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Combustion properties of Scots pine (*Pinus sylvestris* Lipsky) wood impregnated with boron compound doped colophony

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Abstract

The aim of this study was to investigate the combustion properties of Scots pine (*Pinus sylvestris* Lipsky) wood impregnated with boron compound (borax ($Na_2B_4O_7 10H_2O$), boric acid (H_3BO_3) w/w 50:50%) doped colophony ($C_{19}H_{29}COOH$). For this aim, Scots pine wood samples were impregnated with boron compound doped colophony by the method of medium-term dipping according to ASTM D 1413 and producers' definition. Combustion properties of samples after impregnated process were determined according to ASTM E 160. As a part of the research, 6 different combination and contents of impregnation materials have been used in order to especially investigate resistance against combustion of wood material treated with boron compounds in different concentrations. Consequently, retention performance and leaching resistance of boron compounds can be increased through colophony addition. Impregnation materials with colophony decreased the flame sourced combustion (Fsc) temperatures depending on kind of impregnation material, combustion period and weight loss ratio of the test samples decreased in comparison to the control samples. Colophony can be preferred in impregnation with boron compounds depending on kind of wood and using area.

Keywords: Colophony, Boron compounds, Combustion, Impregnation, Scots pine wood

Kolofan reçinesi katkılı borlu bileşiklerle emprenye edilen Sarıçam (*Pinus Sylvestris* Lipsky) odununun yanma özellikleri

Öz

Bu çalışma, kolofan reçinesi ($C_{19}H_{29}COOH$) katkılı borlu bileşiklerle (Boraks ($Na_2B_4O_7 10H_2O$) + Borik asit (H_3BO_3) - ağırlıkça % 50:50) emprenye edilmiş Sarıçam (*Pinus Sylvestris* Lipsky) odununun yanma özelliklerinin belirlenmesi amacıyla yapılmıştır. Bu amaçla, sarıçam deney örnekleri kolofan reçinesi katkılı borlu bileşiklerle ASTM D 1413 standardı esaslarına göre daldırma metodu ile emprenye edilmiştir. Emprenye edilen deney örneklerinin yanma özellikleri ASTM E 160 standardı esaslarına göre belirlenmiştir. Araştırmanın bir parçası olarak, özellikle farklı konsantrasyonlarda hazırlanan borlu bileşikler ile emprenye edilmiş ahşap malzemenin yanma direncini araştırmak için 6 farklı kombinasyon kullanılmıştır. Kolofan reçinesi ilavesiyle borlu bileşiklerin tutunma performansının ve yıkanma direncinin arttığı tespit edilmiştir. Kolofan katkılı borlu bileşiklerle emprenye edilen deney örneklerinde, kontrol örneklerine göre alev kaynaklı yanma (Fsc) sıcaklığı, yanma süresi ve ağırlık kaybı oranlarında düşüş görülmüştür. Kolofan reçinesi, ahşabın cinsine ve kullanım alanına bağlı olarak borlu bileşikler içinde katkı maddesi olarak tercih edilebilir.

Anahtar kelimeler: Kolofon reçinesi, Borlu bileşikler, Yanma, Emprenye, Sarıçam odunu

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1. Introduction

Requirement of protection of wood material was recognized centuries ago and diversity of impregnation techniques was developed by this time (Ayar 2008). Impregnation can be identified as process of embedding of chemical material into gaps of wood structure in order to protect of wood material against fungus, insects, thermites, sea creatures beside dimensional changings and fire (Ors and Keskin 2008). Within this context, organic and inorganic boron compounds appeared as one of mostly preferable protective material on the matter of protection of massive and wood based composite materials (Kartal and Imamura 2004).

Wood protection efficacy of borates against biological agents, flame retardancy, and suitability to the environment is well known. Since borates can be applied to timber as water based solutions, they are preferred economically as well. Even though they are highly mobile in wood, boron compounds are widely used in timber preservation. Borates migrate in liquid and increase the hygroscopicity of wood in damp conditions (Baysal et al. 2006).

