

# Impacts of Varnishes and Impregnation Chemicals on Combustion Properties of Oak (*Quercus petraea* Lipsky)

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Received 14 March 2007; accepted 10 October 2007

DOI 10.1002/app.27548

Published online 10 December 2007 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** This study was performed to determine the effects of varnishing after impregnation with boron compounds on combustion properties of oak. For this aim, the test samples prepared from oak (*Quercus petraea* Liebl.) wood were impregnated according to ASTM D 1413-99 with boric acid (Ba) and borax (Bx) by vacuum technique. After impregnation, surfaces were coated by cellulosic (Cv), synthetic (Sn), polyurethane (Pu), waterborne (Wb), acrylic (Ac), and acid hardening (Ah) varnishes in accordance to ASTM D 3023. Combustion properties of samples after varnishing process were determined, according to ASTM E 160-50. According to material and process types, combustion temperature was the highest in Bx and Cv, the lowest in Ba and Ac. An important difference was not observed between without flame source combustion (WFSC)

and flame source combustion (FSC). According to combustion type, impregnation material and varnish type, combustion temperature was the highest value in WFSC + Bx + acid hardening varnish combination and the lowest in WFSC + Ba + acrylic varnish combination. As a result, the tested varnishes showed an increasing impact but boron compounds showed a decreasing impact on combustion properties of oak. In consequence, for usage areas having a high risk of fire, impregnation of wood material with boron compounds before varnishing will decrease combustion temperature and provide security. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 107: 3981–3986, 2008

**Key words:** combustion; impregnation; European oak; coatings; varnishing

## INTRODUCTION

Wood material burns by itself when its temperature reaches 275°C or with an inflammable material at lower temperatures. Wood materials have better properties than many construction materials, but it is impossible to make it wholly incombustible. It is obvious that, processing wood with some chemical treatments fire resistance and combustion properties. For this purpose, ammonium sulphate, ammonium chloride, borax, boric acid, phosphoric acid etc. are used mostly.<sup>1–3</sup>

Furniture's coated only with paint and varnish has surface protection only for 2 years. So varnishing and painting after the impregnation with materials having appropriate water-repellent is important for long-term utilization against biotic and abiotic effects, photochemical degradation, dimensional changes, biological factors, and fire.<sup>4–6</sup>

Painting and varnishing with water-repellent (Wr) materials after impregnations with boron makes wood more resistant to environmental conditions.<sup>7</sup> Impregnations with the solution of copper chrome and salt make wood material more resistant to envi-

ronmental effects.<sup>8</sup> Because of its combustion and inflammation properties, wood materials must be processed with chemical materials increasing fire resistance.<sup>9</sup>

Keskin declared that, considering the interaction of combustion type and impregnation materials, the lowest values in samples impregnated with Bx and imersol-aqua solutions. Borax in FSC and Imersol-Aqua in WFSC showed a decreasing impact on combustion properties of the Laminated Veneer Lumber (LVL), produced combination of European oak and Lombardy poplar veneers, bonded with Desmodur-VTKA.<sup>10</sup>

This study was performed to determine the effects of coating with various varnishes after impregnation with boric acid (Ba) and borax (Bx) on the combustion properties of oak wood.

## MATERIAL AND METHOD

### Materials

Wood material

European Oak (*Quercus petraea* Liebl.) to be used as test samples were randomly selected from the timber merchants of Ankara, Turkey. Special emphasis is given to the selection of the wood material. Accordingly, nondeficient, proper, knotless, normally grown

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TABLE I  
Some Properties of Varnishes

Type of varnish	pH value	Density (g cm <sup>-3</sup> )	Viscosity (sn DIN Cup/4 mm)	Amount applied (g m <sup>-2</sup> )	Nozzle gap (mm)	Air pressure (bar)
Polyurethane (filler)	5.94	0.98	18	125	1.8	2
Polyurethane (finishing)	4.01	0.99	18	125	1.8	2
Synthetic	–	0.94	18	100	–	–
Wb (primer) <sup>a</sup>	9.17	1.014	18	100	1.3	1
Wb (filler) <sup>b</sup>	9.30	1.015	18	67	1.3	1
Wb (finishing) <sup>c</sup>	8.71	1.031	18	67	1.3	1
Cv (Cv)	2.9	0.955	20	125	1.8	3
Cv (finishing)	3.4	0.99	20	125	1.8	3
Ac (filler)	4.3	0.95	18	125	1.8	2
Ac (finishing)	4.6	0.97	18	125	1.8	2
Ah (finishing)	8.0	0.99	18	100	1.8	3

<sup>a</sup> ASTM D-17.

