WOOD RESEARCH
58 (3): 2013
369-380

EXPERIMENTAL INVESTIGATION OF SOME TECHNOLOGICAL PROPERTIES OF THERMO MODIFIED AND IMPREGNATED WOOD SAMPLES

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(Received February 2012)

ABSTRACT

The study is focused on an experimental investigation of the effect of impregnation and heat treating of oak (*Quercus petraea* L.) and pine (*Pinus sylvestris* L.) samples on bending strength, bonding strength, dowel holds performance, screw holds performance, dowel-joint performance and tenon and mortise-joint performance. The solution of 5 % natural oak tannin was used for impregnation. Heat treatment process was performed at 212°C. Bending and bonding strength have increased on impregnated oak and decreased on pine. A dowel holds, a screw holds, a dowel joints and a tenon and mortise-joint performances of all impregnated samples have been reduced. Thermo process caused a reduction of technological properties for both wood types. A combination of thermo and impregnation processes caused variable performances of oak and pine samples in relation to measured values.

KEYWORDS: Impregnation, natural tannin, heat treatment, thermowood, open air condition, mechanical properties.

INTRODUCTION

Presently used industrial processes are based on a treatment in a gas atmosphere or in a water vapor atmosphere (Rapp 2001). A new industrial development in Menz Company in Germany points to the oil-heat treatment. As known that it can be used in range of 180°C and 220°C (Militz 2002). Also it is known that swelling and shrinking of solid wood can be reduced by a heat treatment (Bächle et al. 2004). The swelling and shrinkage reduction by heat treatment was described by Bächle (2002) and Brox (2003).

By Kollman and Fengel (1965) the duration of treatment has a significant at temperature about 180°C. This knowledge is included in the work of Schanack (2002).

Chemical modification undergoes over the entire cross-section of wood (Kamdem et al. 2000) during the heat process which leads to higher resistance to fungi. Wälchli et al. found out that strength increases at temperature above 240°C (Wälchli et al. 1988).

But the strength of wood has been reduced. It was found that a strong mass loss occurs at 175°C which is caused by the degradation of hemicelluloses (Hakkou et al. 2003).

Based on strong correlation between acidity and thermal degradation in wood reported in previous studies, the effect of borate impregnation as an alkali-buffering medium was investigated on the strength properties of thermally modified wood. The positive effects of borate impregnation as a pretreatment on the strength properties of heat treated wood depend on the degree of heat treatment (Awoyemi and Westermark 2005).

MATERIAL AND METHODS

The impregnation by 5 % solution of natural tannin by dip method and heat treatment by thermowood method to the some test samples (according Tab. 1) were applied in this study. The test samples were kept in open air conditions. A total of 480 test samples were prepared.

Tab. 1: Experimental plan.

<table>
<tr><th rowspan="3">Wood Type</th><th rowspan="3">Treatment</th><th>Bending test</th><th>Adhesion test</th><th>Dowel holds test</th><th>Screw holds test</th><th colspan="2">Diagonal compression test</th></tr>
<tr><th colspan="2" rowspan="2">(N.mm⁻²)</th><th colspan="2" rowspan="2">(N)</th><th>Dowel joint</th><th>Tenon and mortise joint</th></tr>
<tr><th colspan="2">(N)</th></tr>
<tr><td rowspan="4">Oak</td><td>Control</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td>Impregnated</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td>Thermo</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td>Thermo+Impregnated</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td rowspan="4">Pine</td><td>Control</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td>Impregnated</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td>Thermo</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
<tr><td>Thermo+Impregnated</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td><td>10</td></tr>
</table>

Pine (*Pinus sylvestris* L.) and sessile oak (*Quercus petraea* L.) were used. All samples were obtained from the same place of origin. A random method was used for selection of samples. Four layers laminated samples were used for bending and two layers for bonding tests. Laminated samples were glued from 5 mm thick layers using two component D4 adhesive with 200 $g.m^{-2}$ adhesion application according to DIN EN 205 2003. The pressure of 1.2 MPA per 90 minutes was applied.

