

**THE DETERMINATION OF THE RESISTANCE TO
BURNING OF SOME WOOD TYPES IMPREGNATED
WITH SODIUM BORATE SOLUTION**

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ABSTRACT

In this study, Scotch pine (*Pinus sylvestris* L.), Oriental beech (*Fagus orientalis* L.), Oriental spruce (*Picea orientalis* L.), Uludag fir (*Abies bornmülleriana* Mattf.), Sessile oak (*Quercus petraea* Lieble) and Calabrian pine (*Pinus brutia* Ten) woods were impregnated with 5 %, 7.5 % and 10 % sodium borate solution produced by the National Boron Institute (ASTM D 1413-07) and the effectiveness of the chemical substances against burning were determined according to the ASTM E 160-50 standards.

According to the test results, Oriental spruce in the 10 % solution group had the best percentage result in the loss of weight after burning at 39.88 %. This was followed by Uludag fir at 44.74 % and Calabrian pine at 47.70 % in the same solution group. The lowest burning temperatures were obtained at the flame-sourced burning stage in the Scotch pine wood (192.08 °C) impregnated with a 7.5 % sodium borate solution, at the self-burning stage in the Eastern spruce (245.41 °C) with a 10 % solution and at the ember stage in the Uludag fir (129.12 °C) with a 5 % solution. The highest burning temperature occurred in the unimpregnated Scotch pine (554.13 °C). In conclusion, sodium borate caused improvements in all of the burning criteria and it was determined that sodium borate showed an effective burn retardant characteristic, especially in the Oriental spruce and Uludag fir.

KEYWORDS: Impregnation, sodium borate, wooden material, fire retardant, retention.

INTRODUCTION

Wooden materials are subjected to environmental factors in their place of use, biological destruction by bacteria, fungi and insects and by the effects of chemical deformation, such as fires. From this aspect, it appears to be mandatory in many places of use for the wooden materials to be impregnated with chemical substances for increasing their resistances to burning (Le Van and Winandy 1990). Wooden materials do not burn directly. Pyrolyzation occurs prior to burning with flames. In case the conditions continue in this manner, the wooden materials continue to pyrolyze until they are transformed into ashes, which are completely inorganic remains (Browne 1958). It is necessary to raise the temperature to 275 °C for the wooden material to self-ignite. Pyrolyzation in cellulose starts at 350 °C. The gases that are released as the result of pyrolyzation enter into a reaction with each other and with oxygen and at the end of this, ignition and burning start (Hakkarainen et al. 2005). The resistance to burning of a structure can be increased with processes that would be made for making the wooden materials inflammable. These procedures are also necessary for preventing the spreading of flames in structures during a fire (Richardson 1978).

Of the impregnation materials that are still used extensively, chemicals such as pentachlorophenol, copper/chrome/arsenic (CCA), copper/chrome/boron (CCB), acid copper chromate, ammonium and copper/arsenic are substances that are harmful to the environment (Kumar 2006). The impregnation industry is searching for new environmentally friendly chemicals for removing these chemicals from use. In this study, sodium borate, the new chemical produced by the National Boron Research Institute, was tested for resistance to burning.

The aqueous solutions of compounds with boron can penetrate very well into wooden materials. Scotch pine, beech, spruce and fir woods were impregnated with various impregnating materials and it was set forth that the highest retention was provided in all wood types with a 13 % mixture of boric acid and borax (Yalinkilic et al. 1997).

The impregnation materials improved the physical characteristics of the wooden materials at the end of impregnating specimens prepared from Tree-of-Heaven (*Ailanthus altissima*) wood with various water-repellent monomers and borax compounds and subjecting it to density, water intake and burning tests. It was determined that the most positive result from the aspect of weight loss was obtained in the Boric acid (BA) and borax (Bx) mixture with a ratio of 63 % (Baysal et al. 2004).

It was determined that the resistance to burning of wood increased by combinations of aqueous solutions of boric acid and sodium perborate and boron salts and water-repellent monomers (WRM) and that the water-repellent monomers increased the period of time passing until destruction in burning (Yalinkilic et al. 1996).

Along with its being known that compounds with boron improve the burning characteristics of wooden materials, it was also set forth that success was provided in the study of wooden material in which various resin combinations were used, such as melamine formadehyde, and in preventing the impregnated chemicals from washing away the wooden material from its structure (Baysal 2002).

