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Combustion properties of impregnated spruce (*Picea orientalis* L.) wood

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HIGHLIGHTS

- Weight loss was found to be smaller in Wolmanit-CB impregnated specimens.
- Wolmanit-CB and synthetic varnish would be better according to the results.
- The amount of O_2 in control specimens in CWF was observed to be the highest.
- Control and year specimens had close results in the weight tank.

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ABSTRACT

Wooden material is one of the most commonly used construction materials since history of humanity due to its environment friendly and renewable natural source. This structure of wood material is also preferred because it is lightweight, easy to work, resistant to factors such as earthquake and fire, as compared to other used building materials. However, organic structure of the wooden material causes it to be affected negatively because of damages that might occur outdoors under inappropriate conditions. Due to this reason, in this study were investigated effects of various chemicals (impregnated and varnish type), used to prevent environmental damages (biotic, abiotic pests, fire etc.) on combustion resistance of the wood exposed to air conditions for a year. For this purpose, prepared Caucasian spruce (*Picea orientalis* L.) specimens were surface treated (synthetic and water-based varnish) after impregnated according to ASTM-D 1413-76 principles with various chemicals (Tanalith-E and Wolmanit-CB). Test specimens with impregnated, varnished and processless, were left in outdoor conditions for a year. Combustion experiment was conducted according to the principles in ASTM-E 160-50 combustion test standards. According to the results of the experiment, control and year specimens had close results in the weight loss. Weight loss was found be lower Wolmanit-CB impregnated specimens (92.12%) among the impregnated materials to synthetic varnish coated specimens (% 91.89) among the varnished specimens. It was determined that using Wolmanit-CB for impregnating and synthetic varnish for varnishing would be better according to the results. Furthermore, the amount of O_2 (25.84 ppm) in control specimens in combustion with flame; the amounts of CO and CO_2 (31787, 19.92 ppm respectively) in year specimens in combustion without flame and the amount of NO (54.65 ppm) in year specimens in glowing was observed to be the highest.

1. Introduction

Lightness of wooden material and its resistance to physical and mechanical factors are among the most distinct properties of wooden material to other materials [1]. Besides, there is an increasing demand for wood and wood based composites for both interior decoration element, house and outer house building construction applications and in terms of easy processing, physical and mechanical properties, aesthetic appearance, environment and health [2,3]. The biggest advantage of wooden material in case of fire is that it burns slowly and forewarns about collapse resulting in minimizing loss of life. However, wooden material has some undesired properties besides these positive properties. The reason for this is that wooden material undergoes degradation under inappropriate conditions by weathering, fire, bacteria, fungi and destructive insects due to its organic nature. Other negative properties of wooden material cause only financial losses whereas its inflammability may also cause life-threatening situations [4].

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It can be dried, apply preservatives and impregnate with various materials, in order to prevent damages that might occur and to prolong its lifetime [5].

Wooden material is an easily combustible and flammable material. Fire-retardant impregnation materials go into degradation below the degradation temperature of wooden material and rapidly transform cellulose into wood charcoal and water. Thus, volatile and flammable materials that would have been formed in higher temperatures are not formed leading to reduced inflammation of the wood and flames are prevented from spreading around [6].

Due to this reason, inorganic based chemicals and commercial compounds are most commonly used to retard and prevent the combustion of wooden material currently. Most commonly used among these materials are; ammonium sulfate, ammonium chloride, boron, borax, boric acid, phosphoric acid, zinc chloride, chrome and copper compounds [7].

Protection of wood from inner and outer environmental factors, determination of its fire-retardant effect and how it varies annually were studied as the aim of this study.

2. Material and method

2.1. Material

Specimens used in the study were obtained from randomly provided spruce wood in Eastern Black Sea Region by "random selection" method. Selected lumbers were held in a climate chamber with 20 ± 2 °C temperature and 65 ± 3% relative humidity until they reach 12% humidity and constant weight and then rough cutting was performed from wooden material according to conducted experiments.

Chemicals to be used in this study were selected from the commonly used impregnative materials (Wolmanit-CB and Tanalith-E) in our country. Varnish type was selected to be synthetic and water-based.