Diagonal pressure on "L-type" samples were performed on the both wood types for dowel and tenon-mortise joint (Fig. 1).

After gluing all samples were applied the impregnation and heat treatment (Tab. 1).

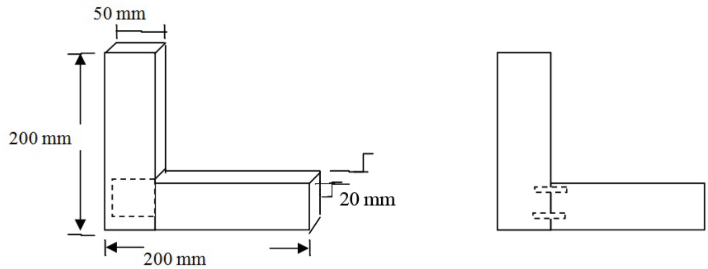


Fig. 1: Tenon-mortise and dowel joined "L-type" samples for a diagonal compression.

Impregnation

All the test samples were impregnated in a 5 % solution of natural oak tannin by dipping method for 2 hours. The test samples were conditioned at 20 ± 2°C temperature and 65 ± 5 % relative humidity conditions until the equilibrium moisture weight (12 %).

Thermowood process

Heat treatment process was applied to test samples with method of thermowood (212°C) according to Tab. 1. The thermowood process is divided into three main phases. These are:

Phase 1

Temperature increasing and high temperature kilning. The kiln temperature rapidly raises on a level around 100°C using steam. There after the temperature increases steadily up to 130°C during the moisture content in the wood reduces to nearly zero.

Phase 2

Intensive heat treatment. Once the high temperature has taken place, the temperature inside the kiln is increased to a level 212°C (Thermo-D) depending on the end-use application. Once the target level has been reached the temperature remains constant for 2 h.

Phase 3

Cooling and moisture conditioning. Temperature decreases using water spray systems and then once the temperature has reached 80–90°C re-moisture and conditioning takes place to adjust the wood moisture content over useable 4 %.

Samples conditioning

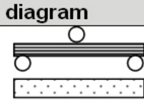
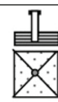
Impregnated, heat treated and heat treated+impregnated samples were conditioned four months (January-April) according to ASTM G7 2011. They were placed in Ankara on southern side under 45° angle. Test sample were placed 50 cm high. The attention was paid to keep the samples out of organic residues in soil, which would increase water content in wood. Climatic conditions have been identified by the Center for Meteorology (www.meteor.gov.tr).

Average maximum temperature (°C)	4.3	6.7	11.9	17.2
Average minimum temperature (°C)	-3.1	-2.0	1.1	5.7
Average of sunshine (hours)	2.6	3.8	5.4	6.4
Average number of rainy days	11.1	10.4	10.6	12.1
Monthly total rainfall amount				
Average ($kg.m^{-2}$)	39.2	33.60	36.1	50.0
UV-B radiation (MED)	46.08	83.62	186.81	266.91

Experimental methods

The laminated samples were investigated according to standards given in Tab. 2.

Tab. 2: Summary of tests performed (L- length, W- width, T- thickness).

Tests	Standards	Samples dim. (L/W/T) (mm)	Sample diagram
Bending strength	DIN EN 310, 1993	360x20x20	
Adhesion strength	DIN EN 205, 2003 DIN 52377, 1978 DIN 53255, 1964	150x50x20	
Dowel holds	DIN EN 320, 2011	50x50x20	
Screw holds	DIN EN 320, 2011	50x50x20	
Diagonal compression	ASTM D1037, 1999	200x50x20	

Evaluation of data

Statistical evaluation of the data obtained from the experiments was performed using program SPSS package. Multiple Analyses of Variance was conducted to determine the significance of the results. In generally the differences are statistically significant if $p < 0.05$ up to Duncan test used. In comparison, the highest average was symbolized by the letter "A".

