

Effects of Impregnation with Imersol-AQUA on Yellow Color Tone of Some Softwoods and Varnishes

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ABSTRACT: This study has been performed for determining impacts of impregnation with Imersol-AQUA (I-AQUA) to yellow color tone of some softwoods and varnishes. For this purpose, samples of scotch pine (*Pinus sylvestris* L.), oriental spruce (*Picea orientalis* L.), and Uludağ fir (*Abies bornmülleriana* L.) wood, prepared according to the ASTM D 358 standards, are impregnated with I-AQUA by short-term (S), medium-term (M), and long-term (L) immersion techniques in accordance with ASTM D 1413 and producer's definition. After impregnation, surfaces have been coated by synthetic (Sv), acrylic (Ac), water-based varnishes (Wb) and polyurethane (Pu) varnishes in accordance with ASTM D 3023 standards. According to ASTM D 2244 standards, yellow color tone value of specimens after varnishing process was determined. As a result, based on the wood material,

varnish type, and impregnation period, yellow color tone was the highest in pine, medium-term dipping, and synthetic varnish and the lowest in fir, long-term dipping, and water-borne varnish. Considering the interaction of wood type, period of impregnation and type of varnish, yellow color tone was the highest in fir + medium-term of dipping + synthetic varnish and the lowest in spruce + long-term of dipping + water-borne varnish. So, impregnation process, impregnation period, and varnishes applied during tests showed increasing impact for yellow color tone value of wood materials. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 103: 1048–1054, 2007

Key words: impact resistance; coatings; compounding; density

INTRODUCTION

Impregnation of wood materials with chemical materials before usage is seemed to be an obligation in many usage areas. Furniture made with unimpregnated wood materials and coated only with paint and varnish have surface protection only for 2 years.¹

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 is also known to cause changes in the natural color of wood.² Furthermore, differences between the specific weights of the growing rings may also result in color distinction. In wood, by the chemical degradation of extractive materials and lignin in wood, yellow and brown colors occur, which accelerates in open-air conditions.³ There is no change in living trees, but when the tree is cut, the color of wood becomes dark or light. Surface process of wood avoids partly or completely this change or degradation and also makes the natural color and pattern of wood much more appa-

rent and makes its living image longer.⁴ Technical surface processes also increase economic, esthetic, and economic value of wood.

The aim of this experimental study is to determine the effect of impregnation scotch pine (*Pinus sylvestris* L.), oriental spruce (*Picea orientalis* L.), Uludağ fir (*Abies Bornmülleriana* L.) woods, which are widely used in furniture industry with I-AQUA on the yellow color tone (YCT) of woods and varnishes.

MATERIAL AND METHODS

Materials

Wood materials

The woods of scotch pine, oriental spruce, Uludağ fir were chosen randomly from timber merchants of Ankara, Turkey. Special emphasis is given for the selection of the wood material. Accordingly, nondeficient, proper, knotless, normally grown (without zone line, without reaction wood, and without decay, insect mushroom damages) wood materials were selected.

Varnishes

Synthetic (Sv), acrylic (Ac), water-borne (Wb), and polyurethane (Pu) varnishes are supplied by merchants in Ankara and used according to the pro-

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ducer's definition. Technical specifications of varnishes are given in Table I.⁵⁻⁷

Impregnation material

I-AQUA used as an impregnation material in this study was supplied from Hemel Hickson Timber Products, Istanbul. I-AQUA is nonflammable, odorless, fluent, water based, completely soluble in water, noncorrosive material with a pH value of 7 and a density of 1.03 g cm^{-3} . It is available as a ready-made solution. It contains 0.5% w/w tebuconazole, 0.5% w/w propiconazole, 1% w/w 3-Iodo-2-propynyl-butyl carbonate, and 0.5% w/w cypermethrin. Before the application of I-AQUA on the wood material, all kinds of drilling, cutting, turning, and milling operations should be completed, and the relative humidity should be in equilibrium with the test environment. In the impregnation process, dipping duration should be at least 6 min, and the impregnation pool must contain at least 15 L of impregnation material for 1 m^3 of wood. The impregnated wood should be left for drying at least 24 h. The wood material can be painted, varnished, or glued after it is fully dried.⁸

Determination of density

The densities of wood materials, used for the preparation of test specimens, were determined according to TS 2472.⁹ For determining the air-dry density, the test specimens with a dimensions of $20 \times 30 \times 30 \text{ mm}^3$ were kept under the conditions of $(20 \pm 2)^\circ\text{C}$ and $(65 \pm 3)\%$ relative humidity until to a stable weight. The weights were measured with an analytic scale of $\pm 0.01 \text{ g}$ sensitivity. Afterwards, the dimensions were measured with a digital compass of $\pm 0.01 \text{ mm}$ sensitivity. The air-dry densities of the specimens were calculated by the formula:

$$\delta_{12} = \frac{M_{12}}{V_{12}} \text{ g cm}^{-3} \quad (1)$$

where, M_{12} is the air-dry weight (g) and V_{12} is the volume (cm^3) at air-dry conditions.

