetic properties in applications where mechanical demands are moderate, such as interior cladding, furniture, joinery, wall panels, and decorative wood products.

5. Conclusions

Overall, the results suggest that bark extract impregnation combined with ammonia fuming is a promising bio-based wood modification approach that can significantly improve fungal durability while simultaneously modifying color and influencing dimensional stability. However, the effects vary among wood species and between extract types. Future studies should evaluate the long-term durability of the treated wood under outdoor conditions, particularly in soil contact, to verify whether the improved fungal resistance observed in laboratory tests is maintained in natural environments. Additional mechanical properties, especially bending strength and modulus of elasticity, should also be investigated to better understand the structural performance of the modified wood. Outdoor weathering tests are recommended to assess the photostability and long-term color stability of the treated material. Furthermore, experiments with larger sample dimensions and cross-sections would help evaluate treatment penetration and the feasibility of scaling up the process for practical applications.

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