

EFFECTS OF SOME IMPREGNATION CHEMICALS ON COMBUSTION CHARACTERISTICS OF LAMINATED VENEER LUMBER (LVL) PRODUCED WITH OAK AND POPLAR VENEERS

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The objective of this research was to investigate the effects of impregnation chemicals on the combustion properties of 3-ply laminated veneer lumber (LVL) made of Oak (*Quercus petraea subsp. Iberica*) and Poplar (*Populus tremula* L.). For this purpose, oak wood was used as the outer ply and poplar used for the core ply in LVL. Borax (BX), boric acid (BA), borax+boric acid (BX+BA), and di-ammonium phosphate (DAP) were used as impregnation chemicals, and urea formaldehyde (UF), phenol formaldehyde (PF), and melamine-urea-formaldehyde (MUF) adhesives as bonding agent were used to produce LVLs. The vacuum – pressure method was used for the impregnation process. The combustion test was performed according to the procedure defined in the ASTM–E 69 standards, and during the test the mass reduction, temperature, and released gas (CO, O₂) were determined for each 30 seconds. As a result, di-ammonium phosphate was found to be the most successful fire retardant chemical in LVL with MUF adhesive. LVL produced from a combination of oak and poplar veneers with MUF adhesive and impregnated with DAP can be recommended to be used as a fire resistant building material where required.

Key Words: Borax (BX); Boric acid (BA); Di-ammonium phosphate (DAP); Fire retardant; Laminated veneer lumber (LVL); Oak; Poplar

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INTRODUCTION

Laminated material (LAM) produced from massive wood is used as a furniture component and is an important building material in the woodworking industry. It is possible to produce many desired forms and shapes of LAM with lamination techniques. According to the wood material, LAM has some technical and economical advantages (Kurt 2006).

TS ENV 12520 describes laminated wood as follows: laminated wood is obtained from wood sheets produced by slicing, sawing, and rotary methods. Between the sheets different adhesives are applied and pressed as smooth and moulding shapes by cold and hot pressing methods.

Laminated veneer lumber (LVL), like plywood panels, is manufactured using different synthetic resins, depending on where they are used. Phenol–formaldehyde (PF) resin is generally used as a binder for exterior grade panel production. Melamine–urea–formaldehyde (MUF) resin is also used for panels to be evaluated under damp conditions.

However, it has been stated that a panel bonded with urea–formaldehyde (UF) resins under exterior conditions is not as resistant as the panel bonded with PF resin (Pizzi 1993).

Increasing concern for fire safety has prompted stricter construction requirements in the form of building codes with stricter fire performance. With greater use of LVL in building construction there is a greater need for the development of effective fire-retardant treatments for this product. It is well known that there are possibilities to improve significantly the fire performance of wood by chemical treatment and to widen its application options. In general the amount of flame retardant uptake to the wood is directly proportional to the improvement of resistance to fire characteristics (Balog 1986).

Long-term retardants consist of flame inhibiting chemicals dissolved in water. They remain effective even after water has been removed by evaporation. The key ingredient in these retardants is the active retardant salt, usually referred to as “active salt,” which is typically either an ammonium sulfate or ammonium phosphate (George and Johnson 1986).

Boric acid (BA) and borax (BX) are the most common boron compounds that have found many application areas in the wood preservation industry in order to obtain the benefit of their biological effectiveness and fire retardancy (Hafizoglu et al 1994).

Kurt and Uysal, (2009) have been investigated that the effects of the impregnation materials di-ammonium phosphate, aluminum sulphate, potassium carbonate, calcium chloride, and zinc chloride on combustion properties of 3-ply LVL produced from oak (*Quercus robur* L.) bonded by PF, or poly(vinyl acetate) (PVAc). According to the test results, zinc chloride was found to be the most successful fire retardant chemical in LVL with PF adhesive. Since zinc chloride diminishes combustion, it is possible to recommend the use of LVL produced from oak.