The specimens were kept at a temperature of $(103 \pm 2)^\circ\text{C}$ in the drying oven till they reach to a stable weight for the assessment of oven-dry density. Afterwards, oven-dried specimens were cooled in the desiccators containing phosphorus pentoxide (P_2O_5) and weighed with an analytic scale of 0.01 g sensitivity. The volumes of the specimens were determined by stereo metric method and the densities calculated by the formula,

$$\delta_o = \frac{M_o}{V_o} \text{ g cm}^{-3} \quad (2)$$

where, M_o is the oven-dry weight (g) and V_o is the oven-dry volume (cm^3) of the wood material.

Determination of humidity

The humidity of test specimens before and after the impregnation process was determined according to TS 2471.¹⁰ Thus, the specimens with a dimension of $20 \times 20 \times 20 \text{ mm}^3$ were weighed and then oven dried at $(103 \pm 2)^\circ\text{C}$ till they reach to a constant weight. Then, the specimens were cooled in desiccators containing P_2O_5 and weighed with an analytic scale of 0.01 g sensitivity. The humidity of the specimens (h) was calculated by the formula:

$$h = \frac{M_r - M_o}{M_o} \times 100 \quad (3)$$

where, M_r is the initial weight of the samples (g) and M_o is the final dry weight (oven-dry) of the samples (g).

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 $190 \times 140 \times 15 \text{ mm}^3$ and conditioned at a temperature of $(20 \pm 2)^\circ\text{C}$ and $(65 \pm 3)\%$ relative humidity for 3 months till they reach 12% humidity distribution according to ASTM D 358.¹¹ The air-dry specimens with a dimension of $150 \times 100 \times 10 \text{ mm}^3$ were cut from the drafts for impregnation and varnishing.

TABLE I
Some Properties of Varnishes

Type of varnish	pH	Density (g cm^{-3})	Viscosity (sn DIN Cup/4 mm)	Amount applied (g m^{-2})	Nozzle gap (mm)	Air pressure (bar)
Polyurethane (filler)	5.94	0.98	18	160	1.8	2
Polyurethane (finishing)	4.01	0.99	18	160	1.8	2
Synthetic	-	0.94	18	120	-	-
Water-borne (primer); ASTM D 17	9.17	1.014	18	150	1.3	1
Water-borne (filler); ASTM D 65	9.30	1.015	18	80	1.3	1
Water-borne (finishing); ASTM D 45	8.71	1.031	18	80	1.3	1
Acrylic (filler)	4.3	0.95	18	120	1.8	2
Acrylic (finishing)	4.6	0.97	18	120	1.8	2

TABLE II
Oven-Dry Densities of Wood Materials (g cm⁻³)

Methods	Statistics	Scotch pine	Oriental spruce	Uludag fir
Control (Co)	x	0.537	0.405	0.380
	Min	0.512	0.388	0.349
	Max	0.572	0.435	0.406
	Ss	0.016681	0.0154602	0.0192202
	v	0.000278	0.0002390	0.0003694
Short-term dipping (S)	x	0.543	0.408	0.382
	Min	0.524	0.393	0.352
	Max	0.566	0.425	0.426
	Ss	0.011758	0.0105399	0.0228182
	v	0.000138	0.0001112	0.0005206
Middle-term dipping (M)	x	0.561	0.409	0.389
	Min	0.525	0.398	0.355
	Max	0.582	0.427	0.431
	Ss	0.017394	0.0091044	0.0214683
	v	0.000302	0.0000828	0.0004608
Long-term dipping (L)	x	0.568	0.414	0.396
	Min	0.542	0.401	0.365
	Max	0.596	0.438	0.444
	Ss	0.014726	0.0111624	0.0223757
	v	0.000216	0.0001246	0.0005006

x , Mean; Min, minimum; Max, maximum; Ss, standard deviation; v , variance.

