

Full Length Research Paper

Effects of impregnation with boron compounds on the surface adhesion strength of varnishes used woods

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The aim of this study was to determine the effects of impregnation with boron compounds on the surface adhesion strength of varnishes used woods. For this purpose, the test specimens prepared from Oriental beech (*Fagus orientalis* Lipsky) and European oak (*Quercus petraea* (Matt.) Liebl.) which met the requirements of ASTM D 358 were impregnated according to ASTM D 1413 with boric acid (Ba) and borax (Bx) by vacuum technique. After impregnation, surfaces were coated with cellulosic, synthetic, polyurathane, water-based, acrylic and acid hardening varnishes in accordance with ASTM D 3023 standards. According to ASTM D 4541 standards, the surface adhesion strength of specimens after varnishing process was determined. Considering the interaction of wood type, impregnation material and varnish type, adhesion strength values were found the highest in Oriental beech + boric acid + synthetic varnish (6.240 Mpa) and the lowest values in European oak + borax + cellulosic varnish (2.080 Mpa). Consequently, impregnation of wood material with boron compounds (boric acid: 3.677 Mpa and borax : 3.732 Mpa) showed increasing impact on the surface adhesion strength of cellulosic, synthetic, polyurathane, water-based, acrylic and acid hardening varnishes.

Key words: Adhesion strength, impregnation, boric acid, borax, varnishes, finishing, wood.

INTRODUCTION

Preserving wood material from environmental effects and providing a long usage period is economically important. Preserving and beautifying covering materials like paint, polish and varnish are used for this reason. Technical surface processes also increase economic, aesthetics and economic value of wood (NZFRI, 1996).

Furnitures coated only with paint and varnish have surface protection only for two years (Evans et al., 1992). So, after the impregnation with materials having appropriate water-repellent, biotic and abiotic effects, varnishing and painting applications are important for long-term utilization against photochemical degradation, dimensional changes, biological factors and fire (Williams et al., 1996).

In painting and varnishing applications with water-repellent materials, wood materials impregnated with boron are more resistant to environmental conditions (Harrow, 1991). Solution of copper, chrome and salt on the surface of wood material impregnated with copper,

chrome and boron make wood material more resistant to environmental effects (Sell and Feist, 1985). Processes like bleaching and impregnation affect the wood structure and specifications such as hardness, color and brightness and surface adhesion strength to some extent. Adhesion strength is one of the most important parameters for limiting the usage of varnishes. Adhesion is determinative in this sense (Atar, 1999).

According to wood and varnish type, adhesion strength value was the highest in Oriental beech + polyurathane varnish and lowest in Uludağ fir + cellulosic varnish (Budakçi, 2003). Adhesion strength of water-based varnish was lower than polyurathane, synthetic, acrylic etc. varnishes (Yakın, 2001). The highest adhesion strength (4.299 MPa) was produced by a combination of scotch pine, medium-duration immersion, and the use of a wood-based varnish. The lowest adhesion strength (2.090 MPa) resulted from the combination of Uludağ fir, long-term immersion, and a water-based varnish (Keskin et al., 2010).

It is reported that borate preimpregnation enhanced surface hardness and gloss of the wood surface but adhesion values of wood pre-impregnated with borates

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Table 1. Some properties of varnishes.

Type of varnish	pH value	Density (g.cm ⁻³)	Viscosity (snDIN Cup/4 mm)	Amount applied (g.m ⁻²)	Nozzle gap (mm)	Air pressure
Polyurethane (filler)	5.94	0.98	18	125	1.8	2
Polyurethane (finishing)	4.01	0.99	18	125	1.8	2
Synthetic	-	0.94	18	100	-	-
Water-based (primer)	9.17	1.014	18	100	1.3	1
Water-based (filler)	9.30	1.015	18	67	1.3	1
Water-based (finishing)	8.71	1.031	18	67	1.3	1
Cellulosic (filler)	2.9	0.955	20	125	1.8	3
Cellulosic (finishing)	3.4	0.99	20	125	1.8	3
Acrylic (filler)	4.3	0.95	18	125	1.8	2
Acrylic (finishing)	4.6	0.97	18	125	1.8	2
Acid hardening (finishing)	8.0	0.99	18	100	1.8	3

before varnish coating were lower than only varnish coated wood. Higher concentration levels of borates resulted in lower adhesion, higher surface hardness, and higher gloss of the wood surface (Toker et al., 2009).

