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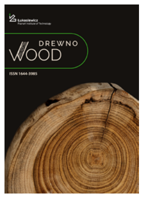

Effects of Artificial Weathering Resistance on the Hardness Value of Ash Wood Surfaces Coated with Polyurethane Modified Water-Based Varnish after Heat Treatment

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Pendulum hardness test is important for determining the mechanical durability and long-term performance of the film layer formed on the surface of wood thermally treated at high temperatures and coated with water-based varnish (WBV). In this study, the effect of artificial weathering conditions on the pendulum hardness (König method) of ash (Fraxinus angustifolia Vahl.) wood samples varnished with polyurethane modified WBV was investigated. The samples were thermally treated under two conditions (190°C for 1.5 h and 212°C for 2 h) and compared with untreated control samples. The samples were exposed to artificial weathering conditions (for periods of 0, 168, 336 and 504 h). Pendulum hardness values were measured at the end of each weathering period. According to the results, increases in hardness values were observed in both heat-treated and untreated ash wood samples treated with varnish after weathering. In the varnished samples treated at 212°C for 2 h, the post-weathering hardness increases were lower compared to the samples treated at 190°C for 1.5 h and the control group. Additionally, analysis of variance for the pendulum hardness test, heat treatment, weathering time and their interaction were determined to be statistically significant. The significant increases in hardness values observed in the unweathered samples highlight the importance of the wood, varnish, and weathering process.

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Introduction

Natural resin varnishes have a complicated and reactive chemical structure, so they can be affected by environmental factors. Chemical changes will take place through light, oxygen, moisture and heat. The varnish is a protective coating that can wear off with use. It follows that these coatings need maintenance, for instance, renewal and removal (van den Berg and Seymour, 2022). Nonetheless, there are

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shortages of the inorganic abrasion wear resistance materials (Ma et al., 2013).

The pendulum hardness test consists of allowing a pendulum with a recess of rotation, which is pre-set and has been struck against the surface of a test specimen, to swing and judging how the specimen damps it (Suzuki et al., 2016). The König stop method involves a 200 g ball with a 5 mm diameter. The test is reported to include a deflection starting at 6° and ending at 3°, and an oscillation period of 1.4 seconds (Schwalm, 2007).

Heat treatment changes many structural properties of wood (Barboutis and Kamperidou, 2019). Heat treatment reduces the mechanical properties of wood due to intensive processing at high temperatures between 150 and 280°C. The mechanical properties of wood are closely related to its moisture content. The strength properties of wood, determined by covalent and intrapolymeric hydrogen bonds, decrease due to the reduction of hydrogen bonds between the main polymer components of the cell wall and the suppression of the increase in bound water content. Heat treatment has a positive effect on the mechanical properties of wood by reducing the maximum bound water content and, consequently, the hygroscopicity of the heat-treated wood (Aytin, 2013). Heat-treated wood surfaces can be painted for aesthetic purposes or to create a distressed appearance. Heat treatment affects the penetration and adhesion of paint systems. Because heat-treated wood is more hydrophobic than untreated wood, water-based paint systems require longer drying times. Therefore, it is recommended to use two thin films instead of one thick one. The required surface tension can be adjusted by adding additives to paint systems (Boonstra, 2008; Korkut and Kocaefe, 2009).

There are studies in the literature that examined the changes in dynamic hardness tests after applying various varnish layers to different wood species and then subjected to artificial or natural aging treatments. Some of these examples include chestnut, Scots pine, and iroko wood treated with WBV and by xenon weathering conditions (Çakıcıer, 2007); Anatolian chestnut, Scots pine, and beech treated with heat, WBV's (Gezer, 2009); iroko, Scots pine, and Anatolian chestnut treated with heat, WBV's by weathering treatments (Çakıcıer et al., 2011b); Uludağ fir, beech, black poplar, oak, and Scots pine, treated with heat, synthetic, parquet, and WBVs (Yalinkiliç, 2013); beech, Scots pine and oak treated with heat, WBV's and weathering treatment (Ayata and Çakıcıer 2021); Scots pine wood treated with heat, WBV's (Kesik et al., 2015); Scots pine treated with WBV and weathering treatment (Gündüz 2018); fir and Scots pine wood with heat, WBV's by natural aging (Kesik et al., 2017); European alder, American ash, white willow, and white poplar with heat, WBV by weathering treatments (Çakıcıer et al., 2017); American ash and scotch pine with heat, WBV's by weathering treatments (Karamanoğlu, 2020); Scots pine, Anatolian chestnut and oriental beech treated with heat, WBV's (Kazan and Özalp, 2021); and European beech and Scotch pine treated with heat, WBV's (Pelit et al., 2023).

