Impact of Impregnation and Bleaching on the Surface Hardness of Oak (*Quercus petraea* L.) Wood

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ABSTRACT: The impact of impregnation and bleaching on the hardness of varnish layers on oak (*Quercus petraea* L.) wood was investigated. A number of solutions [sodium hydroxide (NaOH) and hydrogen peroxide (H_2O_2); NaOH, calcium hydroxide, and H_2O_2; NaOH, magnesium sulfate, and H_2O_2; sodium bisulfate and $H_2C_2O_4 \cdot 2H_2$; sodium silicate and H_2O_2; and potassium permanganate, sodium bisulfate, and H_2O_2] were applied at a concentration of 18% to bleach both impregnated [Tanalith-CBC (T-CBC) or Imersol-WR 2000 (I-WR 2000)] and unimpregnated (natural) wood panels. Subsequently, a water-based varnish (WB) was coated over the samples, and the hardness of the varnished layers was determined in accordance with ASTM D 4366. Among the samples that were varnish-coated without bleaching, T-CBC/WB yielded the highest hardness (59.50), whereas I-WR 2000/WB exhibited the lowest (49.17). However, among the samples varnish-coated after bleaching, the highest (56.50) and lowest (40.83) varnish hardness values were obtained with T-CBC/solution 2/WB and I-WR 2000/solution 4/WB, respectively. All the chemicals used for the bleaching process reduced the surface hardness. However, after the varnish coating, except for solutions 4 and 6, all the solutions showed hardness values similar to those of varnish-coated natural (control) samples. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 498–504, 2004

Key words: coatings; hardness; surfaces

INTRODUCTION

Having numerous superior properties, wood products remain among the most important materials in many applications. The increasing demand for wood-based products and deforestation have obliged researchers to search for methods for the long-term utilization of wood products. It is known that the constitutional components of wood are subjected to some degradation and modification because of the effects of chemical, biological, and physical factors in the surrounding environment. Thus, before they are used, wood materials are exposed to some processes, such as seasoning, chemical treatments, and surface treatments.[1] Varnish is one of most common coverings used to protect the surface against external factors and to make the natural appearance more prominent.[2]

Odors, colors, patterns, and many other physical characteristics of wood species differ. Color distinction may occur because of bruises on living parts of the tree, the formation of dead knots, diseases, and so forth. In addition, the oxidation of some chemicals in wood, the formation of heartwood in older trees, and metal contact with tannin wood are also known to cause changes in the natural color of wood.[3] Furthermore, differences between the specific weights of the growing rings may also result in color distinction.[4]

The color of furniture is as important as its shape, dimensions, form, and balance. In internal decoration, carpets, curtains, and so forth should be in good harmony with wall, ceiling, and base coverings. The natural color of wood materials, in many cases, cannot match these requirements. Therefore, to provide color harmony, wood is bleached before surface treatments.

Bleaching is a process in which some specific chemical solutions are applied to lighten the color of wood. In the furniture industry, this process is carried out on some tree woods (mahogany, oak, etc.) together with surface treatments.[5]

Bleaching and impregnation affect the wood structure and specifications such as the hardness, color, and brightness to some extent. The hardness of the varnished layer is the most important parameter for the protection of wood against external factors.

Impregnated and varnished wood specimens of Scots pine and chestnut were exposed to open air, and the changes in the color, hardness, brightness, and surface bonding strength were measured. For both species, a polyurethane varnish and a synthetic var-

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nish exhibited first and second degrees of hardness, respectively.[6]

Veneer samples taken from oak and Oriental beech were first varnished with celluloid, a synthetic varnish, a polyurethane varnish, and a polyester varnish, and then they were exposed to a treatment in sodium hydroxide (NaOH), acetone, detergent, and acetic acid (CH_3COOH) for 48 h. It was discovered that the CH_3COOH and detergent had no effect on the hardness of the tested varnishes. Moreover, acetone was found to have no effect on the hardness of the polyurethane and synthetic varnishes.[7]

