

Article

The Weathering of the Beech and Spruce Wood Impregnated with Pigmented Linseed Oil

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Abstract: This research aimed to examine the effects of a deep impregnation technique (Royal process) and surface coating using a linseed oil-based product, enhanced with small amounts of brown and grey pigments, on the natural and artificial weathering of wood. The treated and reference samples underwent natural weathering for five years and artificial weathering for 1900 h. Changes in color and surface roughness were assessed during weathering. For the artificially weathered samples, liquid water absorption was measured both before and after exposure. The impregnated and coated samples gradually lost their brown color, turning grey over time. More pronounced differences were observed during natural weathering, with the coated samples showing greater structural changes on the wood surface. In contrast, impregnated samples slowed down structural alterations compared to the reference samples. Both treatments effectively reduced water absorption before weathering, although this effect diminished after exposure. The treatments did not significantly impact the fire resistance of spruce and beechwood.

Keywords: beech; spruce; linseed oil; atmospheric corrosion; irradiation; Royal process; color; roughness; water absorption; hygroscopicity



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1. Introduction

Traditionally, wood protection has relied for centuries on coatings made from linseed oil, either as pigmented coatings or as a clear, hydrophobic treatment [1]. In the past, linseed oil paints often contained additives such as lead white, turpentine, Japan drier, and cobalt or manganese driers, along with earth pigments for color and opacity. These additives influenced the paint's viscosity, durability, drying time, and resistance to biological decay [2]. With growing environmental awareness, there has been a resurgence of linseed oil paint, a centuries-old method for treating wood surfaces, which has gained renewed popularity in the market.

Research in this field continues to develop new and more effective formulations and treatment approaches, such as epoxidized linseed oil or its modification through uretining and silylation reactions or with metal-containing biocides [3]. Linseed oil is one of the nonbonded, leachable impregnating agents, valued for its eco-friendly, non-toxic properties, wide availability, water resistance, and low cost [4].

Generally, surface treatment with linseed oil enhances surface hydrophobicity [5,6]; however, no significant effect on water vapor permeability has been observed [7–9]. Linseed oil impregnation is considered a water-repellent rather than a dimension-stabilizing treatment, as it only affects the rates but not the final values of moisture sorption or swelling [8].

As a drying oil, linseed oil can be applied to wood using conventional impregnation methods, where it subsequently blocks the cell lumens and forms a stable layer on the pore surfaces. The base molecules of linseed oil are considered too large and hydrophobic to

penetrate the cell wall during wood impregnation. Instead, they fill cavities such as cell lumens and cracks formed during the initial drying process [10].

The “Royal treatment” combines the biocide protection and the water-repellent effects of oil-based impregnation for improved resistance to exterior conditions. Several studies have indicated that wood impregnation with plant oils alone does not enhance durability against wood-rotting fungi unless high retention is used [6,11,12]. Due to its water-repellent effect, linseed oil can improve the hydrophobicity of wood surfaces and reduce the leaching of preservative compounds in contact with water [13,14].

During the weathering of wood, solar radiation plays a significant role by causing the photodegradation of the surface layer, primarily due to the photon energy it delivers. Lignin, which absorbs ultraviolet light, is particularly sensitive to decomposition. Besides solar radiation, water also plays a key role in wood weathering. Dimensional changes caused by constant wetting and drying of the wood create surface stresses, leading to checking and warping [15]. Photodegraded lignin and hemicellulose products are leached out from weathered surfaces.

Weathered wood changes color very rapidly. Photodegradation initially darkens the wood, making it yellow or brown due to the accumulation of photodegraded lignin compounds. Wood exposed outdoors can turn grey within six months, as hydrophilic photodegradation products are leached out, and the cellulose-rich surface layers are colonized by dark-colored staining fungi. Not only does the color change, but the wood’s surface structure also degrades. Wind, windblown particles, dust, and atmospheric pollutants act as abrasives, further eroding the wood surface. The weathered surface is characterized by macroscopic checks, often occurring at growth ring boundaries and the interfaces between rays and other longitudinal elements [16].

Linseed oil treatment can limit wood wetting and reduce the effects of leaching by rain in outdoor applications, but it provides only slight or no protection against photodegradation [3]. For instance, [14] demonstrated that *Pinus sylvestris* impregnated with linseed oil experienced reduced cracking and surface discoloration during natural weathering, but noticeable greying still occurred after one year of outdoor exposure.

In fact, linseed oil itself, particularly its polyunsaturated fatty acids, is unstable when exposed to light, as was demonstrated by [17,18].

This study was designed to explore (a) the effects of a combined impregnation process and (b) a surface coating with pigmented linseed oil on the natural and artificial weathering of wood. Impregnation treatment was chosen because it can significantly enhance the durability, dimensional stability, and weathering resistance of the wood material, thereby extending its service life and reducing its maintenance frequency. This method also preserves the wood’s aesthetic appearance and provides an eco-friendly alternative to traditional toxic preservatives, making it a versatile and sustainable solution for various wood products and applications.

A secondary aim of this research was to investigate surface discoloration, roughness, and water uptake, along with the fire resistance of the treated surfaces. Additionally, the grey-colored impregnation was used to imitate the appearance of weathered wood, with the goal of ensuring color stability throughout the exposure period. These properties were selected to evaluate the performance of the tested materials because they provide insight into how quickly wood absorbs moisture from the atmosphere and rainfall and how much, which can lead to warping, cracking, fungal growth, and increased surface roughness—factors that accelerate the degradation process.

These indicators influence the wood’s performance by increasing the risk of dimensional changes, deformations, degradation, reduced structural integrity, susceptibility to biological attack, decreased strength properties, and compromised aesthetic qualities. Monitoring these indicators can help ensure better resistance to weathering and prolong the wood’s lifespan.

2. Materials and Methods

2.1. Impregnation

For this experiment, European beech (*Fagus sylvatica* L.) and Norway spruce (*Picea abies* L. Karst.) were used, with average air-dry densities (at 12% moisture content) of 730 and 495 kg/m³, respectively. The schematic representation of this experiment can be observed in Figure S1.

Four equal sets of each wood species were selected. One set was left uncoated as a reference (denoted as XR), while the other sets were impregnated or coated. The letter “X” in the marking represents the wood species, with “S” representing spruce and “B” representing beech.

The selected samples were straight-grained, free from knots, cracks, and other defects, with the growth rings inclined to the face between 0° and 45°, according to EN 927-6:2019 [19]. The cross sections of all samples were sealed with several layers of two-component epoxy resin (Epolex S1300/Epolex S7300) to prevent the uptake of impregnation solution in the longitudinal direction.

The impregnation process was designed based on the requirements of a company interested in the Royal treatment of spruce for outdoor use, which currently impregnates wood with a copper-based water-borne preservative. The Royal treatment combines biocide protection with water repellency, and the surplus moisture content is removed during the vacuum impregnation phase in hot oil. After storage at 20 °C and 65% relative humidity (RH), the samples were weighed and first impregnated with a water-based biocide (Bochemit Forte Profi based on Cu₂(OH)₂CO₃, 4% solution) to meet the requirements for Use Class 3 according to EN 335: 2013 [20].

Due to the differing permeability of the two wood species, spruce samples were pressure-treated (45 min at 800 kPa) in a laboratory vacuum–pressure impregnation plant (J. Hradecký spol. s r.o., CZ, vessel volume 50 L). The beech samples were impregnated only by dipping for 15 min because the goal was not a full-volume impregnation.

