

EFFECTS OF THERMAL AGING ON THE FILM HARDNESS OF SOME WOOD VARNISHES

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This study was performed to determine the effects of thermal aging on the film hardness of some wood varnishes. For this purpose, Scots pine (*Pinus sylvestris* L.), Eastern beech (*Fagus orientalis* L.), and oak (*Quercus petraea* L.) samples coated with synthetic (alkyd), two-part polyurethane (urethane-alkyd), and waterborne (self cross-linked polyurethane) varnishes were evaluated at a moisture content of 8% and 12%. Afterwards, thermal aging processes were performed for periods of 25, 50, 75, and 100 days at 25 °C, 50 °C, 75 °C, and 100 °C. Hardness changes in the varnish films were measured in accordance with ISO 1522-2006. According to the test results, the samples prepared with polyurethane varnishes at 8% moisture content give the best results.

Keywords: Wood material; Wood varnishes; Thermal aging; Hardness; Moisture content

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INTRODUCTION

Anatomical structure, physical and mechanical properties, as well as chemical composition make wood materials suitable for use in many different products (Bozkurt and Göker 1987). However, a protective coating for the wood material is necessary to ensure sufficient protection against dimensional change under variable atmospheric conditions, such as decay, insect damage, fire, mechanical shocks, and other harmful effects (Hafizoğlu *et al.* 1994). These potential vulnerabilities due to the specific properties of wood can be alleviated by drying, impregnation, and coating with a protective layer of varnish or paint (Kurtoğlu 2000).

Over time, the physical, chemical, and mechanical stresses on wood coatings cause a weakening in the strength of cohesion and adhesion in the film layer and decrease the expected performance (Sönmez 2000). Therefore, the life and resistance of varnish and paints is mainly restricted by aging (Feller 1994). Ultraviolet rays are known to have an aging effect on some polymeric materials (plastic and wood material). It has been stated that temperature in particular plays a key role in the aging process (Andrady *et al.* 1998). In addition, physical and chemical aging causes internal tension in the structure of organic varnishes and paints. The cracking resistance of the top layer exists within 25°C to 60°C, but above 80°C the wood starts to get rigid. While the brightness crossing point is shown as a function of applied temperature and time, it is more important that

temperature and time are applied to characteristics of the varnish in long-term protection (Holzhausen *et al.* 2002).

The photo-and thermal-degradation durability of two- package polyurethane coating, the urethane (NH-COO-) group is the most sensitive to photo-degradation (Decker *et al.* 2004). One of components is aromatic isocyanate, which is sensitive to photo-degradation due to the benzene ring linking the urethane group. As a consequence it is easy to form a quinone imide structure upon exposure to UV light.

When layers of varnish or paint are exposed to various moisture and temperature conditions in a UV test, UV-degradation may be added on top of damages caused by temperature and humidity. As a result of this, micro-cracking occurs (Ochs and Vogelsang 2004). In a different study, a polyurethane topcoat system was exposed to UV aging and it was reported that high temperature plays an important role in the degradation of varnish molecules on the surface. Bubble formation was observed, resulting in an increase in surface roughness and a decrease in surface brightness (Yang *et al.* 2002). While UV radiation carried by the rays of the sun drives photo-oxidation, the sun also creates high temperature and humidity, thermal aging, and hydrolysis. Resistant polymer bonds are also broken as a result of the photo-oxidation (Oosterbroek *et al.* 1991; Perera and Oosterbroek 1994; Perera 1995, 1998, 2001). It has been stated that the varnish in contact with the solid substrate to be coated is hardened by heating at about 120°C for a time not exceeding 2 hours. The thickness of the hardened coating is generally between 1 and 30 micrometers, while a thickness of about 5 to 10 micrometers most often considered to be suitable. After a predrying in the air for 10 to 30 minutes, the varnish is baked at a temperature of about 120°C for a period of an hour or two to assure its hardening (Vantillard *et al.* 1988).

