

## **IMPACT OF VARIOUS CHEMICALS ON COMBUSTION PROPERTIES OF HEAT-TREATED AND IMPREGNATED LAMINATED VENEER LUMBER**

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### **ABSTRACT**

The aim of this study was to investigate the effects of borax and boric acid on the combustion properties of heat-treated and laminated veneer lumber (LVL) produced with Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder) wood. The combustion test method was performed in three stages according to ASTM E 160-50 1975. Consequently, the highest weight loss (WL) was obtained in unimpregnated and unheat treated and glued specimens with D-VTKA (91.75 %) adhesive; the highest temperature of flame source (TFS) was obtained in unimpregnated and heat treated at 210°C and glued specimens with D-VTKA (678°C) adhesive; the highest temperature of without flame source (TWFS) was obtained in unimpregnated and heat treated at 180°C and glued specimens with D-VTKA (673°C) adhesive; and the highest temperature of glowing stage (TGS) was obtained in unimpregnated and unheat treated and glued specimens with MF (224°C) adhesive. In consequence, heat treatment decreased the fire properties of LVL specimens, relatively, and MF gave better results than DVTKA. Additionally, boron compounds had some efficacy in retarding flame spread on wood surfaces, especially borax.

**KEYWORDS:** Heat treatment, combustion, adhesive, lamination, Anatolian black pine, boron.

### **INTRODUCTION**

Laminated veneer lumber (LVL) is one of the most widely used engineered wood products for construction applications. Due to the depletion of forest resources, there is a shortage of wood required by the industry. As such, layered composite materials like vertically and horizontally glued laminated wood and laminated veneer lumber (LVL) may be used as substitute for solid wood as they retain the structural properties of wood (Kamala et al. 1999).

Environmental considerations have increased interest in developing alternative wood modification methods. Heat treatment is an effective method to improve the dimensional stability of wood and resistance against biodegradation. Heat treatments generally take place in an

inert gas atmosphere at temperatures between 180 and 260°C (Leithoff and Peek 1998). However, there is a noticeable reduction of mechanical properties (especially static and dynamic bending strength and also compressive strength) after heat treatment, mainly due to the high temperatures involved (Militz 2002, Sailer et al. 2000, Esteves and Pereira 2009, Bekhta and Niemz 2003, Esteves et al. 2008, Unsal and Ayrilmis 2005, Korkut 2008, Yildiz et al. 2006).

Boron compounds have been used as fire retardants and as wood preservation chemicals against fungi and insects and are especially effective against termites. Boron-based buffers have also been used as additives in fire-retardant treatments and have been found to significantly reduce the severity of thermal degradation. Wood treated with inorganic flame-retardant salts is usually more hygroscopic than untreated wood, particularly at high relative humidity. Increases in the equilibrium moisture content of such treated wood will depend upon the type of chemical, level of chemical retention, and size and species of the wood involved (Hafizoglu et al. 1994, Awoyemi and Westermark 2005, Nagieb et al. 2011, Winandy 1997).

Ozcifci et al. (2007) studied the fire properties of laminated veneer lumber (LVL) prepared from beech (*Fagus orientalis* Lipsky) veneers treated with various fire retardants. They found that the lowest temperature and mass loss were obtained for specimens treated with a diammonium phosphate and boric acid–borax mixture. Baysal (2011) investigated some combustion properties of Calabrian pine (*Pinus brutia* Ten.) wood specimens impregnated with aqueous solutions of commercial fertilizers. Ammonium sulphate (AS) and diammonium phosphate (DAP) were used as commercial fertilizers. The results indicated that fertilizer treatments improved the combustion properties of Calabrian pine specimens. According to Yalinkilic et al. (1998) Douglas wood treated with a mixture of boric acid and borax had higher fire-retardant properties than when treated only with boric acid or borax. In order to reduce flammability and improve safety, wood is treated with fire-retardant chemicals. In other words, the combustibility of wood can be reduced with flame-retardants or fire-retardants (Nussbaum 1988, Ellis and Rowell 1989, Mitchell 1993). Lee and Kim (1982) investigated the fire resistance of meranti plywoods impregnated with various fire retardant chemicals. They found that diammonium phosphate ranked the highest in fire-retardant effectiveness.

This study was done to determine the impact of impregnation chemicals on the combustion properties of the LVL prepared from heat treated Anatolian black pine veneers at 150, 180 and 210°C for 120 min., laminated with MF and D-VTKA.

## MATERIAL AND METHODS

### Wood material

Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder) wood was selected as a test material because of wide usage in industry. Black pine is the second most widely distributed conifer species in Turkey after Calabrian pine (*Pinus brutia* L.), covering a land area of 2 204 381 ha (Kucuk et al. 2007). Special emphasis was given to the selection of wood materials which were non-deficient, proper, knotless, and normally grown (without reaction wood, decay and mushroom damages) according to TS 2470.