<sup>b</sup> ASTM D-65.

<sup>c</sup> ASTM D-45.

(without zone line, without reaction wood and without decay, insect mushroom damages) wood materials were selected according to TS 2476.<sup>11</sup>

#### Varnishes

Cellulosic (Cv), synthetic (Sn), polyurethane (Pu), waterborne (Wb), acrylic (Ac), and acid hardening (Ah) varnishes were used according to the producer's definition. Amount of varnish was determined according to the amount of solid material by the producer's definition. Technical specifications of varnishes are given in Table I.<sup>12</sup>

#### Impregnation material

Boron compounds (boric acid and borax) are obtained from Etibank-Bandırma (Turkey) boric and acid Factory. Properties of boric acid (H<sub>3</sub>BO<sub>3</sub>) is 56.30% ½ B<sub>2</sub>O<sub>3</sub>, 43.70% H<sub>2</sub>O with a molecular weight 61.84, Density 1.435 g cm<sup>-3</sup> and melting point 171°C. Borax (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> · 5H<sub>2</sub>O) content is 21.28% Na<sub>2</sub>O, 47.80% B<sub>2</sub>O<sub>3</sub>, 30.92% H<sub>2</sub>O with a molecular weight 291.3, density 1.815 g cm<sup>-3</sup>, melting point 741°C.<sup>13</sup>

#### Method

##### Preparation of test samples

The rough drafts for the preparation of test and control samples were cut from the sapwood parts of massive woods with a dimension of 20 × 20 × 500 mm<sup>3</sup> and conditioned at 20°C ± 2°C temperature and 65% ± 3% relative humidity till they reach 12% humidity distribution. The air-dry samples with a dimension of 13 × 13 × 76 mm<sup>3</sup> were cut from the drafts for impregnation and varnishing. The test samples were impregnated with 5.5% boric acid and 5% borax according to ASTM D 1413-99.<sup>14</sup> Accord-

ingly, the samples were exposed to a 700 mmHg<sup>-1</sup> prevacuum for 60 min and then were held in a solution under normal atmospheric pressure for 60 min to allow the diffusion of the impregnation material. The processes were carried out at 20°C ± 2°C. Retention of impregnation material (*R*) was calculated by the formula,<sup>15</sup>

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

where, *G* is the amount of impregnation solution absorbed by the samples, *T*<sub>2</sub> is the samples weight after the impregnation, *T*<sub>1</sub> is the samples weight before the impregnation, *C* is the concentration (%) of the impregnation solution, and *V* is the volume of the samples. Impregnated test samples were kept under 20°C ± 2°C temperature and 65% ± 3% relative humidity until reaching to a stable weight.

##### Varnishing

Test samples were varnished according to ASTM D 3023.<sup>16</sup> The surfaces of samples were sanded with abrasive papers (silicon carbide, P180C-QB, waterproof, English abrasives, Atlas Brand, made in England) to remove the fiber swellings and dusts were leaned before varnishing. Producer's definition was taken into care for the composition of solvent and hardener ratio and one or two finishing layers were applied after the filling layer. Spray nozzle distance and pressure were 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°C ± 2°C temperature and 65% ± 3% relative humidity conditions. Layer thickness in varnishes were found 93 μm in Cv varnish, 98 μm in Sn varnish, 100 μm in Pu varnish, 81 μm in Wb varnish, 101 μm in Ac varnish, and 99 μm in Ah varnish.

**TABLE II**  
Retention Amount of Impregnation Material (kg m<sup>-3</sup>)

Impregnation material	Retention amount <sup>a</sup>
Ba	2.879 a
Bx	1.779 a

<sup>a</sup> LSD = 2.012.

**Combustion test**

Combustion tests were done in combustion test devices according to ASTM E 160-50 standards.<sup>17</sup> Accordingly, before the combustion test, impregnated samples were conditioned at 27°C temperature and 30% relative humidity in a conditioning room until reaching to 7% relative humidity. Every sample group were weighted before test and stowed on a wire stand. Samples on every stand were put vertically on stand with respect to the below and above ones. Fire distance from the below burner type outlet at the below was fixed to 25 ± 1.3 cm when the device was empty and gas pressure was fixed to 0.5 kg cm<sup>-2</sup> in manometer. When the gas burned, temperature was set at 315°C ± 8°C in the funnel which has a thermocouple. Flame source was centered below sample pile and flame source combustion was continued for 3 min. After extinguishing of flame source, without flame source combustion was carried out. Temperature changes of combustion were determined with a thermometer.