The long-term durability of varnishes applied to wooden surfaces with respect to mechanical effects, such as friction, abrasion, and impact, depends on the resistance of the varnish layers to these effects. Varnished wooden surfaces are exposed to various effects, depending on the environments in which they are used. Therefore, in order to prevent economic losses, the use of varnish types that supply optimum efficiency according to the usage area is required. The aim of this study is to analyze the importance of temperature on the physical changes in the formation of coatings on woods. Polyurethane, synthetic, and waterborne formulations were applied by spray gun, and the effects of these applications on the surface hardness on the varnish layer were determined, holding other aging factors constant.

EXPERIMENTAL

Materials

Wood material

Wood samples of Scots pine (*Pinus sylvestris* L.), Eastern beech (*Fagus orientalis* L.), and oak (*Quercus petraea* L.) were used during experiment preparation due to their common usage in the furniture and decoration industry in Turkey. The samples were prepared from the sapwood parts of randomly selected first-grade timbers; they were chosen to be regular-fiber, knotless, crack-free, exhibiting no variation in color or

density, and having annual rings perpendicular to the surface, with regard to the principles in TS 2470 (1976).

The samples with a moisture content ensured by air-drying were cut into the dimensions of 110x110x12mm as roughcast. Then, the samples were left in air-conditioning cabinets; at $20 \pm 2^\circ\text{C}$ temperature and $42 \pm 5\%$ relative humidity for 8% moisture content, and at $20 \pm 2^\circ\text{C}$ temperature and $65 \pm 5\%$ relative humidity for 12% moisture content until their mass no longer varied (TS 2471 1976). The samples were then dimensioned to 100x100x10 mm and sanded with 80-grit (on Norton scale) sandpaper and then with 100-grit sandpaper for varnishing. According to the experimental design, a total of 1440 pieces were prepared by creating 4 samples in order to obtain data for each factor such as 3 wood type, 2 moisture content, 3 varnish types, 4 thermal processing temperature, and 5 thermal processing durations.

Varnishes

Synthetic, two-component polyurethane, and water-based varnishes were used to varnish the test samples. Synthetic and two-component polyurethane are reactive finishes. They are composed of small molecules that resemble the blocks in a set of Tinker Toys. In a can of finish these molecules are floating in a thinner. As the thinner evaporates, the molecules approach each other and connect either with the help of oxygen (synthetic varnish) or with the aid of a catalyst, activator, crosslinker, or hardener (two-component polyurethane). Water-based varnishes are the only coalescing finishes. They are composed of droplets (latexes) resembling microscopic soccer balls with plastic covers and solid insides. The insides are reactive finish that has been crosslinked. The droplets are suspended in water and a very slow evaporating solvent. The water evaporates first. The solvent then softens the outside of droplets (as solvent would soften the outer skin on plastic soccer balls). The droplets become sticky and stick together when solvent evaporates (Flexner 2005). The application conditions of varnishes were prepared according to the manufacturer's suggestion and in accordance with the standard ASTM D 3023 (1998). Technical specifications of the varnishes and application systems used are given in Table 1.

Table 1. Technical Specifications of Varnishes and Application Systems Used

Varnish Type	pH	Density (g/cm ³)	Application viscosity (sn DIN Cup/4mm)	Amount of finish application (g/m ²)	Solid content (%)	Spray gun tip diameter (mm)	Air pressure (Bar)
Polyurethane (Filling)	5.94	0.98	18	125	48.1	1.8	2
Polyurethane (Topcoat-Gloss)	4.01	0.99	18	125	44.2	1.8	2
Synthetic (Gloss)	5.51	0.94	18	100	53.2	brush	brush
Waterborne (Primer)	9.17	1.014	18	100	14.20	1.3	1
Waterborne (Filling)	9.30	1.015	18	67	34.13	1.3	1
Waterborne (Topcoat-Gloss)	8.71	1.031	18	67	31.83	1.3	1

Synthetic varnish was applied with a brush as 2 coats filling and 2 coats topcoat. Firstly, polyurethane and water-based filling varnishes were applied on the sample surfaces; then the same type of 2 coats topcoat varnishes were applied on those at room temperature ($\sim 20^{\circ}\text{C}$), with a spray gun. The amount of varnish applied was determined by weighing with a sensitive analytical scale of ± 0.01 g. The samples were then dried at 20°C and at a relative humidity of 65 ± 5 % under laboratory conditions and kept to be reached a constant weight (ASTM D 3023 1998; Budakçı and Sönmez 2010).