Boron compounds gained importance due to its high impacts against to the biological pesticides, diffusion abilities to wood materials, cheap and easy-obtainable features, low toxic impact on mammals and increase combustion resistance of wood materials. However, usage area of these minerals has been restricted to interior applications because of its low leaching resistance against water in atmosphere conditions (Baysal 2003).

In Europe and many other parts of the world, CCA formulations are now being replaced in the lower use classes by preservatives free of arsenic and chromium due to the concerns related to the toxicity of these components. Borate-based preservatives should be taken into account as an effective and safe-to-handle alternative (Mohareb 2005).

Formulations range from being a primarily boron-based formulation to a formulation that contains some amount of boron. The overall efficacy of such compounds may rely more on the other compounds than the borate itself. The advantages of boron preservatives may not be retained as it will result in a change in the mechanism of action and mammalian toxicity (Obanda et al. 2008).

Combustion retardant chemical materials used as impregnation material do not redound wholly incombustible feature to the wood material. However, it can make flammability difficult and retard spread out of flame after combustion started (Ors and Keskin 2001). When impacts of impregnation and upper surface protection materials which are used to protect wood raw materials against interior and exterior ambient (biotic, abiotic: fire etc.) on combustion properties of wood are researched, varnishes applied after impregnation process have no impact on combustion properties of test samples (Ors et al. 1999a).

Lowest mass lost for both of Brazil wood and South Pine samples treated with a mixture of 5% of boric acid and borax solutions with water has been determines between 68.72% and 72.37%, however, borax solution ratio of 5% is the best efficient treatment to extend fire period of wood (Temiz et al. 2008).

Temperature of decomposition by heat of hardwood woods is lower than coniferous woods. This case comes from pentagon content of hardwood woods which is more sensitive against heat (Ors and Keskin 2008).

Some acetone-carried consolidanted for water logged archaeological wood were tested in order to evaluate treatments able to save time and energy. To evaluate the processes, retention of impregnating products was measured; the results highlighted that natural and modified colophony treatments gave the most satisfactory results both in the maintenance of shape. This fact was related to the high retention values of those products that occluded most of the porosity including the microporosity of cell walls (Giachi et al. 2011).

In this study, determination of impacts of colophony used to increase retention amount of boron compounds on Scots pine wood was aimed. Boron compounds are used as healthy and domestic impregnation material but it dissolves in water. Due to this reason, it cannot be used in industry heavily. Within this context, starting from the idea of retention amount of boron compounds can be raised by adding colophony into impregnation liquid, impregnation material produced in the laboratory has been applied into wood material by using dipping method. After weight stabilization, combustion tests of wood samples have been carried out in laboratory.

2. Material and Method

2.1. Materials

2.1.1. Woods

Scots pine (*Pinus sylvestris* Lipsky) wood was chosen randomly from timber merchants of Ankara, Turkey. Special emphasis was given for the selection of the wood material. Accordingly, non-deficient, proper, knotless, normally grown (without zone line, without reaction wood and without decay, insect and fungi damages) wood materials were selected according to TS 2476 (TSE 1976). Test samples selected from sapwood have an average air density of 0.528 g/cm^3 and an annual ring width of 1.9 mm.

2.1.2. Boron compounds

Boric acid (BA) and Borax (BX) were obtained from Etibank–Bandırna (Turkey) boric and acid Factory. Composition of Boric acid (H_3BO_3) was 56.30% B_2O_3 43.70% H_2O with a molecular weight 61.83, density 1.435 g/cm^3 and melting point 171^{o}C. Borax ($Na_2B_4O_7 10H_2O$) consists of 21.28% Na_2O, 47.80% B_2O_3, 30.92% H_2O with a molecular weight 291.35, density 1.815 g/cm^3, melting point 741 ^{o}C (Ors et al. 2006).