**TABLE III**  
Average Temperature Values of Different Combustion Processes and Materials

	Temperature (°C)
Types of combustion <sup>a</sup>	
Flame source combustion (I)	436.0 a
Without flame source combustion (II)	437.4 a
Impregnation materials <sup>b</sup>	
Control (Co)	473.3 a
Boric acid (Ba)	393.8 c
Borax (Bx)	443.0 b
Varnishes <sup>c</sup>	
Unvarnished (Uv)	460.6 ab
Cellulosic (Cv)	458.5 ab
Synthetic (Sn)	474.6 a
Polyurethane (Pu)	410.9 c
Waterborne (Wb)	440.8 b
Acrylic (Ac)	349.3 d
Acid hardening (Ah)	462.1 a

<sup>a</sup> Different letters in the columns refer to significant changes among types of combustion at 0.05 confidence level (LSD<sub>0.5</sub> = 10.41).

<sup>b</sup> Different letters in the columns refer to significant changes among impregnation materials at 0.05 confidence level (LSD<sub>0.5</sub> = 12.71).

<sup>c</sup> Different letters in the columns refer to significant changes among varnishes at 0.05 confidence level (LSD<sub>0.5</sub> = 19.47).

**TABLE IV**  
Average Values for the Combination of Combination Processes and Materials

Types of material	Temperature (°C)
Types of combustion + impregnation materials <sup>a</sup>	
I	469.7 a
I + Ba	415.3 b
I + Bx	423.1 b
II	476.8 a
II + Ba	372.4 c
II + Bx	462.9 a
Impregnation materials + types of varnish <sup>b</sup>	
Co	506.4 b
Ba	455.7 cdef
Bx	419.8 fgh
Cv	466.3 cde
Ba + Cv	427.2 efg
Bx + Cv	482.1 bc
Sn	586.4 a
Ba + Sn	382.5 l
Bx + Sn	455.0 cdef
Pu	430.5 efg
Ba + Pu	328.8 j
Bx + Pu	473.4 bcd
Wb	492.2 bc
Ba + Wb	429.6 efg
Bx + Wb	400.8 ghi
Ac	389.6 hi
Ba + Ac	378.7 k
Bx + Ac	379.7 i
As	441.5 def
Ba + Ah	454.5 cdef
Bx + Ah	490.3 bc
Types of combustion + types of varnish <sup>c</sup>	
I + Cv	508.1 a
I + Sn	465.4 bcd
I + Pu	442.2 de
I + Wb	470.2 bcd
I + Ac	378.1 g
I + Ah	460.9 c
II + Cv	409.0 f
II + Sn	483.9 abc
II + Pu	379.5 g
II + Wb	411.5 f
II + Ac	420.6 ef
II + Ah	463.3 cd

<sup>a</sup> Different letters in the columns refer to significant changes among combustion and impregnation materials types at 0.05 confidence level (LSD<sub>0.5</sub> = 18.02).

<sup>b</sup> Different letters in the columns refer to significant changes among impregnation materials and varnish types at 0.05 confidence level (LSD<sub>0.5</sub> = 33.72).

<sup>c</sup> Different letters in the columns refer to significant changes among combustion and varnish types at 0.05 confidence level (LSD<sub>0.5</sub> = 27.73), I: flame source combustion, II: Without flame source combustion, Co: unimpregnated samples, Ba: Boric acid, Bx: Borax, Cv: Cellulosic, Sn: synthetic, Pu: polyurethane, Wb: waterborne, Ac: acrylic, Ah: Acid hardening.

**TABLE V**  
**Multiple Variance Analysis for Impact of Combustion Type, Impregnation Material and Varnish Type for Temperature Value**

Source	Degrees of freedom	Sum of squares	Mean square	F value	<i>P</i> < % 5 (Sig)
Factor A <sup>a</sup>	1	57.425	57.425	0.0659	Ns
Factor B <sup>b</sup>	6	206137.382	34356.230	39.4544	0.0000
AB	6	190353.579	31725.596	36.4334	0.0000
Factor C <sup>c</sup>	2	134950.517	67475.259	77.4880	0.0000
AC	2	36465.434	18232.717	20.9383	0.0000
BC	12	170833.443	14236.12	16.3486	0.0000
AB	12	105330.466	8777.539	10.0801	0.0000
Error	84	73145.788	870.783		
Total	125	912274.034			

Ns, Not significant.