In a study, the structural decay and fire retardancy of wooden materials as a natural polymer was considered. Wooden materials impregnated with various compounds were subjected to burning tests and it was determined that the boron compounds showed a perfect fire retardancy (Ishikawa et al. 2005).

The boron compounds come to a flaring up stage when they are heated. Since boron compounds block the cell lumens, they prevent the release of decomposed compounds that are the reason for the fire. Thus, burning is retarded. The boron compounds prevent the movements of the products of thermal destruction. These compounds, due to the crystallization of water and the gases, which are

the products of aqueous, flammable thermal destruction, cause the water to heat and vaporize and have an endothermic effect (Nishimoto and Mokuizai 1992).

Wooden materials that have a high level of boric acid shorten to a significant degree the periods of ignition and self-burning. The addition of 25 ml of silicic acid to 100 ml of boric acid-methanol solution completely prevented the ignition and self-burning stages (Amaguchi 2003).

Fire-retardants are also effective in the protection of panel products. Solid wooden materials and plywood acquired an absolute retention and a first class fire retardancy with a 7.5 % borax and boric acid impregnation. It was determined that a 5 % mixture of the solution only provided a second class protection (Le Van and Tran 1990).

It was attempted to strengthen the fire retardant characteristics of boric acid and borax with vegetal tanning materials, but the vegetal tanning materials negatively affected all of the burning parameters at all of the stages of burning (Baysal et al. 2003).

The aim of this study was to determine the effects of burning strength on the samples of Scotch pine, Oriental beech, Oriental spruce, Uludag fir, Sessile oak and Calabrian pine woods, impregnated with the sodium borate solution, produced by the National Boron Institute.

MATERIAL AND METHODS

Wooden materials

Scotch pine (*Pinus sylvestris* L.), Oriental beech (*Fagus orientalis* L.), Oriental spruce (*Picea orientalis* L.), Uludag fir (*Abies bornmülleriana* Mattf.), Sessile Oak (*Quercus petraea* Lieble) and Calabrian pine (*Pinus brutia* Ten) wood types were used for the experimental study. Care was taken that the wooden materials did not have fungal rot, knots, mildew and discoloring, that they had regular fibers and regular annual rings and that had not been inflicted with damage from fungi and insects.

Impregnation substance

In the study, the sodium borate ($\text{Na}_2\text{B}_{10}\text{O}_{16}\cdot 10\text{H}_2\text{O}$) chemical produced by the National Boron Research Institute was used as an impregnation material. Sodium borate is a chemical, which contains a high percentage of boron and has the capability of being dissolved at the ratio of 16 % at a room temperature of 20 °C. Sodium borate is produced in the form of powder. It has a pouring density of 700 kg.m⁻³ and a neutral pH. The pH can be adjusted at a value between 7-8, if wanted. The molecular weight of the compound is 590 g.mol⁻¹. Boric acid forms approximately 58 % of the molecular weight of the compound.

Preparation of the test specimens

Prisms were extracted from the body logs of the wooden materials by cutting in a radial direction according to TS EN 345 and TS 4176 and were kept in an environment that did not receive direct sunlight until they became air dry. Subsequently, a total of 1.728 experimental specimens were prepared from these prisms for each wood type with the dimensions of 13x13x76 mm for the solution concentrations and with 72 each (6x4x72) for the controls. Prior to impregnation, the specimens were kept in a drying oven at a temperature of 103±2°C until they were completely dry. They were subjected to the impregnation process after measuring their weights and dimensions.

Preparation of the impregnation solutions and the method of impregnation

The impregnation solutions used in the experiments were prepared with distilled water (DW) and 5 %, 7.5 % and 10 % of sodiumborat. The temperature was kept at 20 °C during the preparation of the solution. The impregnation of the test specimens was made under the conditions specified in the ASTM D 1413-07 standards. The temperature of the solution and process was 20±2°C for all of the impregnations. A preliminary vacuum at a value of 760 mm Hg and for a period of 30 minutes was applied for each impregnation process. Subsequently, the specimens were left to diffuse for a period of 30 minutes within the solution at atmospheric pressure. After impregnation, the specimens, whose completely wet weights and dimensions were specified, were kept in a drying oven at a temperature of 103±2°C until they became completely dry. The impregnability and burning tests of the test specimens for each one of the six wood types at completely dry measurements were made. Subsequently, the absolute retention for the experimental groups (R), for the B₂O₃ (kg.m⁻³) retention ratios (R_p) and the percentage ratio of retention (R %) were calculated by making use of the following equations (Yalinkilic et al. 1997, Yalinkilic et al. 1996, Ors et al. 2006, Keskin and Atar 2007, Keskin 2009, Sogutlu and Dongel 2009):