These studies provide information on the aging effects of the natural or artificial environment, the extent of interactions between different varnish types, and the various wood species.

This study was conducted to investigate the effects of aging standards on the pendulum hardness (König method) values of ash wood coated with polyurethane-modified WBV, both untreated and heat-treated at high temperatures, compared to a laboratory-based artificial weathering device. Investigating the long-term performance of varnishes used for protective purposes on wood surfaces, particularly when exposed to outdoor conditions, and affecting durability and quality, is crucial.

The objectives of this study are:

- To determine the stages at which changes begin before and after weathering in ash wood varnished using the ThermoWood method, which was heat-treated at 190°C for 1.5 h and 212 °C for 2h, and then varnished with WBVs in accordance with company recommendations, in accordance with industrial practices. These materials were then exposed to UV light for 168, 336, and 504 h in an accelerated QUV aging device.
- To identify the opportunities and limitations for the applications of materials obtained after heat-treating ash wood with WBVs at different temperatures and durations.
- To contribute to the determination of the performance of WBVs on heat-treated wood materials and to the widespread adoption of these varnishes.

Materials and methods

In this study, ash (*Fraxinus angustifolia* Vahl.) logs were used. Test samples were purchased from a private commercial establishment. Test samples of this tree were obtained with a thickness of 8–10 cm, a width of 20–25 cm, and a length of 2–2.5 m, and were prepared with dimensions of 75 mm x 320 mm x 16 mm.

Care was taken to ensure that the wood material was purchased in accordance with the standards, without knots, splinters, growth defects, with smooth fibers, and a mix of heartwood and sapwood.

Experimental samples were brought to the ThermoWood Lumber Factory of Nova Orman Ürünleri Sanayi ve Ticaret A.Ş. in Gerede, Bolu, Turkey, for thermal treatment. The subsequent heat treatment was computer-controlled. The process was carried out in two stages: the samples were heated at 212 °C for 2 h and 190 °C for 1.5 h. After heat treatment, the samples

Table 1. Some important information about the varnish used in the study and the application conditions during application to wood materials

Varnish Type	**Solid Matter Ratio (%)**	**pH Level**	**Applied Varnish Amount (gr/m²)**	**Sample Surface gr/m²**	**Number of Layers**
Primer varnish	22.0	9.12	110	2.64 gr	1
Topcoat varnish	39.0	9.30	150	3.60 gr	2
Varnish properties	Number of Layers		Application Amount		Amount of Solid Matter
Primer varnish 10 sec. immersion method (22%)	1 time application		110 g/m²		24.2 g/m²
Polyurethane modified water based varnish with spray gun Solid Matter (39%)	first layer application		150 g/m²		58.5 g/m²
	second layer application		150 g/m²		58.5 g/m²
					Total Solids Content = 141.2 g/m²

were stored in a specially prepared room for four weeks to reach an equilibrium moisture content of 6–8% (at 20±2 °C and 65±5% relative humidity).

The polyurethane-modified wood varnish, supplied by Dual Paint, was applied to the wood samples in the quantities specified in Table 1 and according to the manufacturer's instructions. A spray gun with a 2.0 mm nozzle was used for application. The gun was positioned parallel to the sample surface, at a distance of 20 to 25 cm. The varnish was initially applied transversely to the wood grain, followed by subsequent coats parallel to it. The air pressure in the gun was 2 bar. The first coat was applied and allowed to dry for 3 h at room temperature (20 °C). Then, the surface was lightly sanded with 400 to 600 grit sandpaper. The second coat was then applied. Table 1 provides basic information about varnishes and their application.