### Impregnation materials

The properties of the boric acid ( $H_3BO_3$ ) was 56.30 %  $\frac{1}{2} B_2O_3$ , 43.70 %  $H_2O$  with a molecular weight 61.84, density 1.435 g.cm<sup>-3</sup> and melting point of 171°C. Borax ( $Na_2B_4O_7 \cdot 5H_2O$ ) content was 21.28  $Na_2O$ , 47.80  $B_2O_3$ , and 30.92 %  $H_2O$  with a molecular weight of 291.3, density 1.815 g.cm<sup>-3</sup>, and melting point of 741°C (Keskin 2009a).

## Adhesives

Melamine formaldehyde (MF) and Desmodur-VTKA (D-VTKA) (Desmodur-Vinyl Triacetonol Acetate) adhesives were used for the bonding of the laminations on top of each other. These adhesives are usually preferable for assembly or furniture production in the woodworking industry and therefore, were chosen as the adhesives for this study as adhesives. Specific characteristics are given in Tab. 1.

Desmodur-VTKA (D-VTKA) is a one-component (without any solvent), polyurethane based and moisture cured adhesive. The bonding surface should be clean, dry, dust and oil free. Dry surfaces should be moistened so as to increase the hardening speed of the glue. Adhesive is directly applied to one of the surfaces and the bonding process is conducted at  $20 \pm 2^\circ\text{C}$  and  $65 \pm 5\%$  relative humidity conditions (Ors et al. 2004). MF adhesive is most commonly used for exterior and semi-exterior plywood and particleboard, and for finger joints. The limitation of the MF adhesives is their high cost due to the cost of the melamine. This has led to the use of melamineurea-formaldehyde (MUF) resins that have much of the water resistance of MF resins, but at a substantially lower cost (Ozcifci et al. 2007).

Tab. 1. Characteristics of adhesives used.

Adhesive	Density (g.cm <sup>-3</sup> )	pH	Viscosity (MPa s)	Time to solidify at 100-110 °C	Gel time at 20 °C	Spread amount (g.m <sup>-2</sup> )
D-VTKA	1.12-1.13	6-7	13.000 ± 2.000	1 min	5-10 min	200
MF	1.22-1.24	9	12.000 ± 3.000	2-3 min	15-20 min	200

## Heat treatment

Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder) wood was cut in parallel with the grain and cut into specimens measuring 50 x 70 x 500 mm (tangential, radial, longitudinal) long. Specimens were air dried for two months until they reached approximately 12 % MC. The samples were heat-treated at 150, 180 and 210°C for 120 min. Heat treatment was carried out under atmospheric pressure, with water vapour as a shielding gas. The total duration of the heat treatment (pre-treatment period + actual heat treatment period + cooling and moisture conditioning period) was 34 hours and actual times at high temperature were 120 min. After the heat treatment, specimens dimensions were re-measured by digital caliper and weights were recorded on a digital weight scale. Then specimens were cut parallel to grain direction and cut into specimens measuring 3 x 70 x 500 mm (tangential, radial, longitudinal) long. Heat-treated specimens were then reconditioned at  $20 \pm 2^\circ\text{C}$  and  $65 \pm 5\%$  relative humidity for 3 weeks.

## Preparation of test specimens

Boric acid (BA), and borax (BX) were dissolved in distilled water to a concentration of 4 %. Before testing the bonded samples, the impregnating process was carried out according to the procedure described in the ASTM-D 1413-76 standard. The dipping method was employed for sample impregnation. For this purpose, a container that was 80 cm in width and 80 cm in length was filled with the impregnating material. The test samples were then dipped into the impregnating material for 360 min. After the impregnation process, the test samples were removed from the container and conditioned at  $20 \pm 2^\circ\text{C}$  and at  $65 \pm 3\%$  relative humidity in the conditioning room until their weights became constant. Finally, the samples were bonded

with adhesives. The MF and D-VTKA adhesives were applied on a single bonding surface of veneers at approximately  $200 \text{ g.m}^{-2}$  by using a roller coater. A pressure of  $0.8 \text{ N.mm}^{-2}$  was applied on the bonding line according to adhesive manufacturer instructions. Solidification time was approximately 30 min. The LVL samples were kept under a temperature of  $20 \pm 2^\circ\text{C}$  and  $65 \pm 5\%$  relative humidity until reaching a constant weight. Combustion test samples with dimensions of  $13 \times 13 \times 76 \text{ mm}$  were prepared from the laminated veneer lumber according to ASTM E 160-50 (1975). The impregnation test plan is given in Tab. 2.

Tab. 2: Impregnation test plan.

