Impacts of Coating with Various Varnishes After Impregnation with Boron Compounds on the Combustion Properties of Uludag Fir

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ABSTRACT: This study was performed to determine the effects of varnishing after impregnation with boron compounds on combustion properties of Uludag Fir. For this purpose, the test samples prepared from Uludag fir (*Abies Bornmülleriana* Mattf.) 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 type, combustion temperature was highest in without flame source combustion (WFSC), Bx, and Wb but the lowest in flame source combustion (FSC), Ba, and Pu.

INTRODUCTION

Wood and wood-based materials are mainly composed of carbon and hydrogen. For this reason, they are combustible.¹ When heated, wood burns by producing flammable volatiles that may ignite. For wood to spontaneously combust, the temperature must be raised to 275°C. However, if there is a flame source, it can become flammable at lower temperature.² For wood ignition, oxygen, O₂ flame source and flammable material are necessary. However, wood has excellent natural fire resistance as a result of its remarkably low thermal conductivity and the fact that wood char is formed when wood is burned. To reduce flammability and provide safety, wood is treated with fire-retardant chemicals. In other words, the combustibility of wood may be reduced with flame-retardants or fire-retardants.^{3,4}

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For the combination of combustion type, impregnation material and varnish type, combustion temperature was the highest in WFSC + Ba + Wb combination but the lowest in WFSC + Ba + Cv combination. Consequently, the tested varnishes showed an increasing impact but boron compounds (Ba and Bx) showed a decreasing impact on combustion properties of Uludag fir wood. 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 106: 4018–4023, 2007

Key words: coatings; flame retardance; compounding; structure

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 increase fire resistance and combustion properties. For this purpose, ammonium sulfate, ammonium chloride, borax, boric acid, phosphoric acid, etc., are used mostly.⁵

Massive constructions and furniture, coated only with paint and varnish, have surface protection only for 2 years. So, varnishing and painting after the impregnation is important for long-term utilization against biotic and abiotic effects photochemical degradation, dimensional changes, biological factors, and fire.⁶

Painting and varnishing with water-repellent chemicals after impregnating with boron compounds makes the wood more resistant to environmental conditions.⁷ Impregnating with the solution of copper, chrome and salt makes wood more resistant to environmental effects.⁸ For combustion properties, the most suitable impregnated wood material is 15% solution of paraphine + boric acid + borax.⁹

It was assessed that the effects of impregnation materials, sodium perborate, sodium tetraborate, Imersol-WR 2000, and Tanalith-CBC, on combustion

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properties of three-ply laminated wood material produced from Uludag fir (*Abies bornmülleriana* M.) were investigated. As a result, the highest mass reduction in massive wood samples impregnated with Tanalith-CBC was determined.¹⁰

In another research, it was carried out to determine the bonding strength of phenol–formaldehyde and melamine–formaldehyde adhesives to impregnated wood materials. For this purpose, pine and elm woods were impregnated with boron compounds, diammonium phosphate, and Tanalith-C 3310 using the vacuum method according to ASTM D 1413. The effects of wood species, impregnating material and type of adhesive on the bonding strength were determined. The highest shear strength (11.09 N mm⁻²) was obtained from elm wood control (i.e., without any impregnating materials) samples with melamine–formaldehyde; thus, the impregnation process negatively affected the adhesive bonding strength.¹¹

The investigation of Kolmann yielded pertinent information on the thermal degradation of the hardwood species is lower than sapwood species for hardwood contains more sensitive pentozans.¹²

Goldstain declared that the lignin of spruce started degradation at $130-145^{\circ}$ C and its cellulose at $156-170^{\circ}$ C. When the dust of beech wood was held at 160° C for 28 days, it lost its cellulose as 80% and within 14 days it lost its lignin as 2-3%.¹³

Uysal and Ozcifci¹⁴ carried out 3 layered laminated veneer lumber (LVL), produced from PVAc adhesive and lime-tree and consisting of different core ply and tested according to the procedure of ASTM E 69 combustion standards. The highest amount of ash and unburned pieces were obtained in LVL consisting of lime-tree.

Yalınkılıc and Ors¹⁵ studied impregnation with boron compounds and the groups of the PEG-400 of Douglas (*Pseudotsuga menziesii* Franco) wood, the test samples were applied to the combustion tests. Although the groups of the PEG-400 had a negative impact on combustion however, boron compounds were shown more effective results.

This study was performed to determine the impacts of coating with various varnishes after impregnation with boron compounds (boric acid and borax) on the combustion properties of Uludag fir wood.