Varnishing was applied according to ASTM D3023-98 (2024). After application of the WBV coating to the wood surfaces, the coated samples were conditioned for four weeks at 20 ± 2 °C and 65 ± 3% relative humidity according to TS 642 ISO 554 (1997). Subsequently, the coating layers of the cured samples were exposed to artificial weathering in a laboratory café under modified conditions of ISO 16474-1 (2013) for various durations (0, 168, 336, and 504 h). The weathering cycle

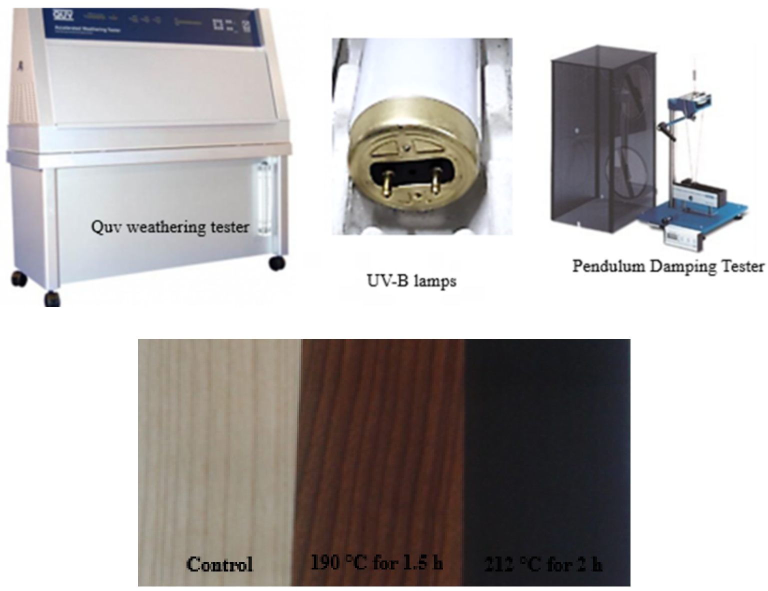

Fig. 1. Images of the devices, equipment, and varnished and heat-treated materials used in the study before weathering application

included 15 min of water spraying, a light intensity of 0.67, 4 h of UV irradiation, and an ambient temperature of 50 °C. UV-B 313 EL fluorescent lamps were used in a QUV weathering unit (Figure 1).

Using ASTM D4366-16 (2021), a pendulum hardness tester model 299/300 Erichsen (Hemer, Germany) was used to determine oscillatory hardness values on coated test specimens with and without weathering treatment, according to the Köning method (Figure 1).

In our study, the experimental setup included 10 samples per group. Oscillatory stiffness values were planned to be ten measurements from each group. In this study, standard deviations, maximum and minimum values, mean results, percentage changes (%), and multivariate coefficients of variation were determined using SPSS software.

Results and discussion

Table 2 shows the results of the analysis of variance. As a result of these results, all factors created for pendulum stiffness values and the interactions determined for these factors were determined to be significant (Table 2).

The results of the pendulum hardness test are shown in Table 3. An increase in all hardness values was observed following artificial weathering. The study confirmed that the pendulum hardness values increased with increasing heat treatment duration of the wood. The lowest pendulum hardness values were found in the samples of the test group without artificial weathering (Table 3).

According to the results determined on samples without heat treatment, the hardness value increases significantly with increasing wear. While the average of the unworn samples was 18, it increased to 39 after 168 h of wear. The value, which was 42 at 336 h, increased again to 40 at 504 h (Table 3).

The hardness results of the 190 °C specimens are higher than those of the control one at each stage of heat treatment, as shown in Table 5: under a weathering time of 1.5 h, while the hardness improves with increasing weathering exposure time, it still does not decrease to the level for control.

The average value of the untreated sample is 20 and becomes 40 after 168 h of weather exposure. At 336 h, this has fallen slightly (38), and at 504 h, there is an increase to 41 (Table 3). Results from the heat treatment group 2 h/212 °C: The highest mean hardness value was obtained with the untreated sample at 24. The average hardness after 168 h of weathering was 38, after 336 h it decreased slightly to 37, and finally reached 41 after 504 h. Even after weathering, the hardness values were higher than those of the control samples and the samples heat-treated for 1.5 h at 190 °C (Table 3).

The control samples and all heat-treated samples in our study showed an increase in hardness with increasing weathering. However, the group treated for 2 h at 212 °C showed a smaller percentage change compared to the other two treatments. The control sample showed the largest percentage increase in hardness at 117% after 504 h of weathering, indicating that the applied artificial weathering significantly improved its hardness. The heat treatments (212 °C for 2 h and 190 °C for 1.5 h) generally resulted in lower percentage increases compared to the control samples; however, an improvement in oscillation hardness was also observed during the weathering treatment, and an increase of 105% was achieved after 504 h at 190 °C (Table 3).