Impregnation chemicals	Solution concentration (%)	Solvent	Temperature	pH	Density $\text{g.ml}^{-3}$
Borax	4	DW	$20 \pm 2^\circ\text{C}$	9.45	1.035
Boric acid	4	DW	$20 \pm 2^\circ\text{C}$	5.73	1.030

DW: Distilled water.

The processes was carried out at  $20 \pm 2^\circ\text{C}$ . The retention content for each treatment was calculated by the following formula:

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

where:  $G$  - the amount of impregnation solution absorbed by the sample,  
 $T_2$  - the sample weight (g) after the impregnation,  
 $T_1$  - the sample weight (g) before impregnation,  
 $C$  - the concentration of the impregnation solution,  
 $V$  - the volume ( $\text{cm}^3$ ) of the samples.

Impregnated test samples were kept at a temperature of  $20 \pm 2^\circ\text{C}$  and  $65 \pm 3\%$  humidity content until they reached a stable weight.

### Combustion test

Combustion tests were carried out according to ASTM E 160-50 (1975) standards in a combustion test device. Specimens were conditioned at  $27 \pm 2^\circ\text{C}$  and 30-35 % relative humidity to the targeted equilibrium moisture content of 7 % prior to the fire test. Samples were weighed before the combustion tests. Twenty-four specimens were stowaged to make 12 layers that shaped a square prism. The fire distance from the maker type outlet at the lower bound of the funnel was fixed to  $25 \pm 1.3 \text{ cm}$ . When the device was empty the gas pressure was fixed to  $0.5 \text{ kg.cm}^{-2}$ . During burning, the temperature was set at  $315 \pm 8^\circ\text{C}$  in the funnel. The combustion test method was then performed (flame stage (FS), without flame stage (WFS), and glowing stage (GS)) according to ASTM E 160-50 (1975). Temperatures were recorded at the combustion column by thermocouples at 15, 30, and 30 second intervals for combustion with a flame stage, at the without-flame stage, and during the glowing stage, respectively. The weight losses (WL) of test specimens after the combustion test were calculated from the following equation:

$$\text{WL} = [(W_{\text{bf}} - W_{\text{af}}) / W_{\text{bf}}] \times 100 \quad (\%) \quad (2)$$

where:  $W_{\text{bf}}$  - the weight (g) of the wood specimen before fire test,  
 $W_{\text{af}}$  - the weight (g) of the wood specimen after the fire test.

Four replicates were used for each variation.

## Statistical analyses

The impact of heat treatment and impregnation with boron compounds (borax and boric acid), on the combustion properties of LVL-produced Anatolian black pine veneers bonded with D-VTKA and MF adhesives was analysed by analysis of variance (ANOVA). When the differences between groups were found to be significant, a Duncan test was used to determine the differences between means at a prescribed level of  $\alpha = 0.05$ .

## RESULTS AND DISCUSSION

The results of the retention quantities for impregnation materials were summarized by using descriptive statistics such as maximum, minimum, mean, standard deviation. The retention quantities of impregnation chemicals are given in Tab. 3.

Tab. 3: Retention quantities of impregnation materials ( $\text{kg}\cdot\text{m}^{-3}$ ).

Statistical values	Borax				Boric acid			
	Unheat treated	150°C	180°C	210°C	Unheat treated	150°C	180°C	210°C
X	22.81	21.22	18.36	17.64	21.78	20.18	17.60	16.81
Mim.	20.89	20.80	17.16	16.73	19.17	18.67	16.54	15.75
Max.	24.99	22.84	20.41	18.99	23.63	21.62	19.25	18.61
Sd.	1.2069	0.7804	1.0102	0.7424	1.2492	0.8492	0.8723	0.9442

x: Mean, Min: Minimum, Max: Maximum, Sd: Standard deviation.

The retention quantities of impregnation materials decreased with an increase in heat treatment temperature and the heat-modified samples absorbed less impregnation materials than un-heated samples. Theoretically, the available OH groups in hemicellulose have the most significant effect on the physical properties of wood. Heat treatment slows water uptake and wood cell walls absorb less water because of the decrease in the amount of the wood's hydroxyl groups (Gunduz et al. 2008). Kortelainen et al. (2006) reported that heat treatment decreased the water absorption of spruce and pine heartwood. They found that the higher the heat-treating temperature, the lower the amount of absorbed moisture.

Average weight losses (WL) after the combustion test and average temperature values during the combustion test according to impregnation chemical type, heat treatment, and type of adhesive are given in Tab. 4, 5, 6, and 7 respectively. The results of a Duncan test are displayed in Tabs. 8, and 9.

Tab. 4: Average weight losses according to type of process.

