Effect of Walnut Tannin on Wood Surface Hardness: A Study on Pine and Walnut Samples

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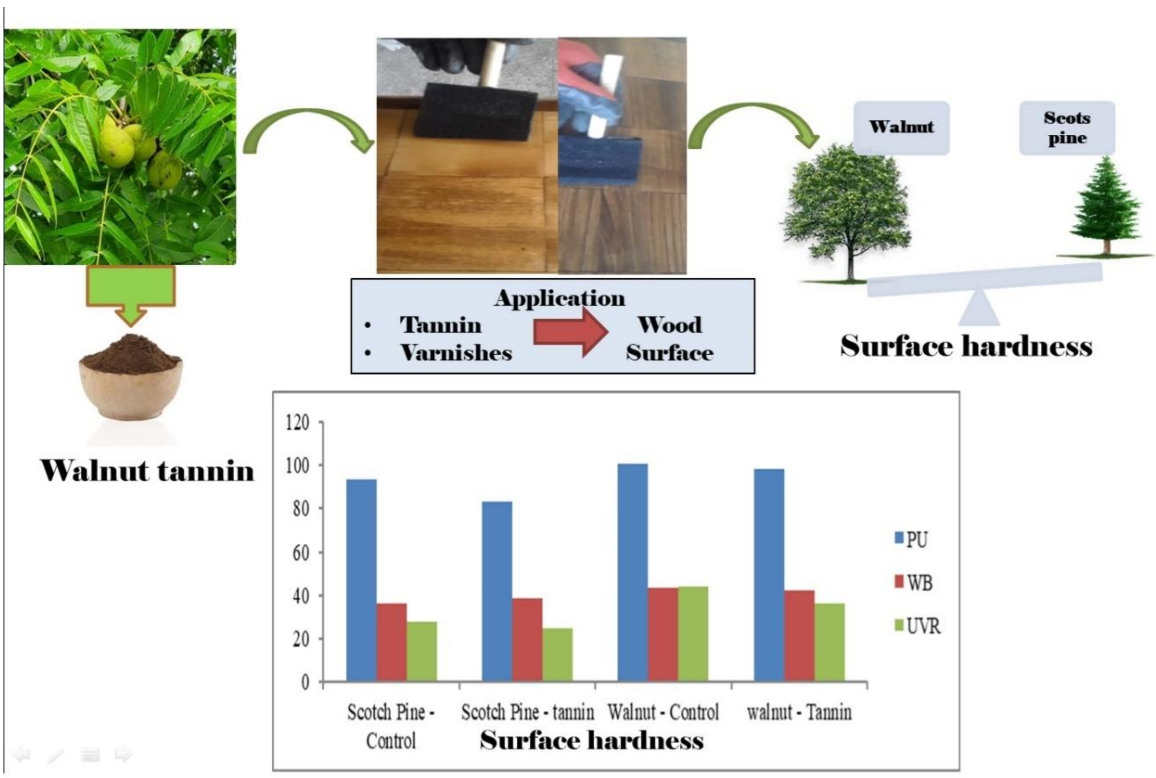
GRAPHICAL ABSTRACT



Effect of Walnut Tannin on Wood Surface Hardness: A Study on Pine and Walnut Samples

Sekip Sadiye Yasar,[a,*] Ahmet Cihangir Yalinkilic,[b] and Mehmet Yasar[a]

The hardness of the wood surface determines its wear resistance and resistance to mechanical damage. This study aimed to determine the effect of walnut tannin used in wood preservation on the surface properties of wood. For this purpose, Scots pine (*Pinus sylvestris* L.) and walnut (*Juglans regia* L.) wood test specimens were treated with walnut tannin with a brush. The specimens then were coated with water-based and polyurethane varnishes. After drying, the surface hardness was measured. As a result of the research, it was determined that the highest surface hardness was in the control samples without tannin application and the highest value was in walnut wood.

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Keywords: Wood materials; Tannin; Scots pine; Walnut; Surface hardness