$$R = \frac{GxC}{V} \cdot 10^3 \text{ (kg.m}^{-3}\text{)} \quad \begin{array}{l} T_2 : \text{Specimen weight after impregnation (g)} \\ T_1 : \text{Specimen weight before impregnation (g)} \\ C : \text{Concentration of the solution (\%)} \\ V : \text{Volume of the specimen (cm}^3\text{)} \end{array} \quad (1)$$

$$G = T_2 - T_1$$

$$Rp = \frac{GxC}{V} \cdot 10^3 \times p \text{ (kg.m}^{-3}\text{)} \quad \begin{array}{l} p = 0,58 \\ p : \text{B}_2\text{O}_3 \text{ ratio in impregnation substance} \end{array} \quad (2)$$

$$R = \frac{Mai - Mbi}{Mai} \cdot 100 \text{ (\%)} \quad \begin{array}{l} Mai : \text{Completely dry weight of the specimen after impregnation (g)} \\ Mbi : \text{Completely dry weight of the specimen before impregnation (g)} \end{array} \quad (3)$$

Burning tests

The burning tests were made according to the ASTM E 160-50 standards. Prior to the burning tests and the burning process of the control specimens, they were brought to a 7 % humidity proposed in the standards in a climatization chamber with a temperature of 27±2 °C and a relative humidity of 30-35 %. In the test, 24 specimens in each group were lined up in the form of a 12 layer square prism and burned. Throughout the burning process the gas pressure was kept constant at the level specified in the standards. The burning test parameters were measures for the three burning stages as burning from flames, self-burning and burning as embers. The ratios of all of these data to the control group values were calculated.

Statistical evaluations

The average values of each burning group were used as data in the statistical evaluations. The Multiple Analyses of Variance (MANOVA) was determined for the reciprocal influence among the wood species and retention ratio factors for the resistance to burning. In cases where the difference among groups was found to be statistically significant at the level of 0.05, they were separated into homogeneity groups according to the critical values of the least significant difference (LSD) by using the Duncan test. The data was evaluated at an 0.95 level of reliability in the MSTAT-C package program written for the PC.

RESULTS AND DISCUSSION

The burning test specimens were weighed and their dimensions were measured after being impregnated with 5 %, 7.5 % and 10 % sodium borate solutions. The ratios of absolute and percentage retentions are given in Tab. 1 and Fig. 1.

Tab. 1: The absolute and percentage retention ratios obtained from the burning test specimens

Type of wood	Concentration (%)	Average retention			Type of wood	Concentration (%)	Average retention		
		(kg.m ⁻³)	(%)	B ₂ O ₃ (kg.m ⁻³)			(kg.m ⁻³)	(%)	B ₂ O ₃ (kg.m ⁻³)
Scotch pine	5.00	16.732	0.683	9.704	Oriental spruce	5.00	20.934	2.681	12.143
	7.50	29.610	2.340	17.173		7.50	30.502	3.950	17.691
	10.00	41.904	3.027	24.304		10.00	46.652	8.331	27.058
Uludag fir	5.00	39.086	4.860	22.669	Oriental beech	5.00	34.845	1.981	20.210
	7.50	49.440	9.395	28.675		7.50	52.865	4.393	30.661
	10.00	67.261	10.819	39.011		10.00	70.239	6.330	40.738
Calabrian pine	5.00	38.316	3.890	22.223	Oak	5.00	8.29	0.273	4.807
	7.50	57.751	6.543	33.495		7.50	13.552	0.538	7.860
	10.00	78.160	8.876	45.33		10.00	15.15	0.362	8.787