Scots pine and chestnut woods impregnated with Tanalith CBC and Imersol WR-2000 were coated with synthetic and polyurethane varnishes, and weathered for four seasons. It was declared that the adhesion strength at the end of the fourth seasons was found to be the highest in the Scots pine samples coated with synthetic varnishes and lowest in the chestnut samples covered with polyurethane varnishes (Peker, 1997).

For Scots pine (*Pinus silvestris* Lipsky), adhesion strength of varnishes was decreased after lightening color application with 18% solution groups (Özçifçi et al., 1999). For chestnut (*Ulmus Camppestris* Spach.), adhesion strength of polyurethane and polyester varnishes was decreased after lightening color application with 18% solution groups (Sönmez et al., 2002).

The aim of this study was to determine the effects of impregnation of Oriental beech and oak with boric acid (Ba) and borax (Bx) on the surface adhesion strength of varnishes used woods.

MATERIALS AND METHODS

Wood material

The woods of oriental beech (*Fagus orientalis* Lipsky) and European oak (*Quercus petraea* (Matt.) Liebl.) were chosen randomly from timber merchants of Ankara, Turkey. Special emphasis was given to the selection of the wood material. Accordingly, non-deficient, proper, knotless, normally grown (without zone line, reaction wood, decay, and damages to insect mushroom) wood materials were selected according to TS 2470 (TS 2470, 1976).

Varnishes

Cellulosic (Sv), synthetic (Sn), polyurethane (Pu), water-based (Wb), acrylic (Ac) and acid hardening (Ah) varnishes were used

according to the producer's definition. Technical specifications of varnishes are given in Table 1 (DYO, 1996).

Impregnation material

Boron compounds (boric acid and borax) were obtained from Etibank-Bandırma (Turkey) boric and acid Factory. Properties of boric acid (H₃ B O₃) is 56.30% ½ B₂ O₃, 43.70% H₂O with a molecular weight 61.84, Density 1.435 g cm⁻³ and melting point 171°C. Borax (Na₂ B₄ O₇.5H₂ O) content is 21.28 Na₂O%, 47.80% B₂O₃, and 30.92% H₂O with a molecular weight of 291.3, density 1.815 g cm⁻³, and melting point 741°C (Keskin, 2009).

Methods

Preparation of test specimens

The rough drafts for the preparation of test and control specimens were cut from the sapwood parts of massive woods with a dimension of 190×140×15 mm³ and conditioned at a temperature of 20±2°C and 65±3% relative humidity till they reach 12% humidity distribution according to ASTM D 358 (ASTM D 358, 1983). The air-dried specimens with a dimension of 150×100×10 mm³ were cut from the drafts for impregnation and varnishing. The test specimens were impregnated with 5.5% boric acid and 5% borax according to ASTM D 1413 (ASTM D 1413-99, 2005). Accordingly, the samples were exposed to a 700 mm/Hg⁻¹ prevacuum for 60 min and then held in a solution under normal atmospheric pressure for 60 min to allow the diffusion of the impregnation material. The processes were carried out at 20±2°C. Retention of impregnation material (R) was calculated by the formula;

$$R = \frac{G \cdot C}{V} 10^3 \text{ kg m}^{-3} \quad (G = T_2 - T_1) \quad (1)$$

Where, G is the amount of impregnation solution absorbed by the sample, T₂ is the sample weight after the impregnation, T₁ is the sample weight before the impregnation, C is the concentration (%) of the impregnation solution and V is the volume of the samples.

Impregnated test specimens were kept under a temperature of 20±2°C and 65±3 % relative humidity until they reached a stable weight.

Table 2. Retention amount of impregnation material (kg m⁻³).

Wood type	x	HG*
Oriental beech (I)	30.87	A
European oak (III)	2.32	B
Impregnation materials	x	HG**
Boric Acid (Ba)	15.75	A
Borax (Bx)	9.71	B

*LSD = 2.012

Table 3. Retention amounts according to wood and impregnation material (kg m⁻³).

Type of process	X	HG
I+Ba	40.99	A
I+Bx	20.75	B
II+Ba	2.87	C
II+Bx	1.77	C

*LSD = 2.012.