<sup>a</sup> Factor A: combustion type (FSC: flame source combustion, WFSC: without flame source combustion).

<sup>b</sup> Factor B: varnish type (Cv: cellulosic, Sn: synthetic, Pu: polyurethane, Wb: waterborne, Ac: acrylic, Ah: acid hardening).

<sup>c</sup> Factor C: Impregnation material (Ba: boric acid, Bx: borax).

### Data analysis

By using one type of wood, two types of combustion, two types of impregnation material and one control samples, six types of varnish and one control samples, a total of 126 samples ( $2 \times 3 \times 7 \times 3$ ) were prepared with three samples for each parameter. Multiple variance analysis was used to determine the effects of impregnation materials and varnishes on FSC and WFSC. Duncan Test was used to determine the significant difference between the groups.

## RESULTS AND DISCUSSION

### Retention quantities

Retention amount of impregnation material is given in Table II. Retention is the highest in boric acid and the lowest in borax. So, types of impregnation material and wood are effective on the amount of retention. Retention amount of impregnation material was found higher in Ba than Bx. The reason for the higher amount of retention with Ba might be high concentration of solution. Average temperature values in combustion according to combustion, impregnation material, and varnish type are given in Table III.

Temperature of combustion was not so different. According to the type of impregnation material, temperature is the highest in Bx, the lowest in Ba. Temperature is the highest in Sn varnish, the lowest in Ac varnish. Accordingly, impregnation materials decreased combustion temperature. Temperature values showed differences in different varnishes.

The combustion temperature according to control samples in varnishing process; 0.5% higher in Cv, 11% higher in Pu, 4% higher in Wb, 24% lower in Ac, 3% higher in Sn and approximately equal for Ah. Accordingly, combustion temperatures are dif-

ferent for varnishes. This case might be due to structural properties of varnishes. Ac varnish showed an important effect for preventing combustion.

Average temperature values according to combustion type and impregnation material, impregnation material and varnish type, combustion type and varnish type combinations are given in Table IV.

Temperature value was measured as the highest in WFSC and Bx, the lowest in WFSC and Ba according to combustion type and impregnation material, the highest in Bx and Ah but the lowest in Ba and Pu according to impregnation material and varnish type combination. Accordingly, Ba showed decreasing effect while Bx resulted with different values for combustion temperature. It was the highest in WFSC and Sn but the lowest in FCS and Ac, FSC and Pu according to combustion temperature and varnish-type combination. Varnishes showed increasing impact for combustion temperature in FSC except Ac and decreasing impact in FSC.

The combustion temperature according to combustion type and varnish type was the highest in FSC and Cv measured as 508.1°C, the lowest in FSC and Ac measured as 378.1°C. For FSC, combustion temperature of unvarnished samples were lower than varnished samples except Ac. Accordingly, varnishes have increasing effect on combustion. Thus, combustion temperature of FSC was higher in Cv, Sn, Pu, Wb, and Ah varnishes at a rate of 16, 8, 3, 9, and 7% orderly and lower in Ac varnish at a rate of 12%. In WFSC, varnishes showed decreasing effect. Combustion temperature of WFSC according to control samples was found lower in (higher in) Cv, Sn, Pu, Wb, Ac, and Ah varnishes at a rate of 17, 2, 23, 16, 15, and 6% orderly.

Results of multiple variance analysis for impact of combustion type, impregnation material, and varnish type for temperature value is given in Table V.

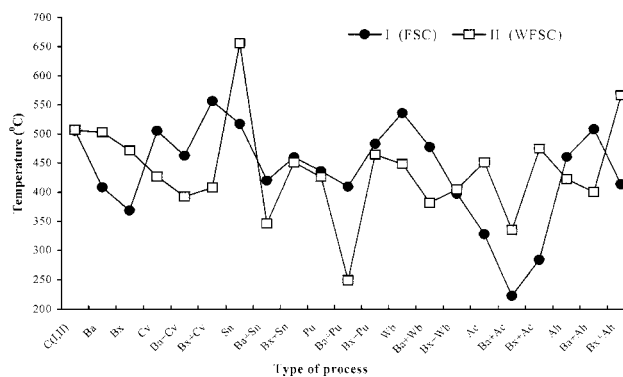
Effects of impregnation material and varnish type (except combustion type) on combustion temperature have been found important for the effect of variance sources ( $\alpha = 0.05$ ) except combustion temperature. The comparisons of the mean values of 42 treatment groups as the result of Duncan's Multiple Range Test are shown in Table VI.

