



### The effects of artificial weathering on the pendulum hardness of chestnut wood applied with polyurethane varnish after heat treatment

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#### Abstract

The effects of artificial weathering (576 h) on the pendulum hardness values of Anatolian chestnut (*Castanea sativa* Mill.) wood applied with polyurethane varnish (PU) after heat treatment at 190°C for 1.5 h and at 212°C for 2 h were investigated. The results revealed that heat treatment, weathering period, and interaction were significant. The hardness values of the heat-treated materials were higher than those of the non-heat-treated test samples. The highest hardness value was determined in the un-weathered group of the test samples that were heat-treated at 190°C for 1.5 h while the lowest value was determined in the group that was heat-treated at 190°C for 1.5 h and weathered for 432 h. The highest reduction rates after weathering were obtained on samples that were heat-treated at 190°C for 1.5 h, followed by heat-treated at 212°C for 2 h and non-heat-treated. It was observed that the heat treatment application caused different pendulum hardness values in chestnut wood samples covered with polyurethane varnish.

**Keywords:** Pendulum hardness, Anatolian chestnut, *Castanea sativa* Mill., artificial weathering, heat treatment, polyurethane varnish

### Isıl işlem sonrası poliüretan vernik uygulanmış kestane odununda salınımsal sertlik üzerine yapay yaşlandırmanın etkileri

#### Öz

Bu çalışmada, 190°C’de 1.5 saat ve 212°C’de 2 saat süre ile ısıl işlem gördükten sonra poliüretan vernikler uygulanan Anadolu kestanesi (*Castanea sativa* Mill.) odununa ait katmanın salınımsal sertlik değerleri üzerine yapay yaşlandırma (576 saat) etkileri araştırılmıştır. Belirlenen sonuçlara göre, tek değişkenli varyans analizi sonuçları için, ısıl işlem, yaşlandırma süresi ve etkileşimi anlamlı olarak belirlenmiştir. Isıl işlem görmüş malzemelere ait sertlik değerleri, ısıl işlem görmemiş deney örneklerinden daha yüksek elde edilmiştir. En yüksek sertlik değeri 190°C’de 1.5 saat süreyle ısıl işlem görmüş deney örneklerine ait yaşlandırma yapılmayan grupta belirlenirken, en düşük değer ise 190°C’de 1.5 saat süreyle ısıl işlem görmüş 432 saat süreyle yaşlandırılmış grupta tespit edilmiştir. Yaşlandırma sonrası azalma oranları en yüksek 190°C’de 1.5 saat süre ile ısıl işlemli örnekler üzerinde elde edilirken, bunu 212°C’de 2 saat süre ısıl işlemli örnekler ve ısıl işlemsiz örnekler izlemiştir. Isıl işlem uygulamasının, poliüretan vernikleri ile kaplanmış kestane odunu örneklerinde farklı salınımsal sertlik değerleri vermesine sebep olduğu görülmüştür.

**Anahtar kelimeler:** Salınımsal sertlik, Anadolu kestanesi, *Castanea sativa* Mill., yapay yaşlandırma, ısıl işlem, poliüretan vernik

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## **1 Introduction**

The method of covering the natural wood surface with a protective material that forms a visibly liquid and shiny layer after drying is called “*varnishing*” (Sümer, 1946). Polyurethane varnishes are available in matte, satin, or glossy surfaces based on water or solvent-based systems (Lyons, 2019).

Polyurethane is transparent and allows working on both sides of its surface. The use of this material may cause different thickness levels to be achieved, giving different results for different uses and techniques (Bebit et al. 2019). The curing rate can vary by system, but pot life is typically 45-60 minutes. This time is reasonable, as polyurethanes can be applied relatively quickly. The rate will change with temperature and large volumes of exothermic reaction will occur. Water pollution and high humidity should be avoided at all costs (Cattell, 2003). Polyurethanes, and especially hydroxylated polyurethanes, can interact with wood via hydrogen bonding (Mubarok et al. 2017).

In artificial weathering devices, three kinds of lamps, namely, the xenon lamp, the ultraviolet fluorescent lamp, and the carbon arc lamp, are widely used as light sources to simulate ultraviolet radiation from sunlight. Periodic condensation or water spray is used to simulate dew or rain (Hu et al. 2009; Cakicier, 2007).