Keskin et al. (2009) investigated the effects of impregnation with the boron compounds BX, BA, BX + BA, Imersol-Aqua (IAQUA), and Timbercare-Aqua (TAQUA) on combustion properties of the laminated wood materials produced from a combination of Oriental beech and poplar veneers bonded with Desmodur-VTKA (DV) and PVAc adhesives. As a consequence, impregnation with boron compounds and Imersol-Aqua of the laminated wood materials, bonded with DV, provides security for the usage under conditions where there is high risk of fire.

Uysal and Ozçifçi, (2004) investigated the combustion properties of LVL from Uludağ fir wood samples impregnated with some chemicals by using dipping process. They found the highest mass reduction in the massive samples impregnated with Tanalith-CBC, and the highest ratio of CO and CO₂ was found in the unprocessed massive control samples. On the other hand the highest temperature variation was obtained in laminated samples impregnated with Tanalith-CBC, the highest consumption ratio of O₂ was in the massive wood samples impregnated with sodium tetra borate, and the highest ash ratio in laminated samples impregnated with sodium perborate.

Kartal and Imamura (2004) reported that boron compounds are considered to be the only wood preservatives capable of acting as both insecticides and fungicides, which and they are widely recognized and accepted to be effective and of low toxicity. Boron mobility is another advantage in most applications; however the natural solubility of

boron limits use in exterior applications of boron treated wood. On the other hand, boron compounds that have low solubility are widely used as wood preservatives and fire retardants in wood-based composite products.

Uysal and Ozçifçi (2000) have obtained laminated wood produced from Uludağ fir for the outer ply, and different veneer materials bonded with PVAc were used for the core ply. The combustion test was applied to the test samples. The highest mass reduction and concentration of O₂ was observed in white mulberry, and the highest heat increase was observed when Scotch pine was used as the core ply

The aim of this paper is to investigate the effect of impregnation with boron compounds BX, BA, BX + BA and DAP on combustion properties of the laminated wood materials produced combination of oak (*Quercus petraea* subsp. Iberica) and poplar (*Populus tremula* L.) veneers, which were used in the middle layer of the lamination forms.

MATERIALS AND METHODS

Wood Species

Oak (*Quercus petraea* subsp. Iberica) and poplar (*Populus tremula* L.) woods were selected as test materials because of their wide usage in industry. Special emphasis was given for the selection of the wood material. Accordingly, non-deficient, proper, knotless, normally grown wood materials (without zone line, without reaction wood and without decay, insect and fungal) was used.

Adhesives

UF, PF, and MUF adhesives were used. The UF, PF, and MUF adhesives were supplied by GENTAS, producer firm in Turkey, and their characteristics are given in Table 1.

Table 1. Characteristics of Adhesives Used

Adhesives	Density (20 °C) (g/cm ³)	pH (20 °C)	Viscosity (20 °C) (cPs)	Solid (2 h, 120 °C) (%)
UF	1.290	8.5	400-600	65.00
PF	1.120	8.4	600	48.00
MUF	1.282	8.2	600	65.00

Impregnation Chemicals

The boron compounds borax (Na₂B₄O₇·5H₂O), boric acid (H₃BO₃), a mixture of BX + BA, and di-ammonium phosphate [(NH₄)₂HPO₄] were used as impregnation chemicals in this study. The chemicals widely available in Turkey and effective against biotic and abiotic damages and have fire-retardant characteristic.

Characteristics of the solutions used in the impregnation process are given in Table 2.

Table 2. Characteristics of Impregnation Chemicals

Impregnation Chemicals	Concentration of solution (%)	Temperature (°C)	Solvent	pH		Density (g/cm ³)	
				B.I.	A.I.	B.I.	A.I.
Borax	5	20	Pure water	9.19	9.21	1.040	1.043
Boric acid	5	20	Pure water	5.20	5.20	1.030	1.035
BX+BA		20	Pure water	7.72	7.74	1.300	1.033
di-ammonium phosphate	5(50:50) 5	20		7.01	7.02	1.070	1.096

B.I.: before impregnation; A.I.: after impregnation

As a result of using fresh solution in every impregnation process, there was no significant change in the acidity and density of the solutions before and after the impregnation.