The test specimens were impregnated according to ASTM D 1413.¹² The specimens were dipped in the impregnation pool immersing 1 cm below the upper surface for 10 min in short-term dipping, 2 h for medium-term dipping, and 5 days for long-term dipping. The specifications of the impregnation solution were determined before and after the process. The processes were carried out at (20 ± 2)°C. Retention of impregnation material (R) was calculated by the formula:

$$R = \frac{GC}{V} 10^3 \text{ kg m}^{-3} \quad (G = T_2 - T_1) \quad (4)$$

where, G is the amount of impregnation solution absorbed by the specimen, T_2 is the specimen weight after the impregnation, T_1 is the specimen weight before the impregnation, C is the concentration (%) of the impregnation solution, and V is the volume of the specimens. Impregnated test specimens were kept under a temperature of (20 ± 2)°C and (65 ± 3)% relative humidity until they reach to a stable weight.

Varnishing

Test specimens were varnished according to ASTM D 3023.¹³ 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 dis-

tance 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. Sv varnish was applied with a hard and strong brush. The varnished samples have been left to drying in a dustless environment having (20 ± 2)°C temperature. The layer thicknesses were mea-

TABLE III
Air-Dry Densities of Wood Materials (g cm⁻³)

Methods	Statistics	Scotch pine	Oriental spruce	Uludag fir
Co	x	0.577	0.420	0.401
	Min.	0.555	0.401	0.385
	Max.	0.592	0.441	0.412
	Ss	0.0121909	0.01435143	0.00922841
	v	0.0001486	0.00020596	0.00008546
S	x	0.579	0.428	0.407
	Min.	0.558	0.407	0.392
	Max.	0.600	0.455	0.415
	Ss	0.0002416	0.01238621	0.00700391
	v	0.0002416	0.00012340	0.00004905
M	x	0.592	0.437	0.410
	Min.	0.578	0.412	0.399
	Max.	0.605	0.460	0.422
	Ss	0.0076324	0.01568961	0.00607379
	v	0.0000582	0.00024616	0.00003689
L	x	0.597	0.440	0.419
	Min.	0.579	0.422	0.408
	Max.	0.612	0.462	0.444
	Ss	0.0098322	0.01436029	0.01106592
	v	0.0000966	0.00020621	0.00012245

Co, Control; S, short-term dipping; M, middle-term dipping; L, long-term dipping.

TABLE IV
Retention Amounts of Wood Materials (kg m⁻³)

Imp. methods	Statistics	Scotch pine	Oriental spruce	Uludag fir
S	X	40.585	32.785	38.745
	Ss	5.1824926	2.4615706	4.4595706
	v	21.486584	4.847464	15.910216
	Min	38.21	29.88	31.79
	Max	51.64	36.1	43.9
M	x	77.891667	51.285	57.593333
	Ss	9.1136628	8.6209048	8.037884
	v	66.44708	59.456	51.686064
	Min	65.44	37.37	52.22
	Max	88.95	59.93	71.13
L	X	176.47167	156.915	177.99667
	Ss	13.613663	3.132566	17.98909
	V	148.26546	7.850376	258.88588
	Min	165.04	151.3	160.2
	Max	197.15	159.49	206.45

S, Short-term dipping; M, middle-term dipping; L, long-term dipping.

sured as 90.19, 92.14, 91.25, and 93.21 μm , respectively, for Sv, Ac, Wb, and Pu. The solid rates were measured as: 54.6, 47.8, 32.4, and 45.7% respectively.

Method of testing

Color measurements

Yellow color tone (YCT) measurements were done according to ASTM D 2244-02 under (20 \pm 2) $^{\circ}\text{C}$ temperature and (50 \pm 5)% humidity conditions before and after the color changes by a color meter having

TABLE V
YCT Mean Values for Wood Types, Varnish Types, and Periods of Impregnation

Types of materials	Color ^a
Wood materials*	
Scotch pine (I)	39.60 a
Oriental spruce (II)	38.33 b
Uludag fir (III)	38.31 b
Varnishes**	
Unvarnished (Uv)	29.97 d
Synthetic (Sv)	44.43 a
Acrylic (Ac)	42.95 b
Water-based (Wb)	33.61 c
Polyurethane (Pu)	42.77 b
Impregnation Methods***	
Control (Co)	29.62 d
Short-term dipping (S)	41.99 b
Middle-term dipping (M)	43.12 a
Long-term dipping (L)	40.26 c

I, Scotch pine; II, oriental spruce; III, Uludag fir; S, short-term; M, medium-term; L, long-term; Sv, synthetic; Ac, acrylic; Wb, water-based varnishes; Pu, polyurethane varnishes.