2.2. Method

Test specimens were cut evenly in 13 × 13 × 76 mm dimensions. Totally 576 (2 × 2 × 2 × 3 × 24) test specimens were prepared; 24 in each group, 3 groups in each test period with 2 different impregnatives from spruce tree, 2 different varnish types, year and control groups. Total number of parts and chemicals to be applied are given in Table 1.

Test specimens were dried under 20 ± 2 °C temperature and 65 ± 5% relative humidity until they reach constant weight before impregnation and then weighed on a precision balance with a 0,01 g precision.

Test specimens were impregnated with vacuum-pressure method in line with ASTM-D 1413-76 [8]. To accomplish this, specimens were vacuumed under pressure equivalent to 60 cm h g^{-1} for 60 min and then placed in a solution under standard atmosphere pressure for 60 min. Impregnated specimens were held in an environment with air circulation for 15–20 days to evaporate the solvent and then exposed to drying process under 20 ± 2 °C temperature and 65 ± 3% relative humidity until they reach 12% humidity.

Retention amount of the impregnatives in test specimens (r-kg/m^3) was calculated for before and after impregnation using the equations below TS 5724 [9].

$$R = \left[\frac{G \cdot C}{V}\right] \times 10^3\ \mathrm{kg/m^3}$$

Wherein

$G = T_2 - T_1$;
T1 = weight before impregnation (g),
T_2 = weight after impregnation (g);
V = specimen volume (cm^3);
C = solution concentration (%);

Statistical values regarding the retention amount (kg/m^3) of test specimens are given in Table 2.

After impregnation, surfaces of the acclimatized specimens were slightly sanded with a No. 220 sander and cleaned of dust to become ready for varnishing. Varnish amount was weighed with an electronic scale with 0,01 precision and hardening agent, thinner or diluent amounts in order to make varnish applicable were prepared taking into account advised amounts by the manufacturer. Varnishing was performed in line with the requirements in ASTM-D 3023 [10]. Varnished specimens were left to dry in room temperature.

Varnished test specimens were then exposed to external factors on stands previously prepared depending on periods. Specimens were positioned facing south with a 45° angle. In this study, effects on the combustion resistance of the wooden material exposed to outer environmental conditions were investigated. For this reason specimens were periodically left in an outer environment together with control groups for a year. After the periodic waiting duration of test parts, combustion tests were conducted.

Impregnated and not impregnated, varnished and not varnished wooden materials were taken from the external medium at the end of their periodic duration and their combustion properties were measured in line with ASTM-E 160-50 in combustion test device shown in Fig. 1 [11]. According to this, each specimen group was weighed and piled on the wire-stand in the device. In the experiment, 24 specimens were put on top of each other in 12 floors in the shape of a square prism and burned. Flame source was placed under the pile and combustion with flame source was pursued for 3 min. Afterwards the flame source was extinguished to proceed with combustion without flame source and glowing stages. The % weight loss was determined using the following formula:

$$WL(\%) = \left[\frac{(W_0 - W_d)}{W_0}\right] \times 100 \quad \text{equation was used}$$

Table 2
Retention amount of test specimens.

Retention (kg/m^3)	Type of wood spruce
Tanalith-E	1.15
Wolmanit-CB	6.35

Table 1
Test parts prepared in the scope of the research.

Wood materials	Groups	Type of impregnate	Type of varnish	Number of samples
Spruce	Year	Wolmanit-CB	Synthetic	24 × 3 = 72
	Control	Tanalith-E	Water-Based	24 × 3 = 72
				24 × 3 = 72
				24 × 3 = 72
				24 × 3 = 72

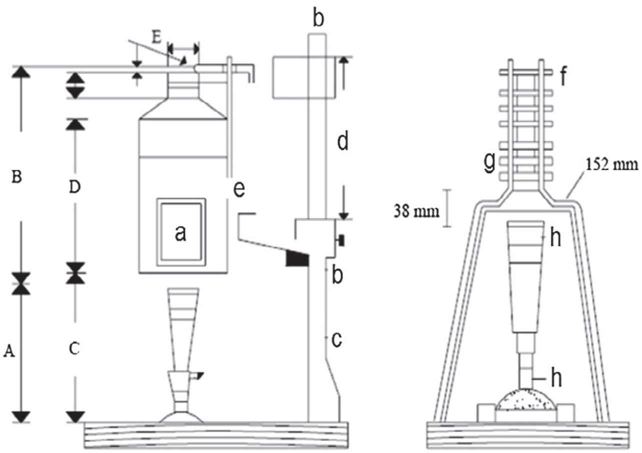

Fig. 1. Combustion test apparatus. a. Mica glass, b. End of slide, c. Flame burner guide, d. Slide, e. Millivoltmeter inlet, f. Wood specimens, g. Wire cage, h. Flame burner (maker type) (ASTM-E 160-50, 1975).