2.1.3. Colophony

Colophony ($C_{19}H_{29}COOH$) was a transparent and Scots pine rosin which was made via distilling tall oil. It dissolves in ether, alcohol, chloride hydrocarbon and other hydrocarbons. Rosin obtained from pine trees via natural way contains 80% colophony and 20% turpentine. As to content of colophony, it contains 90% rosin acids an 10% neutral matter. Moreover, it was softened at 70-80 ^{o}C and it dissolved at 100-130 ^{o}C. Colophony had no negative impact on human health. Colophony was identified as water repellent and combustion resistant in industry (Under et al. 2001). Colophony was obtained from WSSFC – Wuzhou Sun Shine Forestry and Chemicals from China.

2.1.4. Ethanol

Ethanol (CH_3CH_2OH) which was used as solvent in development of impregnation material obtained from industry with the purity ratio of 96%. Its density was 789 kg/m^3, boiling point is 78.5 ^{o}C, freezing point is -114.5 ^{o}C and dissolves in water completely. Ethanol was colourless and made of plant alcohol.

2.1.5. Composition of impregnation material

Compositions of the new generation impregnation materials developed in laboratory are shown in Table 1. Six different combination and contents of impregnation materials were used to especially investigate resistance against combustion of wood material treated with boron compounds in different concentrations.

Table 1. Composition of impregnation materials

Impreg. Material Codes	COMPOSITION OF IMPREGNATION MATERIALS				
	Borax ($Na_2B_4O_7 \cdot 10H_2O$) (%)	Boric Acid (H_3BO_3) (%)	Colophony ($C_{19}H_{29}COOH$) (%)	Distilled water (H_2O) (%)	Ethanol (C_2H_6O) (%)
E1	-	3	10	17	70
E2	3	-	10	17	70
E3	1.5	1.5	10	17	70
E4	3		-	97	-
E5		3	-	97	-
E6	1.5	1.5	-	97	-
C	Control Samples un-impregnated				

Colophony ($C_{19}H_{29}COOH$) that was natural resin and used widely in paint and adhesive industries were added into this combination to increase retention capability of boron compounds. Determined ratios during preparing different concentrations of impregnation material were calculated according to results of pilot applications, determination of dispersion ratios of colophony into ethanol and soluble amount of boron compounds into water. 70% of total weight of impregnation material was ethanol and 10% was colophony. These ratios provided clean and brilliant impregnation liquid without sludge. Furthermore, 3% of boron compounds in this impregnation material completely dissolved into the liquid with no sludge.

2.2. Methods

2.2.1. Determination of density

Weights of impregnated samples were weighed through analytical balances with sensitivity of ±0.01g and volumes are calculated after measure of dimensions through digital calliper with the sensitivity ±0.01mm. Air-dry densities (δ_{12}) were calculated using following formula (1);

$$\delta_{12} = \frac{M_{12}}{V_{12}} \left(\frac{g}{cm^3}\right) \quad (1)$$

In this formula; M_{12} : weight at air-dry condition (g), V_{12} :volume at air-dry condition (cm^3).

2.2.2. Preparation of test samples

Scots pine test samples obtained from industrial companies were shaped according to the ASTM E 160-50 (ASTM 1975) principles using special equipment in Gazi University Technology Faculty Department of Wood Products Industrial Engineering Laboratories. After this process, best samples were reserved as test samples. Shapes and dimensions of test samples to be used to combustion tests were shown in Fig. 1.