^a Different letters in a column refers to significant differences among types of materials at 0.05 confidence level (*LSD_{0.5}: 0.4548; **LSD_{0.5}: 0.5871; ***LSD_{0.5}: 0.5251).

calibration values a = 4.91, b = -3.45, c = 6.00 and H = 324.9.¹⁴

Statistical analyses

Bu using three different types of wood, 3 methods of impregnation + 1 control specimen, 4 types of varnish + 1 control specimen a total of 300 specimens (3 \times 4 \times 5 \times 5 mm⁴) were prepared with five specimens for each parameter. Multiple variance analysis was used to determine the differences in YCT of specimens. Duncan Test was used to determine the significant difference between the groups.

RESULTS AND DISCUSSION

Oven-dry density

Statistical values for oven-dry densities of test specimens impregnated with I-AQUA are given in Table II. Oven-dry densities changes by type of wood and

TABLE VI
YCT Mean Values for Double Combination of Wood Type, Varnish Type, and Period of Impregnation

Types of material	Color ^a
Wood materials + dipping periods*	
I	32.01 f
I + S	41.65 c
I + M	43.81 a
I + L	40.92 cd
II	29.53 g
II + S	41.35 c
II + M	42.77 b
II + L	39.66 e
III	27.31 h
III + S	42.96 ab
III + M	42.79 b
III + L	40.18 de
Dipping periods + varnishes**	
Sv	37.15 jk
S + Sv	47.15 k
M + Sv	49.27 jk
L + Sv	44.14 fg
Ac	30.72 a
S + Ac	46.66 de
M + Ac	49.12 l
L + Ac	45.31 b
Wb	25.64 cd
S + Wb	36.34 m
M + Wb	37.78 gh
L + Wb	34.68 f
Pu	35.56 hi
S + Pu	46.34 bc
M + Pu	46.08 bc
L + Pu	43.13 e

I, Scotch pine; II, oriental spruce; III, Uludag fir; S, short-term; M, medium-term; L, long-term; Sv, synthetic; Ac, acrylic; Wb, water-based varnishes; Pu, polyurethane varnishes.

^a Different letters in a column refers to significant differences among different interactions of wood material, varnish, and impregnation method (**LSD: 0.9095; **LSD: 1.174).

TABLE VII
MANOVA for the Effect of Wood Type, Period of Impregnation,
and Varnish Type on YCT

Source	Degrees of freedom	Sum of squares	Mean square	F-value	Probably 5% (Sig.)
Factor A ^a	2	109.257	54.628	20.4620	0.0000
Factor B ^b	4	10175.289	2543.822	952.8325	0.0000
AB	8	217.487	27.186	10.1829	0.0000
Factor C ^c	3	8646.689	2882.230	1079.5889	0.0000
AC	6	241.299	40.217	15.0638	0.0000
BC	12	595.901	49.658	18.6004	0.0000
ABC	24	447.170	18.632	6.9790	0.0000
Error	240	640.739	2.670		
Total	299				

^a Factor A: I, scotch pine; II, oriental spruce; III, Uludag fir.

^b Factor B: Sv, synthetic; Ac, acrylic; Wb, water-based varnishes; Pu, polyurethane varnishes.

^c Factor C: S, short-term; M, medium-term; L, long-term.

impregnation periods and increased with the increase of impregnation period.

Air-dry density

Statistical values for the air-dry densities of wood materials impregnated with I-AQUA are given in Table III.

Air-dry densities were found different depending on type of wood and duration of impregnation. Air-dry densities of impregnated woods are increased with dipping duration.

Peculiarities of impregnation solutions

The pH value and density of I-AQUA, used for the impregnation process, did not change either before or after the impregnation. This may be due to the use of fresh solution in each impregnation process.

Retention quantities

Retention amounts of woods for different impregnation periods are given in Table IV.