The effects of external factors on the varnish layer hardness of Oriental beech, Scots pine, and chestnut wood samples were investigated. Applying synthetic, cellulose, polyurethane, and acid-hardening varnishes and white opacity paint to the samples and storing the samples under outdoor conditions for 22 months resulted in increased hardness of the varnished layers, except for the synthetic paint.[8]

Uysal et al.[9] studied the effects of the wood species, varnish, varnish concentration, and bleaching on the hardness of the varnish layer. The effects of the wood species were found to be insignificant. However, the varnish-type-induced effects were considerable in unbleached samples. Evaluations after the bleaching process indicated that the effects of the wood species, varnish type, and varnish concentration on the hardness of the varnish layer were significant.

In a study determining the effects of the varnish layer thickness on the hardness, brightness, and surface bonding strength, Oriental beech, Scots pine, and oak samples were coated with synthetic, polyurethane, and acrylic varnishes in different thicknesses. The highest hardness was obtained with a single-layer of polyurethane varnish applied to birch wood, whereas the lowest was obtained with a synthetic varnish applied to pine wood.[10]

After the application of cellulose, synthetic, polyurethane, and acrylic varnishes to massive oak and oak-coated wood panels, the varnish-coated layers were exposed to apple, orange, lemon, sodium hypochloride, and coke. Hardness loss was observed for all the varnishes tested, but coke and sodium hypochloride induced the maximum hardness loss.[11] After exposure to outdoor conditions for 12 months, yellow oak (*Pinus sylvestris* L.) wood panels were treated with NaOH/hydrogen peroxide (H_2O_2), calcium hydroxide [$Ca(OH)_2$]/H_2O_2, and sodium silicate ($NaSiO_3$)/H_2O_2 at a concentration of 18% for bleaching. Although the outdoor conditions reduced the hardness of yellow oak, bleaching eliminated the problem of reduced hardness.[12]

The aim of this study was to determine the effects of different impregnation and bleaching methods on the surface hardness of oak (*Quercus petraea* L.) wood, which is extensively used in the furniture industry.

TABLE I
Properties of the Color-Bleaching Chemicals

Chemical	Density (g cm^{-3})	Viscosity (cP/4 mm at 20°C)	pH
NaOH	1.131	65	13
H_2O_2	1.081	55	5
$Ca(OH)_2$	1.032	45	14
$MgSO_4$	1.001	50	7
$NaHSO_3$	1.130	45	5–6
$NaSiO_3$	1.068	50	12
$KmnO_4$	1.029	40	12
CH_3COOH	1.019	45	2
$H_2C_2O_4$	1.037	55	1–1.5

EXPERIMENTAL

Materials

Wood materials

Oak (*Q. petraea* L.), commonly used in the furniture, decoration, veneer, and floor-covering industries, was chosen as the experimental material. Wood samples were randomly selected from timber merchants of Ankara, Turkey, and were prepared in accordance with TS 1476.[13]

Bleaching chemicals

NaOH, H_2O_2, $NaSiO_3$, magnesium sulfate ($MgSO_4$), oxalic acid ($H_2C_2O_4$), $Ca(OH)_2$, sodium bisulfate ($NaHSO_3$), potassium permanganate ($KMnO_4$), and CH_3COOH were used for the bleaching treatments. These chemicals were obtained from commercial suppliers. The properties of the chemicals used for bleaching are given in Table I.

Impregnation materials

Tanalith-CBC (T-CBC) and Imersol-WR 2000 (I-WR 2000), used in the impregnation process, were supplied by Polisan (İzmit, Turkey). According to the manufacturer, T-CBC contains 38% $Na_2Cr_2O_7 \cdot 2H_2O$, 37.5% $CuSO_4 \cdot 5H_2O$, and 24% H_3BO_3, and the pH ranges from 1.6 to 3. I-WR 2000, an organic solvent impregnation substrate, is available as a ready-made solution. It is appropriate for the BS 5707[14] class I F2/NI and BS 5268[15] standards. The properties of the chemicals used for the impregnation of wood panels are presented in Table II.