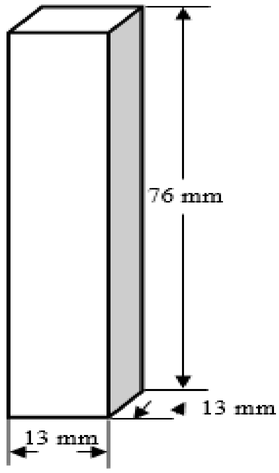

Figure 1. Dimensions of fire samples (R:13 x T:13 x L:76 mm)

2.2.2. Impregnation process

Scots pine wood samples were impregnated with boron compound doped colophony by the method of medium-term dipping (24 hours) at room temperature according to ASTM D 1413 (ASTM 2005) and producers' definition. Test samples at 10% moisture content were kept for 24 hours in 5 litres volume bottles coded with number of related impregnation material. Prepared test samples were put into these bottles sensitively and filled up prepared impregnation materials. It was not allowed to air gaps in the covered bottles to provide full contact of impregnation material to the test samples during impregnation process. Also, the bottles tightly closed were shaken at periodic intervals in order to increase the impregnation impact of the impregnated material. After impregnation process, test samples put on a table vertically and leave to dry until its weights were stable. Moreover, the impregnation liquids were kept in coded bottles.

Amount of impregnation material is calculated according to following formula;

Vn x N = Vt ⟶ 12.844 cm^3 x 72 = 924,76 cm^3
1 cm^3 = 0.001 Liter ⟶ 924,76 cm^3 = 0.924 L
Volume of impregnation bottle = 5 L
Im: 5 – 0.924 = 4.076 L
Vn: Volume of test samples (R:13 x T:13 x L:76 mm)
N: Number of test samples
Vt: Total volume of test samples
Im: Amount of impregnation liquid needed for each of impregnation bottle

2.2.3. Data Analysis

Analysing of data gained from test samples was carried out using SPSS 22. ANOVA analysis was applied for Flame Sourced Combustion (Fsc), Without Flame Source Combustion (WFsc) data, weight loss and combustion period within the context fire test. Moreover, Duncan test was applied among groups at the end of analysis in case of differences are seen in order to test homogeneity of the groups.

3. Results and Discussion

3.1. Retention quantities

Retention performance of developed impregnation material was investigated within the context minimum, maximum, average retention amounts and standard deviation. Retention amounts of impregnation material were shown in Table 2.

Table 2. Retention amounts of wood preservatives in Scots pine samples

Statistical Values	E1	E2	E3	E4	E5	E6
Min (kg/m^3)	14.926	24.781	14.696	13.177	14.663	15.627
Max (kg/m^3)	20.045	29.229	22.394	26.543	29.385	30.131
Mean (kg/m^3)	17.070	26.584	17.424	19.293	21.334	20.605
Standard deviation (sd)	2.6585	2.3404	4.3110	6.7546	7.4573	8.2522
Number of tests (N)	3	3	3	3	3	3

Retention amounts showed that highest amount in terms of average retention amount values were measured from test samples impregnated with E2 coded impregnation material and test samples impregnated with E5 and E6 coded impregnation materials follow respectively. This case can be explained with positive impact of colophony on retention performance of borax and thus penetration of much more amount of impregnation materials to the wood material.

Retention performance of colophony added impregnation materials were up to composition of impregnation liquids which contain different percentages of boron compounds. This case can be explained with compatibility of colophony with boron compounds and its fast retention features that decrease penetration performance of impregnation material during impregnation process which is dipping method. This is preferred case when prevention of easy leaching of boron compounds from wood materials is needed depending on usage area.

3.2. Combustion properties

Combustion resistance of materials was classified according to its usage area. While using of wood material as heat resource, good combustion performance is determined through indicators such as long combustion period, low ash amounts, high heat and light spread out, etc. In industrial materials, this issue showed direct opposition. Best combustion resistance of wood material to be used in industry means long combustion period, high ash amount, low heat and light emission. In this study, longer combustion period, lower weight loss amount, flame source combustion (Fsc) and without flame source combustion (WFsc) temperatures of test samples according to control samples indicate better combustion resistance. In other words, samples that fire in long period and sustain lower weight loss, have better combustion resistance. In this section, data obtained from Scots Pine test samples used in combustion test are investigated separately. Values of Fsc and WFsc temperatures, maximum, minimum and average temperature values, mass loss amount and combustion period of test samples according to the impregnation compositions were given in Table 3.