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INTRODUCTION

Wooden material has been used for the production of many tools and useful structures from the past to the present. Today, the usage of wood as a raw material has decreased with the increase in technological development and the emergence of new materials. Despite this, wood material is still used by many wood-based operation fields and continues to be a biologically sustainable raw material (Aydemir and Gunduz 2009). It is a natural building element that is highly preferred in interior and exterior decoration because of its easy processing, its light and durable structure, its easy transportation, its various colors and texture, its ability to retain heat and sound, the fact that it can insulate electricity with a high aesthetic value, and because it is not affected by chemicals (Kurtoglu 2000). In addition to these positive advantages of wood material, the wood has disadvantages such as burning easily due to being a natural material, being destroyed by insect pests, being decomposed by fungi, changing its sizes due to the equilibrium humidity that changes with respect to the temperature and relative humidity of the environment, and its color fading due to the effect of the sun (Usta 1993; Kurtoglu 2000). As a result of the usage of wood material that was exposed to outdoor climatic conditions, physical and mechanical deterioration has been observed in the places of use, and it has been reported that artificial-natural wood preservatives and top surface treatment materials such as paint and varnish are needed for protection against outdoor conditions (Evans *et al.* 1992; Sen 2001; Hill 2006; Mantanis and Lykidis 2015; Yasar *et al.* 2016; Sandberg *et al.* 2017; Gao *et al.* 2019; Yasar and Altunok 2019; Doruk 2022).

Budakci (1997) applied acrylic, synthetic, and polyurethane varnishes in different film thicknesses to Scots pine, beech, and oak test samples. The film thickness, surface hardness, and bonding to the surface and their effects were investigated. With regard to the research results, it was determined that increasing the varnish film thickness did not have an effect on the surface hardness in the three-coat applications. The highest hardness value was in the beech wood, and in polyurethane varnish, in a single layer application in layer thickness; the lowest hardness value was obtained in pine wood, and in synthetic varnish and 3-layer varnish application.

Ceylan (2016) studied the suitability of woods modified with some herbal extracts for surface treatment, and accordingly, vegetable extracts, Quebracho wood, and Mimosa bark extracts were used in impregnation solution against fungi and insects. The beech wood samples were modified with herbal extracts, and then polyurethane, cellulosic, and water-based varnishes were applied. Pendulum hardness tests were conducted. It was determined that polyurethane varnish gave more positive results than other varnishes in all tests in terms of varnish types, and that the pendulum hardness property was affected adversely by the modification process with extracts.

Okut (2019) applied the walnut tannin solution for impregnation of chestnut, Scots pine, and oak woods by brushing on 3 to 6 layers and dipping for 4 to 8 h. Decay effects were investigated in soil. When compared with the control samples, all untreated samples had a material loss of 8% after exposure to the soil conditions and a decrease in hardness of about 30%. The hardness of the samples treated with tannin and kept in the soil was higher than the untreated samples. The best result in this experiment was observed in the 8-h immersion method, and it was also determined that the natural tannin solution prevents rotting.

Unver (2019) kept the Scots pine and oak treated samples by modifying with natural wood preservers in outdoor conditions for a year and examined some mechanical and physical properties. As a result of the research, it has been determined that natural wood preservatives prevent the density loss in wood material and reduce the hardness loss due to retention by 8.8%. It was also determined that tannin modifiers have a versatile positive effect on the protection of wood and they increase the physical and mechanical properties of wood.

Yalınkilic (2013) investigated the effects of the heat treatment on top surface quality properties of the varnished fir, oak, beech, and poplar woods with the ThermoWood method. As a result of the research, the highest surface hardness was observed in water-based varnished fir heat treated for 2 h in 175 °C and the lowest surface hardness was observed in synthetic varnished oak wood heat treated for 4 h in 165 °C.

Yasar and Altunok (2019) used pine tannin and acorn tannin as natural preservatives, and Imersol Aqua and timber care Aqua as chemical preservatives to determine the retention amount, air dry density, bending strength, modulus of elasticity, compression resistance parallel to the grains, bonding strength parallel to the grains, and screw withdrawal values. It was observed that natural preservers are as effective as chemicals in many aspects. Better results were obtained when compared with the control method.

Hosseini *et al.* (2008) investigated whether walnut extracts (concentrations percent: 1.5, 2.5, and 3.5%) could be successfully used to protect against white-rot fungi of poplar blocks. Untreated poplar specimens had mean weight losses of 55.4% for white-rot fungi, the 1.5% concentration was 43.75%, and the 3.5% concentration had only 48.2%.

In this study, it was aimed to determine the values of hardness that are surface properties of wood treated with tannin that is present in many trees as a natural preservative. For this purpose, walnut tannin was applied to Scots pine (*Pinus sylvestris* L.) and walnut

(*Juglans regia* L.) woods with brush method and the sample surfaces then were covered with water-based and polyurethane varnishes. As a result, it can be evaluated that tannin, which is found in common wood types of our furniture and woodworking industry, can be used by managements in terms of its surface application quality properties, its sustainability, and its economic value.