Impregnation Process

The veneers (520 mm long by 55 mm wide by 3 mm thickness), were cut radially from the heartwood and sapwood region of the planks, because oak has a low sapwood content, to obtain veneer by radially and acclimatized at 20 ± 2 °C and with 65 ± 3 % relative humidity. The veneers were used for making 3-ply laminated veneer lumber. The impregnation process was applied to the veneers before the manufacturing of LVL panels. A vacuum-pressure method was applied to the veneers for impregnation. For that reason, a vacuum chamber, 31 cm diameter and 55 cm depth, was used. Before the impregnation process, the average moisture content of the samples was about 12%. The samples were weighed before impregnation and placed in a vacuum chamber. After the vacuum period (710 mm-Hg, 30 min), the vacuum chamber was filled with the impregnating solution containing BA, BX, BX+BA, or DAP until the samples were covered. Pressure was increased to 3 bar and maintained for 1 h. After the internal pressure was decreased to atmospheric pressure, the impregnation solution was unloaded and the final vacuum, 710 mm-Hg, was applied for 10 min. Then, the samples were removed and re-weighed. The net uptake of impregnation chemicals was calculated from the difference between the last weight and initial weight. The amount of retention (R , kg/m³) was calculated as follows:

$$R = \frac{G \times C}{V} \times 10 \quad (1)$$

where $G = M_2 - M_1$, M_2 = sample mass after impregnation [g], M_1 is sample mass before impregnation [g], V is volume of sample [cm³], and C is concentration of solution [%].

The proportion of impregnation chemicals is given in Table 3.

Table 3. Proportion of Retention (kg/m³)

impregnation chemicals	Retention (kg/m ³)			
	Oak		Poplar	
	\bar{X}	Sd	\bar{X}	Sd
BX	6.63	1.41	22.84	4.35
BA	5.45	0.61	20.04	3.71
BX+BA	6.87	1.62	31.51	3.12
DAP	6.32	0.45	28.07	5.39

\bar{X} : Average , Sd: standard deviation, BA: Boric acid, BX: Borax, BA + BX: Boric acid + Borax, DAP: di-ammonium phosphate

The highest retention proportion was observed from the BA+BX mixture, and the lowest from the BA.

LVL Panel Manufacture

The oversized test samples were climatized until they were stable at 20 ± 2 °C and with 65 ± 3 % relative humidity in a climate-controlled room. Experimental LVL panels were produced from oak and poplar veneers, which were used in the middle layer of the lamination forms. The adhesives were spread at the rate of about 180-200 g/m² on a single bonding surface of the veneers as recommended by the manufacturer. Glues were spread uniformly on the veneers by manually hand brushing. After the gluing process, three veneers were pressed, with the grain directions of all veneers being the same. By considering the general curing temperatures recommended by their manufacturers, the press pressure, temperature, and duration were applied as 7 N/mm², 140 °C, and 12 min for UF and 70 bar, 130 °C, and 6 min for MUF, respectively. Finally the pressed panels were cut with the dimensions of 9x19x1016 mm³ according to the procedure of ASTM E-69. 10 samples were manufactured for each test sample, and 100 test samples were prepared in total.

Determination of Density

The air-dry densities of LVL samples were determined according to EN 323. For gathering the air-dry density, the test samples were kept under the conditions of 20 ± 2 °C and 65 ± 5 % relative humidity until they reached to a constant weight. The weights were measured with an analytical balance of ± 0.01 g sensitivity. Afterwards, the dimensions were measured with a digital compass of ± 0.01 mm sensitivity. The air-dried densities (δ_{12}) of the samples were calculated as follow:

$$\delta_{12} = \frac{W_{12}}{V_{12}} \text{ g/cm}^3 \quad (2)$$

Here, W_{12} is the air-dry weight (g) and V_{12} is the volume (cm³) under air-dry conditions.