According to the data, lowest Fsc temperature was obtained from Scots Pine test samples impregnated with E2 coded impregnation material (490.08 ^oC). While highest Fsc temperature was investigated in impregnation free control samples, using boron compounds and colophony addition could decrease Fsc temperature values in Scots Pine test samples as a result of experiment. Fsc temperature values according to kind of impregnation composition are given in Fig. 2.

Table 3. Combustion values of pine samples according to the impregnation compositions

CombustionType	Statistics	E1	E2	E3	E4	E5	E6	C
	x	520.58	490.08	529.66	507.41	524.58	513.38	555,32
Fsc°C	Sd	17.856	9.152	14.375	5.343	4.006	14.435	28,765
	Min	509.25	483.00	513.08	501.25	521.75	503.25	522,14
	Max	541.16	500.41	538.58	510.66	529.16	529.91	573,25
	x	574.05	610.28	595.43	567.32	591.35	568.55	597,92
WFsc°C	Sd	19.36	17.25	12.03	14.87	11.86	23.39	28,44
	Min	561.84	591.92	581.80	550.16	581.22	554.60	566,50
	Max	596.38	626.18	604.60	576.45	604.40	595.55	621,88
Weight Loss (%)		89.77	89.53	89.58	87.58	89.50	87.30	94.55
Combustion Period (min)		25.00	25.00	24.00	27.00	23.33	21.00	20.79

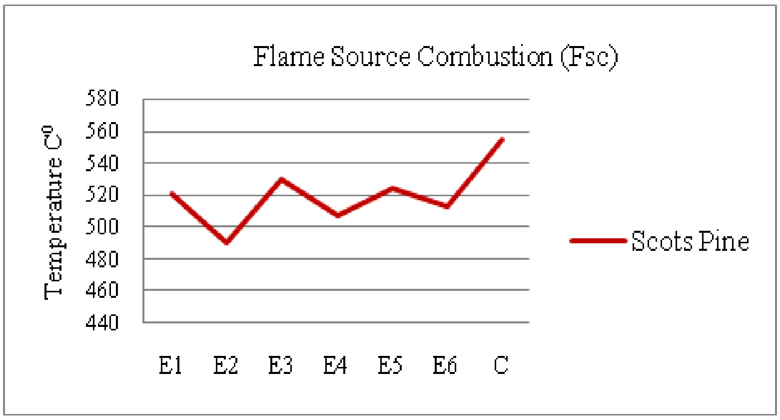

Figure 2. Fsc temperature values of Scots pine test samples according to impregnation compositions

The lowest WFsc temperature values (550.16 °C) had been obtained from test samples treated with E4 coded impregnation material when average WFsc temperature values of test samples investigated. Impregnation materials coded E6 and E1 follow E4 respectively. Altough lowest WFsc temperature values were obtained from E4 that contains Borax and colophony free, according to (Uysal and Kurt 2005) Borax-Boric acid composition used as fire retardant and/or inhibitor impregnation material provides better performance in Scots Pine wood which is coniferous wood (Uysal and Kurt 2005). E6 shows almost equal performance in comparison to E1 when lowest WFsc temperatures compared. WFsc temperature values of pine test samples are shown in Fig. 3.