The hardness of a paint layer is related to its molecular cohesion. The coating's hardness increases when larger molecules are brought closer together. Larger molecules harden through polymerization reactions and cross-linking between the paint molecules. The direction and intensity of this reaction are significantly influenced by the molecular weight and intermolecular cohesion. UV light can alter molecular cohesion and, due to aging, can cause a certain increase in surface hardness (Çakıcıer et al., 2011b).

Table 2. Analysis of variance results for pendulum hardness values

Source	α≤0.05	Sum of Squares	df	F Values	Mean Square
Heat Treatment (A)	0.000*	118.117	2	12.497	59.058
Weathering Period (B)	0.000*	7708.167	3	543.679	2569.389
Interaction (AB)	0.000*	145.283	6	5.124	24.214
Error		510.400	108		4.726
Corrected Total		8481.967	119		
Total		151450.000	120		

*: Significant

Table 3. Results for pendulum hardness values (number of measurements: 10)

Heat Treatment	Weathering Period	Mean	Change (%)	Homogeneity Group	Minimum	Maximum	Standard Deviation	Coefficient of Variation
Control (non-heat-treatment)	Non-weathered	18.40	-	G**	16.00	21.00	1.58	8.57
	168 h	39.60	↑115.22	ABCD	30.00	45.00	4.35	10.99
	336 h	34.60	↑88.04	E	31.00	37.00	1.71	4.95
	504 h	40.00	↑117.39	ABC	36.00	44.00	2.36	5.89
190 °C for 1.5 h	Non-weathered	20.10	-	G	17.00	23.00	2.08	10.34
	168 h	40.20	↑100.00	AB	38.00	42.00	1.23	3.06
	336 h	38.00	↑89.05	CD	35.00	41.00	1.70	4.47
	504 h	41.40	↑105.97	A*	38.00	45.00	2.12	5.12
212 °C for 2 h	Non-weathered	24.10	-	F	22.00	26.00	1.37	5.69
	168 h	38.90	↑61.41	BCD	34.00	44.00	2.73	7.01
	336 h	37.80	↑56.85	D	35.00	41.00	1.69	4.46
	504 h	41.10	↑70.54	A	39.00	43.00	1.20	2.91

*: Highest result, **: Lowest result

It has been said that the heat treatment method is more important than the heat treatment temperature for the hardness values of coatings with different properties applied to wood-based materials, and that both the properties of the wood species and the type of coating affect the hardness properties of coated surfaces (Koç, 2023). According to the literature, changes in stiffness after heat treatment could be related to the degradation of hemicellulose, which is less heat-resistant than cellulose and lignin. Changes in the composition of hemicellulose lead to different strength properties of wood heated at high temperatures (Hillis, 1984).

In a study conducted by Esser et al. (1999), they reported that aging applications had an effect on increasing the hardness value of the layers they used in their studies.

In laboratory research conducted by Baysal et al. (2021), they exposed pine tree surfaces to outdoor environmental conditions for 6 months after coating them with WBV. As a result, they found that the hardness value increased.

Özen and Sönmez (1999) examined the changes that occurred when some woods were coated with some varnishes and then exposed to outdoor conditions for 22 months. They reported that the pendulum hardness of the varnished wood increased over time.

Çakıcıer (2007) reported that increases in pendulum hardness values were obtained in samples treated with acrylic-modified and WBVs applied to iroko, Scots pine, and chestnut wood in his artificial aging study using xenon lamps.

Gezer (2009) investigated the effects of heat treatment on the hardness of wood-based materials. He applied WBV coatings to samples of beech, Scots pine, and Anatolian chestnut and heated them for 2, 4, and 6 h at 100, 150, and 200 °C, respectively. The results showed that two-component coatings performed better than one-component coatings for all treated wood species. The hardness resistance of the samples heat-treated for 2 h at 100 °C, 4.6 h at 100 °C, and 2, 4, and 6 h at 150 °C and 200 °C was better than that of the samples heat-treated for only 2, 4, and 6 h at 100 °C.

Cakıcıer et al. (2011a) reported that the hardness values of iroko, chestnut, and ash wood samples with heat treatment were higher than those of the same wood species with WBV but without heat treatment.

Çakıcıer et al. (2011b) reported that single- and two-component WBVs were applied in two and three coats to iroko, Scots pine, and Anatolian chestnut woods. The varnish-coated materials were then exposed to QUV weathering devices for 216 and 432 h. The results indicated that the surface hardness values varied across all three wood species in all treatment combinations.