Type of process		WL (%)	
		Mean	HG
Impregnation chemicals*	Unimpregnated	87.87	A
	Borax	80.40	C
	Boric acid	82.62	B

Heat treatment**	Unheat treated	83.34	A
	150 °C	83.46	A
	180 °C	83.76	A
	210 °C	84.04	A
Types of adhesive***	Solid wood	85.13	B
	MF	78.20	C
	D-VTKA	87.56	A

MF: Melamine formaldehyde, D-VTKA: Desmodur-Vinyl Triacetonol Acetate, HG: Homogeneity groups, Different letters in a column refers to significant differences among types of processes at a 0.05 confidence level, WL: Weight loss, HG: Homogeneity groups, \*LSD: 0.7610, \*\*LSD: 0.8787, \*\*\*LSD: 0.2722.

According to Tab. 4, the highest weight loss (WL) value was obtained in unimpregnated (87.87 %) wood, at 210°C (84.04 %), with D-VTKA (87.56 %) and was lowest in borax (80.40 %), unheat treated (83.34 %), with MF (78.20 %) adhesive.

Tab. 5: Average temperature of the flame source values according to type of process.

Type of process		TFS (°C)	
		Mean	HG
Impregnation chemicals*	Unimpregnated	649	A
	Borax	494	C
	Boric acid	541	B
Heat treatment**	Unheat treated	547	A
	150 °C	561	A
	180 °C	568	A
	210 °C	570	A
Types of adhesive***	Solid wood	565	A
	MF	546	B
	D-VTKA	573	A

TFS: Temperature of flame source, \*LSD: 18.27, \*\*LSD: 21.10, \*\*\*LSD: 18.46.

According to Tab. 5, the highest temperature of the flame source (TFS) value was obtained in unimpregnated (649°C) wood, at 210°C (570°C), with D-VTKA (573°C) and the lowest in borax (494°C), unheat treated (547°C), with MF (546°C) adhesive.

Tab. 6: Average temperature of the without flame source values according to type of process.

Type of process		TWFS (°C)	
		Mean	HG
Impregnation chemicals*	Unimpregnated	647	A
	Borax	495	C
	Boric acid	548	B
Heat treatment**	Unheat treated	565	A
	150 °C	563	A
	180 °C	568	A
	210 °C	558	A

Types of adhesive***	Solid wood	572	A
	MF	534	B
	D-VTKA	584	A

TWFS: Temperature without flame source, \*LSD:16.48, \*\*LSD:19.03, \*\*\*LSD:16.48.

According to Tab. 6, The highest temperature of the without flame source (TWFS) value was obtained in unimpregnated (647°C) wood, at 180°C (568°C), with D-VTKA (584°C) and was lowest in borax (495°C), at 210°C (558°C), with MF (534°C) adhesive.

Tab. 7: Average temperature for the glowing stage values according to type of process.

Type of process		TGS (°C)	
		Mean	HG
Impregnation chemicals*	Unimpregnated	205	A
	Borax	176	B
	Boric acid	169	B
Heat treatment**	Unheat treated	191	A
	150 °C	184	B
	180 °C	179	C
	210 °C	172	D
Types of adhesive***	Solid wood	185	A
	MF	171	B
	D-VTKA	189	A

TGS: Temperature of glowing stage, \*LSD:5.224, \*\*LSD:6.032, \*\*\*LSD:5.224.

According to Tab. 7, The highest temperature for the glowing stage (TGS) value was obtained in unimpregnated (205°C) wood, unheat treated (191°C), with D-VTKA (189°C) and was lowest in boric acid (169°C), at 210°C (172°C), with MF (171°C) adhesive.

Weight loss values of all the specimens increased depending on the heat treatment temperature. This is probably related to release of volatile compounds during the heat treatment. Similar results have been reported by the Finnish ThermoWood Association (2003). However, there were no significant differences between the groups. According to type of adhesive, the laminated with D-VTKA adhesive samples had a weight loss value higher than the laminated with MF adhesive samples and solid woods. This may be due to delamination in laminated with D-VTKA adhesive samples during the fire test. The weight loss of specimens impregnated with borates lower than unimpregnated specimens. In other words, boron compounds reduces weight of LVL, especially borax. Ozciftci et al. (2007) determined fire properties of laminated veneer lumber (LVL) prepared from beech (*Fagus orientalis* Lipsky) veneers treated with some fire retardants. Their results indicated that the lowest mass loss was found for specimens glued with MF and treated with a DAP and (BA-BX) mixture.

TFS values of all the specimens increased depending on the temperature. According to this; heat treatment decreased the fire properties of LVL specimens. This may be due to a lower equilibrium moisture content. However, there were no significant differences between the groups. According to Finnish ThermoWood (2003), the RHR (Rate of Heat Release) level of heat-treated pine was about 10 kW greater than that of untreated pine. The TFS values of specimens impregnated with borates lower than unimpregnated specimens. According to this; both of impregnation materials showed fire-retardant effect in LVL samples. The laminated

specimens with MF adhesive samples had a weight loss value lower than the laminated with D-VTKA adhesive samples and solid woods. This may be due to the resistive property of MF against combustion.

