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Impact of impregnation with boron compounds on combustion properties of oriental beech (*Fagus orientalis* Lipsky) and varnishes

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This study examined the impacts of varnishing after impregnation with boron compounds on combustion properties of oriental beech. The test samples prepared from oriental beech (*Fagus orientalis* Lipsky) wood were impregnated according to ASTM D 1413-76-99 with boric acid (Ba) or borax (Bx) using a vacuum technique. After impregnation, one of six varnishes (cellulosic (Cv), synthetic (Sn), polyurethane (Pu), water-based (Wb), acrylic (Ac) or acid-hardening (Ah)) was applied in accordance with ASTM D 3023 coated surfaces. After treating, varnishing and conditioning, the combustion properties of samples were determined according to ASTM E 160-50. Combustion temperatures for Bx and Wb were highest when testing without flame source combustion (WFSC), while Ba and Pu exhibited the lowest temperature when testing for flame source combustion (FSC). For the combination of combustion type, impregnation material and varnish type, combustion temperature was the highest for WFSC + boric acid + water-based varnish, but the lowest for WFSC + boric acid + synthetic varnish. As a result, the tested varnishes showed an increasing impact, but boron compounds (Ba and Bx) showed a decreasing impact on the combustion properties of beech. In conclusion, for usage areas having a high risk of fire, impregnation of wood material with boron compounds before varnishing will decrease combustion temperature and provide some degree of security.

Key words: Combustion, flame retardance, coatings, boron compounds, varnish.

INTRODUCTION

Due to the sensitivity of wood materials to fire, architects, construction element designers and furniture and wood producers seek methods of increasing the resistance of wood material against fire. In addition to known

combustion properties, it is important to determine the effects of impregnation and other processes on fire resistance.

Wood and wood-based materials are primarily composed of carbon and hydrogen. For this reason, they are combustible (Chin-Mu and Wang, 1991). When heated, wood burns by producing flammable volatiles that may ignite. For wood to spontaneously combust, the temperature must be raised to 275°C (Levan and Widany, 1990). Two types of "combustion" are often evaluated with woody materials, low temperature charring (non-flaming combustion) and higher temperature flaming combustion. The former usually occurs without an open flame and at temperatures in the 260 to 290°C range, the later occurs at higher temperatures.

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Abbreviations: Ba, Boric acid; Bx, borax; FSC, flame source combustion; WFSC, without flame source combustion; LVL, laminated veneer lumber; PVAc, polyvinyl acetate; PEG, polyethylene glycol; Cv, cellulosic; Sn, synthetic; Pu, polyurethane; Wb, water-based; Ac, acrylic; Ah, acid-hardening.

For wood ignition, oxygen (O₂), a flame source and flammable material are necessary. However, wood actually has excellent natural fire resistance because of its remarkably low thermal conductivity and the fact that, wood char is formed when wood is burned. To reduce flammability and provide additional fire safety, wood is sometimes treated with fire-retardant chemicals. That is, the combustibility of wood can be reduced with the use of flame- or fire-retardants (Goldstein, 1973; Browne, 1963)

Wood materials have better properties than many construction materials, but it is not possible to make them incombustible. It is well recognized that processing wood with some chemical treatments increases fire resistance and combustion properties. For this purpose, ammonium sulphate, ammonium chloride, dicyandiamide, borax, boric acid and various phosphorous compounds (for example, phosphoric acid, monoammonium and diammonium phosphates) have historically been used (Atar et al., 2004).

Large-scale construction and furniture, coated only with paint and varnish, have surface protection for only about 2 years. Thus, varnishing and painting after impregnation is important for long-term resistance against biotic and abiotic effects, photochemical degradation, dimensional changes, biological factors and fire (Evans et al., 1992; Williams et al., 1996). Painting and varnishing with water-repellent chemicals after impregnating with boron compounds makes wood more resistant to in-service environmental degradation (Harrow, 1991). Impregnating with a solution of copper, chrome and salt can make wood more resistant to degradation from environmental effects (Sell and Feist, 1985). For many years, before recent concerns about combustion properties, a common method for protection wood material was to use a 15% solution of paraffine + boric acid (Ba) + borax (Bx) (Feist, 1988). High doses of boron compounds are the preferred method today.