Methods

Thermal aging

Varnished experimental samples were exposed to thermal aging at 25°C , 50°C , 75°C , and 100°C temperatures in dry air sterilizers (ovens) for a period of 25 days, 50 days, 75 days, and 100 days, respectively, and kept in the air-conditioned cabinet until reaching an 8% to 12% equilibrium moisture content.

Pendulum hardness test

The changes in hardness of the varnish films were determined using the pendulum hardness tester according to the principles of ISO 1522 as shown in Fig 1. The device is placed on the sample, and the hardness is determined according to the pendulum oscillations. The pendulum swings with two balls that have a hardness of $\text{HRC } 63 \pm 3.3$ and are 5 ± 0.0005 mm in diameter. Surfaces that have more oscillations are harder surfaces, and those with fewer oscillations have lower hardness (Sönmez 1989; ISO 1522 2006).

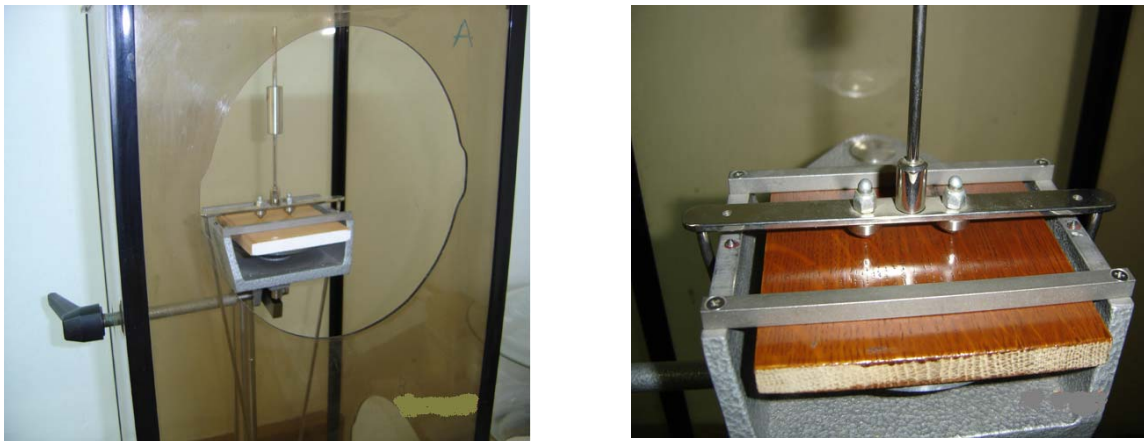


Fig. 1. Pendulum hardness tester and the application of experiments

Statistical evaluation

In the evaluation of data, the statistical package software MSTATC was used. In the analysis, the values of factors were determined as a result of multiple variance analysis. Factor effects were considered significant with $\alpha = 0.05$ error rate. According to variance analysis “ANOVA” results, Least Significant Difference (LSD) critical values were used, and causing factors were determined.

RESULTS AND DISCUSSION

Hardness Value Results of Scots Pine Samples

The arithmetic mean values of hardness for the Scots pine samples were obtained, taking into account the following factors: moisture content, type of varnish, thermal processing temperature, and thermal processing time. To determine which factor(s) caused difference, multiple variance "ANOVA" analysis was carried out. The results are given in Table 2.