Hardness also has a close relationship with other mechanical properties such as strength, ductility, and fatigue resistance, and therefore hardness testing can be used in industry as a simple, fast, and relatively inexpensive method of material quality control (Broitman, 2017).

Different test methods are available to measure the surface hardness properties of materials and material surface layers. One of these is the König pendulum method.

The König pendulum consists of an open frame connected by a crossbar and has two balls with a hardness of  $5+0.005$  mm and a diameter of  $63+3$  HRC placed on its lower face to serve as a fulcrum. The lower end of the frame is formed in the form of a marker. In balancing the pendulum, a weight sliding on a rod in the vertical direction is used, depending on the crossbar. The total weight of the pendulum is  $200+0.2$  g (ASTM D 4366-95, 1984).

Two test methods based on different types of pendulums are discussed: the König pendulum stiffness test (time in seconds for the oscillation amplitude to decrease from  $6^\circ$  to  $3^\circ$ ) and the percus pendulum stiffness test (the oscillation amplitude to decrease from 12 to  $4^\circ$  in seconds). In general, the extinction time of the König pendulum is about half of that of the rivet pendulum (ASTM D 4366-95, 1984).

In laboratories, coatings are artificially etched in apparatus specially designed to simulate or measure aging processes that occur during natural wear. Artificial weathering involves fewer parameters than natural weathering. However, it allows for more homogeneous, controllable, and accelerated test conditions (Simms, 1987; Kropat et al. 2020).

In this study, the effects of artificial weathering on the pendulum hardness values of chestnut wood applied with polyurethane varnish after heat treatment at different temperatures and times were investigated. In the literature, pendulum hardness properties research was not found after 576 h of artificial aging using UV-B 313 EL type lamp on heat-treated chestnut wood covered with polyurethane varnish. The obtained results aim to reveal the interaction between heat treatment, polyurethane varnish and chestnut wood.

## 2 Material and Method

### 2.1 Material

#### 2.1.1 Obtaining wood material

Anatolian chestnut (*Castanea sativa* Mill.) woods were used in the study. Wooden samples were obtained from a lumberman by purchasing method. The samples were prepared to 320x75x16 mm in net dimensions. Air-conditioning (20±2°C and 65% relative humidity) processes of the materials were performed (TS 642 ISO 554, 1997).

### 2.2 Method

#### 2.2.1 Heat treatment

The heat treatment modification of chestnut woods with an initial moisture content of 12% was carried out in a commercial facility with special computer-aided closed kiln application for 1.5 h at 190°C and 2 h at 212°C.

#### 2.2.2 Application of polyurethane varnish on wood material surfaces

In this study, topcoat glossy polyurethane (polyurethane resin-based two-component) varnishes belonging to a commercial company were applied to the heat-treated and non-heat-treated test samples. The technical information on the varnishes used is given in Table 1.

**Table 1.** Technical information on the varnishes used

Varnish Type	Sample Surface (m <sup>2</sup> )	Amount of Varnish Applied (gr/m <sup>2</sup> )	Solids Ratio (%)	pH	Number of Layers
Polyurethane Filler	2.40	103	46.20	6.55	1
Polyurethane lossy	2.40	100	46.90	6.25	2

It was applied according to industrial applications and the recommendations of the varnish manufacturer as per (ASTM D3023-98, 2017) in the amounts given in Table 2. Care was taken to ensure that the film layer thicknesses were close to each other and in line with the determined solid matter amounts of the varnishes.

**Table 2.** Information on the application of polyurethane varnish on wooden surfaces

Features of Varnish	Number of Layers	Application Quantity	Solids Ratio (%)
Using the sealant spray gun	1	99 g/m <sup>2</sup>	47 g/m <sup>2</sup>
Using the topcoat varnish spray gun	1	101 g/m <sup>2</sup>	45 g/m <sup>2</sup>
	2	101 g/m <sup>2</sup>	47 g/m <sup>2</sup>
Total Solids Amount =			139 g/m <sup>2</sup>

#### 2.2.3 Artificial weathering

After the varnishes were applied to the test samples, they were left to dry in the air-conditioning room according to ISO 554, (1997) (65±3% relative humidity and 20±2°C temperature). Subsequently, UV-B 313 type fluorescence was applied in a QUV accelerated aging device adjusted to ISO 16474-1, (2013) standards (ambient conditions: 50°C ambient temperature cycle, 15 min water spray, 0.67 light intensity, 4 h UV). It was decomposed by exposure to lamps for 144, 288, 432, and 576 h. They were exposed to lamps for 144, 288, 432, and 576 h for decomposition.