Different methods were used for each species because of their distinct anatomical structures, which affected the ease with which they absorbed treatment agents. For beech, dipping is sufficient due to its diffuse-porous structure, high water uptake, and permeability [21]. Spruce, however, requires vacuum–pressure impregnation to allow for deeper penetration and more effective protection.

As the goal of this research was to imitate weathered wood with stable properties and without quality deficiencies, a biocide was applied before oil impregnation. Oil impregnation alone would not have provided sufficient protection against insects and fungal decay. This creates a synergistic effect, addressing both biotic and abiotic degradation factors.

The subsequent impregnation of the still-wet samples with hot oil (ProcessOil 203 Ready-To-Use, Koppers Sweden AB, Helsingborg, Sweden) was performed under vacuum and at controlled temperatures according to the process parameters shown in Table 1.

Two types of oil pigmentation were used: grey (XIG) and brown (XIB). Brown-colored oil was also used for surface coating by brushing in two layers without previous biocide treatment (XCB).

Table 1. Oil impregnation process parameters (parameter marked with “–” indicates no control).

Process Phase	Time for Spruce (min)	Time for Beech (min)	Absolute Pressure (kPa)	Temperature (°C)
Initial vacuum	1	1	50	–
Filling	5	5	–	–
Heating	60	60	–	75
Drying 1	90	15	50	80
Drying 2	90	–	40	80
Drying 3	30	–	30	80
Drying 4	30	–	20	80
Air equalization	1	1	75	–

Table 1. Cont.

Process Phase	Time for Spruce (min)	Time for Beech (min)	Absolute Pressure (kPa)	Temperature (°C)
Emptying	5	5	–	–
Final vacuum	15	5	50	–
Air equalization	1	1	–	–
Final emptying	5	5	–	–
Total	333	98	–	–

After each step of impregnation, the samples were weighed to determine the retention of the impregnation agent (R; kg/m³), although oil uptake was only indicative due to concurrent water loss during the drying phases. After impregnation, all samples were cooled and stored for several weeks at standard climate (20 °C, 65% RH) before testing, allowing them to reach constant mass and equilibrium moisture content.

2.2. Natural and Artificial Weathering

The natural weathering test (NW) began on 1 July 2019 and lasted for five years. Samples measuring 375 mm × 100 mm × 20 mm (length × width × thickness) were exposed horizontally on racks inclined at 45° and facing south in Útěchov (465 m a.s.l., 49°17′29.9″ N 16°38′08.4″ E). Surface color measurements and visual evaluations were performed annually. The samples were conditioned indoors for 24 h before measurements to stabilize their surface moisture content. Climate data (air temperature, RH, global irradiance, and precipitation) were continuously recorded and are presented in Supplementary Material, Figure S2.

This region experiences warm, wet summers (May–September), with average temperatures between 11 °C and 21 °C, sometimes reaching up to 35 °C, and monthly precipitation between 12 mm and 193 mm. Winters (October–April) are drier, with monthly precipitation between 6 mm and 115 mm and mean temperatures ranging from −1 °C to 12 °C, with a maximum of 25 °C.

During the test period, the average annual rainfall was around 615 mm, ranging from 398 to 878 mm. The average annual temperature was 10 °C, ranging from 8.5 °C to 11.8 °C. Relative humidity ranged from 13% to 100%. The average annual global irradiance was 4.3 MW/m², with a daily average of 11.7 kW/m².

In parallel to the natural weathering test, an artificial weathering test (AW) was conducted in a climate test chamber (CL-30/600BF Sun, CTS GmbH, Hechingen, Germany) using a 2500 W metal halide lamp (HMI® 2500 W/DXS, OSRAM, Vienna, Austria) for sunlight simulation. The temperature of the black panel was 63 ± 3 °C, and the total light irradiance was approximately 1060 W/m². Each cycle lasted 2 h, consisting of a 102-minute dry phase, followed by an 18-minute wet phase with water spray. RH in the chamber was not controlled but remained above 85% during the dry phase.

The exposed sides of the samples (150 mm × 74 mm × 20 mm) were scanned, and color measurements were taken before exposure and at intervals of 4, 14, 24, 50, 100, 200, 300, 400, 500, 900, and 1900 h.

2.3. Color Measurement

Surface color was measured using Konica Minolta CM-2500 spectrophotometer (Konica Minolta, Inc., Tokyo, Japan; d/8 measuring geometry, 10° standard observer, D65 standard illuminant, measurement area 8 mm, CIEL*a*b*, SCE) at wavelengths between 360 nm and 740 nm (10 nm resolution). Color coordinates include lightness (*L**, ranging from 0 for black to 100 for diffuse white), redness/greenness (*a**, negative values indicating green and positive values red), and yellowness/blueness (*b**, negative values indicating blue and positive values yellow).

The overall color change (ΔE^*) was calculated using Equation (1):

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}}, \quad (1)$$

where ΔL^* , Δa^* , and Δb^* represent the value differences before exposure and at the end of the weathering interval. Measurements were taken on three spots on each sample type (natural and artificial weathering), and the final color coordinates were the average of these points.

The color difference in treated samples was compared to the actual color of the reference sample (ΔE_{year}^*) to see if the tested treatments resulted in the same color as weathered wood. It was calculated using Equation (2):

$$\Delta E_{year}^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}}, \quad (2)$$

where ΔL^* , Δa^* , and Δb^* represent the value differences between the reference and treated sample at a given year.

2.4. Roughness

The surface roughness was measured before and after five years of exposure using a 3D Profiler KEYENCE VR 6000 (KEYENCE, Osaka, Japan), with a resolution of 0.1 μm on the Z axis. Measurements were taken perpendicular to the wood fibers on each sample from five areas of 10 \times 12 mm² using a 50 \times magnification camera using multiline roughness with 11 lines and 80-pixel intervals. The arithmetic mean height (R_a) and the maximum height (R_z) were determined.

2.5. Hygroscopicity

Conditioned impregnated samples measuring 74 \times 70 \times 20 mm³ (length \times width \times thickness) with sealed cross sections were placed in a desiccator containing a saturated solution of K₂SO₄ (RH 97%) at 20 °C. The mass of each sample was recorded regularly for four months. The samples were then kept at standard climate at 20 °C and 65% RH to monitor the rate and degree of sorption and desorption. Mass changes were expressed as the mass difference in grams before and after exposure due to the different densities of the materials after impregnation.

2.6. Liquid Water Absorption

The absorption test was performed using the free-floating method according to EN 927-5:2007 [22]. The pre-leaching procedure consisted of a repeated cycle of 10-h-long floating followed by conditioning at 20 °C and 65% RH until reaching the equilibrium moisture content. Samples were of the same dimension as those used for artificial weathering. They were stored at standard climate (20 °C, 65%). After weighing, they were placed on the surface of the water and removed regularly (after 2, 4, 6, 8, 10 h). Excess water was gently wiped off with a paper towel before weighing. The same procedure was repeated with samples after 1900 h of artificial weathering. Water absorption was determined as the mass increased (g) per wetted area and calculated using Equation (3):

$$\Delta m = (m_w - m_i) / A, \quad (3)$$

where m_w is the mass (g) of the wet sample; m_i is the initial mass (g) of the sample before submersion, and A is the wetted area (m²).