RESULTS AND DISCUSSION

Bending strength test, bonding (shear) strength test, dowel holds performance test, screw holds performance test and diagonal compression test were applied on four layers laminated samples. Statistical evaluation is given in Tab. 3. Statistical averages and standard deviations were evaluated for control, impregnated, heat treated-thermo and thermo+impregnated oak (*Quercus petraea* L.) and pine (*Pinus sylvestris* L.).

Tab. 3: Statistical averages and standard deviations.

Wood Type	Treatment	Mean	Bending strength	Bonding strength	Dowel holds performance	Screw holds performance	Diagonal compressions perform. Dowel-joint performance	Diagonal compressions perform. Tenon and Mortise -joint performance
			($N.mm^{-2}$)		(N)		(N)	
Oak	Control	X	81.106	6.615	3743.124	2634.872	2090.542	4564.107
		ss	19.22	0.9	264.4	247.29	150.5	291.51
	Impregnated	X	90.375	7.959	3044.580	2691.802	1832.752	3826.176
		ss	6.61	1.04	210.58	216.21	186.14	573.48
	Thermo	X	47.419	5.128	1422.366	1334.096	1359.013	2138.884
		ss	3.68	0.23	102.69	83.35	161.39	464.04
	Thermo + Impregnated	X	49.875	4.691	1545.837	1307.739	1351.254	2792.413
		ss	5.74	0.22	70.03	31.5	95.64	240.76
Pine	Control	X	80.174	4.523	2710.906	1701.718	1349.693	4417.099
		ss	15.94	04	341.65	78.3	96.05	57.86
	Impregnated	X	72.737	3.626	2077.989	1685.408	1313.894	3363.29
		ss	8.5	026	165.82	66.05	77.89	317.72
	Thermo	X	42.439	2.235	641.866	1120.043	1139.822	1548.902
		ss	2.58	0.14	73.75	36.57	114.35	244.88
	Thermo + Impregnated	X	34.967	3.166	738.819	1152.905	1286.421	1900.362
		ss	2.55	0.29	119.83	45.19	120.96	324.4

Bending strength ($N.mm^{-2}$)

Wood types and treatments affect a bending strength in most cases. In addition, wood types, treatment and their interactions appear to be significant (Multiple Variance Analysis). Multiple Variance Analysis of bending strength and homogeneity of the groups of the bending strength is given in Tab. 4 and Tab. 5.

Tab. 4: Multiple variance analysis of bending strength.

Factor	D.F	Sum of squares	Mean squares	F Value	P≤0.05
Wood type (A)	1	1848.772	1848.772	18.4946	0.0001
Treatment (B)	3	28080.910	9360.303	93.6376	0.0000
Interactive (AB)	3	946,310	315.437	3.1555	0.0299
Error	72	7197.342	99.963		
Total	79	38073.334			

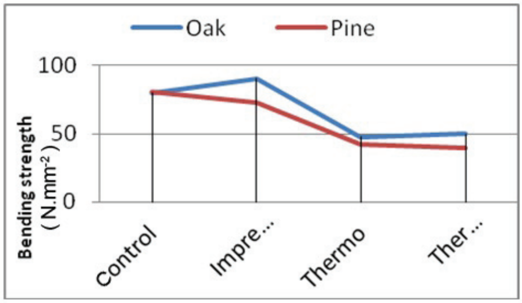


Fig. 2: Bending strength of a treated wood.

Tab. 5: Bending strength of a treated wood.

Wood type and treatment	Bending strength	HG
Oak+impregnated	90.38	A
Oak control	81.11	B
Pine control	80.17	B
Pine+impregnated	72.74	B
Oak+Thermo+impregnated	49.88	C
Oak+thermo	47.42	C
Pine+thermo	42.44	CD
Pine+Thermo+impregnated	34.97	D

LSD: ± 8.900

The highest value of the bending strength was obtained on impregnated oak (90.38 $N.mm^{-2}$) and the lowest value on thermo+ impregnated pine (34.97 $N.mm^{-2}$) as Tab. 5 shows. Comparing to the control samples the impregnation process decreases bending strength of the pine and increases a bending strength of the oak on other side. Thermo process reduces a bending strength of both wood types (Fig. 2). The impregnation increases a bending strength of oak. The reason may be in tannin obtained in oak and in impregnation also.