Table 2. Results of Variance Analysis of Scots Pine Samples

Source of Variance	Degrees of freedom	Sum of squares	Mean square	F value	Prob. $\alpha=0.05$
Factor A	1	5148.300	5148.300	286.2154	0.0000*
Factor B	2	107112.188	53556.094	2977.4062	0.0000
Interaction AB	2	1748.413	874.206	48.6008	0.0000
Factor C	3	9595.408	3198.469	177.8162	0.0000
Interaction AC	3	8080.850	2693.617	149.7494	0.0000
Interaction BC	6	10941.279	1823.547	101.3785	0.0000
Interaction ABC	6	7277.488	1212.915	67.4310	0.0000
Factor D	4	48270.029	12067.507	670.8830	0.0000
Interaction AD	4	4661.346	1165.336	64.7859	0.0000
Interaction BD	8	15456.958	1932.120	107.4146	0.0000
Interaction ABD	8	3293.817	411.727	22.8896	0.0000
Interaction CD	12	23919.737	1993.311	110.8165	0.0000
Interaction ACD	12	23624.421	1968.702	109.4483	0.0000
Interaction BCD	24	15613.325	650.555	36.1671	0.0000
Interaction ABCD	24	12942.867	539.286	29.9812	0.0000
Error	360	6475.500	17.988		
Total	479	304161.925			

Factor A: Moisture content, B: Varnish type, C: Thermal processing temperature, D: Thermal processing time

* Meaningful (according to $\alpha=0.05$)

According to the results in Table 2, all of the main factors and their interactions were statistically significant at $\alpha=0.05$. Then, using the critical value of LSD, comparison results of the Duncan test are given in Table 3.

According to Table 3, the difference between the varnish layer and hardness values of the samples with 8% and 12% level of moisture content was significant. The hardness value of samples with 8% moisture content was found to be higher. It was found that the highest hardness value was obtained from polyurethane varnish and the lowest hardness was from synthetic varnish. The highest hardness values were obtained from the experimental samples exposed to 100°C temperature, and the samples aged 75 and 100 days were not significantly different. These samples showed higher values than the test samples aged 25, 50 days, and the control.

Table 3. Comparison Results of Duncan Test of Scots Pine Samples

Moisture content	\bar{x}	HG
% 8	56.79	A*
% 12	50.24	B
LSD \pm 0.7608		
Varnish type	\bar{x}	HG
Synthetic	38.58	C
Polyurethane	73.92	A*
Water borne	48.04	B
LSD \pm 0.9317		
Thermal processing temperature ($^{\circ}$ C)	\bar{x}	HG
25	47.14	D
50	51.53	C
75	57.14	B
100	58.23	A*
LSD \pm 1.076		
Thermal processing time (Days)	\bar{x}	HG
Control	36.77	D
25	47.45	C
50	58.25	B
75	62.36	A*
100	62.73	A*
LSD \pm 1.203		

\bar{x} : Average value HG: The homogeneous group * : The highest hardness value.

Table 4. Results of Variance Analysis of Eastern Beech Samples

Source of Variance	Degrees of freedom	Sum of squares	Mean square	F value	Prob. $\alpha=0.05$
Factor A	1	3234.408	3234.408	308.2428	0.0000*
Factor B	2	107311.717	53655.858	5113.4637	0.0000
Interaction AB	2	2526.017	1263.008	120.3661	0.0000
Factor C	3	9893.467	3297.822	314.2862	0.0000
Interaction AC	3	7493.625	2497.875	238.0503	0.0000
Interaction BC	6	12242.933	2040.489	194.4609	0.0000
Interaction ABC	6	7064.700	1177.450	112.2123	0.0000
Factor D	4	47409.012	11852.253	1129.5331	0.0000
Interaction AD	4	6909.071	1727.268	164.6106	0.0000
Interaction BD	8	23515.512	2939.439	280.1318	0.0000
Interaction ABD	8	2362.129	295.266	28.1392	0.0000
Interaction CD	12	18255.304	1521.275	144.9793	0.0000
Interaction ACD	12	14178.479	1181.540	112.6021	0.0000
Interaction BCD	24	19744.171	822.674	78.4017	0.0000
Interaction ABCD	24	8617.821	359.076	34.2203	0.0000
Error	360	3777.500	10.493		
Total	479	294535.867			

Factor A: Moisture content, B: Varnish type, C: Thermal processing temperature, D: Thermal processing time
* Meaningful (according to $\alpha= 0.05$)

Hardness Value Results of Eastern Beech Samples

The arithmetic mean values of hardness measurements of the Eastern beech samples were obtained as affected by moisture content, type of varnish, thermal processing temperature, and thermal processing time. Multiple variance "ANOVA" analysis was used to determine which factors affected film hardness. The results are given in Table 4.