2.7. Fire Resistance

The tests were conducted using equipment compliant with ISO 11925-2:2020 [23]. (single-flame source test) under prescribed conditions. The flame was applied to the surface of the panels for 30 s. The surface of the samples measured 90 mm \times 250 mm (width \times length). The flame tip was monitored to see if it reached or exceeded 150 mm above the flame application point, and if it did, the time it took to reach this height was recorded. Sample surfaces were scanned after the test.

2.8. Statistical Analysis

Statistical analysis was performed using Statistica software (version 14.0.0.15, TIBCO Software Inc.). Factorial analysis of variance (ANOVA) combined with Tukey's HSD test was conducted, and differences were considered significant at $p < 0.05$.

3. Results

3.1. Impregnation

The retention and spreading rate of biocide, grey oil, and brown oil were determined to assess the possible performance of these treatments and evaluate how much was absorbed by the tested wood species. The results can be found in Table 2.

Table 2. Retention of impregnation agents and spreading rate of oil for spruce and beech samples.

Sample Size (mm)	Biocide Retention (kg/m ³)		Grey Oil Retention (kg/m ³)		Brown Oil Retention (kg/m ³)		Brown Oil Spread Rate (g/m ²)	
	Spruce	Beech	Spruce	Beech	Spruce	Beech	Spruce	Beech
70 × 74 × 20	92.8	30.3	14.1	56.6	13.7	50.0	58.6	53.4
150 × 74 × 20	93.5	33.3	20.6	89.4	17.4	87.6	60.4	56.9
375 × 100 × 20	85.5	34.1	36.3	140.2	23.8	93.5	53.6	64.9

The retention of both oils shows a marked difference between spruce and beech samples. However, it is important to point out that these values are only indicative due to the prior impregnation with the water-based biocide and subsequent loss of water during the vacuum impregnation process, which affected the results. Beech samples exhibited significantly higher retention rates, particularly in the larger sample sizes. This can be attributed to its better impregnability (Class 1, EN 350:2017 [24]) compared to spruce (Class 3–4). The higher retention in the longest samples, especially in beech, is also linked to its good permeability and fiber orientation in the longitudinal direction, allowing the oil to penetrate a substantial part of the sample volume even from the longitudinal surface (Figure 1). The oil spreading rate for brushing was comparable not only between different sample sizes but also between the wood species.

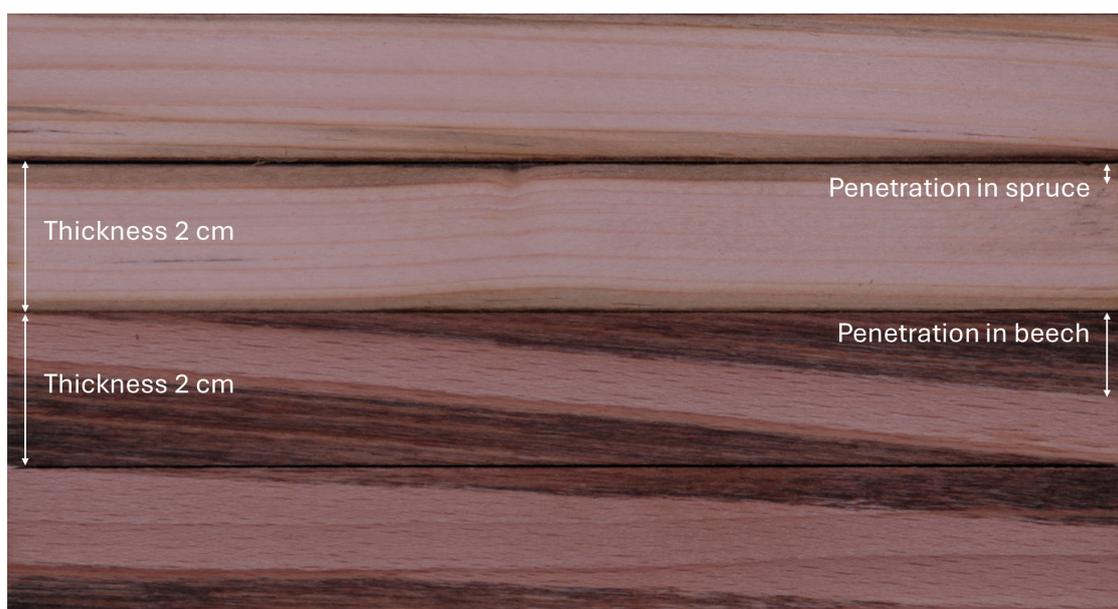


Figure 1. The depth of oil penetration in spruce (**above**) is smaller than that of beech (**below**) after impregnation. The figure shows longitudinal sections of four samples with a 20 mm thickness. The wood appears darker where the oil has penetrated.

3.2. Natural and Artificial Weathering

Scans of weathered samples are shown in Figure 2. During weathering, both impregnated and coated samples lost their brown hue, and the wood turned grey. Despite a significant color difference at the start of the test, the distinction between the reference and treated samples became less noticeable to the naked eye over time. While all groups' colors turned grey, this change was most distinct in the reference samples, even after the first year. Due to the dark initial color being close to the final state of the weathered surface, the discoloration was less visible in the impregnated samples, particularly in the SIG samples.

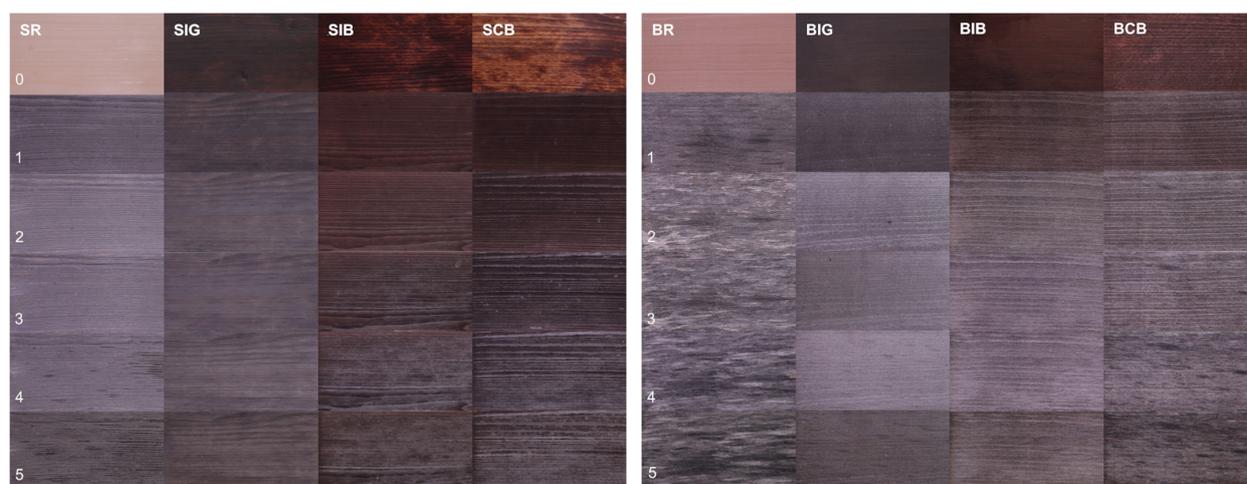


Figure 2. Surface discoloration of spruce (S, left) and beech (B, right) samples before and during exposure to natural weathering (from top to bottom 0 to 5 years). R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

Treated samples exhibited changes similar to those of the reference sample; on the other hand, more significant changes were found in the wood surface structure compared to the impregnated samples. Many deep and wide cracks formed on the exposed surfaces, though their location varied depending on the wood species. In beech, the cracks were primarily located where the wide pith rays were present, while in spruce, the cracks typically occurred along the growth ring boundaries. Additionally, the relief texture due to erosion and mold growth was more distinct in untreated samples. Fewer and smaller cracks were apparent in oil-impregnated wood.