MATERIALS AND METHODS

Materials

Wood material

Uludag fir (Abies Bornmülleriana Mattf.) wood was selected as test the materials because of wide usage

of industry. Special emphasis was given for the selection of wood materials which are nondeficient, proper, knotless, normally grown (without reaction wood, decay and mushroom damages) according to TS 2476.¹⁶

Impregnation materials

Boron compounds (boric acid and borax) are obtained from Etibank-Bandırma (Turkey) Borax and Acid Factory. Properties of boric acid (H₃BO₃) is 56.30%, 1/2 B₂O₃ 43.70%, H₂O with a molecular weight 61.84, density 1.435 g cm⁻³, and melting point 171°C. Borax (Na₂B₄O₇5H₂O) content is 21.28%, Na₂O 47.80%, B₂O₃ 30.92%, H₂O with a molecular weight 291.3, density 1.82 g cm⁻³, and melting point 741°C.¹⁷

Varnishes

Cellulosic (Cv), synthetic (Sn), polyurethane (Pu), waterborne (Wb), acrylic (Ac), and acid hardening (Ar) varnishes were used according to the producers definition. Amount of varnish was determined according to the amount of solid material by the producer's definition. Technical specifications¹⁸ of varnishes are given in Table I.

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 mm \times 20 mm \times 500 mm and conditioned at a temperature of 20°C \pm 2°C and 65% \pm 3% relative humidity till they reached 12% humidity distribution. The airdry samples with a dimension of 13 mm \times 13 mm \times 76 mm were cut from the drafts for impregnation and varnishing. The test samples were impregnated with 5.5% Ba and 5% Bx according to ASTM D 1413-99.¹⁹

Accordingly, the samples were exposed to a 700 mmHg 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 $20^{\circ}C \pm 2^{\circ}C$. Retention of impregnation material (*R*) was calculated by the formula;

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

where *G* is the amount of impregnation solution absorbed by the sample, T_2 is the sample weight after the impregnation, T_1 is the sample weight

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Type of varnish	pH value	Density (g cm ⁻³)	Viscosity (snDINCup/4 mm)	Amount applied (g m ⁻²)	Nozzle gap (mm)	Air pressure (bar)
Pu (filler)	5.94	0.98	18	125	1.8	2
Pu (finishing)	4.01	0.99	18	125	1.8	2
Sn	_	0.94	18	100	-	_
Wb (primer) ^a	9.17	1.014	18	100	1.3	1
Wb (filler) ^b	9.30	1.015	18	67	1.3	1
Wb (finishing) ^c	8.71	1.031	18	67	1.3	1
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

TABLE I Technical Properties of Some Varnishes

^a ASTM D 17.

^b ASTM D 65.

^c ASTM D 45.

before the impregnation, *C* the concentration (%) of the impregnation solution, and *V* the volume of the samples. Impregnated test samples were kept under a temperature of 20° C ± 2° C and 65% ± 3% humidity content until they reach to a stable weight.

The test samples were varnished according to ASTM D 3023.²⁰ The surfaces of samples were sanded with abrasive paper (Silicon carbide, P180C-QB, waterproof, English Abrasives, Atlas Brand, made in England) to remove the fiber swellings and dusts were cleaned 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 noz-zle distance and pressure were adjusted according to the producer's definition and moved in parallel to the samples surface at a distance of 20 cm. Varnishing was done under $20^{\circ}C \pm 2^{\circ}C$ temperature and $65\% \pm 3\%$ relative humidity conditions.

Layer thickness in varnishes were found 98 μ m in Cv varnish, 99 μ m in Sn varnish, 102 μ m in Pu varnish, 88 μ m in Wb varnish, 103 μ m in Ac varnish, and 100 μ m in Ah varnish.

Combustion test

Combustion tests were done in combustion test devices according to ASTM E 160-50.²¹ Accordingly, before 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 weighted before the test and stowed on a wire stand. Samples on every stand were put vertically on the stand with respect to the below and above ones. Fire distance from maker type outlet below was fixed to 25 ± 1.3 cm when the device was empty and gas pressure was fixed to 0.5 kg cm⁻² in manometer. When the gas burned, temperature was set at $315^{\circ}C \pm 8^{\circ}C$ in the funnel which has a thermocouple. Flame source was centered below sample pile and FSC was continued for 3 min. After extinguishing of flame source, WFSC was carried out. Temperature changes of combustion (°C) were determined with a thermometer.

Statistical analyses

By using one type of wood, two types of combustion, two types of impregnation material and one control samples, six types of varnish + 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 impacts of impregnation materials and varnishes on WSFC and FSC. Duncan test was applied to determine the significant difference between the groups.