Adhesion (Bonding) strength ($N.mm^{-2}$)

Wood types affect the adhesion strength in most cases, treatment and their interactions less. In addition, wood types, thermo process and their interactions appear to be significant. Multiple Variance Analysis of Adhesion strength and homogeneity of groups of the adhesion strength averages is given in Tab. 6 and Tab. 7.

Tab. 6: Multiple variance analysis of adhesion strength.

Factor	D.F	Sum of squares	Mean squares	F value	P≤0.05
Wood type (A)	1	146.963	146.963	496.3595	0.0000
Treatment (B)	3	71.478	23.826	80.4713	0.0000
Interactive (AB)	3	22.269	7.423	25.0705	0.0000
Error	72	21.318	0.296		
Total	79	262.028			

Tab. 7: Adhesion strength of a treated wood.

Wood type and treatment	Adhesion strength	HG
Oak+impregnated	7.959	A
Oak control	6.615	B
Oak+therrno	5.128	C
Oak+Thermo+impregnated	4.691	CD
Pine control	4.523	D
Pine+impregnated	3.626	E
Pine+Thermo+impregnated	3.166	E
Pine+thermo	2.235	F
LSD: ±0.4843		

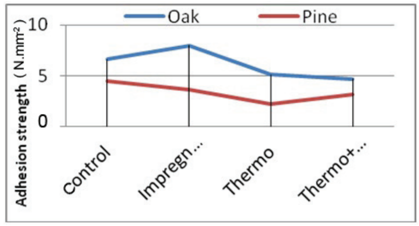


Fig. 3: Adhesion strength of a treated wood.

The highest value of a bonding strength was achieved on impregnated oak (7.959 $N.mm^{-2}$), the lowest value on the thermo processed pine (2.235 $N.mm^{-2}$) as Tab. 7 shows.

Comparing to the control samples an impregnation decreases a bonding strength of the pine and increases the bonding strength of the oak. Thermo-process reduces a bonding strength of both wood types (Fig. 3). The impregnation process increases the bonding strength of the pine. This is caused because the impregnation increases more the density of pine than oak.

Dowel holds performance (N)

Dowel holds performance was reduced in all cases. In addition, wood types and thermo process appear significant. Multiple Variance Analysis of dowel holds performance and homogeneity of the groups of dowel holds performance is given in Tab. 8 and Tab. 9.

Tab. 8: Multiple variance analysis of adhesion strength.

Factor	D.F	Sum of squares	Mean squares	F value	P≤0.05
Wood type (A)	1	146.963	146.963	496.3595	0.0000
Treatment (B)	3	71.478	23.826	80.4713	0.0000
Interactive (AB)	3	22.269	7.423	25.0705	0.0000
Error	72	21.318	0.296		
Total	79	262.028			

Tab. 9: Dowel holds performance.

Wood type	Dowel holds	HG	Treatment	Dowel holds	HG
Oak	2439	A	Control	3227	A
Pine	1542	B	Impregnated	2561	B
			Thermo+ impregnated	1142	C
			Thermo	1032	C
LSD: ± 85.59			LSD: ± 121.00		

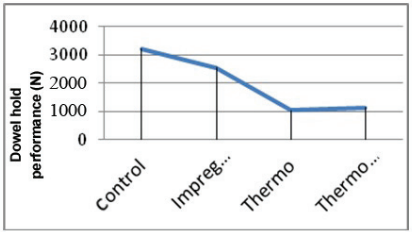


Fig. 4: Dowel holds performance of a treated wood.

The highest value of dowel holds performance is for oak (2439 N), the lowest for pine (1542 N), see Tab. 9. Thermo process more reduces the dowel holds performance. Thermo process may negatively affect to the cross connection between wood and glue molecules. For this reason the molecular structure of wood material deteriorates during the thermo process (Hakkou 2003).