Combustion Testing Procedure

The combustion test was carried out according to the principles of the ASTM E – 69 (ASTM-E 69. 1975). But some changes were made in the stand of the combustion. For this purpose, a digital balance with 0.01 g sensitivity was used for determination of the

mass reduction of materials when they are burnt. Butane gas was used to make an ignition flame. According to the gas flow standard where the height of the flame is 25 cm, the temperature must be 1000 °C. The distance, between the bottom of the test samples hung inside the fire tube and the top of the gas pipe must be adjusted as 2.54 cm. During the test, mass reduction, temperature, and released gas (CO, O₂) were determined in every 30 seconds. The test was made under a chimney where the flow of air blown was drawn with natural draft. At the beginning of the combustion test flame source was maintained for 4 minutes, and then the flame source was taken away and it was continued for 6 minutes. The test lasted a total of 10 minutes. The schematic arrangement of the apparatus can be seen in Fig. 1.

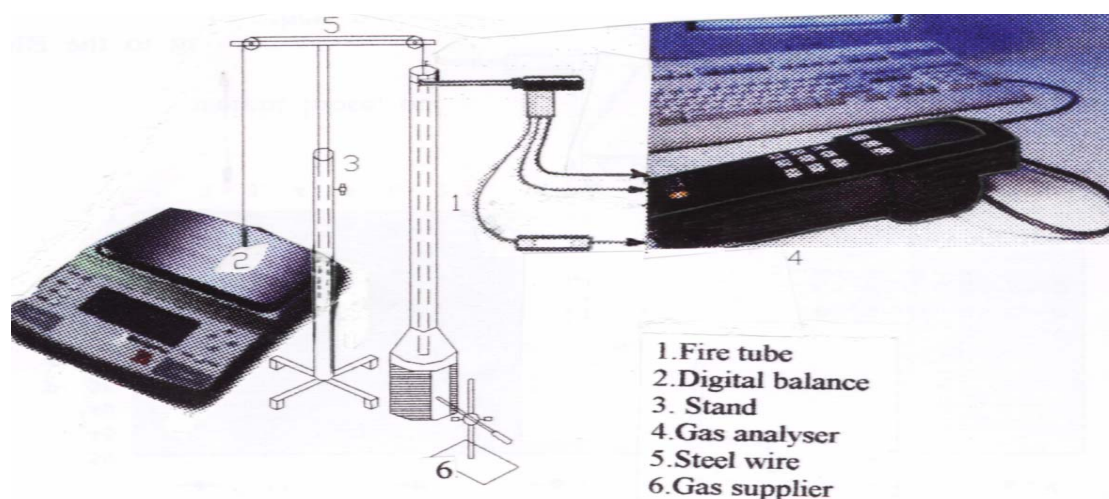


Fig. 1. Schematic arrangement of apparatus

Statistical Procedure

Multi-variance analysis was applied to determine both the amount of retention in the prepared laminated samples and the effects of impregnation material on combustion. By use of the Duncan test, each significant test group was compared with one another and itself.

RESULT AND DISCUSSION

Air Dry Density

The average air-dry densities (δ_{12}) of LVL samples, containing 12 % humidity are given in Table 4.

The highest density was observed in the LVL samples impregnated with BA+BX. According to the control samples, it is possible to say that impregnation chemicals and glue increased the density of LVL.

Table 4. Air Dry Density of Wood Samples (g/cm³)

Samples	δ_{12} (With UF)	δ_{12} (With PF)	δ_{12} (With MUF)
Control	0.595	0.597	0.606
Borax	0.643	0.616	0.651
Boric acid	0.641	0.650	0.676
BA+BX	0.657	0.661	0.638
Di-ammonium phosphate	0.641	0.650	0.677

Combustion Properties

The obtained average values concerning the impregnation chemicals are given in Table 5.

Table 5. Average Combustion Values of Impregnation Chemicals

Adhesive Type	Impregnation chemicals	CO (ppm)	Temperature (°C)	O ₂ (%)	Mass reduction (%)
UF	Control	3378.37 c ^a	280.04 b	14.79 a	93.19 a
	BX	1437.42 a	208.76 a	17.84 c	80.49 c
	BA	3334.42 c	260.69 b	15.97 ab	88.90 b
	BX+BA	2439.08 b	281.17 b	15.60 ab	91.85 a
	DAP	1043.71 a	241.73 ab	16.65 b	86.17 b
PF	Control	4820.07 b	289.69 a	15.46 a	94.24 b
	BX	2558.90 a	273.47 b	16.16 b	84.24 b
	BA	2614.43 a	256.99 ab	16.69 bc	89.69 a
	BX+BA	2391.57 a	250.25 b	16.58 c	90.61 b
	DAP	3031.98 a	259.41 b	16.11 c	86.85 ab
MUF	Control	3188.85 c	267.03 b	15.84 a	91.86 b
	BX	4092.63 d	302.02 c	15.02 a	84.10 b
	BA	2391.90 b	281.57 bc	15.90 a	87.68 b
	BX+BA	3318.73 c	269.56 b	15.33 a	90.83 b
	DAP	699.62 a	204.82 a	17.61 b	64.40 a