Based on a visual examination of artificially weathered samples (Figure 3), it is evident that the surface lightness of both the reference (BR) and oil-treated wood (BCB, BIB) increased over time due to artificial weathering. After the first 24 h, the color became more vivid. Compared to natural weathering, the reference samples were significantly lighter than the treated samples, and their visual appearance was different.

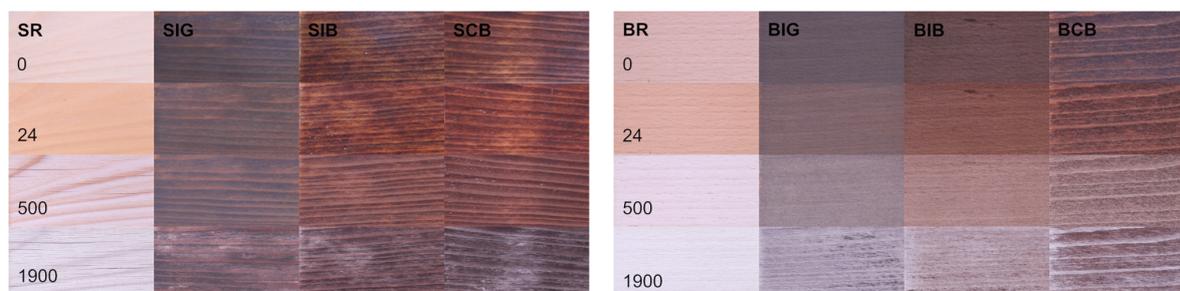


Figure 3. Surface discoloration of spruce (S) and beech (B) samples before and during irradiation (0–24–500–1900 h). R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

3.3. Color Measurement

The color measurement results of naturally weathered samples can be found in Table S1 and Figure 4.

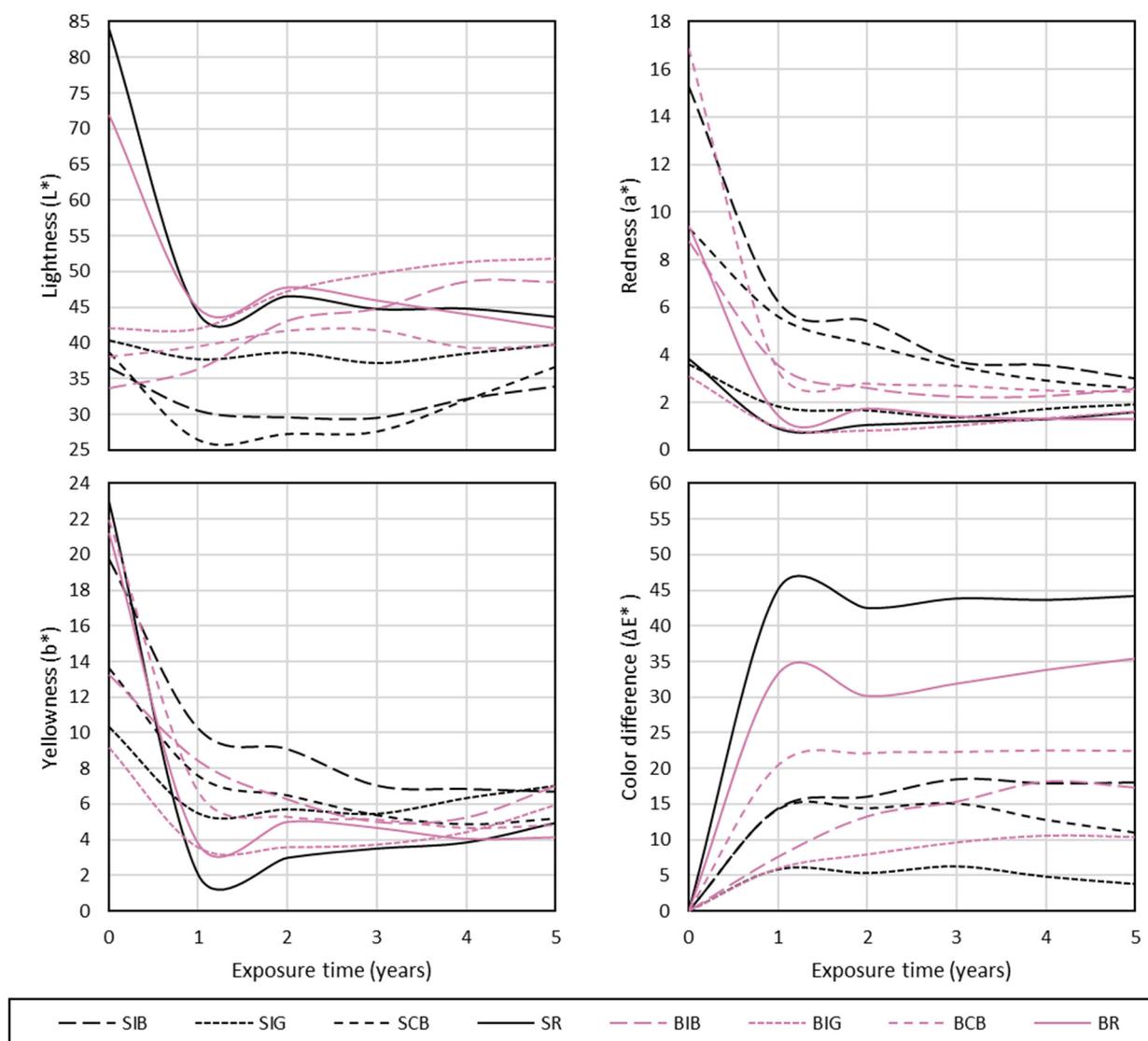


Figure 4. Changes in lightness (L^*), redness (a^*), yellowness (b^*), and total color difference (ΔE^*) of spruce (S) and beech (B) samples during a 5-year weathering exposure. R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

For spruce samples, the L^* values show a general decrease over time, particularly in the reference sample (SR), which dropped from 83.9 to 43.7, indicating significant darkening. The impregnated spruce with brown oil (SIB) also darkened but to a lesser extent than the reference. Impregnation with grey oil (SIG) showed a relatively stable L^* value after an initial drop.

For beech samples, the reference (BR) exhibited a sharp decline in L^* from 71.9 to 42.0, similar to spruce. Beech impregnated with brown oil (BIB) initially darkened but then lightened over time. The grey oil-treated beech (BIG) remained relatively stable, similar to its spruce counterpart.

Both spruce and beech reference samples (SR, BR) showed a reduction in a^* , indicating a loss of red color over time. Spruce samples treated with brown oil (SIB, SCB) exhibited a

more significant decrease in a^* than those impregnated with grey oil (SIG). Beech samples also had more stable a^* values when impregnated with grey oil (BIG).

When examining the change in b^* values, similar trends can be observed. The reference samples (SR, BR) showed a significant reduction during greying. Samples treated with brown oil (SIB, SCB, BIB, BCB) showed a marked reduction in b^* values, while impregnation with grey oil (SIG, BIG) exhibited a slower rate of decline in b^* . The shifts in a^* and b^* values can be observed in Figure S3.