Keskin (2007) stated that, considering the interaction of combustion type and impregnation materials, the lowest thermal degradation values were obtained in samples impregnated with Bx and imersol-aqua solutions, containing 0.5% w/w tebuconazole, 0.5% w/w propiconazole, 0.1% w/w 3-iodo-2-propynyl-butyl carbamate and 0.5% w/w cypermethrin. Borax in flame source combustion (FSC) and imersol-aqua in without flame source combustion (WFSC) showed a decreasing impact on the combustion properties of the laminated veneer lumber (LVL), produced by a combination of European oak (*Quercus petraea* Liebl.) and Lombardy poplar (*Populus nigra* Lipsky) veneers, bonded with Desmodur-VTKA (Desmodur-vinyl triethyl ketonol acetate) (Keskin, 2007).

The effects of impregnation materials, including sodium perborate, sodium tetraborate, imersol-WR 2000 and tanalith-CBC, on the combustion properties of three-ply laminated wood material produced from Uludag fir (*Abies bornmülleriana* Mattf.) were investigated. The highest mass reduction in massive wood samples impregnated

with tanalith-CBC was determined (Uysal and Ozciftci, 2004). The investigation of Kolmann (1960) showed that, the thermal degradation of hardwood species was lower than sapwood species for hardwoods containing more sensitive pentozans (Kolmann, 1960)

Goldstain (1973) found that, the lignin of spruce started to degrade at 130 to 145°C and cellulose at 156 to 170°C. He also found that, when powdered beech was held at 160°C for 28 days, it lost 80% of its cellulose and in 14 days it lost 2 to 3% of its lignin.

Uysal and Ozciftci (2000) tested three layered LVL, produced from polyvinyl acetate (PVAc) adhesive and lime tree, consisting of different core ply, according to the procedure of ASTM E 69 combination standards. They found the highest amount of ash and unburnt wood were obtained in LVL consisting of lime tree.

Yalinkilic and Ors (1996) studied impregnation of Uludag fir (*A. bornmülleriana* Mattf.) wood with boron compounds and polyethylene glycol (PEG)-400. They tested those samples using standard laboratory combustion tests. PEG-400-treatment had little influence on combustion properties, while the boron compounds were more effective.

In this study, we determined the influence of coating with various varnishes after impregnation with two common fire-resistant boron compounds (boric acid and borax) on the FSC and WFSC properties of oriental beech.

MATERIALS AND METHODS

Wood material

Oriental beech (*Fagus orientalis* Lipsky) wood was selected as a test material because of its wide use in industry. Special attention was given to the selection of wood materials, which were straight-grained, free of knots and exhibited no reaction wood, decay or insect damage as specified in TS 2470 (1976).

Varnishes

Cellulosic (Cv), synthetic (Sn), polyurethane (Pu), water-based (Wb), acrylic (Ac), and acid-hardening (Ah) varnishes were used. Application procedures followed the manufacturer's recommendations. The amount of varnish applied was determined on a solids basis, as defined by the manufacturer's label. Technical specifications of varnishes are given in Table 1 (Dewilux et al., 1996).

Impregnation materials

Boron compounds (boric acid and borax) were obtained from Etibank-Bandırma (Turkey) boric acid factory. The properties of boric acid (H₃BO₃) are 56.30% ½ B₂O₃, 43.70% H₂O, with a molecular weight of 61.84 g/mol, a density of 1.435 gcm⁻³ and melting point of 171°C. Borax (Na₂B₄O₇·5 H₂O) content was 21.28% Na₂O, 47.80% B₂O₃ and 30.92% H₂O, with a molecular weight of 291.3 g/mol, a density of 1.815 gcm⁻³ and a melting point of 741°C (Ors et al., 2006).