Similarly, according to impregnation chemicals, both of impregnation materials showed fire-retardant effect in LVL samples for TWFS. MF adhesive gave better results than D-VTKA adhesive for TWFS. This may be due to the resistive property of MF against fire. TWFS values of all the specimens decreased depending on the heat treatment temperature except for 180°C. TGS values of all the specimens decreased depending on the heat treatment temperature and boron compounds. The laminated specimens with MF adhesive had lower TGS than laminated specimens D-VTKA and solid wood. This may be due to the dispersal of the laminated samples with D-VTKA and solid wood samples during the combustion test.

Tab. 8: Results of the Duncan test for weight loss and temperature of the flame source.

Impregnation chemicals	Heat Treatment	Type of adhesive	WL (%)*			TFS (°C)**		
			Mean	HG	Sd	Mean	HG	Sd
Unimpregnated	Unheat treated	Solid Wood	88.45	BCDE	1.306	635	A	46.396
		MF	86.95	CDEF	2.052	611	ABC	46.482
		D-VTKA	91.75	A	2.840	625	AB	35.693
	150°C	Solid Wood	86.85	CDEF	1.462	657	A	54.522
		MF	85.76	DEFG	2.356	638	A	51.439
		D-VTKA	90.25	AB	1.241	644	A	58.963
	180°C	Solid Wood	88.65	BCD	1.568	667	A	46.568
		MF	86.25	DEFG	2.603	642	A	34.409
		D-VTKA	88.85	ABC	1.283	671	A	41.856
	210°C	Solid Wood	88.75	BCD	1.703	675	A	50.622
		MF	82.18	HIJ	2.242	645	A	36.340
		D-VTKA	89.73	ABC	1.382	678	A	45.332
Borax	Unheat treated	Solid Wood	80.05	JK	0.632	485	DEF	49.012
		MF	73.18	L	1.823	466	F	36.746
		D-VTKA	83.37	GHI	0.911	495	DEF	53.841
	150°C	Solid Wood	83.11	GHI	1.386	491	DEF	54.433
		MF	73.43	L	0.870	475	EF	35.311
		D-VTKA	85.46	EFG	2.059	503	DEF	44.078
	180°C	Solid Wood	83.25	GHI	1.717	498	DEF	36.792
		MF	72.78	LM	1.312	485	DEF	32.659
		D-VTKA	87.05	CDEF	2.237	510	DEF	33.536
	210°C	Solid Wood	85.85	DEFG	1.895	493	DEF	36.986
		MF	70.16	M	2.078	485	DEF	53.674
		D-VTKA	87.11	CDEF	1.966	545	CDE	49.665



Boric acid	Unheat treated	Solid Wood	82.12	IJ	1.206	546	CDE	45.028
		MF	79.92	JK	1.862	521	DEF	23.895
		D-VTKA	84.48	FGHI	1.906	538		38.423
	150°C	Solid Wood	85.25	FGH	1.901	551	CDE	56.747
		MF	74.21	L	1.629	536		48.359
		D-VTKA	85.79	DEFG	0.850	558	BCD	41.709
	180°C	Solid Wood	84.79	FGHI	1.740	547	CDE	49.345
		MF	75.29	L	2.545	541		33.011
		D-VTKA	86.95	CDEF	2.906	552	CDE	39.152
	210°C	Solid Wood	84.45	FGHI	1.977	533	DEF	46.636
		MF	78.27	K	2.606	512	DEF	55.263
		D-VTKA	89.90	ABC	2.324	556	BCD	33.678

WL: Weight loss, TFS: Temperature of flame source, Sd: Standard deviation, \*LSD: 2.636, \*\*LSD: 63.30.

Tab. 9: Results of the Duncan test for temperature of the without flame source and temperature for the glowing stage.

Impregnation chemicals	Heat Treatment	Type of adhesive	TWFS (°C)*			TGS (°C)**		
			Mean	HG	Sd	Mean	HG	Sd
Unimpregnated	Unheat treated	Solid Wood	646	A	41.516	221	ABC	12.124
		MF	616	AB	76.227	224	A	17.823
		D-VTKA	637	A	52.694	215	A	15.557
	150°C	Solid Wood	665	A	45.389	219	AB	13.820
		MF	631	A	44.597	197	CDEFG	12.556
		D-VTKA	656	A	34.477	213	ABCD	12.328
	180°C	Solid Wood	667	A	46.166	208	ABCDE	20.984
		MF	626	A	50.460	185	FGHIJ	16.337
		D-VTKA	673	A	41.355	199	BCDEF	17.969
	210°C	Solid Wood	660	A	36.432	192	EFGH	13.952
		MF	629	A	42.602	173	HIJKLM	19.364
		D-VTKA	667	A	26.166	216	ABC	16.132
Borax	Unheat treated	Solid Wood	499	CDEF	38.99	189	EFGHI	11.958
		MF	452	F	31.626	171	HIJKLM	8.057
		D-VTKA	512	CDEF	36.399	183	FGHIJK	13.275
	150°C	Solid Wood	502	CDEF	31.042	174	HIJKLM	12.257
		MF	475	EF	26.191	161	KLMN	9.810
		D-VTKA	511	CDEF	34.912	185	FGHIJ	15.077
	180°C	Solid Wood	505	CDEF	43.034	160	LMN	9.810
		MF	470	EF	41.577	157	MN	10.214
		D-VTKA	517	CDEF	36.669	193	DEFGH	17.682
	210°C	Solid Wood	512	CDEF	37.797	155	MN	8.164
		MF	465	EF	49.216	149	N	6.976
		D-VTKA	520	CDEF	38.274	167	JKLMN	6.683