Water-based varnish (WB)

WB is a colorless and odorless varnish that does not turn yellow with time and does not cause any changes in the properties of woody materials. It dries chemically and forms hard layers. It can be applied several times in the same day. It is resistant against acidic

TABLE II
Properties of the Impregnation Materials

Impregnation material	Viscosity (cP/4 mm at 20°C)	Solvent	Temperature (°C)	pH		Density (g/mL)	
				BI	AI	BI	AI
T-CBC	65	Distilled Water	23	3.05	3.05	1.080	1.080
I-WR 2000	45	—	23	6.75	6.75	0.82	0.82

BI = before impregnation; AI = After impregnation.

foods, such as mustard and vinegar. The layer thickness is between 70 and 80 μm under wet conditions and between 25 and 35 μm under dry conditions. The density is 1.00 ± 0.05 at 20°C, and it is a milky liquid.

The surfaces onto which WB is applied should be dry and cleaned of dust, dirt, and oily stains by sandpapering. WB can be applied in one to three layers to previously untreated surfaces and in one to two layers to previously painted surfaces. It takes about 30 min for the first layer to have touching resistance. For multilayer applications, the subsequent layers should be allowed approximately 4–6 h to dry. One liter of varnish has been reported to be sufficient to treat a 10-m^2 surface.[16]

In this study, Jansen WB (Jansen Farben and Lackhersteller, Ahrweiter, Germany), a single-compound polyurethane–acrylic resin (produced according to DIN 53160), was applied in accordance with ASTM D 3023.[17]

During the varnish preparation, to avoid inferior effects on the layer performance, the emulsion ratios were carefully calculated on the basis of the product specifications. The varnish viscosity was determined to be 18 s/4-mm-diameter flow cup at 20 ± 2°C and 60 ± 5% relative humidity (RH). The varnish was applied to the surfaces of test samples with hard-hair brushes. The solid content and dry film thickness of WB were determined to be 34.3% and 38 μm, respectively.

Methodology

Preparation of the test samples

Wood panels 190 mm (longitudinal) × 140 mm (tangential) × 15 (radial) mm were prepared from the air-dried sapwood of oak (*Q. petraea* L.) so that the growing rings were vertical to the surface. The samples, initially conditioned at 20 ± 2°C and 65 ± 3% RH, remained unprocessed until the moisture content was 12%. Subsequently, the sample dimensions were adjusted to 150 mm × 100 mm × 10 mm. The specimens were mainly divided into three experimental groups: impregnated with T-CBC, impregnated with I-WR 2000, and unimpregnated (natural control). All of these groups were exposed to bleaching (with six different solutions) and varnish-coating processes. In addition, differences in the surface hardness between the varnish-coated wood after bleaching and the same varnish-coated wood without bleaching were also considered to reflect the effects of WB on the impregnated and unimpregnated specimens. In all, 42 treatment groups were obtained.

Preparation of the impregnation solutions

T-CBC at a concentration of 13% was dissolved in distilled water. I-WR 2000 was used in its pure form. The temperature, density, and pH values of the prepared solutions were recorded before and after the impregnation process.

Impregnation method

ASTM D 1413-76 was considered for the impregnation process. Accordingly, the samples were exposed to a 60 $cmHg^{-1}$ prevacuum for 60 min and then were held in a solution under normal atmospheric pressure for 60 min to allow the diffusion of the impregnation material.[18]