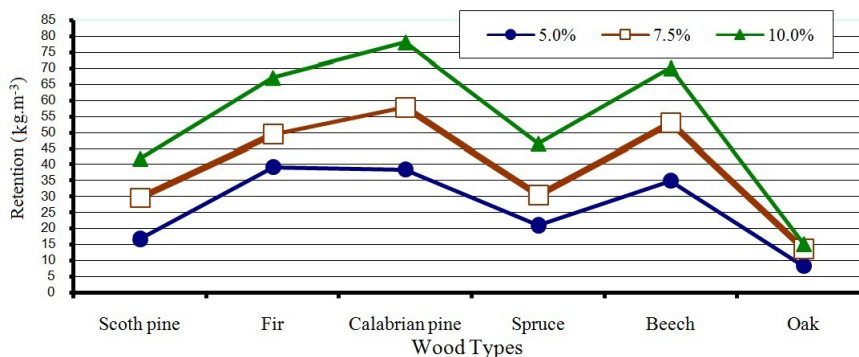


Fig. 1: The absolute retention ratios realized in the burning tests

According to the table, the highest result in the absolute retention ratios was obtained with the 10 % solution in the Calabrian pine wood (78.160). The results obtained from all types of wood are way above the protective value of 4.5 kg.m⁻³, which is required against the biological decayers in the most effective wood material. The lowest kg.m⁻³ retention values were obtained in the oak wood specimens. It is thought that the low retention value in oak wood was caused by the frequently encountered formation of a membrane. According to the percentages of concentration,

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when the ratios of wood type retention are evaluated, the Uludag fir, Calabrian pine, Oriental spruce and Oriental beech woods showed a retention increase in direct proportion to the concentration. This ratio was very little in Scotch pine and it was not realized proportionally in oak. In general, the percentage retention values were in parallel with the $\text{kg}\cdot\text{m}^{-3}$ retention values. Only in the Uludag fir this order was not followed and it had the highest result from the aspect of completely dry impregnation substance (percentage retention).

The MANOVA related to the loss of weight values of the burning tests is given in Tab. 2. The average and homogeneity groups formed at the end of the LSD test are given in Tab. 3 and Fig. 2.

Tab. 2: The results of the multiple analysis of variance related to the weight loss values

Source of variance	Degree of freedom	Sum of the squares	Average of the squares	F-value	Significance level
Wooden material (A)	5	4 506.477	901.295	152.8870	0.0000
Concentration of the solution (B)	3	10 855.046	3 618.349	613.7814	0.0000
Interaction (AB)	15	3 149.886	209.992	35.6211	0.0000
Error	48	282.968	5.895		
Total	71	18 794.377			

When Tabs. 2 and 3 are examined, it is observed that the highest weight losses were in the control specimens and these were followed by the 5 %, 7.5 % and 10 % solutions, respectively. In all type of wood other than the oak, as the ratio of concentration increased, the loss of weight fell to a significant extent in parallel to this. The loss of weight of oak wood in all of the concentration levels was at the level of the control group. In Oriental spruce, the 10 % solution group, with a 39.88 % weight loss acquired an increase in resistance to burning at a ratio of 57 % compared to the control group. At the same time, it also had the best result in all of the burning tests.

The results of the moment of destruction and the type of destructions also supported the loss of weight results. A complete destruction was not realized in any of the impregnated groups in the Oriental spruce specimens in which the best results were obtained. In contrast to this, complete destruction was observed in all of the concentration groups of the oak specimens.

The MANOVA related to the temperature values at the burning stages is given in Tab. 4. The average and homogeneity groups formed at the end of the LSD test are given in Tab. 5 and Fig. 3.

Tab. 3: Comparison of the weight loss averages according to the interaction of the wood type - solution concentration, the moment of destruction and the results of the type of destruction

Type of wood	Concentration	Loss of weight (%)		Moment of destruction (sec.)	Type of destruction
		Average	HG*		
Scotch pine	Control	90.83	LM	Self-burning 810**	Complete
	5.00	81.19	J	Self-burning 810	Destruction started
	7.50	75.62	I	Self-burning 675	Destruction started
	10.00	67.20	F	-	-
Uludag fir	Control	91.47	MN	Self-burning 780	Complete
	5.00	55.85	CD	-	-
	7.50	52.84	C	-	-
	10.00	44.74	B	-	-
Calabrian pine	Control	87.13	KL	Self-burning 420	Complete
	5.00	68.75	FG	Self-burning 360	Destruction started
	7.50	56.33	CD	Self-burning 465	Destruction started
	10.00	47.70	B	-	-
Oriental spruce	Control	94.81	N	Self-burning 750	Complete
	5.00	71.35	GH	Self-burning 900	Destruction started
	7.50	69.87	FG	-	-
	10.00	39.88	A	-	-
Oriental beech	Control	91.86	MN	Burning as embers 600	Complete
	5.00	84.04	JK	Self-burning 900	Complete
	7.50	83.54	JK	Self-burning 990	Complete
	10.00	82.70	J	Self-burning 915	Complete