Table 1. Some properties of varnishes.

Type of varnish	pH value	Density (g cm ⁻³)	Viscosity (DIN Cup/4mm)	Amount applied (g m ⁻²)	Nozzle gap (mm)	Air pressure (bar)
Polyurethane (filling)	5.94	0.98	18	125	1.8	2
Polyurethane (last)	4.01	0.99	18	125	1.8	2
Synthetic	-	0.94	18	100	-	-
Wb (primer)*	9.17	1.014	18	100	1.3	1
Wb (filler)**	9.30	1.015	18	67	1.3	1
Wb (finishing)***	8.71	1.031	18	67	1.3	1
Cv (filler)	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

ASTM D 17*, ASTM D 65**, ASTM D 45***.

Preparation of test samples

The rough-cut beech drafts used for the preparation of test and control samples were cut from the sapwood parts of beech lumber with dimensions of 20 × 20 × 500 mm and conditioned at 20±2°C and 65±3% relative humidity until they maintained a constant weight. The final test samples, with a dimension of 13 × 13 × 76 mm, were cut from these drafts before impregnation and varnishing. The test samples were impregnated with 5.5% boric acid or 5% borax, according to ASTM D 1413-76-99 (2005). Accordingly, the samples were exposed to a 700 mmHg prevacuum for 60 min and then held in either a Ba or a Bx solution at normal atmospheric pressure for 60 min to allow the treating solutions to diffuse into the impregnated materials. The processes were carried out at 20±2°C. Retention of impregnation material (R) was calculated by the formula:

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

Where, G is the amount of impregnation solution absorbed by the samples; T₂ is the sample weight after impregnation; T₁ is the sample weight before impregnation; C is the concentration (%) of the impregnation solution; V is the volume of the sample.

Impregnated test samples were kept at 20±2°C and 65±3% relative humidity until they reached constant weight (moisture equilibrium). Test samples were varnished according to ASTM D 3023 (1998). The surfaces of the samples were sanded with abrasive papers (silicon carbide, P180C-QB, waterproof, English abrasives, Atlas Brand, England) to remove the fiber swellings and dust was removed before varnishing. The manufacturer's recommendations were followed for the composition of solvent, hardener ratio and whether one or two finishing layers were applied after sanding. Spray nozzle distance and pressure were adjusted according to the manufacturer's instructions and moved in parallel to the specimen's longitudinal surface at a distance of 20 cm. Varnishing was done at 20±2°C and 65±3% relative humidity. Layer thicknesses of varnishes were determined to be 98 µm in cellulosic varnish, 99 µm in synthetic varnish, 102 µm in polyurethane

varnish, 88 µm in water-based varnish, 103 µm in acrylic varnish and 100 µm in acid-hardening varnish.

Combustion test

Combustion tests were done in combustion test devices according to ASTM E 160-50 (1975) standards. Accordingly, before the combustion test, impregnated samples were conditioned at 27°C and 30% relative humidity in a conditioning room until reaching 7% relative humidity. Every sample group was weighed before the test and stored on a wire stand. Samples on every stand were placed vertically on the stand with respect to the samples below and above. Distance between samples and the fire-flame outlet was fixed at 25±1.3 cm when the device was empty and gas pressure was fixed at 0.5 kg cm⁻² in the manometer. When ignited, the test temperature was set at 315±8°C in the funnel, using a calibrated thermocouple. The flame source was centered below the sample pile and the flame source combustion was continued for three minutes. After extinguishing the flame source, subsequent evaluation of flame spread (charring) without open-flame source combustion was carried out. Temperatures during combustion (°C) were determined with a thermometer (Figure 1).