Screw holds performance (N)

Wood types and treatment processes affect the screw holds performance in most cases including their interactions. Their case is reduction. Wood types, treatment and their interactions appear significant. Multiple Variance Analysis of screw holds performance and homogeneity of the groups of a screw holds performance is given in Tab. 10 and Tab. 11.

Tab. 10: Multiple variance analysis of screw holds performance.

Factor	D.F	Sum of squares	Mean squares	F value	P≤0.05
Wood type (A)	1	6660882.564	6660882.564	410.7778	0.0000
Treatment (B)	3	18045242.716	6015080.905	370.9511	0.0000
Interactive (AB)	3	3105742.805	1035247.602	63.8439	0.0000
Error	72	1167501.234	16215.295		
Total	79	28979369.319			

Tab. 11: Screw holds performance (N).

Wood type and treatment	Screw hold	HG
Oak control	2692	A
Oak+impregnated	2635	A
Pine control	1702	B
Pine+impregnated	1685	B
Oak+thermo	1334	C
Oak+ Thermo+impregnated	1308	C
Pine+Thermo+impregnated	1153	D
Pine+thermo	1120	D

LSD: ±113.4

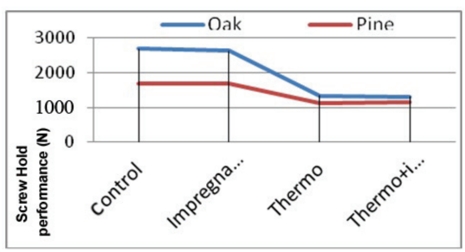


Fig. 5: Screw holds performance of a treated wood.

Comparing to the control samples the highest value is obtained for the impregnated oak (2692 N), the lowest for the heat treated pine (1120 N), see Tab. 11. Thermo process reduces the screw holds performance more. For this reason, the molecular structure of wood deteriorates during the thermo process (Hakkou et al. 2003).

Dowel joint performance (N)

Wood types and treatment and their interactions reduce the dowel joint performance in most cases. Wood types, treatment and their interactions appear significant. Multiple Variance Analysis of dowel joint performance and homogeneity of the groups of the dowel joint performance averages is given in Tab. 12 and Tab. 13.

Tab. 12: Multiple variance analysis of dowel joint performance.

Factor	D.F	Sum of squares	Mean squares	F value	P≤0.05
Wood type (A)	1	2994334.997	2994334.997	178.1982	0.0000
Treatment (B)	3	2872827.129	957609.043	56.9890	0.0000
Interactive (AB)	3	1386965.609	462321.870	27.5136	0.0000
Error	72	1209844.243	16803.392		
Total	79	8463971.978			

Tab. 13: Dowel joint performance of a treated wood.

Wood type and treatment	Dowel joint perform.	HG
Oak control	2091	A
Oak+impregnated	1833	B
Oak+thermo	1359	C
Oak+Thermo+impregnated	1351	C
Pine control	1346	C
Pine+impregnated	1314	C
Pine Thermo+impregnated	1286	C
Pine+thermo	1140	D

LSD: ±115.40

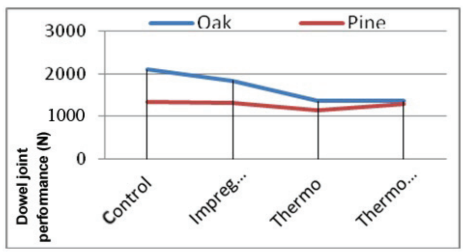


Fig. 6: Dowel joint performance of a treated wood.

While comparing the control samples the highest value is for the impregnated oak (1833 N), the lowest for the heat treated pine (1140 N), see Tab. 13.

Impregnation and heat treatment reduced the performance of both wood types (more on the oak). The results in Tab. 8 are suitable with the results of Awoyemi and Westmarck 2005's findings. However, after the heat-treatment impregnation the performance of pine was increased (Fig. 6).