Boric acid	Unheat treated	Solid Wood	555	BC	41.444	181	FGHIJKL	17.682
		MF	527	CDE	44.399	162	KLMN	6.683
		D-VTKA	644	A	28.581	173	HIJKLM	13.638
	150°C	Solid Wood	550	C	32.659	177	GHIJKLM	12.832
		MF	511	CDEF	55.764	160	LMN	10.801
		D-VTKA	565	BC	39.092	174	HIJKLM	11.045
	180°C	Solid Wood	563	BC	28.593	173	HIJKLM	13.142
		MF	532	CDE	31.101	161	KLMN	6.244
		D-VTKA	556	BC	26.956	172	HIJKLM	7.483
	210°C	Solid Wood	545	CD	40.959	168	IJKLMN	9.899
		MF	477	DEF	36.340	157	LMN	5.737
		D-VTKA	552	BC	40.439	175	HIJKLM	11.842

TWFS: Temperature of without flame source, TGS: Temperature of glowing stage, \*: LSD: 57.10, \*\*:LSD:18.10.

According to Tab. 8, considering the interaction of impregnation chemicals, heat treatment, and types of adhesive the highest weight loss (WL) was obtained in unimpregnated and unheat treated and glued specimens with D-VTKA (91.75 %), and the lowest weight loss (WL) was obtained in impregnated specimens with borax and heat treated at 210°C and glued specimens with MF (70.16 %) adhesive. Baysal (2003) reported that *Fagus orientalis* wood specimens treated with a mixture of BA and BX lost around 68 % of mass during combustion. Yalınkılıç et al. (1998) found that douglas fir specimens treated with a mixture of BA and BX lost around 58 % of mass during combustion. The highest temperature of flame source (TFS) was obtained in unimpregnated and heat treated specimens at 210°C and glued specimens with D-VTKA (678°C), and the lowest TFS was obtained in impregnated specimens with borax and unheat treated and glued specimens with MF (466°C) adhesive.

According to Tab. 9, considering the interaction of impregnation chemicals, heat treatment, and types of adhesive the highest temperature of without flame source (TWFS) was obtained in unimpregnated specimens and heat treated at 180°C and glued specimens with D-VTKA (673°C), and the lowest TWFS was obtained in impregnated with borax and unheat treated and glued specimens with MF (452°C) adhesive. The highest temperature of glowing stage (TGS) was obtained in unimpregnated and unheat treated and glued specimens with MF (224°C), and the lowest TGS was obtained in impregnated with borax and heat treated at 210°C and glued specimens with MF (149°C) adhesive.

Rowell (2005) stated that wood material treated with boric acid had a weight loss of 81 % and borax a loss of 89 % at 500°C, which is not as effective as phosphorus compounds. Similarly, Atar et al. (2011) reported varnishes showed an increasing impact, but boron compounds (Ba and Bx) showed a decreasing impact on the combustion properties of beech wood. Indeed, boron compounds showed a decreasing impact on the combustion properties of the laminated wood materials, produced by a combination of beech and poplar veneers, bonded with D-VTKA (Keskin et al. 2009).

Borax and Boric acid treatment had a lower heat release rate at almost all combustion stages indicating inhibitor effects as a fire retardant on combustion. For this reason, borax and boric acid treatments for these trials of combustion had a diminishing effect on heat release rate. These results are consistent with previous studies that have shown similar effects (Baysal et al. 2003, Yuksel et al. 2014, Atar 2008, Atar and Keskin 2007, Atilgan et al. 2012, Candan et al. 2010, Keskin 2009b).

## CONCLUSIONS

Combustion properties of heat treated and laminated (with MF and D-VTKA) Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder) treated with water borne solutions of borax and boric acid were studied.