Amounts of retention were found different depending on wood type and impregnation period. Retention

TABLE VIII
Some Properties of Varnishes

Materials	Color ^a	Materials	Color ^a	Materials	Color ^a
I + S + Ac	52.29 a	I + Sv	43.89 ghijklm	III + Sv	35.27 pqrstuvw
I + M	50.45 ab	II + S	43.81 hijklmn	III + M + Pu	35.23 pqrstuvw
I + S + Sv	49.74 bc	II + L + Pu	43.80 ijklmno	II + Pu	34.67 qrstuvwxy
I + Wb	49.18 cd	II + Wb	43.01 jklmnop	II + M	34.64 rstuvwxyz
I + L + Pu	48.47 cd	I + M + Wb	42.92 klmnop	II + S + Wb	34.57 stuvwxyz
I + S	48.35 de	III + Wb	42.80 klmnopq	III + M + Wb	33.38 tuvwxzya
I + M + Sv	48.16 de	II + M + Pu	41.16 lmnopq	III + L + Ac	32.92 uvwxyzab
I + Ac	47.94 def	III + L + Pu	41.08 lmnopq	I + M + Ac	32.66 vwxyzabc
I + S + Pu	47.65 defg	II + L + Wb	39.70 lmnopqr	III + M	32.46 wxyzabc
I + L + Wb	47.47 defgh	III + L	37.16 lmnopqrs	I + L + Sv	31.70 xyzabc
I + Pu	47.45 efghi	III + L + Wb	37.01 lmnopqrst	I + Co	31.48 yzabc
I + L	46.95 efghi	III + S	36.59 lmnopqrst	III + S + Pu	31.27 zabcd
I + L + Ac	46.68 efghi	II + Ac	36.49 mnopqrstu	III + S + Wb	31.20 zabcd
I + S + Wb	46.58 efghij	III + Ac	36.49 nopqrstu	III + Pu	27.98 zabcd
II + S + Sv	46.42 efghij	II + L + Ac	36.49 nopqrstu	II + Co	27.54 ABCD
I + M + Pu	46.25 efghijk	II + S + Pu	36.27 nopqrstu	III + Co	25.67 ABCD
III + S + Sv	45.51 fghijkl	II + M + Wb	36.15 nopqrstu	II + M + Ac	23.28 ABCD
III + S + Ac	45.39 fghijkl	II + Sv	35.61 opqrstuv	II + L + Sv	22.43 BCD
II + S + Ac	45.10 fghijkl	III + M + Sv	35.56 pqrstuvw	III + M + Ac	18.21 CD
II + M + Sv	44.18 ghijklm	II + L	35.54 pqrstuvw	III + L + Sv	16.42 D

I, Scotch pine; II, oriental spruce; III, Uludag fir; S, short-term; M, medium-term; L, long-term; Sv, synthetic; Ac, acrylic; Wb, water-based varnishes; Pu, polyurethane varnishes.

^a Different letters in a column refers to significant differences among the different interactions of wood materials, varnish, and impregnation method at 0.05 confidence level (LSD_{0,5}: 1.027).

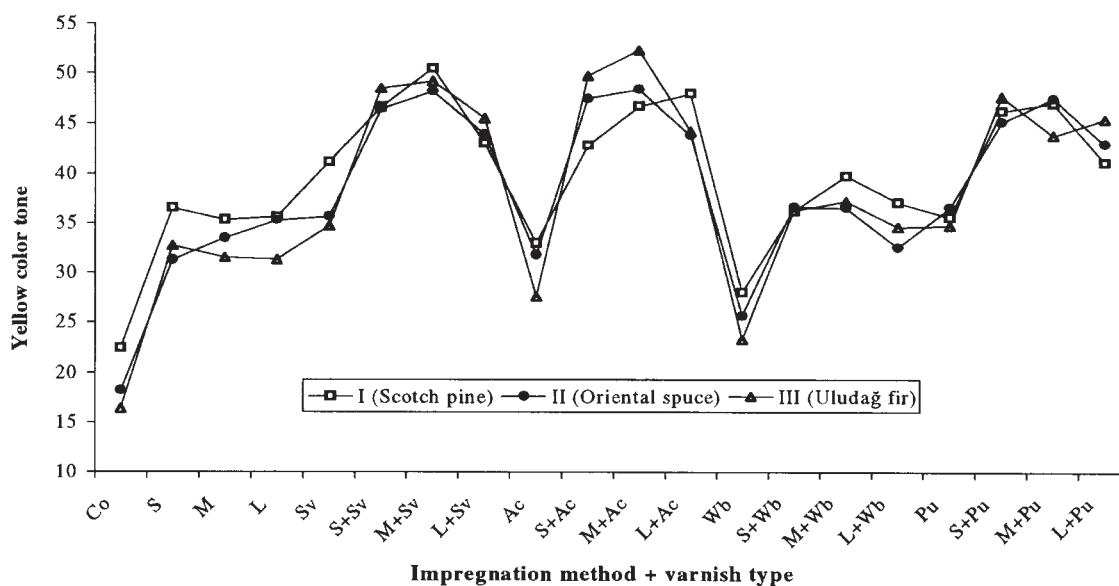


Figure 1 Change of yellow color tone (YCT) according to wood species impregnation periods and varnishes.

is the highest in Uludağ fir and the lowest in oriental spruce. When the dipping period increased, the amount of retention increased.