BA: Boric acid, BX: Borax, BA + BX: Boric acid + Borax, DAP: Di-ammonium phosphate

^a Groups with same letters in column indicate that there was no statistical difference ($p < 0.001$) between the samples according to the Duncan's multiply range test.

According to Table 5, the impregnation chemicals affected the combustion properties. Boron compounds and DAP showed a decreasing impact on combustion properties of laminated wood materials.

The multi-variance analysis applied on the data obtained from the combustion test is given in Table 6.

Concerning the variance analysis, the effects of adhesive type, impregnation chemical type on mass reduction, released gas (CO, O₂), and temperature were statistically significant. The interaction between the factors was statistically identical ($p \leq 0.05$). The mean values of the variation sources that were found to be significant were compared using Duncan's test, and the results are summarized in Table 5.

Table 6. Multiple Variance Analysis for the Effect of Impregnation Chemical Type and Adhesive Type on the Combustion Properties

Source	Mass Reduction		O ₂		CO		Temperature	
	P- value	Sig.	P- value	Sig.	P- value	Sig.	P- value	Sig.
Corrected Model	4.60	0.000	7.99	0.000	6.66	0.000	19.26	0.000
Intercept	9794.23	0.000	26240.9	0.000	1495.8	0.000	7774.3	0.000
A	3.49	0.036	0.68	0.506	9.70	0.000	1.65	0.193
B	8.75	0.000	5.83	0.000	24.68	0.000	5.89	0.000
A *B	2.80	0.009	5.73	0.000	13.37	0.000	6.45	0.000

Factor A = Adhesive type (UF, PF and MUF), Factor B = Impregnation chemical type (BA , BX, BA + BX, DAP)

The results showed that MUF resin has better fire resistance than UF. This is related to melamine content in the UF resin. Melamine-based flame retardants are commonly used in the forest products industry. When melamine is exposed to heat and flames it decomposes, absorbing heat and creating a cooling effect. The time to ignition is delayed significantly as nitrogen, liberated by melamine during its decomposition, dilutes the oxygen, which in turn inhibits the spread of flames and the generation of smoke. As melamine also acts as a blowing agent, a stable, insulating charred foam layer is formed. One of the effective fire retardant chemicals in wood products is melamine phosphate (Ayrilmis et al. 2005). Melamine phosphate can also be used for plastic industry.

Based on the results presented in the combustion test, the highest reduction of O₂-concentration was measured in UF adhesive LVL, impregnated with BX (14.79 %), and the lowest change of O₂-concentration in the combustion of LVLs with UF adhesive and LVL control samples (17.84 %). If flames were present, fire temperatures were high and more oxygen was available from thermally induced convection. The lower temperatures of the smouldering stage resulted in a lower oxygen supply from diffusion into the fuel bed-gasses in this phase, which leave the fuel bed and are not oxidized further (Lobert and Scharffe 1991).

The highest increase in CO concentration was observed in the experiment of LVL samples with PF adhesive and impregnation unprocessed (4820.07 ppm), and the lowest increase in CO concentration was observed in the experiment of DAP-treated LVL samples with MUF adhesive (699.62 ppm). The reason for the relationship to variations in the CO concentration is the difference in melting behavior of the chemicals used in the present study. The melting point of DAP is lower than borax. The difference in melting behavior leads to the result that the amount of combustible gases which release from surface of the panels treated with DAP differ compared to those of the LVLs treated with borax and boric acid. DAP also provides an earlier charring reaction than borax and boric acid. Previous studies carried out by Myers and Holmes (1975) and Ayrilmis (2005 and 2007) found similar results.