### 2.2.4 Determination of the solid matter amounts

The amount of solid matter in the varnishes has been calculated according to TS EN ISO 3251 (2019).

### 2.2.5 Determination of pendulum hardness

The hardness values of the weathered and un-weathered test samples were made according to the TS 642 ISO 1522 (2022) standard, using the König method on the platform of the pendulum hardness measuring device.

### 2.2.6 Statistical analysis

Standard deviations, univariate coefficients of variation, maximum and minimum results, % change rates after weathering, homogeneity groups, and mean results were calculated by an SPSS program.

## 3 Results and Discussion

The results of the univariate analysis of variance, calculated using the measurement results of the pendulum hardness values, are shown in Table 3. According to these results, the heat treatment (A) factor, the weathering period (B) factor, and the interaction (AB) of these factors were determined to be significant.

**Table 3.** Univariate analysis of variance results for pendulum hardness values

Variance Source	Sum of Squares	Degree of Freedom	F Value	Mean Square	$\alpha \leq 5\%$
Heat Treatment (A)	113.853	2	6.012	56.927	0.003*
Weathering Period (B)	9130.827	4	241.074	2282.707	0.000*
Interaction (AB)	6528.413	8	86.182	816.052	0.000*
Error	1278.300	135		9.469	
Total	2799345.000	150			
Corrected Total	17051.393	149			

Result by \*: Significant  $\alpha \leq 0.05$

The results of the pendulum hardness values measured before and after weathering are presented in Table 4. According to these results, the control measurement result was obtained as 136.10 in the untreated and varnished test samples. In the test performed for this group, a decrease of 4.41% in 144 h of weathering period, an increase of 1.32% in 288 h, a decrease of 3.01% in 432 h and an increase of 4.56% in the last 576 h were recorded. According to Table 4, each period took place in different HG groups among the weathering periods on the surfaces coated with PU varnish of the non-heat-treated samples. However, it was determined that there was no linear change. This is thought to be related to the changes in the UV, temperature, humidity, and PU varnish components and the cell structure of the wood that makes up the aging cycle. It can be attributed to the effect of chemical bonds or thickness forming the varnish layer under the influence of high temperature and UV-B rays.

Ceylan (2016) found the hardness values of beech wood samples applied with polyurethane varnish to be 89.10 before weathering, 121.80 after 100 h of weathering and 118.80 after 300 h of weathering. Peker (1997) determined an increase of 6.43% and 36.90%, respectively, in polyurethane-varnished Scotch pine and chestnut woods after aging in outdoor environmental conditions.

**Table 4.** Measurement results of pendulum hardness values

Treatment	Weat-hering Period	N	Mean (s)	Change (%)	HG	Standard Deviation	Min.	Max.	Coefficient t of Variation
Non-heat-treated	Control	10	136.10	-	DE	1.97	134.00	139.00	1.45
	144 h	10	130.10	↓4.41	G	1.97	127.00	134.00	1.51
	288 h	10	137.90	↑1.32	D	3.18	132.00	142.00	2.30
	432 h	10	132.00	↓3.01	FG	3.02	127.00	136.00	2.29
	576 h	10	142.30	↑4.56	C	2.11	140.00	145.00	1.48
190°C for 1.5 h	Control	10	158.80	-	A*	4.26	152.00	168.00	2.68
	144 h	10	131.40	↓17.25	FG	3.24	127.00	135.00	2.46
	288 h	10	132.00	↓16.88	FG	1.89	130.00	135.00	1.43
	432 h	10	113.30	↓28.65	I**	4.40	106.00	120.00	3.88
	576 h	10	141.90	↓10.64	C	3.67	136.00	147.00	2.58
212°C for 2 h	Control	10	152.80	-	B	3.55	148.00	158.00	2.33
	144 h	10	126.80	↓17.02	H	2.39	124.00	132.00	1.89
	288 h	10	133.60	↓12.57	EF	2.50	130.00	138.00	1.87
	432 h	10	138.00	↓9.69	D	2.94	133.00	142.00	2.13
	576 h	10	135.90	↓11.06	DE	3.51	130.00	141.00	2.58