Impregnated samples were stored under perfectly ventilated conditions for 15–20 days to maintain the complete evaporation of the solvents, and afterward they were kept at 20 ± 2°C and 65 ± 3% RH until they attained air-dry moisture (12%). The retention of impregnation materials (ER; kg m^{-3}) and the retention ratio (R; %) were calculated with the following equations:[19]

$$\mathrm{ER} = \frac{\mathrm{GC}}{\mathrm{V}} \times 10^3$$

$$R = \frac{\mathrm{DW} - \mathrm{DWB}}{\mathrm{DWB}} \times 100 \qquad (1)$$

$$G = T_2 - T_1$$

where G is the difference in the sample weight after impregnation (g), C is the emulsion concentration (%), V is the sample volume (cm^3), DW is the sample dry weight after impregnation (g), DWB is the sample dry weight before impregnation (g), T_1 is the sample weight after impregnation (g), and T_2 is the sample weight before impregnation (g).

TABLE III
Composition of the Bleaching Solutions

Solution	Composition	Neutralization substance
S_1	$NaOH + H_2O_2$	Distilled water
S_2	$NaOH + Ca(OH)_2 + H_2O_2$	Distilled water
S_3	$NaOH + MgSO_4 + H_2O_2$	Distilled water
S_4	$NaHSO_3 + H_2C_2O_4 \cdot 2H_2$	Distilled water
S_5	$NaSiO_3 + H_2O_2$	Distilled water
S_6	$KMnO_4 + NaHSO_3 + H_2O_2$	Distilled water

Bleaching

Six different solutions made of eight different chemicals are presented in Table III. Depending on their properties, the chemicals used for bleaching were prepared at a concentration of 18 vol % or 18 wt %.[20]

The prepared solutions were applied with a sponge to the surface of the samples, first parallel, then vertically, and then again parallel to the fibers at 100 ± 10 mL m^{-2}. The constitutive chemicals of the solutions were individually applied to the wood surfaces at 3-min intervals to allow the interactions of the previously applied chemicals with the wood. After the bleaching, for improved vertical penetration, the treated wood samples were allowed to dry at room temperature for 2 days, and then the neutralization process was performed with CH_3COOH and water. Finally, the sample moisture was adjusted to 12%, and the sample surfaces were slightly sandpapered before the varnish coating.

Varnish coating

After the treatment with bleaching solutions, the samples were coated with WB. Single-component WB was treated according to ASTM D 3023.[17] The surface to be treated was slightly sandpapered to remove protruded fibers, and it was cleaned of dust. Varnish (viscosity = 150 g m^{-2}) was applied to the surfaces with brushes as suggested by the manufacturer's instructions.

Test methods

After the bleaching and varnish coating, both the impregnated and unimpregnated test samples were conditioned at 23 ± 2°C and 50 ± 5% RH for 16 h.[21] The conditioned samples were subsequently subjected to a pendulum damping test to determine the hardness of the varnish layer.[22] The device was calibrated in due course. The device determined the layer hardness by means of the swing of a pendulum. The pendulum had marbles (5 ± 0.0005 mm in diameter) with a 63 ± 3.3 HRC (Conventional Hardness Rockwell). The amount of the swing was directly proportional to the surface hardness. Seven replications were conducted for each treatment group.

TABLE IV
Retention Amounts of the Impregnation Materials

Impregnation material	Retention (kg/m^3)		Retention (%)	
	$\bar{x}$	HG	$\bar{x}$	HG
T-CBC	4.85	B	1.60	B
I-WR 2000	162.56	A	24.78	A

$\bar{x}$ = arithmetic mean; HG = homogeneity groups.

Statistical evaluation

The effects of the color bleaching and impregnation chemicals on the hardness of the varnished layers were subjected to an analysis of variance (ANOVA). A Duncan test was also used when it was appropriate.

RESULTS

The retention of the impregnation materials is indicated in Table IV. The retention was higher for an I-WR 2000 solution. This could be attributed to the relatively lower viscosity of I-WR 2000.

The mean values of the surface hardness, as affected by different treatments, are given in Table V. ANOVA results indicating the effects of the treatment and solution types on the hardness of the varnish layer, including the interactions among the different treatments, are presented in Table VI. All bleaching solutions reduced the surface hardness of wood panels. This negative impact was maximal in solutions S_4 and S_6.