* HG : Homogeneity group

** As of the start of burning, destruction was realized in 810 seconds at the self-burning stage.

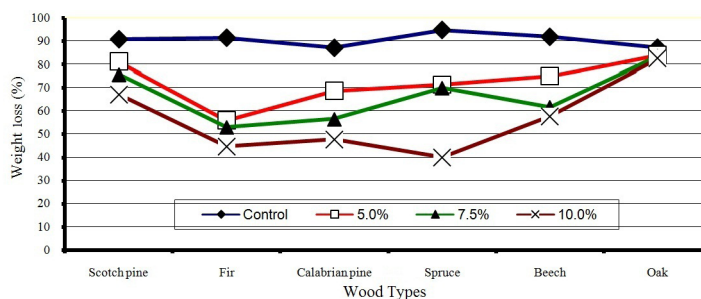


Fig. 2: The weight losses (%) formed at the end of burning

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Tab. 4: The multiple analysis of variance related to the temperature values (°C) at the burning stages

Source of variance	Degree of freedom	Sum of the squares	Average of the squares	F-value	Significance level
BURNING WITH FLAMES					
Wooden material (A)	5	299 733.177	59 946.635	14.8688	0.0000
Concentration of the solution (B)	3	683 879.962	227 959.987	56.5717	0.0000
Interaction (AB)	15	311 028.390	20 735.226	5.1430	0.0000
Error	264	1 064 327.143	4 031.713		
Total	287	2 359 013.672			
SELF-BURNING					
Wooden material (A)	5	286 139.591	57 227.918	5.4450	0.0001
Concentration of the solution (B)	3	1 236 786.630	412 262.210	39.2249	0.0000
Interaction (AB)	15	398 385.373	26 559.025	2.5270	0.0016
Error	264	2 774 698.490	10 510.222		
Total	287	4 696 010.084			
BURNING WITH EMBERS					
Wooden material (A)	5	22 796.038	4 559.208	7.0445	0.0000
Concentration of the solution (B)	3	158 941.040	52 980.347	81.8719	0.0000
Interaction (AB)	15	21 485.430	1 432.362	2.2135	0.0064
Error	264	170 837.791	647.113		
Total	287	374 060.299			

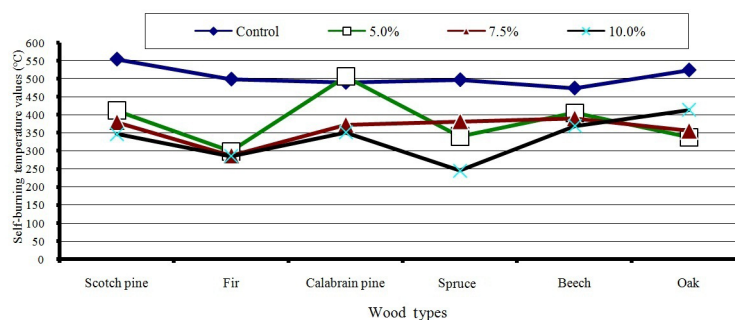


Fig 3: The temperature values (°C) at the self-burning stage according to wood types

Tab. 5: Comparison of the temperature ($^{\circ}\text{C}$) averages at the burning stages according to the wood type - solution concentration interaction and the homogeneity groups (HG)