RESULTS AND DISCUSSION

Retention quantities

Retention quantities of impregnation materials are given in Table II.

TABLE II Retention Quantities of Impregnation Materials (kg m $^{-3}$)

Retention quantities ^a
13.00 A
12.72 AB

 a LSD = 2.012.

TABLE III Average Temperature Values of Different Combustion Processes and Materials

	Temperature (°C) ^a
Types of combustion ^b	
Flame source combustion (I)	475.7 A
Without flame source combustion (II)	411.4 B
Impregnation materials ^c	
Control (Co)	497.7 A
Boric acid (Ba)	386.8 C
Borax (Bx)	446.1 B
Varnishes ^d	
Unvarnished (Uv)	476.9 A
Cellulosic (Cv)	415.0 C
Polyurethane (Pu)	378.3 D
Synthetic (Sn)	450.8 B
Acrylic (Ac)	456.1 B
Water based (Wb)	480.1 A
Acid hardening (Ah)	447.5 B

Co, unimpregnated samples.

^a Different letters in a column refers to significant differences among types of processes and materials at 0.05 confidence level.

^b LSD_{0.5}: 7.464.

^c LSD_{0.5}: 9.141.

^d LSD_{0.5}: 13.96.

Retention quantities are found highest in boric acid and lowest in borax. So, type of impregnation material is effective on the amount of retention.

Combustion temperature

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

Temperature of combustion is measured as the highest in without flame source combustion (FSC), borax, and waterborne varnish and the lowest in boric acid and polyurethane varnish. Accordingly, impregnation materials decreased combustion temperature. Temperature values showed differences in varnishes. Average temperature values according to combustion type + impregnation material, impregnation material + varnish type, and combustion type + varnish type combinations are given in Table IV.

According to combustion type + impregnation material combination, temperature value was measured as the highest in FSC + boric acid but the lowest in without flame source combustion (WFSC) + boric acid. According to impregnation material + varnish type combination, temperature value was the highest in Ba+Cv but the lowest in Ba + Ah. Accordingly, impregnation materials showed a decreasing impact for combustion temperature. It was the highest in FSC + Ac but the lowest in FSC

+ Wb combination according to combustion temperature and varnish type. Combustion temperature was the highest in Wb, the lowest in Sn for WFSC. Varnishes showed an increasing impact for combus-

TABLE IV Temperature Average Values for the Combination of Combustion Process and Materials