At the end of the combustion test, the highest weight loss was found in unimpregnated specimens, unheat treated and laminated with D-VTKA, and the lowest weight loss was obtained in impregnated with borax, heat treated at 210°C and laminated with MF. The highest TFS was founded in unimpregnated specimens, heat treated at 210°C and laminated with D-VTKA, and the lowest TFS was obtained in impregnated specimens with borax, unheat treated and laminated with MF. The highest TWFS was obtained in unimpregnated specimens, heat treated at 180°C and laminated with D-VTKA, and the lowest TWFS was obtained in impregnated specimens with borax, unheat treated and laminated with MF adhesive. The highest TGS was obtained in unimpregnated, unheat treated solid wood, and the lowest TGS was obtained in impregnated with borax specimens, heat treated at 210°C and laminated with MF.

According to these results, borax and boric acid reduced weight loss to a considerable extent. The boron compounds have some efficacy in retarding flame spread on wood surfaces. Heat treatment decreased fire properties of LVL specimens to some extent. However, there were no significant differences between the groups. MF adhesive gave better results than D-VTKA adhesive. This may be due to the resistive property of MF against fire.

In consequence, impregnation with boron compounds, especially with borax in laminated Anatolian black pine (*Pinus nigra* J.F. Arnold subsp. *nigra* var. *caramenica* (Loudon) Rehder) bonded with MF provides additional protection against a high risk of fire.

## REFERENCES

1. ASTM D 1413-76-99, 1976: Standart test method for wood preservatives by laboratory soil-block cultures.
2. ASTM E 160-50, 1975: Standard test method for combustible properties of treated wood by the cribe test.
3. Atar, M., 2008: Impacts of varnishes and impregnation chemicals on combustion properties of oak (*Quercus petraea* Lipsky). Journal of Applied Polymer Science 107(6): 3981-3986.
4. Atar, M., Keskin, H., 2007: Impacts of coating with various varnishes after impregnation with boron compounds on the combustion properties of Uludag fir. Journal of Applied Polymer Science 106(6): 4018-4023.
5. Atar, M., Keskin, H., Korkut, S., Korkut, D.S., 2011: Impact of impregnation with boron compounds on combustion properties of oriental beech (*Fagus orientalis* Lipsky) and varnishes. African Journal of Biotechnology 10(15): 2867-2874.
6. Atilgan, A., Peker, H., Ulusoy, H., 2012: Effects of different impregnation chemicals on combustion characteristics and decay resistance of wood. International Journal of Physical Sciences 7(47): 6149-6157.
7. Awoyemi, L., Westermark, U., 2005: Effects of borate impregnation on the response of wood strength to heat treatment. Wood Sci. Technol. 39(6): 484-491.
8. Baysal, E., 2003: Combustion properties of beech (*Fagus orientalis* Lipsky) wood treated with vegetable tanning extracts and boron compounds. Firat University, Journal of Institute of Science and Technology 15(1): 163-174.

9. Baysal, E., 2011: Combustion properties of wood impregnated with commercial fertilizers. *African Journal of Biotechnology* 10(82): 19255-19260.
10. Baysal, E., Yalinkilic, M.K., Colak, M., Goktas, O., 2003: Combustion properties of Calabrian pine (*Pinus brutia* Ten.) wood treated with vegetable tanning extract sand boron compounds. *Turk. J. Agric. For.* 27(4): 245-252.
11. Bekhta, P., Niemz, P., 2003: Effect of high temperature on the change in color, dimensional stability and mechanical properties of spruce wood. *Holzforschung* 57(5): 539-546.
12. Candan, Z., Ayrilmis, N., Dundar, T., Atar, M., 2010: Fire performance of LVL panels treated with fire retardant chemicals. *Wood Research* 57(4): 651-658.
13. Ellis, W.D., Rowell, R.M., 1989: Flame-retardant treatment of wood with a diisocyanate and an oligomerphosphate. *Wood Fiber Sci.* 21(4): 367-375.
14. Esteves, B.M., Domingos, I.J., Pereira, H.M., 2008: Pine wood modification by heat treatment in air. *BioResources* 3(1): 142-154.
15. Esteves, B.M., Pereira, H.M., 2009: Wood modification by heat treatment: A review. *BioResources* 4(1): 370-404.
16. Finnish Thermo Wood Association, 2003: Thermo wood handbook. Helsinki, Finland.
17. Gunduz, G., Korkut, S., Korkut, D.S., 2008: The effects of heat treatment on physical and technological properties and surface roughness of camiyani black pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood. *Bioresource Technol.* 99(7): 2275-2280.
18. Hafizoglu, H., Yalinkilic, M.K., Yildiz, U.C., Baysal, E., Peker, H., Demirci, Z., 1994: Utilization of Turkey's boron reserves in wood preservation industry. Project of Turkish Science and Tech. Council (TUBITAK), Code: TOAG-875, 377 pp.
19. Kamala, B.S., Kumar, P., Rao, R.V., Sharma, S.N., 1999: Performance test of Laminated Veneer Lumber (LVL) from rubber wood for different physical and mechanical properties. *Holz als Roh- und Werkstoff* 57(2): 114-116.
20. Keskin, H., 2009a: Effects of impregnation solutions on weight loss during combustion of laminated veneer lumber. *G.U. Journal of Science* 22(3): 235-243.
21. Keskin, H., 2009b: Impacts of impregnation chemicals on the flame source combustion light intensity of the laminated veneer lumber (LVL). *Wood Research* 54(4): 67-78.
22. Keskin, H., Atar, M., Izciler, M., 2009: Impacts of impregnation chemicals on combustion properties of the laminated wood materials produced combination of beech and poplar veneers. *Construction and Building Materials* 23(2): 634-643.
23. Korkut, S., 2008: The effects of heat treatment on some technological properties in Uludag fir (*Abies bornmuellerianana* Mattf.) wood. *Build. Environ.* 43(4): 422-428.
24. Kortelainen, S.M., Antikainen, T., Viitaniemi, P., 2006: The water absorption of sapwood and heartwood of Scots pine and Norway spruce heat-treated at 170°C, 190°C, 210°C and 230°C. *Holz als Roh- und Werkstoff* 64(3): 192-197.
25. Kucuk, O., Saglam, B., Bilgili, E., 2007: Canopy fuel characteristics and fuel load in young black pine trees. *Biotechnology & Biotechnological Equipment* 21(2): 235-240.
26. Lee, P.W., Kim, J.M., 1982: Studies on fire-retardant treatment and press drying of plywood. *Kor. Wood Sci. Technol.* 10(1): 5-37.
27. Leithoff, H., Peek, R.D., 1998: Hitzebehandlung - eine Alternative zum chemischen Holzschutz. In: *Tagungsband zur 21. Holzschutz-Tagung der DGFH in Rosenheim*. Pp 97-108 (in German).
28. Militz, H., 2002: Thermal treatment of wood: European Processes and their background. International Research Group on Wood Preservation, Section 4-Processes, N° IRG/WP 02-40241.