Type of wood	Concentration (%)	Temperature ($^{\circ}\text{C}$)					
		Burning with flames		Self-Burning		Burning with embers	
		Average	HG	Average	HG	Average	HG
Scotch pine	Control	350.92	HI	554.15	H	193.36	FGH
	5.00	219.83	AB	410.92	EFG	151.52	BCD
	7.50	192.08	A	380.04	DE	132.13	AB
	10.00	194.75	A	346.88	BCDE	136.92	ABC
Uludag fir	Control	328.29	FGHI	498.53	H	191.94	FGH
	5.00	223.21	ABC	298.16	ABCD	129.12	A
	7.50	204.42	A	287.21	ABC	138.09	ABC
	10.00	229.67	ABC	285.63	AB	148.19	ABCD
Calabrian pine	Control	430.79	J	489.39	GH	199.25	GH
	5.00	299.17	DEFG	505.39	H	155.39	CD
	7.50	211.54	AB	372.85	DE	164.10	DE
	10.00	258.46	BCD	351.75	BCDE	178.10	EF
Oriental spruce	Control	374.54	I	496.88	H	184.55	FG
	5.00	274.13	CDE	339.71	BCDE	136.72	ABC
	7.50	236.17	ABC	332.09	E	148.62	ABCD
	10.00	203.25	A	245.41	A	131.34	AB
Oriental beech	Control	320.96	EFGH	473.71	FGH	222.00	I
	5.00	295.88	DEF	404.75	EF	137.04	ABC
	7.50	198.88	A	390.48	E	147.54	ABCD
	10.00	219.42	AB	368.88	CDE	144.58	ABCD
Oak	Control	329.08	FGHI	523.98	H	211.15	HI
	5.00	293.54	DEF	336.75	BCDE	138.50	ABC
	7.50	355.46	HI	355.83	BCDE	161.18	DE
	10.00	347.96	GHI	413.63	EFG	158.17	DE

When Tabs. 4, 5 and Fig. 3 are examined, it is observed that the average temperatures formed at the flame sourced burning stage of all of the impregnated specimens remained between 200-250 $^{\circ}\text{C}$. This temperature level caused a delay in the ignition. Oak showed a difference from the other wood types in this criterion and reached temperatures values of 290-350 $^{\circ}\text{C}$.

The most positive result from the aspect of temperature at the flame sourced burning stage was 192 $^{\circ}\text{C}$ in the Scotch pine specimens with a 7.5 % solution. In all of the other wood types, other

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than oak wood, the 5 %, 7.5 % and 10 % solution groups produced temperature values very close to each other at this stage.

The lowest temperature values at the self-burning stage were obtained from Uludag fir and Oriental spruce wood. There was no significant difference between the temperature values and the concentrations in Uludag fir wood. There were differences observed in the Oriental spruce wood. The highest temperature values at this stage of burning were realized in the Scotch pine and Calabrian pine types. The reason for this is that both of these wood types have a rather resinous structure.

CONCLUSIONS

The highest absolute and percentage retention values were obtained from the 10 % impregnated solutions. When the weight loss and retention results were evaluated together, it was observed that the Uludag fir wood, which had the highest percentage retention at 10.819, had the most positive result in loss of weight at 44.74 % after Oriental spruce at 39.88 %. The Oriental spruce was among the types of wood that produced the highest percentage retention and the most positive burning result. There was not a complete destruction in any of the Uludag fir wood groups; whereas there was not a complete destruction in the Oriental spruce wood groups, other than in the control groups. These support the results given above.

The lowest temperature values at the self-burning stage were obtained from Uludag fir and Oriental spruce wood. There was no significant difference between the temperature values and the concentrations in Uludag fir wood. There were differences observed in the Oriental spruce wood. The highest temperature values at this stage of burning were realized in the Scotch pine and Calabrian pine types. The reason for this is that both of these wood types have a rather resinous structure. From the results given above, it can be stated that the sodium borate impregnated chemicals in Uludag fir and Oriental spruce woods were rather effective from the aspect of preventing them from burning.

The fact that a complete destruction was realized in all of the oak wood test groups and that all of the impregnated groups of the other wood types reach a high weight loss at the level of the control groups, even if relatively good results were provided in the temperature values, could not prevent it from remaining at a low level from the aspect of resistance to burning. It is thought that the low ratio of retention of oak wood was influential on this result. Consequently, it is thought that it would be better to impregnate the oak wood with the vacuum and high pressure methods, not only with the vacuum method.

In conclusion, sodium borate (agricultural borax) impregnated chemical is environmentally friendly, and moreover, is just like a useful inorganic fertilizer. In case it is used as an impregnation chemical in the Forest Products and Furniture and Decoration Industry, then it acquires a resistance to burning at the level of 50 % in wooden materials. Sodium borate provides improvements in all of the resistance to burning criteria. It was determined that it showed an effective burning retardant characteristic, especially in Oriental spruce and Uludag fir.

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