Statistical analyses

Using one type of wood, two types of combustion, two types of impregnation material and one control sample, six types of varnish and one control sample, a total of 126 samples (2 × 3 × 7 × 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. The Duncan test was used to determine the significance of differences between groups.

RESULTS AND DISCUSSION

Retention and varnish levels

The retention amount of the impregnation material is

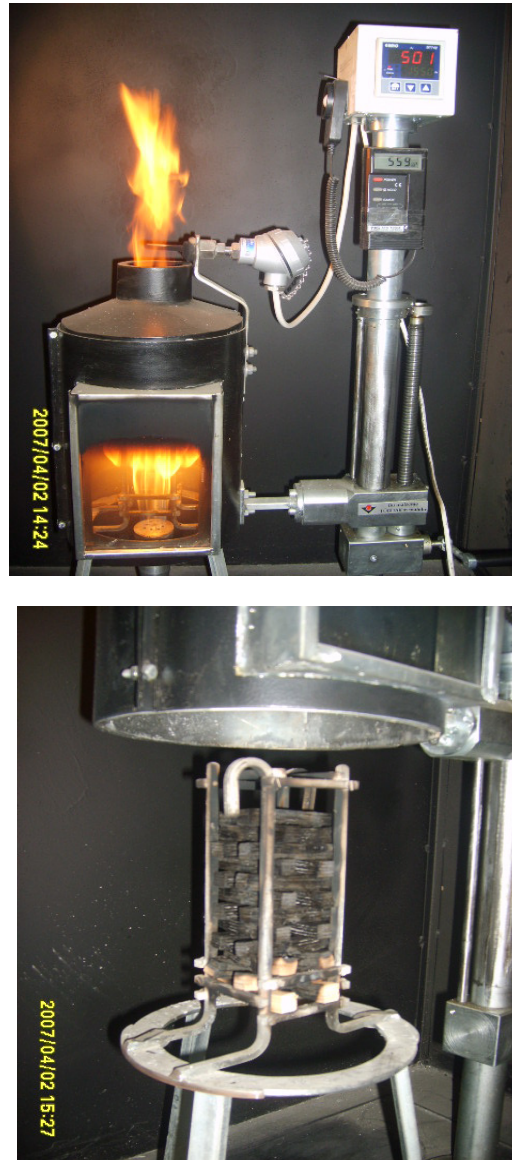


Figure 1. Combustion tests (ASTM E 160-50).

given in Table 2. The retained amount of the impregnation material was found to be higher with boric acid than borax. A reason for the higher amount of retention with boric acid might be the high concentration of the solution.

Combustion temperature

Average temperature values for each combustion type varied according to impregnation material and varnish types and are given in Table 3. The relative WFSC-combustion temperatures were determined as the highest temperature measured without flame source combustion. WFSC temperatures were highest for borax treatment, followed by water-based varnish and the lowest in boric

acid treatment with polyurethane varnish. The combustion temperature in the impregnation treatment was 16% lower in boric acid samples and 11% lower in borax samples, compared with control specimen. Accordingly, impregnation materials can decrease the combustion temperature. 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 desmodur-VTKA (Desmodur-vinyl triethyl ketone acetate) (Keskin et al., 2009). The combustion temperature, according to control specimen in the varnishing process were 5% higher in Cv, 12% higher in Wb, 3% lower in Pu and approximately equal for Ac, Ah and Sn. Temperature values showed differences, depending on the varnish.

Average temperature values according to combustion

Table 2. Retention amount of impregnation materials (kg m^{-3}).

Impregnation material	Retention ^a
Boric acid (Ba)	40.99 ^A
Borax (Bx)	20.75 ^B

^aLeast significant difference (LSD) = 2.012.

Table 3. Average temperature values of different combustion processes and materials.