29. Mitchell, S., 1993: Fire performance of wood: Test methods and fire retardant treatments. In: Proceedings of the 4<sup>th</sup> annual BCC Conference on Flame Retardancy, Stamford, CT Normwalk, CT; Business Communications Co. Pp 36-43.
30. Nagieb, Z.A., Nassar, M.A., El-Meligy, M.G., 2011: Effect of addition of boric acid and borax on fire-retardant and mechanical properties of urea formaldehyde saw dust composites. International Journal of Carbohydrate Chemistry, Article ID 146763, 1-6.
31. Nussbaum, R., 1988: The effect of low concentration fire retardant impregnations on wood charring rate and char yield. J. Fire Sci. 6(4): 290-306.
32. Ors, Y., Atar, M., Keskin, H., 2004: Bonding strength of some adhesives in wood materials impregnated with Imersol-AQUA. International Journal of Adhesion and Adhesives 24(4): 287-294.
33. Ozcifci, A., Toker, H., Baysal, E., 2007: Fire properties of laminated veneer lumber treated with some fire retardants. Wood Research 52(4): 37-46.
34. Rowell, R.M., 2005: Handbook of wood chemistry and wood composites. CRC Press, 2000 N.W. Corporate Blvd., Boca Raton, Florida 33431, USA, 467 pp.
35. Sailer, M., Rapp, A.O., Leithoff, H., Peek, R-D., 2000: Vergütung von Holz durch Anwendung einer Öl-Hitzebehandlung. Holz als Roh- und Werkstoff 58: 15-22.
36. TS 2470, 1976: Wood - Sampling methods and general requirements for physical and mechanical tests.
37. TS EN 386, 1999: Glued laminated timber - performance requirements and minimum production requirements.
38. Unsal, O., Ayrimis, N., 2005: Variations in compression strength and surface roughness of heat-treated Turkish river red gum (*Eucalyptus camaldulensis*) wood. J. Wood Sci. 51(4): 405-409.
39. Winandy, J.E., 1997: Effects of fire retardant retention, borate buffers, and redrying temperature after treatment on thermal-induced degradation. Forest Products Journal, 47(6): 79-86.
40. Yalinkilic, M.K., Baysal, E., Demirci, Z., 1998: Fire resistance of Douglas fir (*Pseudotsuga menziesii* (Mirb.) Franco) wood treated with some chemicals. Pamukkale University, Journal of Engineering Sciences 4: 613-624.
41. Yildiz, S., Gezer, E.D., Yildiz, U.C., 2006: Mechanical and chemical behavior of spruce wood modified by heat. Build Environ 41(12): 1762-1766.
42. Yuksel, M., Baysal, E., Toker, H., Simsek, H., 2014: Combustion characteristics of oriental beech wood impregnated with commonly used borates. Wood Research 59(1): 39-50.

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