Table 3
Analysis of variance results regarding weight loss of spruce wood.

Variation source	Degrees of freedom	Sum of squares	Squares average	F-Value
Year (y)	1	24.9	24.9	20.39**
Impregnate (i)	2	11.1	5.6	4.56*
Varnish Type (vt)	2	9.7	4.8	3.96*
y * i	2	25.5	12.7	10.43**
y * vt	2	1.2	0.6	0.49
i * vt	4	6.8	1.7	1.39
y * i * vt	4	11.7	2.9	2.39
Error	36	44	1.2	
Total	53	134.9		

*, **: Significant at the level of 5% and 1% respectively.

wherein;
WL: Weight Loss (%),
W_0: Dry specimen weight before the test (g),
W_d: Dry specimen weight after the test (g)

After the application period of the parts exposed to outer environmental conditions for a year: oxygen, carbon dioxide, carbon monoxide, nitrogen monoxide (O_2, CO_2, CO, NO) amounts due to combustion with flame source, O_2, CO_2, CO and NO amounts due to self combustion and O_2, CO_2, CO and NO amounts due to glowing were determined as (%).

Gas amount, temperature, combustion duration and weight loss ratios regarding combustion with flame source, combustion without flame source and glowing of the test specimens were determined with analysis of variance in three repetitions using an SAS software according to random blocks test pattern [12].

3. Results and discussion

Analysis of variance results regarding weight loss of impregnated spruce wood in combustion with flame source, combustion without flame source and glowing according to year, impregnatives and varnish type are given in Table 3; average values and least significant difference (LSD) test results are given in Table 4.

Average values and least significant difference (LSD) test results regarding year, impregnatives and varnish types' combustion temperature, light density and combustion durations in impregnated specimens in combustion with flame source, without flame source and glowing according to year, impregnative and varnish type are given in Table 4.

As a result of analysis of variance regarding the weight loss of impregnated specimens, differences in a significance level of 0.01 between year specimens and 0,05 between impregnative and varnish type were found (Table 3).

Weight loss of control specimens (91.78%) was found out to be lower compared to "year" (93.17%) as a result of spruce combustion test. Furthermore, the lowest average values in impregnated

Table 4
Groups formed according to average values (%) and least significant difference (LSD) test results regarding weight loss and combustion temperature, light density and combustion duration of impregnated spruce wood in combustion with flame source, without flame source and glowing according to year, impregnite and varnish type.

Factor	Combustion with flame source		Combustion without flame source		Glowing		Combustion duration		Weight loss
	CT (°C)	LD (lux)	CT (°C)	LD (lux)	CT (°C)	LD (lux)	FT (sn)	TCT (sn)	(%)
Year									
Control	480 a	289 a	603 a	289 b	333 a	290 b	351 a	626 a	91.78 b
Year	482 a	320 a	562 b	320 a	306 b	324 a	350 a	625 a	93.17 a
Avg.	481	305	582	305	319	307	351	625	92
S_x	1.41	21.92	28.99	21.92	19.09	24.04	0.71	0.71	0.98
LSD	6.22	2.22	6.30	1.14	12.88	1.17	7.17	30.6	0.61
Varnish type									
Control	481 ab	307 a	589 a	305 a	329 a	307.6 a	346 a	602 b	92.89 a
Water-based	487 a	305.5 b	583 ab	304.4 a	316 ab	306.6 a	354 a	657 a	92.59 ab
Synthetic	474 b	304 ab	575 b	304 a	313 b	306.8 a	352 a	618 b	91.89 b
Avg.	6.22	2.22	6.30	1.14	12.88	1.17	7.17	30.6	0.61
S_x	4.04	1.73	8.08	0.58	9.24	177.59	3.46	9.24	0.58
LSD	7.62	2.72	7.72	1.39	15.79	1.44	8.78	37.46	0.75
Impregnate									
Control	479 b	305.3 a	578 b	304.6 ab	302 b	307 a	352 ab	649 a	92.14 b
Tanalith-E	477 b	304 a	580 b	304 b	326 a	306.9 a	355 a	604 b	93.10 a
Wolmanit-CB	487 a	305.2 a	589 a	305 a	330 a	306.8 a	344 b	623 ab	92.12 b
Avg.	6.22	2.22	6.30	1.14	12.88	1.17	7.17	30.6	0.61
S_x	4.62	0.06	6.35	176.09	16.17	0.12	4.62	15.01	0.01
LSD	7.6	2.72	7.72	1.39	15.78	1.44	8.78	37.46	0.75