The ΔE^* values were the highest for the reference samples (SR, BR). Samples treated with grey oil (SIG, BIG) had the lowest ΔE^* values, indicating that this treatment provided better color stability during weathering exposure. The visual analysis results were confirmed by these color measurements.

The colors can also be compared to the reference at each state. This reveals that while initially, the treatments resulted in distinct color differences from the untreated reference samples, these differences diminished significantly over time due to natural weathering. After one year, the color difference between the reference and treated samples decreased dramatically (Figure 5). The color difference was the lowest in beech samples, in some cases decreasing below 2, being nearly indistinguishable to the naked eye. Spruce samples did not decrease below 4, being similar to weathered wood but having distinctive differences [25]. Impregnation and coating with brown oil retained the color better in spruce, imitating weathered wood less.

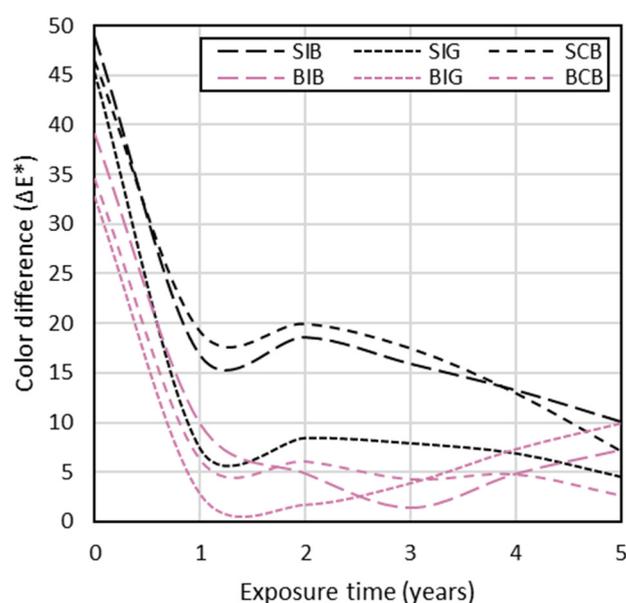


Figure 5. Color difference in treated samples (ΔE_{year}^*) compared to the reference samples of spruce (S) and beech (B) during each year of natural weathering exposure. The actual color difference is compared to the reference sample's color for the respective year. IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

By the end of the five-year period, all treated samples exhibited a color difference compared to the reference, with a maximum color difference of 10. This indicates that they grey similarly to untreated wood. Given their relatively stable color during exposure (e.g., SIG, BIG), these treatments are able to imitate the color of weathered wood quite well.

The color measurement results of artificially weathered samples can be found in Table S2 and Figure 6. The lightness (L^*) of both reference groups showed a slight decrease at the beginning of irradiation. For spruce, this reduction stabilized after 4–100 h, while for beech, it was shorter, stabilizing within 4–50 h. L^* values then increased and almost stabilized after 1000 h. SIG samples exhibited a relatively stable L^* value with minimal change throughout the irradiation period. When comparing the initial L^* to the final L^* (after 1900 h), spruce samples showed no remarkable change ($\Delta L^* < 2$), although there were

greater differences during the artificial weathering process. Beech samples exhibited larger changes in L^* (12.7–23.0) during photobleaching, with the most remarkable changes seen in brown-pigmented impregnated samples for both species.

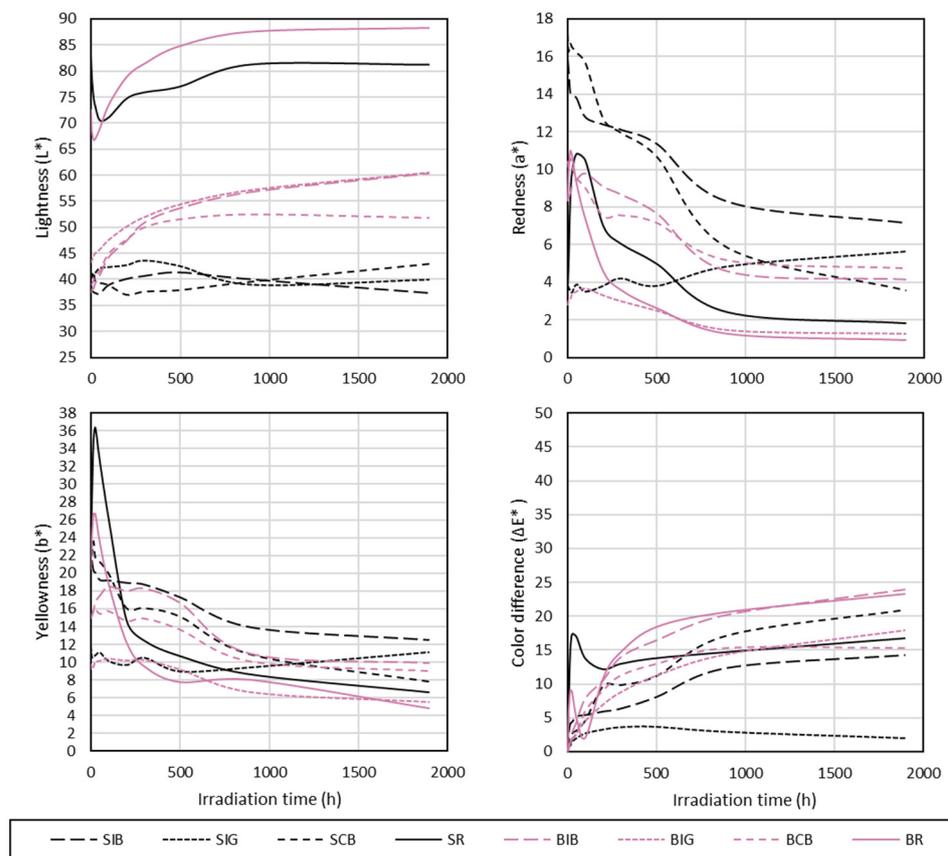


Figure 6. Changes in lightness (L^*), redness (a^*), yellowness (b^*), and total color difference (ΔE^*) of spruce (S) and beech (B) samples during a 1900 h of artificial weathering. R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

The redness (a^*) of the reference samples initially increased during the first 14–50 h, rising by 7.1 in spruce and 2.7 in beech. The treated samples did not show this peak, or only to a small extent, before exhibiting a decline in a^* . The most stable a^* values were found in the grey oil-impregnated samples for both species.

Similarly, the yellowness (b^*) of the reference samples showed an initial increase during the first 24 h, with a rise of 13.1 in spruce and 6.3 in beech. The treated samples had smaller peaks before also declining in b^* . The most stable b^* values were also observed in grey oil-impregnated samples. The initial increase in a^* and b^* can be observed in a horseshoe-shaped curve (Figure S4).

The ΔE^* values indicate that grey oil impregnation provides the highest color stability among the tested treatments. Reference samples showed significant color changes, especially in the early stages of irradiation, with the most noticeable changes in the first 14 h.

3.4. Roughness

Surface roughness is an important wood performance parameter that influences its aesthetics as well as the adhesion of coatings and adhesives. It also plays a significant role in the durability of wood, as higher roughness can promote the growth of mildew and wood-rotting fungi while also accelerating water uptake into the internal structure of the wood.