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$\begin{array}{llllllllllllllllllllllllllllllllllll$	Ba+Ah	386.3 H			
I 479.8 B I+Cv 453.4 CDE I+Pu 478.0 B I+Sn 451.1 DE I+Ac 518.5 A I+Wb 446.7 DE I+Ah 502.2 A II 474.1 BC II+Cv 376.5 G II+Pu 423.6 F II+Sn 305.5 H II+Ac 441.7 EF II+Wb 465.5 BCD	Bx+Ah	460.1 CD			
$\begin{array}{cccc} I+Cv & 453.4 \ \text{CDE} \\ I+Pu & 478.0 \ \text{B} \\ I+Sn & 451.1 \ \text{DE} \\ I+Ac & 518.5 \ \text{A} \\ I+Wb & 446.7 \ \text{DE} \\ I+Ah & 502.2 \ \text{A} \\ II & 474.1 \ \text{BC} \\ II+Cv & 376.5 \ \text{G} \\ II+Pu & 423.6 \ \text{F} \\ II+Sn & 305.5 \ \text{H} \\ II+Ac & 441.7 \ \text{EF} \\ II+Wb & 465.5 \ \text{BCD} \\ \end{array}$	Types of combustion + types of varnish	es ^d			
$\begin{array}{cccc} I+Pu & 478.0 \text{ B} \\ I+Sn & 451.1 \text{ DE} \\ I+Ac & 518.5 \text{ A} \\ I+Wb & 446.7 \text{ DE} \\ I+Ah & 502.2 \text{ A} \\ II & 474.1 \text{ BC} \\ II+Cv & 376.5 \text{ G} \\ II+Pu & 423.6 \text{ F} \\ II+Sn & 305.5 \text{ H} \\ II+Ac & 441.7 \text{ EF} \\ II+Wb & 465.5 \text{ BCD} \\ \end{array}$	Ι	479.8 B			
$\begin{array}{ll} I+Sn & 451.1 \ DE \\ I+Ac & 518.5 \ A \\ I+Wb & 446.7 \ DE \\ I+Ah & 502.2 \ A \\ II & 474.1 \ BC \\ II+Cv & 376.5 \ G \\ II+Pu & 423.6 \ F \\ II+Sn & 305.5 \ H \\ II+Ac & 441.7 \ EF \\ II+Wb & 465.5 \ BCD \\ \end{array}$	I+Cv	453.4 CDE			
$\begin{array}{ll} I+Ac & 518.5 \ A \\ I+Wb & 446.7 \ DE \\ I+Ah & 502.2 \ A \\ II & 474.1 \ BC \\ II+Cv & 376.5 \ G \\ II+Pu & 423.6 \ F \\ II+Sn & 305.5 \ H \\ II+Ac & 441.7 \ EF \\ II+Wb & 465.5 \ BCD \end{array}$	I+Pu	478.0 B			
$\begin{array}{cccc} I+Wb & & 446.7 \ DE \\ I+Ah & & 502.2 \ A \\ II & & 474.1 \ BC \\ II+Cv & & 376.5 \ G \\ II+Pu & & 423.6 \ F \\ II+Sn & & 305.5 \ H \\ II+Ac & & 441.7 \ EF \\ II+Wb & & 465.5 \ BCD \\ \end{array}$	I+Sn	451.1 DE			
I+Ah 502.2 A II 474.1 BC II+Cv 376.5 G II+Pu 423.6 F II+Sn 305.5 H II+Ac 441.7 EF II+Wb 465.5 BCD	I+Ac				
II 474.1 BC II+Cv 376.5 G II+Pu 423.6 F II+Sn 305.5 H II+Ac 441.7 EF II+Wb 465.5 BCD	I+Wb	446.7 DE			
II+Cv 376.5 G II+Pu 423.6 F II+Sn 305.5 H II+Ac 441.7 EF II+Wb 465.5 BCD	I+Ah	502.2 A			
II+Pu 423.6 F II+Sn 305.5 H II+Ac 441.7 EF II+Wb 465.5 BCD	II	474.1 BC			
II+Sn 305.5 H II+Ac 441.7 EF II+Wb 465.5 BCD	II+Cv	376.5 G			
II+Ac 441.7 EF II+Wb 465.5 BCD	II+Pu	423.6 F			
II+Wb 465.5 BCD	II+Sn	305.5 H			
	II+Ac	441.7 EF			
II+Ah 392.8 G	II+Wb	465.5 BCD			
	II+Ah	392.8 G			

I: FSC, flame source combustion; II: WFSC, without flame source combustion; Co, unimpregnated samples; Ba, boric acid; Bx, borax; Cv, cellulosic, Sn, synthetic; Pu, polyurethane; Wb, water based; Ac, acrylic; Ah, acid hardening.

^a Different letters in a column refers to significant differences among different interactions of types of combustion, varnish and impregnation materials, level.

^b LSD_{0.5}: 12.93.

^c LSD_{0.5}: 24.18.

^d LSD_{0.5}: 19.75.

TABLE	V
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Source	Degrees of freedom	Sum of squares	Mean Square	F value	P < % 5 (Sig.)
Factor A ^a	1	130136.805	130136.805	290.5014	0.0000
Factor B ^b	6	139445.725	23240.954	51.8802	0.0000
AB	6	87306.614	14551.102	32.4821	0.0000
Factor C ^c	2	258903.506	129451.753	288.9721	0.0000
AC	2	190669.018	95334.509	212.8130	0.0000
BC	12	65210.566	5434.214	12.1307	0.0000
ABC	12	151405.617	12617.135	28.1649	0.0000
Error	84	37629.742	447.973		
Toplam	125				

MANOVA for Impact of Combustion Type, Impregnation Material, and Varnish Type on Combustion Temperature

^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, water based; Ac, acrylic; Ah, acid hardening). ^c Factor C: impregnation materials (Ba, boric acid; Bx, borax).

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

Impacts of combustion type, impregnation material, and varnish type on combustion temperature have been found important for the impact of variance sources ($\alpha = 0.05$). Duncan Test results are given in Table VI to indicate the importance of differences between the groups.

Combustion temperature value was the highest in WFSC + Sn varnish combination but the lowest in WFSC + Pu varnish for varnished wood material without impregnation. For samples varnished after impregnation, combustion temperature was the highest in WFSC + Wb varnish after impregnation with

Ba but the lowest in WFSC + Sn varnish after impregnation with Ba (Fig. 1).