Varnishing

Test specimens were varnished according to ASTM D 3023 (ASTM D 3023, 1998). The surfaces of specimens were sanded with abrasive papers to remove the fiber swellings and dusts are leaned before varnishing. Producer's definition is taken into care for the composition of solvent and hardener ratio and one or two finishing layers were applied after the filling layer. Spray nozzle distance and pressure are adjusted according to the producer's definition and moved in parallel to the specimen surface at a distance of 20 cm. Varnishing was done under 20±2°C temperature and 65±3% humidity conditions.

Adhesion strength measurements

Adhesion strength measurements were done according to ASTM D 4541 with adhesion test device working with pneumatic system (ASTM D 4541, 1995). Surfaces of test specimens were coated with protective layer and full-dried. Test cylinders with Ø 20 mm were adhered to these surfaces by using mold under normal room temperature.

Two hours later, gelled adhesive residues were removed by a spatula and left for drying for 24 h. Before tests, samples were conditioned at 23±2°C and 50±5% RH for 24 h according to TS EN 24624 (TS EN 24624, 1996). Later on, varnish layer was cut by a cutter till the wood material surface. In pneumatic adhesion device with 2 bar pressure, samples were pulled off from cylinders adhered to surface. Pulling process was completed in 90 s. Adhesion strength (σ_c) was calculated with the equation

$$\sigma_c = \frac{4F}{\pi d^2} \text{ MPa} \quad (2)$$

Where, F is maximum force (Newton), d is diameter of test cylinder (mm).

Table 4. Adhesion strength average values (MPa)

Wood material type	x	HG*
Oriental beech (I)	3.792	A
Oak (II)	3.323	B
Impregnation material	x	HG**
Control (C)	3.265	B
Boric Acid (Ba)	3.677	A
Borax (Bx)	3.732	A
Varnishes	x	HG***
Cellulosic varnish (Sv)	2.227	D
Synthetic varnish (Sn)	4.490	A
Polyurethane varnish (Pu)	3.823	B
Water-borne varnish (Wb)	3.730	B
Acrylic varnish (Ac)	3.737	B
Acid hardening varnish (Ah)	3.340	C

*LSD:0.1568 ** LSD:0.1927 *** LSD: 0.2725.

Statistical analysis

By using 2 different types of wood, 2 types of impregnation + 1 control specimen, 6 types of varnish a total of 180 specimens (2×3×6×5) were prepared with 5 specimens for each parameter. Multiple variance analysis was used to determine the differences in surface hardness values of specimens. Duncan Test was used to determine the significant difference between the groups.

RESULTS

Retention

Retention amount of impregnation material is given in Tables 2 and 3. R is found highest in oriental beech and boric acid and lowest in oak and borax.

Retention amount of impregnation materials according to wood material type and impregnation material is highest in oriental beech+boric acid and lowest in oak + borax. Accordingly, wood material type and impregnation material type are important for retention amounts.

Adhesion strength

Adhesion strength average values according to wood material type, varnish type and impregnation material are given in Table 4.

Adhesion strength was higher in Oriental beech than oak. According to varnish type, adhesion strength was highest in synthetic varnish and lowest in cellulosic varnish. Adhesion strength was highest in borax according to impregnation material type. Adhesion strength was higher in impregnated samples. Accordingly, impregnation materials have increasing effect for adhesion strength of varnishes. Average values

Table 5. Hardness average values according to wood, impregnation material and varnish type.

Types of material	x	HG
Impregnation + wood materials*		
I	3.213	B
I+Ba	4.157	A
I+Bx	4.007	A
II	3.317	B
II+Ba	3.179	B
II+Bx	3.457	B
Impregnation + varnishes**		
Sv	2.230	F
Ba+Sv	2.190	F
Bx+Sv	2.260	F
Sn	4.150	BC
Ba+Sn	4.860	A
Bx+Sn	4.460	AB
Pu	3.410	DE
Ba+Pu	3.700	CD
Bx+Pu	4.360	B
Wb	3.350	DE
Ba+Wb	4.130	BC
Bx+Wb	3.710	CD
Ac	3.520	D
Ba+Ac	3.550	D
Bx+Ac	4.140	BC
Ah	2.930	E
Ba+Ah	3.630	CD
Bx+Ah	3.460	D
Wood materials + varnishes		
I+Sv	2.287	E
I+Sn	5.433	A
I+Pu	4.047	B
I+Wb	3.727	BC
I+Ac	3.807	BC
I+Ah	3.453	CD
II+Sv	2.167	E
II+Sn	3.547	CD
II+Pu	3.600	CD
II+Wb	3.733	BC
II+Ac	3.667	BC
II+Ah	3.227	D

according to wood material type+impregnation material, impregnation material + varnish type and wood material type+ varnish type are given in Table 5.