Both the arithmetic mean height (R_a) and the maximum height (R_z) increased significantly in treated samples compared to reference samples. This increase in roughness parameters was more pronounced in spruce wood (Table 3). An increase in surface roughness was also

observed in all investigated groups after five years of exposure, which was statistically significant. The reference samples exhibited the largest difference in roughness after weathering, with the mean surface roughness increasing by 863% for spruce and 979% for beech.

Table 3. Roughness parameters of surface before and after 5 years of natural weathering. S—spruce; B—beech; R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil; R_a —arithmetic mean height; R_z —maximum height; CV—coefficient of variation.

	R_a (μm)	Non-Exposed			Exposed			
		CV (%)	R_z (μm)	CV (%)	R_a (μm)	CV (%)	R_z (μm)	CV (%)
SR	12.0	15.9	90.4	24.8	116.0	19.0	866.5	23.6
SIG	19.7	23.2	126.5	19.3	57.7	17.8	354.8	17.5
SIB	16.2	13.6	121.3	19.1	59.2	17.4	415.7	19.1
SCB	16.4	13.0	136.3	29.9	71.3	20.0	560.0	24.0
BR	8.0	10.7	63.5	12.7	86.2	26.3	616.0	28.2
BIG	9.0	12.3	71.8	12.3	38.4	11.4	317.0	16.8
BIB	11.5	13.6	81.7	10.0	33.9	16.3	274.1	23.6
BCB	8.8	8.6	65.3	10.5	52.0	24.7	377.9	24.7

The results indicate that deep impregnation is able to protect wood from the surface roughness changes caused by weathering factors. By the end of the exposure period, the roughness was significantly lower, both in terms of measured parameters and visual evaluation, compared to reference samples (Figures S5 and S6). Even the simple brushing led to a statistically significant decrease in surface roughness for both wood species, although the effect was not as pronounced as with impregnation.

3.5. Hygroscopicity

For outdoor applications, impregnation and coating can modify wood–water interactions, affecting the wood’s behavior under changing humidity and, consequently, its service life. It is evident that oil-based impregnation slows down the sorption process, which is particularly noticeable during long-term exposure (Figure 7). Beech wood absorbed more water (6.2–6.5 g) under the same conditions than spruce wood (4.6–5.0 g).

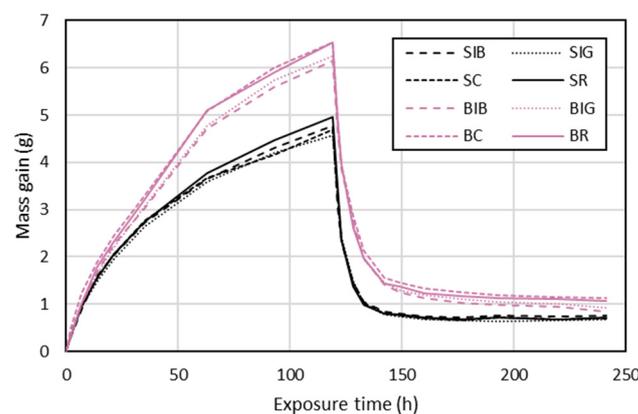


Figure 7. Mass change during water sorption and desorption process of spruce (S) and beech (B) samples. R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

The sorption process stopped after 119 days, with the average difference between the reference samples and the treated samples being up to 0.4 g. This indicates that the difference in moisture content did not exceed 0.5%. During the subsequent desorption phase, water loss occurred more rapidly in the beginning, and the samples’ mass settled at a slightly higher value compared to their original mass before exposure. The increase in

mass was approximately 1.2% for both wood species. All spruce sets had the same average mass when equilibrium was achieved.

3.6. Liquid Water Absorption

The reference samples for both spruce and beech show the highest water uptake (Figure 8), given that untreated wood is highly susceptible to water absorption. After ten hours, samples impregnated with grey oil had lower water uptake compared to the reference samples by 46% and 82%, samples impregnated with brown oil by 47% and 81%, and samples coated with brown oil by 41% and 75% for spruce and beech, respectively. The water uptake was the lowest for impregnated samples, which exhibited the highest hydrophobicity for the tested wood species.

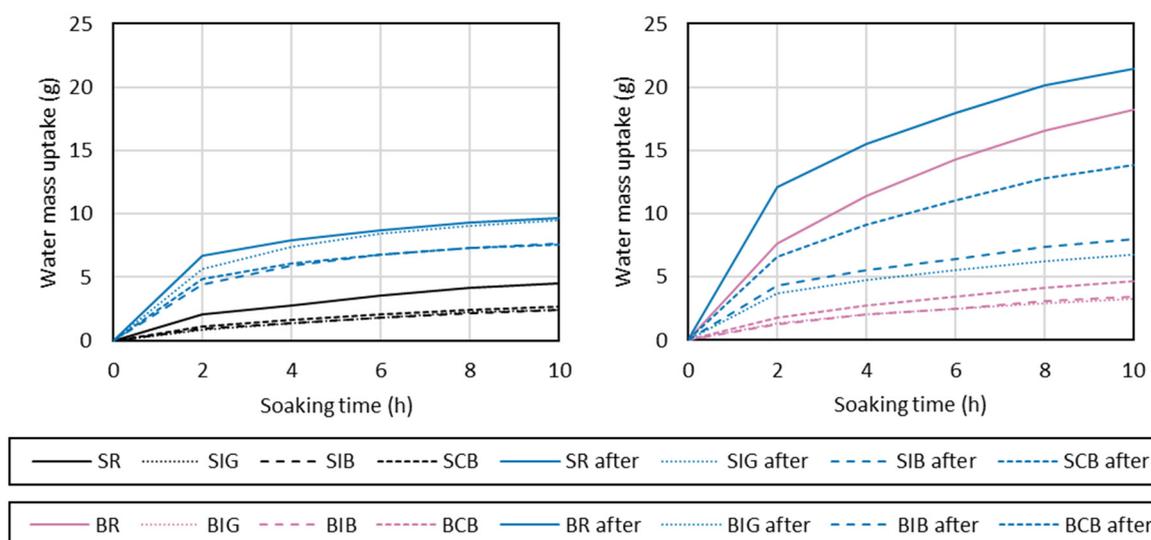


Figure 8. Water uptake of spruce (S) and beech (B) samples before and after artificial weathering. R—reference; IG—impregnated with grey oil; IB—impregnated with brown oil; CB—coated by brown oil.

After 1900 h of artificial weathering, all samples demonstrated an increased water uptake compared to their pre-weathered counterparts. The increase in water uptake was more pronounced in the treated samples compared to the reference samples. The mass of the reference samples increased by 5.1 g and 3.2 g, while that of samples impregnated with grey oil increased by 7.0 g and 3.5 g, samples impregnated with brown oil by 5.2 g and 4.5 g, and samples coated with brown oil by 4.9 g and 9.2 g for spruce and beech, respectively. The BCB samples showed distinctively higher water absorption compared to oil-impregnated beech samples.

Weathered samples impregnated with grey oil had lower water uptake after ten hours of soaking compared to the reference samples by 2% and 69%, samples impregnated with brown oil by 21% and 63%, and samples coated with brown oil by 21% and 35% for spruce and beech, respectively.

3.7. Fire Resistance

The aim of this test was to evaluate whether oil impregnation or coating decreased the fire resistance of wood, as these materials could be utilized as facades, among others. The measurement results can be found in Table 4. Figure S7 shows the scans of selected samples before and after testing.