The combustion temperature in impregnation treatment was 22% lower in Ba and 11% lower in Bx than control samples. Accordingly, impregnation materials have a decreasing impact on combustion temperature. Indeed after the treatment with boron compounds, combustion temperature of Uludag fir wood was decreased.¹⁴

CONCLUSIONS

Retention quantity of impregnation material was found higher in Ba than Bx. The reason for the higher quantity of retention with Ba might be due to high concentration of solution.

TABLE VI **Duncan Test Results**

Process type	Temperature (°C) ^a	Process type	Temperature (°C) ^a	Process type	Temperature (°C) ^a
Π	633.6 A	I+Bx	477.9 EFGHIJ	I+Ba+Pu	433.0 LMNO
II+Wb	620.0 A	I+Bx+ Wb	475.3 EFGHIJK	II+Bx+Ah	426.7 MNO
I+Ba+Wb	571.5 B	II+Sn	471.9 EFGHIJKL	I+Bx+Cv	409.4 NOP
II+Ac	533.6 C	I+Ba+Sn	464.4 FGHIJKLM	II+Cv	401.4 OP
I+Sn	533.3 C	I+Pu	463.4 FGHIJKLM	II+Ba	400.3 OP
II+Bx+Ac	527.3 C	II+Bx+Sn	461.5 GHIJKLM	II+Bx	388.3 P
Ι	520.6 CD	I+Ac	459.1 HIJKLM	II+Ba+Sn	337.3 Q
I+Ah	509.6 CDE	I+Bx+Pu	456.9 HIJKLM	II+Ba+Ac	335.6 Q
I+ Wb	508.8 CDE	I+Ba+Cv	455.6 HIJKLM	II+Pu	335.0 Q
I+Ba+Ah	503.4 CDEF	I+Ba+Ac	447.4 IJKLMN	II+Bx+Pu	314.4 Q
II+Bx+Cv	502.0 CDEFG	II+Bx+ Wb	442.3 IJKLMN	II+Ba+Ah	269.1 R
I+Cv	495.2 CDEFGH	I+Ba	440.7 JKLMN	II+Ba+Pu	267.2 R
I+Bx+Ah	493.4 CDEFGH	I+Bx+Sn	436.3 KLMNO	II+Ba+Wb	262.7 R
II+Ah	482.5 DEFGHI	I+Bx+Ac	433.5 LMNO	II+Ba+Cv	226.2 S

Ba, boric acid; Bx, borax; Sv, Cellulosic; Sn, synthetic; Pu, polyurethane; Wb, water based; Ac, acrylic; Ah, acid hardening.

^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}:34.20), I: flame source combustion (FSC), II: without flame source combustion (WFSC).

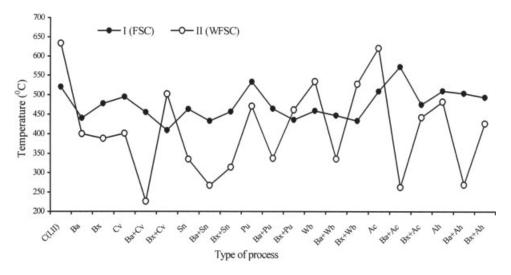


Figure 1 Combustion temperature changes of Uludag fir wood according to type of treatment.

The combustion temperature according to control samples in varnishing process; 13% lower in Cv, 5% lower in Sn, 11% lower in Pu, 4% lower in Ac, 8% lower in Ah, but 1% higher for Wb. Accordingly, the combustion temperature of varnishing samples are lower than the combustion temperature of control samples.

Impregnation materials showed a decreasing effect for combustion temperatures in Pu, Sn, Ac, Wb, and Ah varnishes 3%, 11%, 3%, 19%, and 2% for borax (Bx), 24%, 12%, 20%, 21%, and 26% for Boric acid (Ba). In Cv varnish, 24% decrease for Ba, 5% increase for Bx was observed. Besides, it is important to take into care the increasing effect of varnishes for FSC.

The combustion temperature according to combustion type + varnish type combination was the highest in FSC + Wb (518°C), the lowest in WFSC + Pu measured as 305.5°C. For FSC, combustion temperatures of unvarnished samples were lower than varnished samples except Wb and Ah. Thus, combustion temperature of FSC was higher in Cv, Pu, Sn, and Ac varnishes at a rate of 5%, 6%, 1%, 7% orderly. In WFSC, all varnish showed a decreasing effect. For wood material and impregnation material and varnish interaction, combustion temperature was the highest in FSC + Ba + Wb (571.5°C) and the lowest in WFSC + Ba + Cv (226.2°C).

Consequently, the tested varnishes showed an increasing impact but boron compounds showed a decreasing impact on combustion properties of Uludag fir (*Abies Bornmülleriana* Mattf.) wood.

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