N: Number of Measurements, \*: Highest result. \*\*: Lowest result

Considering the pendulum hardness values of wood species with polyurethane varnish applied in the literature; it was 73.60 for scotch pine, 62.60 for chestnut (Peker, 1997), 109 for black pine, 82.60 for beech (Sarı, 2012), 62 for Scots pine, 60 for oak, 67 for chestnut (Kılıç, 2019), 115 for Scotch pine, 133 for beech, 104 for mahogany (Akdemir, 2022), 100.40 for walnut, 93.20 for Scots pine (Uzun, 2021), 72.40 for fir, 72.70 for chestnut (Soylamış, 2007), 69.00 for beech (Baysal, 2011), and 38.00 for Scots pine (Baysal et al. 2014). It is reported in the literature that surfaces with higher oscillations are hard, and surfaces with less oscillation have lower hardness (Sönmez, 1989). It is thought that the reason for the different hardness values in the polyurethane varnish studies may be due to the use of chemicals from different companies, the use of different types of wood, and the different layer thicknesses obtained after the stages during the application.

In the literature, after exposure to UV rays for 500 h in an accelerated weathering device consisting of UVA-340 lamps, the pendulum hardness values of wood materials on which polyurethane varnishes were applied were found to increase 21% in varnished oriental beech wood (Baysal, 2011) and 24% in varnished Scotch pine wood (Baysal et al. 2014). In this study, it was observed that the hardness value increased after 576 h of weathering on non-heat-treated and varnished materials.

Genç, (2019) determined that the hardness value of polyurethane varnishes was 115 seconds on average before weathering. This was reported to be 79 seconds after 500 h of weathering of UVA-340 lamps.

In addition, the highest value was determined in the control experimental sample group of the experimental samples that were heat-treated and varnished at 190°C, for 1.5 h while the lowest value was determined in the experimental samples that were heat-treated at 190°C for 1.5 h and varnished for 432 h obtained in the experimental sample group. In addition, the reason why heat-treated wood material and non-heat-treated wood material behave differently against the same varnish type may be due to the change in the structure of the wood with the heat treatment.

It was observed that the hardness value of the heat-treated materials decreased after weathering. While the reduction rates after weathering were lowest without heat treatment, this was followed by samples with heat treatment at 212°C for 2 h and heat treatment at 190°C for 1.5 h. In pendulum hardness, the results of the heat-treated materials were higher than those of the untreated test samples. In addition, the hardness values of the heat-treated materials decreased after weathering.

It was reported by Ulay (2018) that similar results were obtained after aging applications on polyurethane varnishes applied to heat-treated and untreated iroko and ash woods of 212°C for 2 h and 190°C for 1.5 h. In addition, this varied depending on the degrading effect of the heat treatment process and aging factors on lignin and wood components such as cellulose and hemicellulose (Kropat et al. 2020). It has been reported in many studies that they cause many changes such as color change (Karamanoğlu and Akyıldız, 2013; Ulay and Çakıcıer, 2017), glossiness (Ulay, 2018), roughness (Yang et al. 2002), crack formation (Holzhausen et al. 2002; Oosterbroek, et al. 1991), pits and new cracks (Akyıldız and Karamanoğlu, 2016), and varnish layer structure (Jaic and Zivanovic, 1997). It should be taken into account that the factors affecting these can also affect the surface hardness feature.

#### 4 Conclusions

According to data obtained;

- According to the results of univariate analysis of variance, treatment, weathering period, and the interaction were found to be significant.
- The hardness results of the heat-treated materials were higher than those of the non-heat-treated test samples.
- The lowest rate of decrease after weathering was determined to be for the group without heat treatment (between 3 and 4%), followed by samples with heat treatment at 212°C for 2 h (between 9 and 17%) and heat-treated at 190°C for 1.5 h. (between 10 and 17%) followed.

It is recommended to add various additives to the polyurethane varnish in order to prevent a decrease in the hardness value.

#### Author Contributions

**Göksel Ulay:** Creating the research idea, conducting the laboratory work, taking the measurement data, writing and edit **Ümit Ayata:** writing the article, performing the statistical operations.

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#### Conflict of interest statement

The authors declare no conflict of interest

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