Adhesion strength value for wood material type+impregnation material was highest in Oriental beech+boric acid and lowest in oak+boric acid. For impregnation material+varnish type, adhesion strength

was highest in boric acid+synthetic varnish, lowest in boric acid+cellulosic varnish. Impregnation materials have increasing impact for adhesion strength of varnishes. For wood material+varnish type, adhesion strength was highest in Oriental beech+synthetic varnish, lowest in oak+cellulosic varnish. Statistical values of interaction of wood material+varnish type+impregnation material are given in Table 6.

Adhesion strength was increased in impregnated Oriental beech samples. Amount of increase in oak was found different according to varnish type and impregnation material type. This may be due to convenience of Oriental beech surface for specific adhesion. Besides, low retention amount in oak might have negative effect. Results of multiple variance analysis for impact of wood material type+varnish type+ impregnation material for adhesion strength is given in Table 7.

Impact of wood material type, impregnation material and varnish type on adhesion strength have been found important for the effect of variance sources on the adhesion strength ($\alpha=0,05$). Duncan Test results are given in Table 8 to indicate the importance of differences between the groups.

Adhesion strength value was highest in Oriental beech+synthetic varnish and lowest in oak+cellulosic varnish for varnished wood material without impregnation. For samples varnished after impregnation, hardness was highest in Oriental beech varnished with synthetic after impregnation with boric acid and lowest in oak varnished with cellulosic varnish after impregnation with borax (Figure 1).

Conclusion

Retention amount of impregnation materials according to wood and impregnation material is highest in beech+boric acid and lowest in oak+borax. Reason of higher amount of retention with boric acid might be due to high concentration of solution. This provides advantages for applications requiring high retention quantity. On the other hand, higher retention value in oriental beech than oak may be due to tyll formation in oak wood.

Layer thickness in varnishes were found 78 μm in cellulosic varnish, 92 μm in synthetic varnish, 120 μm in polyurathane varnish, 66 μm in water-based varnish, 128 μm in acrylic varnish and 100 μm in acid hardening varnish. The difference between varnishes might be due to different solid materials.

Adhesion strength value was highest in Oriental beech, borax and synthetic varnish and lowest in oak, boric acid and cellulosic varnish. Accordingly, wood material type and varnish type is effective in adhesion strength, main effective group is firstly varnish type and secondly, impregnation material.

Adhesion strength value in impregnation treatment was 13% higher in boric acid, 14% higher in borax than

Table 6. Adhesion strength values related to interaction of wood material, varnish type and impregnation material.

Type of wood materials	Impregnation materials	Stat.	Varnish type					
			Sv	Sn	Pu	Wb	Ac	Ah
Oriental beech	Co	x	2.24	4.7	3.34	3.12	3.18	2.7
		Ss	0.23	0.64807	0.472229	0.609918	0.67	0.23
		v	0.0544	0.336	0.1784	0.2976	0.4536	0.052
		Min	1.9	4	2.9	2.6	2.5	2.4
		Max	2.6	5.3	3.9	4.1	4.3	3.1
	Ba	x	2.18	6.24	3.88	4.24	4.18	4.22
		Ss	0.21	0.2881	0.238747	1.18	0.73	0.44
		v	0.0456	0.0664	0.0456	1.3864	0.5336	0.1976
		Min	1.9	5.9	3.6	3.2	3	3.5
		Max	2.5	6.6	4.2	6.2	5	4.8
	Bx	x	2.44	5.36	4.92	3.82	4.06	3.44
		Ss	0.439318	0.6269	0.67602	0.44	0.57	0.72
		v	0.1544	0.3144	0.3656	0.1896	0.3304	0.5184
		Min	2.1	4.7	4.1	3.3	3.5	2.6
		Max	3.2	6.2	5.6	4.5	4.9	4.7
Co	x	2.22	3.6	3.48	3.58	3.86	3.16	
	Ss	0.07	0.21909	0.643117	0.222711	0.287054	0.2416609	
	v	0.0056	0.048	0.4136	0.0496	0.0824	0.0584	
	Min	2.1	3.2	2.6	3.4	3.5	2.9	
	Max	2.3	3.8	4.3	4	4.3	3.6	
European oak	Ba	x	2.2	3.46	3.52	4.02	2.92	3.04
		Ss	0.21	0.31369	0.172047	0.31241	0.116619	0.4498889
		v	0.044	0.0984	0.0296	0.0976	0.0136	0.2024
		Min	1.9	3.1	3.2	3.6	2.8	2.6
		Max	2.4	4	3.7	4.5	3.1	3.9
	Bx	x	2.08	3.56	3.8	3.6	4.22	3.48
		Ss	0.21	0.13565	0.42426	0.167332	0.337046	0.3187475
		v	0.0456	0.0184	0.18	0.028	0.1136	0.1016
		Min	1.7	3.4	3.2	3.4	3.9	3.1
		Max	2.3	3.8	4.3	3.8	4.8	3.9