In the case of the spruce samples, sustained burning was observed after the removal of the ignition flame during the observation period. However, the beech samples did not ignite. During the ignition and inspection period, the flames did not reach the 150 mm reference limit for any of the samples, and no burning particles or droplets were observed.

The burn patterns on the surfaces were uniform; the samples did not deform, and there was no significant smoke production.

Table 4. Fire resistance results of reference (R), coated (CB), and oil-impregnated (IB and IG) specimens of spruce (S) and beech (B).

	Flame Application Time (s)	Did It Ignite?	Did It Pass the 150 mm Limit?	Flame Length (mm)	Mass Loss (g)
SR	30	yes	no	74.8	0.224
SIG	30	yes	no	70.4	0.168
SIB	30	yes	no	64.4	0.194
SCB	30	yes	no	53.2	0.164
BR	30	no	no	34.8	0.136
BIG	30	no	no	26.4	0.220
BIB	30	no	no	32.8	0.095
BCB	30	no	no	35.6	0.148

4. Discussion

According to the weather data, mildew was anticipated to develop during the summer months. In dry seasons, degradation is more significantly influenced by cracking, insect activity, and UV radiation, while in wet seasons, fungal growth and leaching intensify. Fungi require oxygen, moderate temperatures (25–35 °C), optimal air humidity (35%–50%), nutrients, specific pH levels, vitamins, and minerals, but water is the key factor that drives their growth [26]. Water directly influences several critical physiological processes of fungi, like their metabolic activities and energy production, enzyme function and catalyzation, the structural integrity of fungal cells and growth of hyphae, spore germination, reproduction, and growth [26].

Comparing the retention properties and depth of oil in the tested wood species, differences can be found between the agents, as non-polar liquids penetrate the wood by bulk flow and polar liquids by both bulk flow and diffusion through the wood cell wall [27]. Retention and spread rate can vary depending on density, porosity, moisture content, grain structure, cellular structure, wood extractives, and, of course, surface preparation. Although beech is better impregnable than spruce [21], our results indicate that spruce samples consistently retain higher amounts of biocide compared to beech samples across all sizes. This is due to the fact that different impregnation methods were applied to different wood species due to their different permeability. Higher retention values indicate a more effective treatment, ensuring improved properties. While beech retains more brown oil, the distribution of the oil on the surface is relatively uniform across both wood types.

Impregnated wood offers better photostability compared to natural wood, but it is not immune to weathering. The chemicals used in impregnation can leach out over time, reducing their protective efficacy. Additionally, the aesthetic appeal of impregnated wood can be compromised as the surface treatments wear off, necessitating regular maintenance and reapplication of protective coatings. The protective mechanism and durability issues of clear coatings [26], resins, solvents, pigments, additives [28], oil-based varnishes and paints [29], acetylated wood [30], and oil-heat-treated wood [31], among others, have been reviewed in the literature. For untreated wood, the surface lightens, while its redness and yellowness decrease during weather exposure because of the degradation of chromophores in wood [26,32].

In the case of linseed oil, the oil substance degrades under UV light, and it has a low amount of pigment, which does not provide enough photostability like, e.g., opaque coatings. This observation aligns with the findings of [29], who noted rapid degradation of linseed oil under light exposure due to photo-oxidation processes. Degradation products have been leached out during rain. Previous studies [30–33] suggest that while surface treatments can initially alter the color of wood, prolonged exposure to environmental

factors such as UV light, rain, and temperature variations ultimately leads to a uniform greying of both treated and untreated wood surfaces, which can be further deteriorated by wind, dust, fungi, and insect attack.

There is a growing interest in the aesthetic value of weathered wood. Designers and architects increasingly incorporate naturally weathered wood into their projects for its unique, rustic appearance. This trend has led to the development of controlled weathering techniques that accelerate the aging process to achieve the desired look without compromising the wood's structural integrity. In this test, the initial reduction in L^* and increment of a^* and b^* were probably the same for weathered samples at the beginning of the test, but the color was only measured after one year. The color measurement data of artificially weathered samples could be linked to the weathered samples to see approximately to what weathering exposure time irradiation for 1900 h corresponds. In [30], the color after 200-h-long xenon lamp irradiation corresponded to the color acquired after one-month-long weather exposure. The initial reduction in L^* and increment of a^* and b^* is a common phenomenon that was observed in natural wood [34], as well as in acetylated [30], oil-heat-treated [31], steamed [35], and impregnated wood [36].

The surface roughness slightly increased after treatment, which was related to the wetting of the surface associated with subsequent swelling and shrinkage with residual deformation, increasing the initial surface roughness [37]. The roughness of untreated wood increased significantly after 5 years of outdoor exposure, with many deep and wide cracks occurring on the exposed surface. All wood species show a systematic trend to higher surface roughness values with prolonged natural weathering progress [38]. It has already been explained in many studies that the cyclic wetting and drying of wood exposed outdoors lead to rough surfaces caused by raised grain [39]. The wood interacts with outdoor conditions in complex ways, resulting in the gradual breakdown of wood constituents and the weakening of fiber bonds. The constant stress in surface layers due to moisture fluctuation leads to the failure of weakened fiber bonds and the occurrence of surface erosion and cracks [38]. The tested treatments reduced the rate of degradation and roughing to some extent, having the most favorable results for the grey oil-impregnated samples. The treatment with linseed oil acts as a water repellent, thereby limiting the influence of water on weathered wood and also the emerging stresses in the superficial layer. Thanks to this, only fewer and smaller cracks are present on the surface of the treated wood. Also, for artificially weathered Scots pine treated with different vegetable seed oils, [40] presented that treated samples developed fewer and smaller cracks compared to the untreated samples. In our study, the coated samples showed a considerably lower protection effect based on roughness changes due to lower oil retention and penetration depth. The low pigment concentration in oil is able to protect the surface against photodegradation and wood erosion and slow down the whole process of weathering [41]. Semitransparent stains are penetrating wood finishes that allow for partial penetration of light into the wood, thereby causing photodegradation of the wood layer under the stain. The consequence is the failure of the wood and finish, which is visible on coated surfaces (SCB, BCB).

The hygroscopicity of treated samples was similar to that of reference samples in both wood species. It is known that plant oils are non-polar substances with large molecules that limit the penetration into the cell wall [4,10]. Considering that the cell wall is still able to absorb water, the rate of sorption is slowed down. Although the samples were conditioned before the experiment, it cannot be guaranteed that the process of linseed oil autoxidation has been completed. Linseed oil oxidation, which incorporates a cross-linking reaction, involves oxygen consumption and induces increasing sample mass [42]. This mass-increasing phase can take a few days, depending on the linseed oil type [43], but it can be significantly delayed if the thick layer of oil is applied due to the lack of oxygen or if it is held in wet conditions [12]. The effect of oil treatment on the wood sorption rate can be partly covered by the gradual change in the oil mass. The difference in sample mass before and after exposure is apparently due to the sorption hysteresis when the moisture content is not influenced only by the ambient climate but also by the moisture history. The

moisture content is higher during the desorption phase than absorption under the same ambient climate conditions [44]. Obviously, the hygroscopicity, which is based on mass changes, is not a suitable parameter for the evaluation of the drying oil treatment effect on wood behavior if complete cross-linking of the oil is not ensured.