Among the T-CBC and I-WR 2000 impregnated and natural (unimpregnated) wood panels, the highest surface hardness was obtained in the T-CBC impreg-

TABLE V
Average Surface Hardness as Affected by Different Treatments

Treatment	$\bar{x}$
N	46.00
T-CBC	54.00
I-WR 2000	47.50
N + WB	50.17
T-CBC + WB	59.50
I-WR 2000 + WB	49.17
N + S	43.75
T-CBC + S	50.52
I-WR 2000 + S	42.89
N + S + WB	48.14
T-CBC + S + WB	52.33
I-WR 2000 + S + WB	44.49

$\bar{x}$ = arithmetic mean; N = natural; S = bleaching solution groups.

TABLE VI
ANOVA Indicating the Effects of the Treatment and Solution Types

Source	Degrees of freedom	Sum of squares	Mean of squares	*F* value
Treatments (A)[a]	5	3399.079	679.816	110.2688*
Solutions (B)[b]	6	1543.079	257.180	41.7156*
A × B	30	402.254	13.408	2.1749*
Error	210	1294.667	6.165	
Total	251	6639.079		

*p < 0.01 (p: level of significance).
[a]Natural, natural + WB, T-CBC impregnated, T-CBC + WB, I-WR 2000 impregnated, I-WR 2000 + WB.
[b]Control group (without bleaching treatment) and 6 different bleaching solutions.

nated samples. Thus, T-CBC tended to enhance surface hardness.

After the varnish coating, the mean hardness values were maximal in T-CBC/WB-treated samples and minimum in I-WR 2000/WB-treated samples. In fact, the main agent for the hardness of the varnish layer was the structure of the varnish, and the initial surface hardness of the natural wood panels had no effect on the hardness of the varnish-coated layer.

Bleaching solutions reduced the surface hardness by 6.5% in T-CBC-treated samples, by 5% in natural (unimpregnated) samples, and by 9.0% in I-WR 2000-treated samples. However, after the varnish coating, the reduction due to bleaching solutions could be considered ineffective.

In relation to the effects of the treatment type and bleaching solution on the surface hardness, the differences among the impregnated, unimpregnated, bleached, and nonbleached samples were significant ($p < 0.01$). The mean values for different combinations are shown in Table VII.

The average highest hardness of the varnish layer was observed in samples treated with T-CBC and WB, whereas the lowest was observed in samples treated with I-WR 2000, S_6, and WB. The variations of the final surface hardness due to different treatment combinations are shown in Figure 1.

As for the treatment types, the highest surface hardness was obtained from samples treated with T-CBC, and the lowest was obtained from samples varnished after I-WR 2000 impregnation. Bleaching solutions and I-WR 2000 had a reducing impact on the surface hardness. Nevertheless, T-CBC improved the surface hardness. The results reflected the interactions of bleaching, varnishing, and impregnation.

TABLE VII
Mean Comparison of the Final Surface Hardness at Different Treatment Combinations