EXPERIMENTAL

First-class Scots pine (*Pinus sylvestris* L.) and walnut (*Juglans regia* L.) lumber pieces were used in the study. Randomly selected lumber was acclimatized at 20 ± 2 °C and the relative humidity of 65 ± 3% until it reached a constant moisture content of 12% before rough cutting according to ISO 3129 (2019) principles. Attention was paid to select samples that were resin-free, had a smooth fiber structure, and were free of knots and cracks. The surface of the prepared parts was sanded with 80, 100, and 120 grit sandpaper, respectively, until they became smooth. Radial cross-section samples, which were brought to 10 × 10 × 1 cm^3 dimensions according to the test standards, were kept in the air conditioning device at 20 ± 2 °C and equilibrium humidity of 10 to 12% until they reached a constant weight. Walnut tannin supplied from AR-TU KİMYA Acorn and Valex Factory (ARTU 2022) was prepared by boiling at 70 °C for 1 h to be 10 g/L in accordance with the manufacturer's instructions. Tannin application was carried out with a brush first parallel to the fibers, then perpendicular to the fibers, and finally parallel to the fibers again, and each layer was dried at room temperature by waiting 1 hour between layers, as shown in Fig. 1.

a b

Fig. 1. Tannin application with brush (a: Scotch pine tannin (WT) application; b: Walnut tannin (WT) application)

Water-based varnish (WB) and polyurethane varnish (PU) were used to create the upper protective layer. The WB was preferred because it does not release volatile chemicals and PU was chosen because it is a widely used varnish type. The used varnishes were applied at a level of 120 g/m^2 (Budakci and Sonmez 2010; Yalınkilic *et al.* 2021).

The dry film thickness of the varnishes was measured with a digital micrometer, which is capable of measuring with a sensitivity of 0.001 mm = 1 µm (micrometer) with respect to ASTM D1005-95 (2001) and TS EN ISO 2808 (2019) standards (Sonmez 1989).

Varnish layer hardness is an important element that determines the durability of varnishes against external factors (Kilic and Sogutlu 2022). Before starting the measurements with the hardness test device and regularly during the measurements, it is adjusted to make 100 oscillating movements in 40 s using the caliber glass of the device. Hardness was measured by counting the oscillating movements from 6° to 3° with two balls, which have 5 ± 0.0005 mm diameters, in a hardness scale of 63 ± 3.3 HRC, according ASTM D4366 (2021). With respect to the instructions, there is more oscillation movement on hard surfaces and less on soft surfaces (Sonmez 1989; Kurtoglu 2000). The Koning Pendulum hardness test device was used in the experiments.

Two wood types in the study were prepared into a total of 120 test pieces [2 woods x 2 (tannin + control) x 3 (2 varnishes + control) x 10] consisting of 10 samples from each group.

In the study, interaction variations such as wood type, varnish type, treatment type, as well as the interaction terms wood type - varnish type, wood type - treatment type, varnish type - treatment type were considered; surface hardness tests were carried out. The SPSS statistical package (IBM SPSS, version 21.0, Chicago, IL, USA) program with 95% confidence interval was utilized in statistical data evaluation, and multiple analysis of variance (ANOVA) was conducted between all modifications and top surface application groups.

RESULTS AND DISCUSSION

The pH values of the tannin and varnishes used in the experiments were measured with a pH meter, and values are given in Table 1.

Table 1. The pH Values

Materials	pH Values (20 ± 2 °C)
Tannin	8
PU Glossy Varnish	5
PU Hardener	6
WB Gloss Varnish	7

In summary, the pH value of the water-based glossy varnish (WB) was neutral, the polyurethane varnish and hardener (PU) were acidic, and the tannin was basic (Kilic and Sogutlu 2022).

Dry layer thicknesses of the varnishes are given in Table 2.

Table 2. Dry Film Thickness of Varnishes

Dry Film (Layer) Thickness (µm)	
PU varnish (2 coats)	104.5
WB varnish (2 coats)	104.25

As a result of the conducted measurements, the tendency of polyurethane varnish and water-based varnish to contribute to layer thickness was found to be approximately equal.

The multiple variance analysis was performed to determine the significance of wood type, treatment type, varnish type, and their interactions on hardness values. The data are given in Table 3.

Table 3. Multiple Variance Analysis on the Effects of Wood, Treatment, and Varnish Type on Surface Hardness

Source	Degrees of Freedom	Sum of Squares	Mean Squares	F- value	P (α < 0.05)
Wood (A)	1	3224.033	3224.033	49.835	0.001*
Treatment (B)	1	396.033	396.033	6.122	0.015*
Varnish (C)	2	87772.067	43886.033	678.359	0.001*
Interaction (AB)	1	0.533	0.533	0.008	0.928
Interaction (AC)	2	338.467	169.233	2.616	0.048*
Interaction (BC)	2	258.067	129.033	1.995	0.141
Interaction (ABC)	2	261.267	130.633	2.019	0.138
Error	108	6987.000	64.694		
Total	119	99237.467			

*: Statistically significant difference P ≤ 0.05

The interactions of wood type, treatment type, varnish type and wood type-varnish type were found to be significant, while the interaction of wood type-treatment type, treatment type-varnish type, and wood - treatment -varnish type was found to be insignificant ($P \leq 0.05$). Surface hardness value variations with respect to wood type, treatment type, varnish type are given in Table 4.