One important property of the performance of treated wood in outdoor conditions is water exclusion efficiency. Wood that stays dry for longer periods is better protected against fungal attacks and the formation of cracks. All treatments reduced water uptake through the longitudinal surface, with the water-repellent effect being more pronounced in beech wood than spruce, likely due to the latter's lower water permeability.

Historically, linseed oil has been commonly used to reduce the effects of water on wood [3]. The hydrophobicity of linseed oil was confirmed by [45], though it was noted that the surfaces took days or even weeks to become fully hydrophobic. A study [6] demonstrated that linseed oil performs well as a water repellent when the transverse side of the wood has short-term contact with water, which aligns with the findings by [46]. However, during long-term water exposure (7 days), the water uptake of linseed oil-treated spruce was comparable to that of untreated control spruce specimens.

Regarding the water uptake of irradiated wood, our results indicate that weathering processes, such as UV radiation, moisture, and temperature fluctuations, degrade the wood surface, leading to increased water absorption [47]. This suggests that while weathering affects all samples, the degradation of oil-based substances and superficial layers might lead to a relatively higher increase in water uptake compared to initially untreated wood. The species-related properties, e.g., depth of impregnation and layer thickness, also influence the water absorption after weathering. Regular maintenance or reapplication of treatments for wood is inevitable for outdoor usage to maintain its protective properties over time.

Based on the single flame source tests, the tested treatments did not significantly influence the ignition or mass loss of spruce and beech wood. The impregnated spruce samples showed slightly lower mass loss and slower flame spreading. It is known that during the linseed oil oxidation in air, sufficient heat is liberated to ignite whatever was used to apply the linseed oil formulation [48]. On the other hand, information about wood fire resistance after the application and autoxidation of linseed oil-based stains and paints is scarce. A study [7] showed that linseed oil-impregnated *Ailanthus* did not show fundamentally different thermal stability compared to the reference. Also, the thermal behavior of clear linseed oil, based on the thermogravimetric profile and its derivative, is comparable to wood degradation. Linseed oil begins to decompose slowly from 100 °C and then sharply from 300 °C to 550 °C [49]. The full cell impregnation of wood, especially in spruce, can play an important role in the fire resistance of the material. The density of the surface layer considerably increased, and at the same time, the volume of available oxygen decreased. The flame spread rate, mass loss rate, and flame height decreased with the increased wood density in densified wood [50]. Considering the similar thermal behavior of both materials, the presence of linseed oil in cell lumens can also affect these parameters in a similar way, especially in low-density spruce wood. The pigment itself can also influence thermal behavior, as observed in coated spruce (SCB), which exhibited the best performance parameters. In spruce, the pigment tends to remain concentrated on the surface due to its relatively impermeable structure. In contrast, in beech, the pigment penetrates more evenly throughout the wood's depth.

5. Conclusions

In this study, spruce and beech wood were treated with pigmented stain based on linseed oil combined with a copper-based biocide using the Royal process. The influence of the treatment on enhancing weathering and fire resistance was evaluated.

The findings indicate that linseed oil treatment significantly affected the color and surface properties of the wood during weathering. Both impregnation and coating provided greater color stability during artificial and natural weathering compared to the reference samples. However, graying, characteristic of weathered wood, was observed on all treated surfaces. The color

stability was, therefore, determined more by the color of the treatment—which, with its parameters, closely resembled weathered wood—than by actual protection against photodegradation. Notably, gray oil impregnation best imitated weathered wood and maintained a stable color throughout the natural weathering process.

Weathering resistance was further evaluated based on changes in surface roughness, water absorbency, and hygroscopicity. All treatments reduced roughness changes caused by weathering, and fewer minor cracks formed on the exposed surfaces compared to the untreated wood. This improvement is attributed to the water-repellent nature of the treatment, which significantly reduced water uptake into the wood and minimized moisture-induced stress in the surface layers, thus limiting crack formation. Even after 1900 h of artificial aging, the water repellency effect was still visible, particularly in oil-impregnated beech wood.

The treatment did not significantly affect the hygroscopicity of the wood. However, it should be noted that during the measurements, the weight of the impregnated samples may have continued to change due to the ongoing autoxidation of the oil. Based on the results, it can be concluded that coating leads to poorer weathering performance than impregnation.

In terms of fire resistance, the single-flame source test showed no significant change after the application of the linseed oil-based stain. There was a slight improvement in spruce wood, where mass loss and flame length were lower compared to untreated wood.

Overall, this study revealed that Royal process treatment with a linseed oil-based stain is a promising alternative for wood protection in outdoor applications. However, regular maintenance is still necessary due to the degradation of linseed oil in the surface layer. Further tests on various wood species will be conducted to gather more comprehensive data following this research.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/coatings14111374/s1>, Figure S1. Schematic representation of the test methodology, highlighting the main steps and materials used. Figure S2. Monthly mean climatic data for Útěchov (Czech Republic) for test period (July 2019–July 2024). Precipitation (mm), global irradiance ($W \cdot m^{-2}$), relative humidity (%) and air temperature ($^{\circ}C$) were tracked every month. Unfortunately, the rain gauge was non-functional from January 2023. Table S1. Lightness (L^*), redness (a^*), yellowness (b^*) parameters and overall color difference (ΔE^*) change during 5 years of natural weathering. S—spruce, B—beech, R—reference, IG—impregnated with grey oil, IB—impregnated with brown oil, CB—coated by brown oil. Figure S3. Chromacity diagram of reference and treated spruce and beech samples. The diagram shows the color shifting of redness (a^*) and yellowness (b^*) during natural weathering of five years. Each dot represents the color of each year, showing the initial color point with filled circle. As the values of a^* and b^* decrease, the color of each sample become more pale and grey. Spruce (S) is marked with black, beech (B) is marked with grey. R—reference, IG—impregnated with grey oil, IB—impregnated with brown oil, CB—coated by brown oil. Table S2. Lightness (L^*), redness (a^*), yellowness (b^*) meters (L^* , a^* , b^*) and overall color difference (ΔE^*) change during 1900 hours of irradiation. S—spruce, B—beech, R—reference, IG—impregnated with grey oil, IB—impregnated with brown oil, CB—coated by brown oil. Figure S4. Chromacity diagram of reference and treated spruce and beech samples. The diagram shows the color shifting of redness (a^*) and yellowness (b^*) during natural weathering of five years. Each dot represents the color of each year, showing the initial color point with filled circle. As the values of a^* and b^* decrease, the color of each sample become more pale and grey. Spruce (S) is marked with black, beech (B) is marked with grey. R—reference, IG—impregnated with grey oil, IB—impregnated with brown oil, CB—coated by brown oil. Figure S5. Surface roughness profiles of the reference spruce wood (up) and oil impregnated wood (below) before (left) and after exposure (right). The color coding indicates the height or depth of the peaks and valleys with respect to a reference plane of the roughness profile. Figure S6. Surface roughness profiles of the reference beech wood (up) and oil impregnated wood (below) before (left) and after exposure (right). The color coding indicates the height or depth of the peaks and valleys with respect to a reference plane of the roughness profile. Figure S7. Fire resistance samples of spruce (above) and beech (bottom) before (left) and after (right) the fire resistance test. The samples are from left to right: reference, coated with brown oil, impregnated with brown oil,

impregnated with grey oil. The flame height was bigger for spruce than beech, but the treatments did not influence the ignition or mass loss significantly.

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