Types of combustion*	Temperature ($^{\circ}\text{C}$) ^a
Flame source combustion (I)	449.5 ^B
Without flame source combustion (II)	456.6 ^A
Impregnation materials**	
Control (Co)	497.6 ^A
Boric acid (Ba)	419.4 ^C
Borax (Bx)	442.1 ^B
Varnishes***	
Unvarnished (Uv)	443.4 ^C
Cellulosic (Cv)	464.7 ^B
Synthetic (Sn)	441.7 ^C
Polyurethane (Pu)	429.5 ^D
Water-borne (Wb)	502.1 ^A
Acrylic (Ac)	444.4 ^C
Acid hardening (Ah)	445.4 ^C
Impregnation materials**	
Control (Co)	497.6 ^A
Boric acid (Ba)	419.4 ^C
Borax (Bx)	442.1 ^B

*Different letters in the columns refer to significant changes among types of combustion at 0.05 confidence level ($\text{LSD}_{0.5} = 2.141$); **different letters in the columns refer to significant changes among impregnation materials at 0.05 confidence level ($\text{LSD}_{0.5} = 2.622$); ***different letters in the columns refer to significant changes among varnishes at 0.05 confidence level ($\text{LSD}_{0.5} = 4.05$). I: Flame source combustion; II: without flame source combustion; Co: unimpregnated samples; Ba: boric acid; Bx: borax, Uv: unvarnished; Cv: cellulosic; Sn: synthetic; Pu: polyurethane; Wb: waterborne; Ac: acrylic; Ah: acid hardening.

type + impregnation material, impregnation material + varnish type and combustion type + varnish type combinations are shown in Table 4. According to combustion temperature and impregnation material combination, combustion temperature was highest in the FSC (I) + Bx combination (443.1°C) and lowest in the WFSC (II) + Ba (413.2°C). Impregnation materials showed a decreasing effect on the combustion temperature values of FSC and WFSC at a rate of 11 and 20% in Ba and 8 and 15% in Bx, respectively.

The combustion temperatures based on impregnation material and varnish types were highest in Bx + Cv (523.30°C) and lowest in Ba + Sn (369.6°C). The combustion temperatures of varnished samples without impregnation were higher than impregnated and varnished samples, except Bx + Cv. Accordingly, impregnation materials decreased the increasing properties of varnishes.

Impregnation materials showed a decreasing effect for combustion temperatures in Sn, Pu, Wb, Ac and Ah varnishes, measured at 29, 1, 8, 22 and 28% for Ba and 15, 8, 9, 14 and 12% for Bx, respectively. With the Cv varnish, a 14% decrease for Ba and a 10% increase for Bx was observed.

Results of multiple variance analysis for the impact of combustion type, impregnation material and varnish type on combustion temperature value are given in Table 5. Effects of combustion type, impregnation material and varnish type on combustion temperature have been found to be important for the effect of variance sources ($\alpha = 0.05$). Duncan test results, given in Table 6, indicated the differences between the groups.

The combustion temperature value was the highest in the II + synthetic varnish combination but the lowest in the II + polyurethane varnish for varnished wood material

Table 4. Average temperature values for the combination of combustion process and materials.

Type of material	Temperature (°C) ^a
Types of combustion + impregnation materials*	
I	479.75 ^B
I+Ba	425.59 ^D
I+Bx	443.10 ^C
II	515.518 ^A
II+Ba	413.248 ^E
II+Bx	441.043 ^C
Impregnation materials + types of varnishes**	
Co	514.67 ^{BC}
Ba	445.45 ^{FG}
Bx	370.150 ^L
Cv	468.34 ^E
Ba+Cv	402.59 ^J
Bx+Cv	523.30 ^A
Sn	517.90 ^{ABC}
Ba+Sn	369.60 ^L
Bx+Sn	437.73 ^{HI}
Pu	441.84 ^{GH}
Ba+Pu	440.20 ^{GH}
Bx+Pu	406.56 ^J
Wb	521.11 ^{AB}
Ba+Wb	511.65 ^{CD}
Bx+Wb	473.504 ^E
Ac	505.75 ^D
Ba+Ac	394.66 ^K
Bx+Ac	432.72 ^J
Ah	513.82 ^{BC}
Ba+Ah	371.81 ^L
Bx+Ah	450.43 ^F
Types of combustion + types of varnishes***	
I	409.72 ^H
I+Cv	488.53 ^B
I+Sn	463.33 ^D
I+Pu	431.76 ^F
I+Wb	439.36 ^E
I+Ac	482.53 ^C
I+Ah	431.10 ^F
II	477.15 ^C
II+Cv	440.95 ^E
II+Sn	420.15 ^G
II+Pu	427.27 ^F
II+Wb	564.84 ^A
II+Ac	406.22 ^H
II+Ah	459.64 ^D