Table 4. Average Surface Hardness Values with Regard to Wood, Treatment, and Varnish Type

Source		Means	95% Confidence Interval		Std. Error
			Lower Limit	Upper Limit	
Wood	Scotch pine	50.6	48.5	52.6	1.038
	Walnut	61.0*	58.9	63.0	
Treatment	Control	57.6*	55.5	59.6	
	Tannin	54.0	51.9	56.0	
Varnish	WB	40.3	37.7	42.8	1.272
	PU	93.8*	91.3	96.3	
	UVR	33.3	30.7	35.8	

*: The highest surface hardness PU: polyurethane, WB: water based, UVR: unvarnished

With regard to the wood type, the highest average surface hardness value was found in walnut (61.0) and the lowest was in Scots pine (50.6). Walnut is known to be an extractive-rich material, and its anatomical structure may have increased the values. Density of wood has a critical importance for mechanical features. As the density values in wood decrease, the surface hardness value also decreases (Bozkurt and Goker 1987; Kutnar and Šernek 2007; Percin *et al*. 2017).

With regard to the treatment type, the highest values in surface hardness were found in the control samples (57.6), and the lowest values were found in the tannin samples (54.0). It can be understood that the reason for this decrease in hardness of tannin samples is that the walnut tannin solution has a negative effect on the surface hardness of the wood material.

With respect to the varnish type, the average surface hardness values from the highest to the lowest were, respectively, polyurethane varnish (93.8), water-based varnish (40.3), and unvarnished samples (33.3). With regard to the Tukey test, varnish types formed different groups in terms of hardness, as can be deducted from the HG graph. In the HG chart, the surface hardness values of water-based varnish and unvarnished samples were low and relatively close to each other. It is observed that the surface hardness value was quite high in polyurethane varnish.

Average surface hardness value changes according to wood, treatment, and varnish type are given in Table 5 and its graph in Fig. 2.

Table 5. Average Surface Hardness Values Comparisons of Wood, Treatment, Varnish Type

Source			Means	95% Confidence Interval	
Wood	Treatment	Varnish		Lower Limit	Upper Limit
Scotch Pine	Control	WB	36.2	31.158	41.242
		PU	93.2	88.158	98.242
		UVR	27.6	22.558	32.642
	Tannin	WB	38.4	33.358	43.442
		PU	83.2	78.158	88.242
		UVR	24.9	19.858	29.942
Walnut	Control	WB	43.8	38.758	48.842
		PU	100.4*	95.358	105.442
		UVR	44.3	39.258	49.342
	Tannin	WB	42.6	37.558	47.642
		PU	98.4	93.358	103.442
		UVR	36.2	31.158	41.242

*: The highest surface hardness Std. Error: 2.544

With regard to wood-treatment-varnish type, the highest average surface hardness value was found in walnut + control + polyurethane varnish (100.4), and the lowest was in Scots pine + tannin + unvarnished (24.9) samples.