*LSD=0.2725, **LSD=0.4719, ***LSD= 0.3853

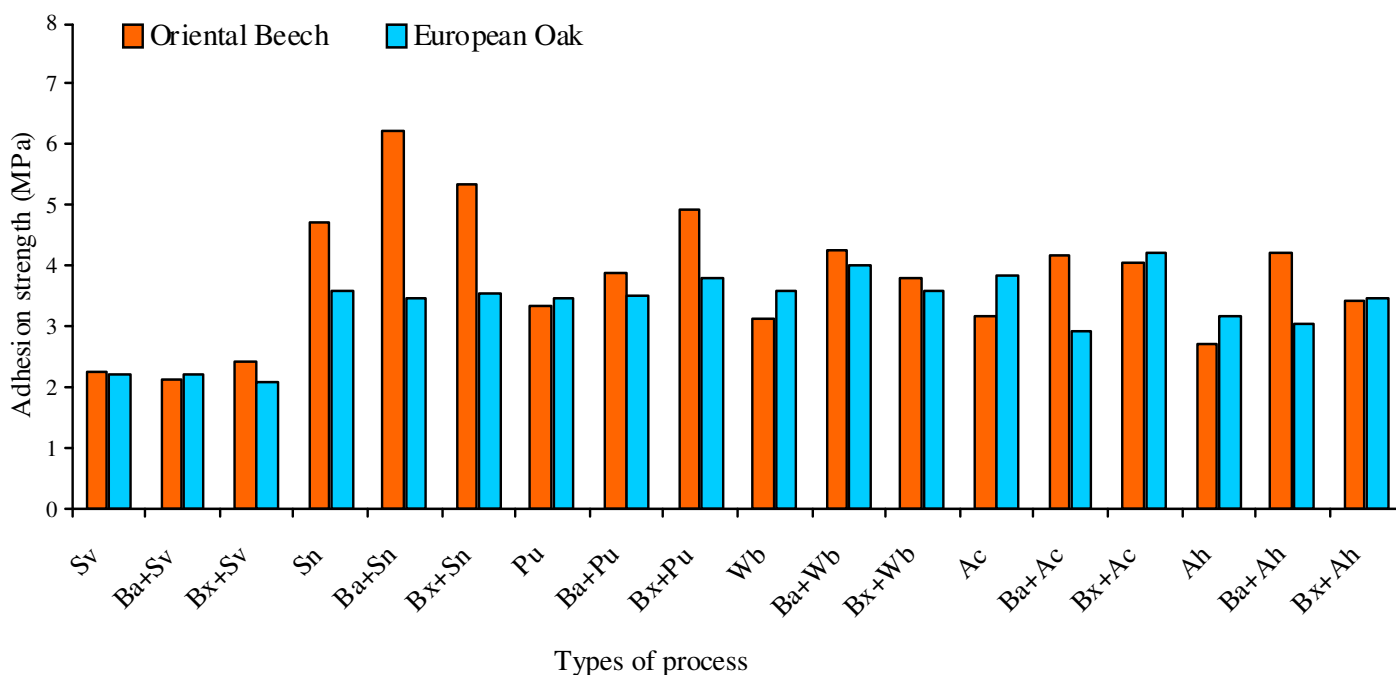
Table 7. Multiple variance analysis for impact of wood material type, varnish type and impregnation material for adhesion strength.