*Different letters in the columns refer to significant changes among combustion and impregnation materials types at 0.05 confidence level (LSD_{0.5}= 3.708); **different letters in the columns refer to significant changes among impregnation materials and varnish types at 0.05 confidence level (LSD_{0.5}= 6.937); ***different letters in the columns refer to significant changes among combustion and varnish types at 0.05 confidence level (LSD_{0.5}= 5.664).

Table 5. Multiple variance analysis for impact of combustion type, impregnation material and varnish types on combustion temperature.

Source	Degrees of freedom	Sum of squares	Mean square	F value	P < % 5 (Sig)
Factor A ^a	1	1601.58	1601.583	43.4488	0.0600
Factor B ^b	6	62124.106	10354.018	280.8908	0.0000
AB	6	138254.823	23042.470	625.1118	0.0000
Factor C ^c	2	136054.340	68027.170	1845.4872	0.0000
AC	2	13476.898	6738.449	182.8052	0.0000
BC	12	147944.074	12328.673	334.4606	0.0000
ABC	12	202135.435	16844.620	456.9723	0.0000
Error	84	3096.354	36.861		
Total	125	704687.614			

a,Factor A: combustion type (FSC: flame source combustion, WFSC: without flame source combustion); b: factor B: varnish type (Cv: cellulosic, Sn: synthetic, Pu: polyurethane, Wb: waterborne, Ac: acrylic, Ah: acid hardening); c: factor C: Impregnation materials (Ba: boric acid, Bx: borax).

Table 6. Duncan test results.

Process type	Temp. (°C) ^a	Process type	Temp. (°C) ^a	Process type	Temp. (°C) ^a
II+Ba+Wb	595.8 ^A	I	494.2 ^{FG}	I+Bx+Ah	414.1 ^M
II+Sn	573.5 ^B	II+Ba	493.8 ^{FG}	I+Bx+ Wb	412.1 ^{MN}
II+ Wb	563.7 ^B	I+Ac	492.3 ^G	II+Bx	402.5 ^{NO}
II+Bx+Cv	543.1 ^C	II+Ba+Cv	491.6 ^G	I+Ba	397.2 ^{OP}
II+Ah	540.3 ^C	II+Cv	488.2 ^{GH}	I+Ba+Ah	391.8 ^{PQ}
II	535.2 ^C	I+Ah	487.3 ^{GH}	II+Pu	388.5 ^{PQ}
II+Bx+ Wb	535 ^C	II+Bx+Ah	486.8 ^{GH}	II+Bx+Pu	383.2 ^Q
II+Ac	519.2 ^D	I+ Wb	478.5 ^H	II+Bx+Ac	370.7 ^R
I+Ba+Cv	513.5 ^{DE}	I+Sn	462.3 ^I	I+Ba+Pu	370.2 ^R
II+Ba+Pu	510.1 ^{DE}	I+Ba+Ac	460.6 ^I	II+Bx+Sn	366.1 ^R
II+Bx+Sn	509.4 ^{DE}	I+Cv	448.5 ^J	II+Ba+Ah	351.8 ^S
I+Bx+Cv	503.5 ^{EF}	I+Bx+Pu	429.9 ^K	I+Bx	337.8 ^T
I+Pu	495.1 ^{FG}	I+Ba+ Wb	427.5 ^{KL}	II+Ba+Ac	328.7 ^{TU}
I+Bx+Ac	494.7 ^{FG}	I+Ba+Sn	418.3 ^{LM}	II+Ba+Sn	320.9 ^U