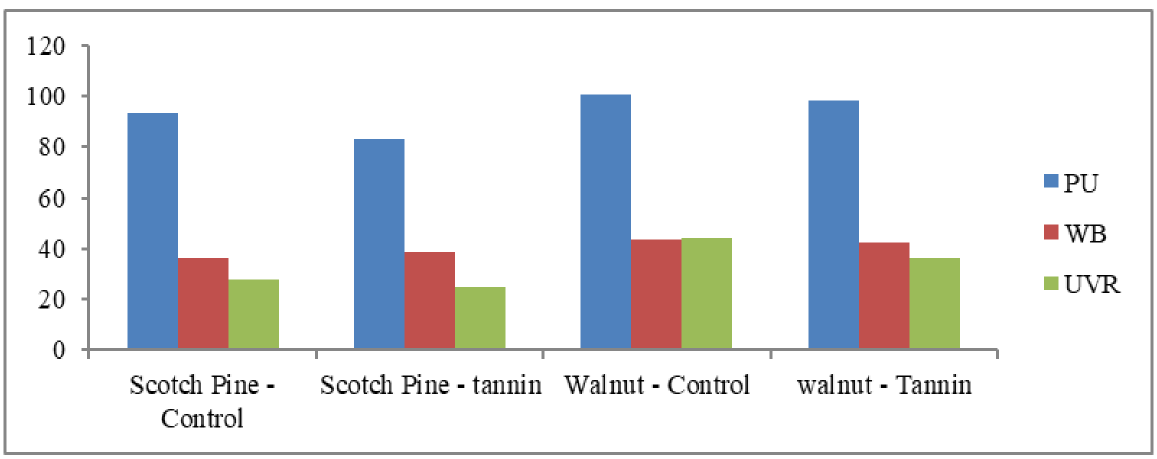

Fig. 2. Average surface hardness values corresponding to wood-treatment-varnish type

Polyurethane varnish had the highest average surface hardness values corresponding to wood-treatment-varnish type. When the surface hardness bar graph was analyzed, it is observed that there was an exceptional and reverse case in the walnut + control + unvarnished test samples when compared with the water-based varnish. When this situation was evaluated in terms of the other samples, the effect appears to have been caused by the tannin.

In this study, it was aimed to determine the surface layer thickness and hardness values of the varnishes as a result of the top surface applications. The results of this study are at a level that will shed light on future scientific research and this study can be a reference. The current study focused on materials that contribute to the sustainability of the wood material without disturbing the life cycle of the product. This provides a potential alternative to toxic preservatives. Such alternative products have become widespread thanks to consumer preferences and increasing environmental awareness. The findings obtained as a result of these studies will be investigated for alternative preservatives and top surface materials in wood protection to create economic value and to increase their usability by enterprises.

The layer thicknesses of the used varnishes were close to each other, and when the pH values of the used varnishes were studied, it was found that polyurethane varnish had a more acidic nature than the water-based varnish. The pH of almost all tree species is acidic (Zelinka and Stone 2011). It has been reported in the literature that acid and base variations between the varnish and the wood surfaces on which the varnish is applied can affect the hardness of the layer (Allen 1987; Mittal 1995; Nelson 1995; Budakci 2006; Budakci and Sonmez 2010; Kawalerczyk *et al*. 2022; Mai and Militz 2023).

The top surface hardness values of the test samples were examined before and after the tannin application. Before applying tannin and varnish, surface hardness measurements were carried out on Scots pine and walnut test samples. According to the wood type, the hardness value was 17.0% higher in walnut than in Scots pine samples.

After the varnish application, the highest increase rate in hardness was observed in the test samples applied with polyurethane varnish. With respect to the data obtained as a result of the study, it was determined that the application of tannin adversely affected the hardness values.

When the varnish layer hardness values were analyzed with regard to the interaction of tannin application and varnish type of both wood types, the highest values were obtained in control + polyurethane varnish (Yakin 2001; Sonmez and Budakci 2004).

Even though the approximately same layer thicknesses were measured in the applied varnishes, the water-based varnish had lower hardness. The reason may be related to both the chemical structure of the resin and greater penetration into the porous structure of wood. Such penetration may have reduced the surface hardness (Sonmez 1989; Budakci 2003; Ceylan 2016).

After the applied varnish processes, it was observed that the hardness values of the varnishes mostly increased, and the percentage increase was higher for hardness in Scots pine than walnut. It is believed that this may have resulted from the fact that that the used woods have different anatomical structures, extractive material contents, and permeability differences.

In this study, walnut tannin at a certain concentration was applied to the Scots pine and walnut wood surfaces in three layers with the brush method. In other studies, various analyses can be carried out on tannins. Different application methods with different tannins and various concentrations can be tested on widely used wood types, especially those that are used outdoors.

CONCLUSIONS

This study investigated the surface hardness on Scots pine (*Pinus sylvestris* L.) and walnut (*Juglans regia* L.) woods modified with walnut tannin.

1. Walnut exhibited higher average surface hardness values compared to Scots pine. This can be attributed to the extractive-rich nature and anatomical structure of walnut, which enhanced its properties.
2. Tannin application generally reduced the surface hardness values for both wood species. This reduction is likely due to the tannin particles affecting the surface structure of the wood.
3. Varnishes increased surface hardness values compared to control samples. Polyurethane varnish resulted in the highest surface hardness values across all samples, significantly outperforming water-based varnish and unvarnished samples. The chemical composition and better penetration capabilities of polyurethane varnish contribute to its superior performance.

These findings highlight the importance of considering the characteristics of the wood, the treatment process, and the choice of varnish when aiming to achieve desired surface hardness to produce more durable and resistant wood products. Further research may be needed to explore the specific mechanisms behind these interactions and to optimize the use of tannin and varnishes in wood applications.

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