Treatment	$\bar{x}$	HG[a]	Treatments	$\bar{x}$	HG[a]
T-CBC + WB	59.50	A	I-WR 2000 + S1 + WB	46.83	GHIIJKL
T-CBC + S2 + WB	56.50	B	I-WR 2000 + S3 + WB	46.33	HIJKL
I-WR 2000 + S5 + WB	55.00	B	N	46.00	IJKLM
T-CBC + S1 + WB	54.67	B	T-CBC + S5 + WB	45.83	JKLM
T-CBC	54.00	BC	I-WR 2000 + S2 + WB	45.50	KLM
T-CBC + S3 + WB	53.67	BC	I-WR 2000 + S1	45.17	KLMN
T-CBC + S2	51.50	CD	N + S2	45.00	KLMNO
T-CBC + S3	51.50	CD	N + S3	44.83	KLMNO
T-CBC + S5	51.17	CD	N + S5	44.67	KLMNO
T-CBC + S1	50.83	CDE	N + S1	44.17	LMNOP
T-CBC + S6 + WB	50.33	DEF	I-WR 2000 + S3	44.17	LMNOP
N + WB	50.17	DEFG	I-WR 2000 + S2	44.17	LMNOP
N + S2 + WB	50.00	DEFG	T-CBC + S4 + WB	43.83	LMNOP
N + S3 + WB	49.83	DEFG	I-WR 2000 + S5	43.67	LMNOPQ
N + S1 + WB	49.50	DEFGH	N + S6	42.83	MNOPQR
N + S5 + WB	49.50	DEFGH	N + S4 + WB	42.33	NOPQR
I-WR 2000 + WB	49.17	DEFGHI	I-WR 2000 + S6 + WB	41.67	OPQR
T-CBC + S4	49.17	DEFGHI	N + S4	41.00	PQR
T-CBC + S6	49.00	EFGHIJ	I-WR 2000 + S4 + WB	40.83	PQR
N + S6 + WB	47.67	EFGHIJK	I-WR 2000 + S6	40.50	QR
I-WR 2000	47.00	FGHIJKL	I-WR 2000 + S4	39.67	R

[a] Different letters in a column refer to significant differences among treatment groups at 0.05 confidence level. Least significant difference ($LSD_{0.5}$) = 2.823.
N = natural; S = bleaching solution group.

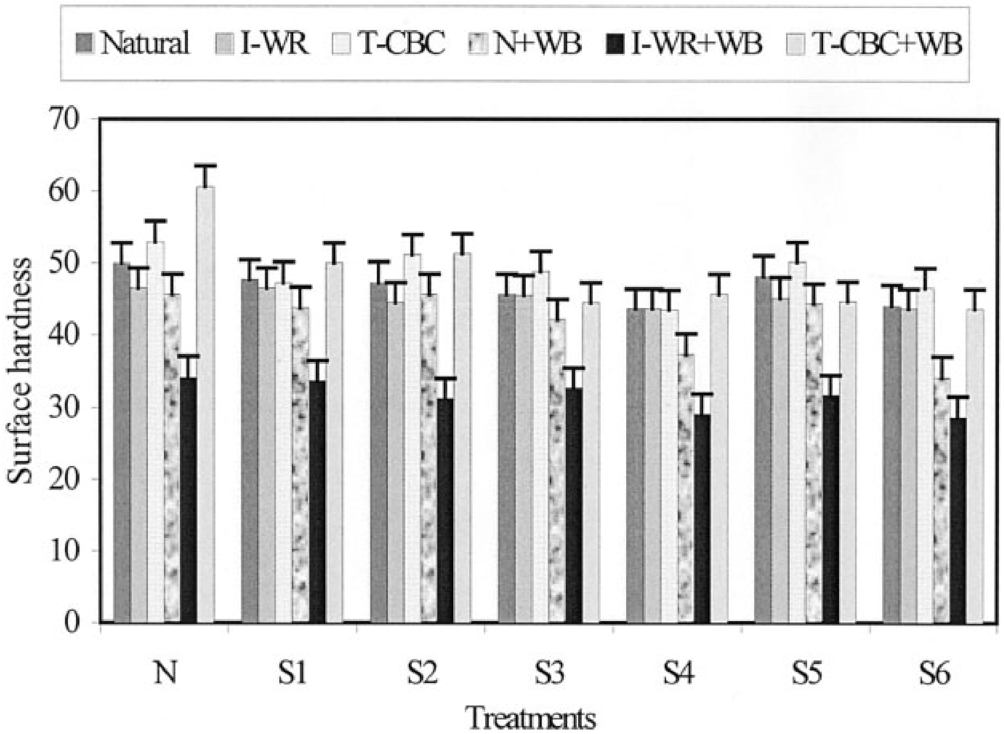

Figure 1 Variations of the surface hardness due to different treatment methods.