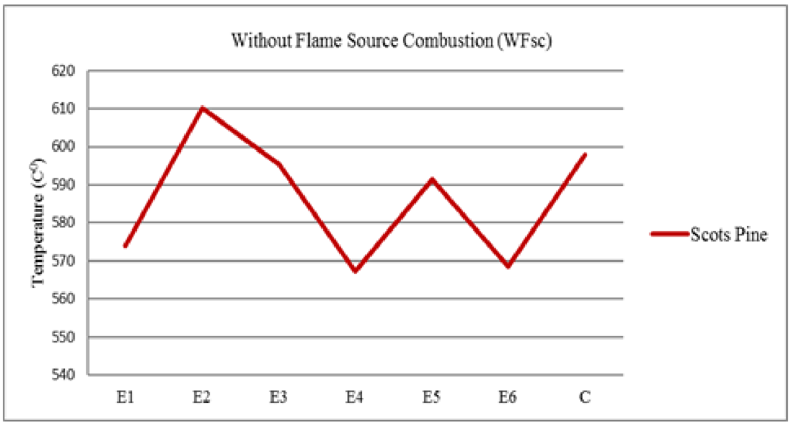

Figure 3. WFsc combustion temperature values of Scots pine test samples according to impregnation compositions

Weight loss and combustion period were another factor on the issue determination of combustion resistance of test samples after impregnation process. Weight loss ratios of test samples according to impregnation compositions are given in Fig. 4.

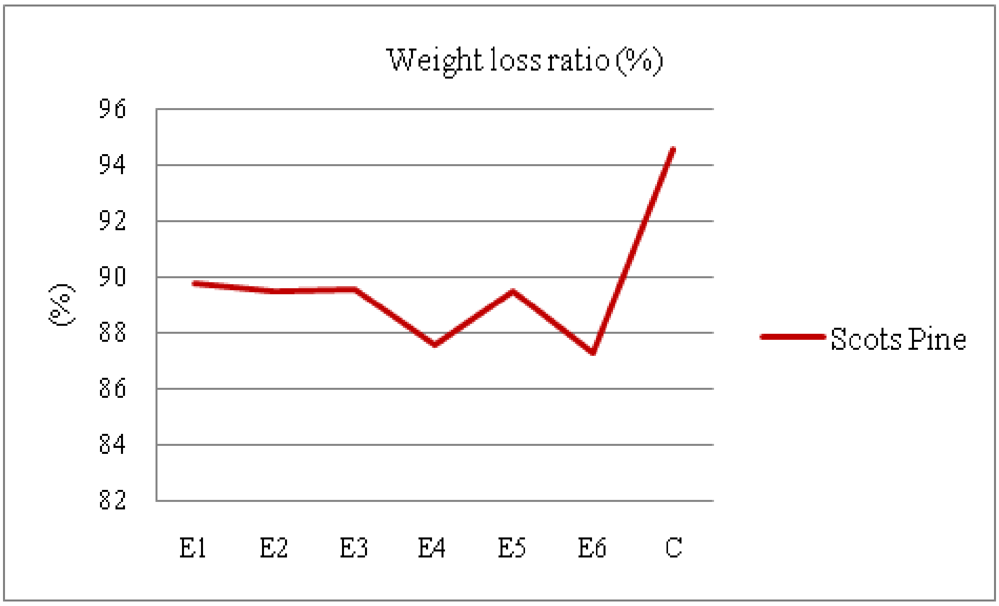

Figure 4. Weight loss ratios of Scots pine test samples according to impregnation compositions

Depending on the data, highest weight loss ratios were investigated on control samples which were not impregnated. As to investigation of combustion period of test samples, higher combustion periods were investigated on impregnated test samples in comparison to control samples, hence, impregnation process extends combustion period of test samples was concluded. Combustion periods of Scots Pine test samples according to the impregnation material compositions were given in Fig. 5.