Source	Degrees of freedom	Sum of squares	Mean square	F value	Probably 5% Sign.
Factor A	1	9.894	9.894	39.8309	0.0000
Factor B	5	84.615	16.923	68.1312	0.0000
AB	5	18.940	3.788	15.2501	0.0000
Factor C	2	7.805	3.903	15.7121	0.0000
AC	2	8.628	4.314	17.3682	0.0000
BC	10	7.653	0.765	3.0809	0.0014
ABC	10	6.517	0.652	2.6235	0.0058
Error	144	35.768	0.248		
Total	179	179.819			

Table 8. Duncan test results.

Process type	x	HG	Process type	x	HG	Process type	x	HG
I+Ba+Sn	6.24	A	I+Bx+ Wb	3.82	EFGH	I+Ac	3.18	GHIJK
I+Bx+Sn	5.36	B	II+Bx+Pu	3.80	EFGH	II+Ah	3.16	GHIJK
I+Bx+Pu	4.92	BC	II+Bx+Wb	3.60	EFGHI	I+ Wb	3.12	GHIJK
I+Sn	4.70	BCD	II+Sn	3.60	EFGHI	II+Ba+Ah	3.04	HIJK
I+Ba+ Wb	4.24	CDE	II+ Wb	3.58	EFGHI	II+Ba+Ac	2.92	IJKL
II+Bx+Ac	4.22	CDE	II+Bx+Sn	3.56	EFGHI	I+Ah	2.70	JKLM
I+Ba+Ah	4.22	CDE	II+Ba+Pu	3.52	EFGHI	I+Bx+Sv	2.44	KLM
I+Ba+Ac	4.18	CDE	II+Pu	3.48	EFGHIJ	I+Sv	2.24	LM
I+Bx+Ac	4.06	DEF	II+Ba+Sn	3.48	EFGHIJ	II+Sv	2.22	LM
II+Ba+ Wb	4.02	DEF	II+Bx+Ah	3.48	EFGHIJ	II+Ba+Sv	2.20	LM
I+Ba+Pu	3.88	EFG	I+Bx+Ah	3.44	EFGHIJ	I+Ba+Sv	2.18	LM
II+Ac	3.86	EFG	I+Pu	3.34	FGHIJ	II+Bx+Sv	2.08	M

LSD value = 0.6674

**Figure 1.** Adhesion strength value changes in varnished surfaces according to wood material and treatment type.

control specimen. Accordingly, impregnation materials have increasing effect on adhesion strength values. This case might be due to increase of wood material density because of impregnation material. After treating with different impregnation materials, density of Scotch pine increased (Örs et al., 1999). In this regard, impregnation with boron compounds provides advantage for decreasing surface hardness and this increases specific adhesion between varnish and wood material (Örs et al., 2006). In this regard, impregnation with boron compounds provides advantage for applications and usage areas where adhesion strength is important. Besides, low

retention amounts for the impregnation materials and decreasing of specific adhesion because of big annular tracheid structure were the reasons for the low adhesion strength of oak.

Adhesion strength for wood material + impregnation material was highest in I+Ba (4.157 MPa) and lowest in II+Ba (3.179 MPa). Impregnation materials affected the adhesion strength of Oriental beech and oak at a rate of 29% increase, 14% decrease in boric acid and 25% increase, 4% increase in borax orderly.

Adhesion strength value according to impregnation material+varnish type combination was the highest in

Ba+Sn (4.860 MPa) and the lowest in Ba+Sv (2.190 Mpa). Adhesion strength value of varnished samples which were not impregnated was lower than impregnated and varnished samples except Sv+Ba. Impregnation materials showed increasing effect in the adhesion strength values of synthetic, polyurethane, water-borne, acrylic and acid hardening at a rate of 15, 8, 23, 1 and 24% in boric acid and 7, 22, 11, 18 and 18% in borax orderly. With cellulosic varnish, adhesion strength decreased by 2% in boric acid and increased by 2% in borax. Adhesion strength value was lowest in cellulosic varnish. This might be due to completion of cellulosic varnish formation during production phase. Accordingly, boron compounds have positive impact for adhesion strength of varnishes.

Adhesion strength according to wood material + impregnation, material+varnish combination was highest in I+Ba+Sn (6.240 Mpa) and lowest in II+Bx+SV (2.080 Mpa). For adhesion strength of varnishes, impregnation material and varnish type was effective in addition to wood material type, but impregnation material and varnish type were the main sources of this effect. More effect was observed for the wood material with big annular tracheid.

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