DISCUSSION

During the impregnation process, impregnation solutions were individually applied. The measured pH and density values of the solutions before and after the impregnation showed no significant variation. This was probably dependent on the freshness of the prepared solutions. The acidic nature of T-CBC prepared at a 13% concentration would probably have a negative effect on polysaccharides in wood and increase the possibility of hydrolysis.[23]

The highest retention was maintained with I-WR 2000, whereas the lowest was noted with T-CBC. Because of the lower viscosity and higher concentration, I-WR 2000 could penetrate into the cell wall better than T-CBC.[19]

The surface hardness of unvarnished samples impregnated with T-CBC and I-WR 2000 was higher than that of unvarnished natural samples, and this confirmed the findings of Örs et al.,[24] who claimed that impregnation increased the hardness values of both Scots pine and oriental beech. This may be attributable to the increase in the wood density upon the impregnation of chemicals.

Bleaching solutions had a reducing impact on the surface hardness. It was reported that an average reduction of 35% in the pressure strength and an average reduction of 30% in the bonding strength of Scots pine sapwood occurred after a treatment with 10% NaOH and a sulfuric acid solution.[25] These reductions were 10 and 15%, respectively, in the heartwood of Scots pine. In addition, Atar[26] determined that color bleaching chemicals caused the loss or reduction of O—H peaks in Scots pine, chestnut, oak, and oriental beech woods.

Among the bleached and unvarnished samples, the surface hardness values were maximum in the second solution (45.00) and minimum in the sixth solution (41.00). The hardness values of the samples treated with the bleaching solutions were lower than that of the control. With respect to the recorded hardness levels, all the bleaching solutions softened the wood panels.

In the unimpregnated and varnish-coated samples, after a treatment with bleaching solutions, the maximum surface hardness was observed in the samples treated with solution 2 (50.00), and the minimum was observed in the samples treated with solution 4 (42.33). After the varnishing, the effects of the bleaching solution groups on the hardness of the varnish layer were not significant, and the main impact was due to the varnish structure. Therefore, the average hardness of unimpregnated (natural) samples treated and not treated with bleaching solution groups were found to be 48.14 and 50.00, respectively. Bleaching solutions reduced the surface hardness of wood materials by 4.10% and the hardness of varnish layers by 1.32%.

In the T-CBC impregnated and unvarnished samples, the surface hardness was highest (51.50) in the second and third bleaching solutions, whereas it was lowest in the sixth solution (43.67). However, among the T-CBC impregnated samples varnished after the treatment with bleaching solutions, the highest surface hardness (56.50) was recorded for the second solution, whereas the lowest (43.83) was recorded for the fourth solution. Bleaching chemicals reduced the surface hardness of wood materials by 6.42% and the hardness of the varnish layer by 8.72%.

I-WR 2000 impregnation without varnishing resulted in a higher average surface hardness value (45.17) in the samples treated with the first bleaching solution. Along with this treatment group, the lowest hardness (39.67) was determined in the fourth solution. Among the I-WR 2000 impregnated samples varnished after the bleaching treatment, the maximum hardness (46.83) was obtained from the first solution, and the lowest (40.83) was obtained from the fourth solution. The hardness values of the varnished samples were lower than those of the unvarnished samples. This may be attributable to the monomer structure of I-WR 2000. Bleaching chemicals reduced the surface hardness of the wood materials by 8.74% and the hardness of the varnish layer by 9.50%.

Although the solutions affected the surface hardness, this impact was not marked after the varnishing. It was reported that, in unvarnished wood-coated panels and massive woods, the surface hardness decreased after a synthetic varnish coating, and the hardness before varnishing had no considerable effect on the hardness of the varnish layer.[27]

A study should be undertaken to inspect the effects of wood species and common varnish types on the hardness values of varnish layers after bleaching treatments. Furthermore, the influence of different bleaching solvent concentrations and varnish thicknesses on the surface hardness should be examined.

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