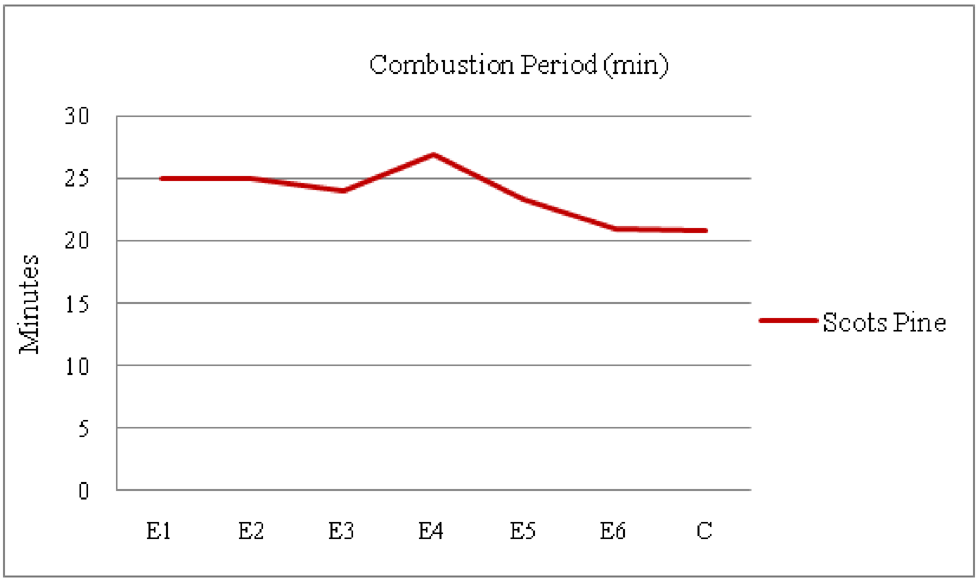

Figure 5. Combustion periods of test samples according to the impregnation material

Fsc, WFsc, weight loss and combustion period values of Scots pine test samples were investigated separately through Duncan test depending on ANOVA test and Sig values. ANOVA results of test samples are given in Table 4.

Table 4. ANOVA results of test samples

FACTOR		Ss	df	Ms	Fv	*SIG
Fsc(°C)	Between Groups	7378.962	6	1229.82	5.095	0.006
	Within Groups	3379.399	14	241.386		
	Total	10758.36	20			
WFsc(°C)	Between Groups	4933.296	6	822.216	2.270	0.097
	Within Groups	5071.538	14	362.253		
	Total	10004.83	20			
Weight Loss(g)	Between Groups	101.500	6	16.917	21.417	0.000
	Within Groups	11.058	14	0.790		
	Total	112.558	20			
Period(min)	Between Groups	90.736	6	15.123	3.907	0.017
	Within Groups	54.191	14	3.871		
	Total	144.928	20			

Ss: Sum of squares, df: Degrees of freedom, Ms: Mean square, Fv: F value, SIG: Significance, *P < 0.05

When combustion resistance of Scots pine test samples impregnated with 6 different impregnation materials investigated, significant differences could be seen among Fsc, weight loss and combustion period values between groups. According to Fsc Duncan test results of Scots Pine test samples, highest Fsc temperature values were investigated in control samples while lowest Fsc temperature values were in test samples impregnated with E2 coded impregnation material. Fsc Duncan test results of Scots Pine test samples were shown in Table 5.

Table 5. Duncan test results for Fsc of Scots pine wood samples

Fsc(°C)	Impregnation Materials	N	Subset for alpha = 0.05		
			1	2	3
Duncan[a]	E2	3	490.08333		
	E4	3	507.41667	507.41667	
	E6	3	513.38900	513.38900	
	E1	3		520.58333	
	E5	3		524.58333	
	E3	3		529.66633	529.66633
	C	3			555.32667
	Sig.		.102	.133	.063

Means for groups in homogeneous subsets are displayed. a. Uses Harmonic Mean Sample Size = 3.000

The highest weight loss ratio values were investigated from Scots Pine control samples (C) after combustion period. Conversely, lowest weight loss ratios were seen in Scots Pine test samples impregnated with E6 coded impregnation material. Control samples were classified under separated homogeneity group. Weight loss Duncan test results of Scots Pine test samples were shown in Table 6.