According to the results in Table 4, interactions among factor and factors were found to be statically significant at $\alpha=0.05$. Using the critical value of LSD, comparison results of Duncan test were done on the levels of thermal processing time, moisture content, varnish type, and thermal processing temperature (Table 5).

Table 5. Comparison Results of Duncan Test of Eastern Beech Samples

Moisture content	\bar{x}	HG
% 8	60.91	A*
% 12	55.72	B
LSD \pm 0.5810		
Varnish type	\bar{x}	HG
Synthetic	41.64	C
Polyurethane	77.91	A*
Water borne	55.40	B
LSD \pm 0.7116		
Thermal processing temperature ($^{\circ}$ C)	\bar{x}	HG
25	51.67	D
50	56.97	C
75	60.83	B
100	63.80	A*
LSD \pm 0.8217		
Thermal processing time (Day)	\bar{x}	HG
Control	43.88	E
25	49.89	D
50	61.09	C
75	66.85	B
100	69.88	A*
LSD \pm 0.9187		

\bar{x} : Average value HG: The homogeneous group *: The highest hardness value

According to Table 5, moisture content significantly affected the hardness of the varnish films. The hardness value of samples with 8% moisture content was higher. It was found that highest hardness value was polyurethane varnish and the lowest hardness was obtained from the synthetic varnish. The hardness value of experimental samples exposed to a temperature of 100 $^{\circ}$ C was found to be higher at the level of thermal processing temperature. When compared at the level of thermal processing time, the highest hardness value was obtained from the experimental samples aged for 100 days.

Hardness Value Results of Oak Samples

The arithmetic mean of hardness values from the Eastern beech samples was obtained as affected by moisture content, type of varnish, thermal processing

temperature, and thermal processing time. Factor(s) showing a significant effect were identified by variance "ANOVA" analysis. The results are given in Table 6.

Table 6. Results of Variance Analysis of Oak Samples

Source of Variance	Degrees of freedom	Sum of squares	Mean square	F value	Prob. $\alpha=0.05$
Factor A	1	4845.052	4845.052	259.0648	0.0000*
Factor B	2	125395.704	62697.852	3352.4528	0.0000
Interaction AB	2	1004.754	502.377	26.8621	0.0000
Factor C	3	8157.190	2719.063	145.3883	0.0000
Interaction AC	3	9605.940	3201.980	171.2098	0.0000
Interaction BC	6	9314.179	1552.363	83.0048	0.0000
Interaction ABC	6	10832.429	1805.405	96.5350	0.0000
Factor D	4	53445.033	13361.258	714.4262	0.0000
Interaction AD	4	5506.667	1376.667	73.6103	0.0000
Interaction BD	8	14249.879	1781.235	95.2426	0.0000
Interaction ABD	8	3158.621	394.828	21.1114	0.0000
Interaction CD	12	16465.300	1372.108	73.3666	0.0000
Interaction ACD	12	17613.300	1467.775	78.4819	0.0000
Interaction BCD	24	14960.988	623.374	33.3318	0.0000
Interaction ABCD	24	8659.113	360.796	19.2918	0.0000
Error	360	6732.750	18.702		
Total	479	309946.898			

Factor A: Moisture content, B: Varnish type, C: Thermal processing temperature, D: Thermal processing time * Meaningful (according to $\alpha= 0.05$)

Table 7. Comparison Results of Duncan Test of Eastern Beech Samples

Moisture content	\bar{x}	HG
8%	58.53	A*
12%	52.17	B
LSD±0.7757		
Varnish type	\bar{x}	HG
Synthetic	36.91	C
Polyurethane	76.27	A*
Water borne	52.86	B
LSD±0.9500		
Thermal processing temperature (°C)	\bar{x}	HG
25	49.96	D
50	53.23	C
75	57.33	B
100	60.88	A*
LSD±1.097		
Thermal processing time (Day)	\bar{x}	HG
Control	37.98	D
25	49.42	C
50	58.05	B
75	66.13	A*
100	65.17	A*
LSD±1.227		

\bar{x} : Average value HG: The homogeneous group *: The highest hardness value

The critical value of LSD by comparison results of the Duncan test identified which levels of thermal processing time, moisture content, varnish type, and thermal processing temperature were significantly different (Table 7).