Combustion temperature value was the highest in WFSC and Sn varnish and the lowest in FSC and Ac

**TABLE VI**  
Duncan Test Results

Process type	Temp. (°C) <sup>a</sup>
II + Sn	655.5 a
II + Bx + Ah	566.8 b
I + Bx + Cv	556.4 bc
I + Wb	535.9 bcd
I + Sn	517.3 bcde
I + Ba + Ah	508.4 cdef
II	507.3 cdef
I + Cv	505.5 cdefg
I	505.4 cdefg
II + Ba	503.3 cdefg
I + Bx + Pu	482.6 defg
I + Ba + Wb	477.5 efghi
II + Bx + Ac	475.2 efghi
II + Bx	471.3 efghij
II + Bx + Pu	464.1 efghijk
I + Ba + Cv	462.4 efghijkl
I + Ah	460.6 efghijkl
I + Bx + Sn	459.4 fghijkl
II + Ac	451.5 fghijklm
II + Bx + Sn	450.7 fghijklm
II + Wb	448.5 ghijklmn
I + Pu	435.4 hijklmno
II + Cv	427.0 hijklmno
II + Pu	425.6 hijklmnop
II + Ah	422.4 ijklmnop
I + Ba + Sn	419.5 ijklmnop
I + Bx + Ah	413.7 jklmnop
I + Ba + Pu	408.7 klmnop
I + Ba	408.2 klmnop
II + Bx + Cv	407.9 klmnop
II + Bx + Wb	404.4 lmnop
II + Ba + Ah	400.5 mnop
I + Bx + Wb	397.1 mnopq
II + Ba + Cv	392.0 nopq
II + Ba + Wb	381.6 opqr
I + Bx	368.2 pqr
II + Ba + Sn	345.5 qrs
II + Ba + Ac	335.0 rs
I + Ac	327.7 st
I + Bx + Ac	284.2 tu
II + Ba + Pu	248.9 uv
I + Ba + Ac	222.4 v
II + Bx	471.3 efghij

<sup>a</sup> Different letters in a column refers to significant differences among the different interactions of combustion, varnishes and impregnation materials at 0.05 confidence level (LSD<sub>0.5</sub>: 9.811), I: flame source combustion, II: Without flame source combustion, Ba: Boric acid, Bx: Borax, Sv: Cellulosic, Sn: synthetic, Pu: polyurethane, Wb: waterborne, Ac: acrylic, Ah: acid hardening.



**Figure 1** Combustion temperature changes of oak wood according to type of treatment.

varnish for varnished wood. For samples varnished after impregnation, combustion temperature was the highest in WFSC varnished with Ah varnish after impregnation with borax and the lowest in FSC varnished with Ac varnish after impregnation with Ba (Fig. 1).

**CONCLUSION**

The combustion temperature in impregnation treatment was 17% lower in Ba and 6% lower in Bx than control samples. Accordingly, impregnation materials have decreasing effect on combustion temperature. Indeed, after the treatment with boron compounds, combustion temperature of Uludag fir was decreased.<sup>18</sup>

The combustion temperatures according to impregnation material and varnish type was the highest in Bx and Ah measured as 490.3°C, the lowest in Ba and Pu measured as 369.6°C. The combustion temperatures of varnished samples were higher than impregnated and varnished samples except Bx and Cv, Bx and Pu, Bx and Ah. Accordingly, impregnation materials decrease the increasing properties of varnishes. Impregnation materials showed decreasing effect for combustion temperatures in Cv, Sn, Pu, Wb, Ac, and Ah varnishes as 8, 35, 24, 13, 3, and 3% for Ba and 3, 8, 19, and 10% increasing effect in Cv, Pu, Wb, Ah and 22 and 3% decreasing effect for Sn and Ac was observed for Bx. This might be due to low retention amount of Bx and its low impact on decreasing combustion.

For wood material and impregnation material and varnish interaction, combustion temperature was the highest in WFSC, Bx and Ah measured as 566.8°C and the lowest in FSC, Ba and Ac measured as 222.4°C. As a result, in high risk of fire, impregnation of wood material with boron compounds before varnishing will increase combustion temperature and provide security.

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