Table 6. Duncan test results for weight loss of Scots pine wood samples

Weight loss (g)	Impregnation Materials	N	Subset for alpha = 0.05		
			1	2	3
Duncan[a]	E6	3	87.3000		
	E4	3	87.5867		
	E5	3		89.5067	
	E2	3		89.5300	
	E3	3		89.5833	
	E1	3		89.7733	
	C	3			94.550
	Sig.		0.699	0.740	1.000

As to combustion period, lowest combustion period was determined in control samples (C) while highest was in test samples impregnated with E4 coded impregnation material which didn't contain colophony additive. However, impregnation materials coded E1 and E2 extended combustion period of Scots Pine test samples as well. Duncan Test results for combustion period of Scots Pine test samples were shown in Table 7.

Table 7. Duncan test results for combustion period of Scots pine wood samples

Combustion Period (min)	Impregnation Materials	N	Subset for alpha = 0.05	
			1	2
Duncan[a]	C	3	20.79	
	E6	3	21.00	
	E5	3	23.33	23.33
	E3	3	24.00	24.00
	E1	3		25.00
	E2	3		25.00
	E4	3		27.00
	Sig.		0.085	0.056

Colophony additive into boron compounds used as impregnation material increased retention performance of impregnation material depending on content of boron compounds and even develops combustion properties of wood and boron compounds. Colophony was used as fire-resistant and water insulator for dry wood material (Under et al. 2001). Fsc temperatures and weight loss ratios could be decreased; combustion period can be extended of wood materials depending on content of impregnation material.

4. Conclusions

Boron compounds are clear and nontoxic impregnation materials for human health by means of its chemical properties and impacts on nature. Major problem related to boron compounds is low leaching resistance from wood material. Impacts of colophony addition into impregnation materials on combustion properties of wood material were investigated. Following results were concluded at the end of test;

- When impact of colophony additive on combustion resistance of boron compounds investigated, Fsc temperatures of Scots pine samples can be decreased using colophony additive depending on the kind of boron compound. The lowest Fsc temperatures

(490.08 ºC) were obtained from colophony additive impregnation material (E2) according to control samples. Fsc temperature values can be decreased by means of colophony additive into boron compounds.

- WFsc temperatures of colophony added impregnation materials were mostly higher than impregnation materials which are composition of boron compounds, no colophony additive. Although this case, lower WFsc temperatures were obtained from E1 and E3 coded impregnation materials (574.05 ºC and 595.43 ºC respectively) according to the control samples (597.92 ºC). Colophony addition can increase average WFsc temperatures in comparison to impregnation materials without colophony. However, WFsc temperatures of impregnation materials with colophony still lower than control samples'.
- According to the test results, colophony added impregnation materials have lower weight loss ratio values when compared with control samples. Using colophony in impregnation of Scots Pine wood with boron compounds can increase combustion resistance of wood material. On the contrary, weight loss ratio values of impregnation materials untreated with colophony were significantly lower than treated compositions. Lowest value of weight loss ratio was investigated on E6 coded impregnation material (87.30%) while highest one on control samples (94.55%). Differences were found statistically meaningful. Control samples were determined in divided in different homogeneity group. Colophony addition may cause to negative impact of weight loss ratios of test samples when the values obtained from colophony-added impregnation materials compared with colophony free impregnation liquids which contain only boron compounds.
- Lowest combustion period was observed on control samples (20.79 min) while higher combustion period was seen on samples impregnated with E2 coded impregnation material (25.00 min). Colophony has positive impact on extending combustion period of wood material. The highest combustion period was seen on E4 coded impregnation material which does not contain colophony, but average values are proof of impact of colophony on combustion period of Scots Pine wood.
- Additive of colophony may be useful for increasing the against fire performance of boron compounds Fsc and WFsc temperatures could be reduced through colophony additive. On the other hand, performance of impregnation materials differs according to its compositions and impregnation technique.
- Protection performance of boron compounds used as impregnation material can be strengthened through colophony addition in industrial material design.

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