According to Table 7, moisture content significantly affected hardness of the varnish films. The hardness value of samples with 8% moisture content was the highest. It was found that highest hardness value was polyurethane varnish and the lowest hardness was for the synthetic varnish. The hardness value of experimental samples exposed to a temperature of 100°C was found to be higher at the level of thermal processing temperature. The surface hardness values obtained from the test samples aged 75 and 100 days were not significantly different and were all higher values than the test samples aged 25, 50 days, and the control.

CONCLUSIONS

According to the findings, the highest pendulum hardness value (60.91) of the samples at 8% moisture content was found on the beech. The pendulum hardness values obtained were 56.79 from scots pine and 58.53 from oak. The average pendulum hardness values of samples at 12% moisture content were 55.72 for beech, 52.17 for oak, and 50.24 for pine samples. It is thought that the reason beech material had the highest hardness is because it has diffused small fibers or pores, a homogeneous structure, and a high density (Berkel 1970; Bozkurt and Erdin 1997). It has been discussed in the literature that woods that have more internal surface area and lower density have decreased hardness values (Sönmez *et al.* 2004). Many mechanical and technological properties of wood are proportional to its density, such as swelling by taking in moisture, shrinking by taking out moisture, the amount of heat value, abrasive effects, hardness, and processing capability (Berkel 1970). In this context, the study is in accordance with the literature.

Pendulum hardness values of samples at 12% moisture were lower than samples at 8% moisture content. It is explained in the literature that because the thermal aging process was applied to the samples, the wood moisture was reduced and hardness increased (Kantay 2007). In addition, thermal processes cause some changes to the chemical and physical properties of wood material, and the cause of change is usually shown by the thermal degradation of hemicelluloses. Theoretically, hydroxyl (OH) groups of hemicelluloses have a significant impact on physical properties of wood material. As a result of thermal processing, a large reduction in amount of hydroxyl groups in wood has been reported (Inoue *et al.* 1993; Sevim Korkut *et al.* 2008; Boonstra 2008; Korkut and Budakçı 2009). The reduction of hemicelluloses occurs in both crystallite zones, in the chemical structure of wood material and the composition of cellulose. When associated with flexibility, more rigid cellulose-cellulose bonds replace the hemicellulose-cellulose-hemicellulose-cellulose bonds. This change in the molecular structure of wood material reduces its elasticity, making it more brittle and giving it a harder structure (Joščák *et al.* 2007; Phuong *et al.* 2007; Kocaefe *et al.* 2008; Korkut and Budakçı 2009).

The control group and experimental samples treated at 25°C were determined to have lowest value of pendulum hardness. The increase in thermal processing duration and temperature causes an increase in hardness value. The highest pendulum hardness value was obtained from polyurethane varnished beech samples subjected to thermal aging for 100 days at 8% humidity at 100°C temperature. The lowest pendulum hardness value was attained from the control samples of untreated yellow pine at 12% moisture with synthetic varnish. The pendulum hardness value also rose as thermal aging time increased. Increasing the temperature during the thermal aging process can increase molecular cohesion among resin molecules in the varnish layer, resulting in a harder film. The layers of polyurethane varnish are composed of large molecules created by copolymerization reactions, and because of higher molecular cohesion the pendulum hardness values were higher (Budakçı 1997; Budakçı 2003; Sönmez and Budakçı 2004). The pendulum hardness values from samples varnished with synthetic varnish were lower than samples with polyurethane varnish and water-based varnish. Due to the composition of synthetic varnishes containing a certain amount of oil alkyd, they are more elastic, resulting in a low hardness, which is consistent with the literature (Kurtoğlu 2000; Sönmez 1989; Sönmez and Budakçı 2004).

Increased thermal processing temperature and duration resulted in increased pendulum hardness values from polymeric varnish layers. The experimental samples prepared with polyurethane varnish at 8% moisture gave the best results.

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