The lowest temperature variation was observed in the experiment with MUF adhesive LVL impregnated with DAP (204.82 °C), and the highest in MUF adhesive LVL impregnated with BX (302.02 °C).

It was reported in the literature that the temperature must be increased up to 275 °C for the wood burning itself. So, it is necessary for wood material to be impregnated with chemical materials in order to increase the resistance of wood against fire (Le Van, and Winandy 1990).

Considering the mass reduction values, MUF adhesive LVL, impregnated with DAP gave the lowest reduction value when compared to control LVL samples (Fig. 3). It was observed that decreases in mass reduction values averaged about 30%. Inorganic materials act as heat sink, lowering the efficiency of combustion. Also, inorganic materials favour the formation of char. Di-ammonium phosphate ranked first in reducing the flame spread, followed by monoammonium phosphate, ammonium chloride, ammonium sulphate, borax, and zinc chloride. Zinc chloride, although excellent as a flame retardant, promoted smoke and glowing (Levan 1984).

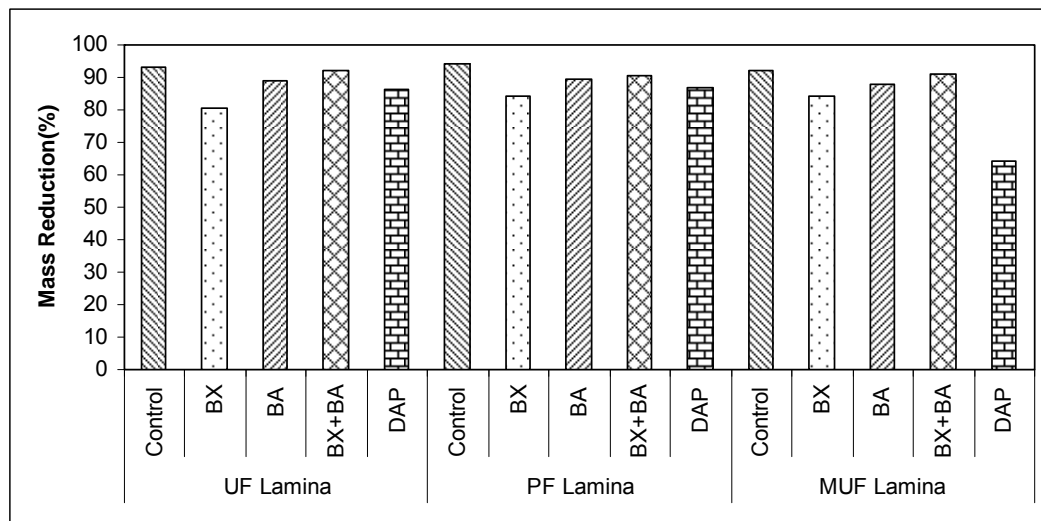


Fig. 3. Mass reduction values

Another result of this study is that boron compounds and DAP showed a decreasing impact on combustion properties of laminated wood materials. DAP showed the best fire resistance as compared to the others. The reason is due to the fact that the FRT chemicals have different chemical composition and mechanism. Phosphate is the most effective in both reducing the amount of volatile products and also reducing the temperature where these products are formed. Ammonium phosphate almost eliminates the glowing exotherm (Rowell 2005). Also, literature informs us that retention levels of at least 48 kg/m³ for borax-boric are need for plywood (pine) and strandboard to obtain a good fire resistance (Levan and Tran 1990, Winandy et al. 2008). The loading levels of the LVLs used in the present study were lower than those of plywood in previous studies, although significant differences were obtained as compared to control LVL.

CONCLUSION

In this study, the effects of fire retardant chemical and resin type on the combustion properties of LVL panels made from oak and poplar veneers were determined. As a result, boron compounds and DAP showed a decreasing impact on combustion properties of laminated wood materials. Considering the adhesive type and impregnation materials, LVL samples bonded with MUF adhesive and impregnated with DAP gave the best results on combustion tests. Consequently, LVL produced from a combination of oak and poplar by using MUF adhesive and impregnated DAP can be recommended to be used as a fire resistant building material, where required.

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