In his research, Yalinkiliç (2013) reported that the highest surface hardness according to the type of wood-varnish-heat treatment-application type was found in the WBV with fir (38.20) test sample, which was heat treated at 175°C for 2 h.

According to Pelit (2014), pine wood treated with WBV coatings showed higher fatigue strength values than untreated varnished samples.

Kesik et al. (2015) examined the hardness of paint and varnish layers on pine boards treated with linseed oil at high temperature (150°C, 2 h). The study showed that the hardness of the paint applied with linseed oil at high temperature was higher than that of the paint layer.

Kesik et al. (2017) examined the surface hardness of spruce and pine boards coated with WBVs after heat treatment and modification after one year of aging. The results showed that the hardness of the heat-treated samples increased more than that of both wood types. According to the surface hardness test, varnished and heat-treated surfaces were more resistant to deformation than unvarnished wood.

Çakıcıer et al. (2017) studied the effect of a WBV coating on the accelerated UV resistance of heat-treated wood floors. The layers are applied at different temperatures and for different times. Wood species used include white willow, American ash, European spruce, and white poplar. The samples were then irradiated with UV-A fluorescent light for 144, 288, and 432 h using the QUV method. This study shows that the weight of the pendulum changes as the atmosphere expands.

Gürleyen et al. (2017) reported divergent results in the pendulum hardness test on thermally treated and non-treated pine wood coated with UV varnishes.

Gündüz (2018) found that the pendulum hardness values of pine wood coated with thermally modified increased compared to the control group after accelerated weathering for 250, 500, 750, and 1000 h.

Karamanoğlu (2020) observed that pendulum hardness values increased after 240 h of weathering in two-component transparent coatings on thermally treated and untreated pine and ash wood.

In a laboratory study conducted by Ayata and Çakıcıer (2021), it was reported that the hardness values of pine, oak and beech wood samples, which were heat treated (190 °C for 2 h and 212 °C for 1–2 h) and coated with WBVs, increased compared to artificial weathering after exposure to ultraviolet radiation for 144, 288 and 432 h using UV-A fluorescent lamps.

In the study by Kazan and Özalp (2021), wood samples of Scots pine, Anatolian chestnut, and oriental beech were treated with WBVs and subsequently exposed to temperatures of 100 °C, 125 °C, and 150 °C for 2, 4, and 6 h, respectively. The results showed an improvement in hardness in all samples exposed to 100 °C for 2 h. Furthermore, all samples treated at 100 °C, 125 °C, and 150 °C for 2, 4, and 6 h, respectively, exhibited high hardness resistance. It has been reported that two-component lacquers deliver better results than one-component lacquers on all types of wood being lacquered.

Aytin et al. (2021) reported that an increase in pendulum hardness was observed in water-based parquet varnish layers applied to oak, eucalyptus, and maple veneers after 144 h of exposure to weathering using artificial media.

In a study conducted by Pelit et al. (2023), it was reported that the hardness values of samples coated with WBV increased compared to unvarnished samples for all different heat treatment methods and different temperatures for European beech and Scotch pine wood.

Conclusions

In this study, all samples in the experimental group were varnished, and changes in fatigue hardness due to artificial aging were investigated. Unglazed samples were not available. The pH and absorption capacity of the varnish by the wood change after heat treatment. Furthermore, the drying and varnish formation systems used may differ and exhibit variable hygroscopic behavior. After weathering, a general increase in hardness values was observed in all experimental groups. The highest fatigue hardness values were measured in samples from the experimental group that were heat-treated for 2 h at 212 °C, followed by those heat-treated for 1.5 h at 190 °C. The lowest average hardness values were generally found in samples from the control group, which were not subjected to any heat treatment. Although artificial aging in the laboratory led to fluctuations in hardness values, particularly after 336 h, a general trend of increasing fatigue hardness was observed, especially in heat-treated and varnished samples. It was found that heat treatment significantly increased the vibrational hardness of WBV coatings applied to ash wood and that the effects of artificial weathering, particularly treatment at higher temperatures (212 °C for 2 h), were enhanced. The results showed that, although the vibrational hardness values of all wood samples improved over time due to weathering, heat treatment at 190 °C produced the most significant improvements in all periods of artificial weathering.

Conflict of interest

The author(s) declare(s) that there is no conflict of interest concerning the publication of this article.

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