Color change

YCT mean values for wood types, varnish types, and periods of impregnation are given in Table V.

YCT was found the highest in scotch pine and the lowest in Uludağ fir. This result may be due to the structural properties of scotch pine. For type of varnishes, it was the highest in Sv varnish and the lowest in Wb varnish. Varnishes showed the effect of increasing YCT. For the period of impregnation, YCT was the highest in medium-term dipping and the lowest in long-term dipping. As the period of dipping increases, YCT decreases. For the combination of wood type + impregnation period, wood type + varnish type, varnish type + impregnation period, the mean values are given in Table VI.

For the combination of wood type + impregnation period, as the period of impregnation increased, YCT increased. This effect was found the highest in medium-term dipping of pine, the lowest in long-term dipping of spruce. For varnish type + impregnation period combination, it showed an increasing effect on all types of varnishes. This effect was found the highest in Sv varnish, the lowest in Wb varnish. Results of multiple variance analysis (MANOVA) for the effect of wood type, varnish type, and impregnation period on YCT are given Table VII.

The difference between the groups have been found important for the effect of variance sources on the YCT ($\alpha = 0.05$). Duncan test results are given in Table VIII

to indicate the importance of differences between the groups.

YCT in unimpregnated woods was measured as the highest in pine and the lowest in fir. YCT was measured as highest in medium-term dipping impregnated pine and the lowest in long-term dipping impregnated oak. In impregnated test specimens, it was measured the highest in medium-term dipping impregnated oak and the lowest in medium-term impregnated fir. In impregnated and varnished test specimens, it was measured the highest in short-term dipping impregnated and Ac varnished pine and the lowest in long-term impregnated and then Sv varnished fir.

YCT values for scotch pine, oriental spruce, and Uludağ fir for different types of varnishes and periods of impregnation are given in Figure 1.

CONCLUSIONS

Air-dry and full-dry densities of test specimens impregnated with different methods were increased as compared with those of control specimens. This case may be due to more absorption of impregnation material as impregnation period increases. Retention was found more in long-term dipping than in short-term dipping method. It was reported that in impregnation of scotch pine and oriental beech with Imersol-WR, retention was increased by the period of impregnation.¹⁵ Impregnation with long-term dipping provides advantages for applications requiring high retention quantity. On the other hand, retention was found higher in scotch pine than in oriental spruce and Uludağ fir. This case may be due to pit aspiration of oriental spruce and Uludağ fir.

YCT was measured the highest in Pine, Sv varnish and medium-term dipping and the lowest in Fir, long-term dipping and Wb varnish. YCT was found 3% higher in scotch pine than in oriental spruce and Uludağ fir. YCT were measured more than control specimen: 42% in short-term, 46% in medium-term, and 36% in long-term dipping. So, it can be said that impregnation material increases YCT.

YCT was measured more than control specimens: 48% in Sv varnish, 43% in Ac varnish, 12% in Wb varnish, 42% in Pu varnish. So, varnishing affects YCT of wood.

For the combination of wood and varnish, YCT was measured the highest in I + M (43.81) and the lowest in II + L (39.66). Efficacy of impregnation time to increase YCT as compared with control specimens of scotch pine, oriental spruce, and Uludağ fir was 30%, 40 and 57% in S, 38, 45, and 57% in M, 28, 34, and 47% in L.

For the combination of impregnation period and varnish, YCT was measured the highest in M + Sv (49.27) and the lowest in L + Wb (34.68). So, YCT of varnished specimens was found lower in unimpregnated specimens than in impregnated specimens. Efficacy of impregnation time to increase YCT as compared with Sv, Ac, Wb, and Pu varnishes was 30, 52, 42, 30% in S, 33, 60, 47, 30% in M, 19, 47, 35, and 21% in L.

For the combination of wood + impregnation material + varnish, YCT was measured the highest in III + M + Ac (52.29) and the lowest in II + L + Wb (32.46). Impregnation material, impregnation period, type of varnish, and type of wood are important in YCT, but the efficacy of varnish type and impregnation material is more important.

As a result, we can say that the type of varnish and impregnation period has a second degree efficacy for a first wood type. This result must be taken into care in the manufacture of wooden furniture and construction elements where YCT is important.

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