a,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_{0.5}: 9.811); I: flame source combustion; II: Without flame source combustion; Ba: boric acid; Bx: borax; Sv: cellulosic; Sn: synthetic; Pu: polyurethane; Wb: water-borne; Ac: acrylic; Ah: acid hardening.

without impregnation. For samples varnished after impregnation, the temperature was the highest in WFSC varnished with water-based varnish after impregnation with boric acid, but lowest in WFSC varnished with synthetic varnish after impregnation with boric acid (Figure 2).

Conclusions

The application of certain varnishes effectively enhanced

fire resistance of two types of borate-treated beech LVL. Combustion temperatures were highest when tested without flame source combustion (WFSC) for boric acid treatments using a water-based varnish (595.9°C). Combustion temperatures were lowest when tested with flame source combustion (FSC) for boric acid treatments with a synthetic varnish (320.9°C). As a result, it appears that impregnation of wood material with appropriate boron treatment, followed by subsequent varnishing, will further increase combustion temperatures and provide additional fire resistance and a degree of security.

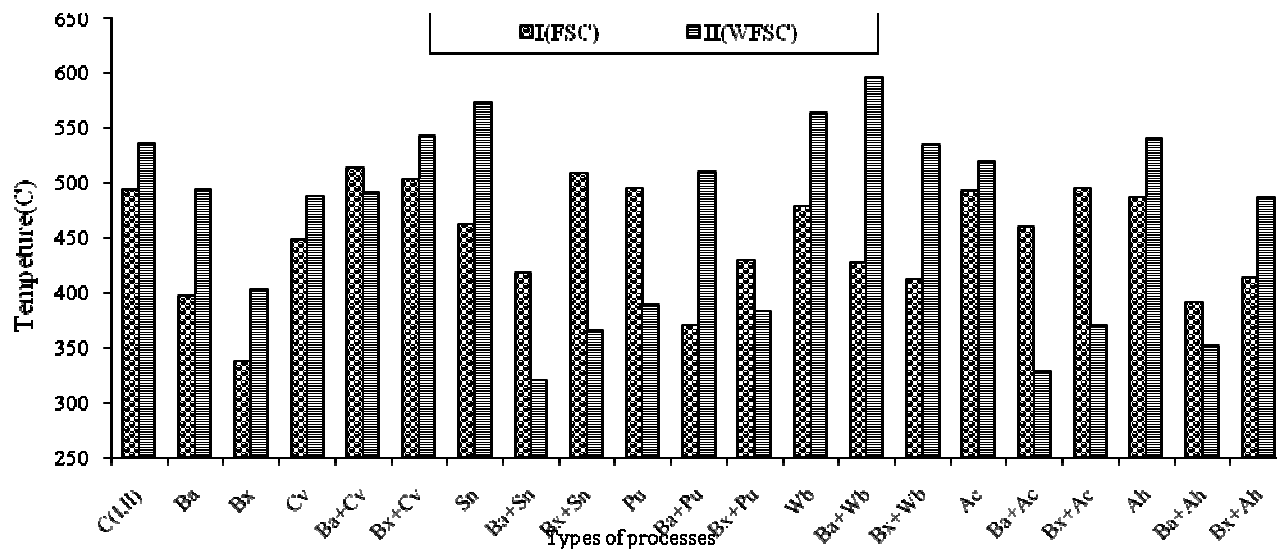


Figure 2. Combustion temperature changes of beech according to the type of treatment.

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