Tenon and mortise joint performance (N)

Wood types and treatment affect the performance in most of their interactions. In addition to wood types, treatment and their interactions appear significant. Multiple Variance Analysis of tenon and mortise joint performance and homogeneity of the groups of the tenon and mortise-joint performance is given in Tab. 14 and Tab. 15.

Tab. 14: Multiple variance analysis of tenon and mortise joint performance.

Factor	D.F	Sum of squares	Mean squares	F value	P≤0.05
Wood type (A)	1	5470203.589	5470203.589	45.7528	0.0000
Treatment (B)	3	86408097.310	28802699.103	240.9060	0.0000
Interactive (AB)	3	1428340.556	476113.519	3.9822	0.0111
Error	72	8608312.883	119559.901		
Total	79	101914954.339			

Tab. 15: Tenon and mortise- joint performance of a treated wood.

Wood type and treatment	Tenon and mortise joint perform. (N)	HG
Oak control	4564	A
Pine control	4417	A
Oak+ımpregnated	3826	B
Pine+ımpregnated	3363	C
Oak+Thermo+ımpregnated	2792	D
Oak+thermo	2139	E
Pine+Thermo+ ımpregnated	1900	E
Pine+thermo	1549	F

LSD: ±127.38

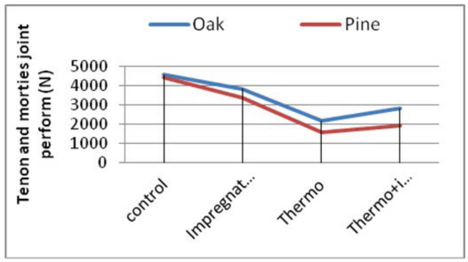


Fig. 7: Tenon-mortise joint perform. of a treated wood.

Comparing to the control samples the highest value is obtained on the impregnated oak (3826 N), the lowest on the heat treated pine (1549 N), (Tab. 15). Impregnation and heat treatment reduces the performance of both wood types. The results in Tab. 9 are also suitable with the results of Awoyemi and Westmarck 2005's findings. However, after the heat-treatment+impregnation the tenon and mortise joint performance was increased on both wood types (Fig. 7).

CONCLUSIONS

In general the bending strength, bonding strength, dowel holds performance, screw holds performance, dowel-joints performance and tenon and mortise joint performance was decreased by impregnation. However, bending strength, bonding strength and screw holds performance were increased on the oak in range 2-20 % (Tab. 16).

The strength and performance of all the properties on the both wood types were decreased by thermo process more than by the impregnation.

Bending strength, dowel holds performance, tenon and mortise performance on the oak, nevertheless bonding strength, dowel holds performance, screw holds performance, dowel joints performance and tenon and mortise-joint performance on the pine was increased due thermo+impregnation process. Percentages change in properties is given in Tab. 16.

Tab. 16: Some selected properties of two wood types and theirs percentage change due treatment. according to control samples (%).

Wood Type	Treatment	Bending strength	Bonding strength	Dowel holds performance	Screw holds performance	Diagonal compressions performance	
						Dowel-joint performance	Tenon and Mortise-joint performance
		(N.mm^{-2})		(N)		(N)	
Oak	Control	-	-	-	-	-	-
	Impregnated	+11.43	+20.32	-18.66	+2.16	-12.33	-16.17
	Thermo	-41.53	-22.48	-62.00	-49.37	-34.99	-53.14
	Thermo + Impregnated	-38.51	-29.09	-58.70	-50.37	-35.44	-38.82
Pine	Control	-	-	-	-	-	-
	Impregnated	-9.28	-19.83	-23.35	-0.96	-2.65	-23.86
	Thermo	-47.07	-50.59	-76.32	-34.18	-15.55	-64.93
	Thermo + Impregnated	-56.39	-30.00	-72.75	-32.25	-4.59	-56.98

In generally the application of the impregnation after the heat-treatment of the oak and pine wood which will used for external environment can be proposed.

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