CT: Combustion temperature, LD: Light density, FT: Failure time, TCT: Total combustion time, Avg.: Average, Sx: Standard deviation.

materials was found out to be in Wolmanit-CB impregnated specimens 92.12% and regarding varnish type in synthetic varnish coated specimens 91.89% (Table 4).

According to combustion test results, highest measured average values regarding weight loss were obtained Tanalith-E impregnated control specimens (69.71%) and lowest values were obtained in borax impregnated control specimens (43.04%) [13]. According to obtained spruce wood results, the highest weight loss was obtained in Tanalith-E impregnated specimens (93.10%) and the lowest was obtained in Wolmanith-CB impregnated specimens (92.12%). The reason for this might be related to boron compounds in Wolmanith-CB.

As a result of performed analysis, weight loss ratios of spruce wood was found out to be lower in Wolmanith-CB impregnated and synthetic varnish coated specimens.

In the performed studies, higher temperature values were obtained in specimens impregnated with vegetable tanning materials compared to control group in combustion with flame source and without flame source stages [14]. When compared to control, our findings show similarity with the findings in the literature.

Weight losses as a result of combustion were given according to year, control group, impregnatives and varnish type in Figs. 1 and 2.

Weight loss of the control group was lower than that of the year group as seen in Fig. 1. As observed in Fig. 2, the lowest weight loss according to impregnative was seen in Wolmanit-CB impregnated specimens and according to varnish type, synthetic varnish type yielded lower results than water-based varnish type (Fig. 3).

As analyzed in Table 4; average value of total combustion and degradation duration is maximum in control specimens 626 s. and 351 s. respectively; degradation duration of Tanalith-E (355 s) is higher than that of Wolmanit-CB (344 s); combustion duration of Wolmanit-CB impregnated specimens (623 s.) is higher than that of Tanalith-E impregnated specimens (604 s.); when considered according to varnish types, combustion and degradation duration of water-base varnish coated specimens (657 s., 354 s. respectively) is higher than those of synthetic varnish coated specimens (618 s., 352 s. respectively).

Temperatures of combustion in combustion with flame source, without flame source and glowing of Tanalith-E impregnative (477 °C, 580 °C, 326 °C respectively) were lower than those of Wolmanit-CB impregnative (487 °C, 589 °C, 330 °C respectively); temperatures of combustion with flame source, without flame source and glowing in water-based varnish type (487 °C, 583 °C, 313 °C respectively) higher than those of synthetic varnish (474 °C, 575 °C, 313 °C respectively).

Average light densities of impregnated cedar wood in combustion with flame source, without flame source and glowing were maximum in year specimens, respectively 320 lux, 320 lux and 324 lux; light densities of Tanalith-E impregnative in combustion with flame source, without flame source and glowing (304 lux, 304 lux, 307 lux respectively) were lower than but close to those of Wolmanit-CB impregnative (305 lux, 305 lux, 307 lux respectively); light densities of water-based varnish type in combustion with flame source, without flame source and glowing (303 lux, 304 lux, 306 lux respectively) were lower than but close to those of synthetic varnish (304 lux, 304 lux, 307 lux respectively) (Table 4).

Average values and least significant difference (LSD) tests of gas analysis amounts belonging to combustion with flame source, without flame source and glowing of spruce wood specimens are calculated in Table 5.

Regarding average O_2 amounts in spruce wood specimens according to impregnative, the highest value was observed in Wolmanit-CB impregnated specimens of combustion with flame source 14% and lowest in Tanalith-E impregnated specimens of combustion without flame source 1.18%; regarding the average value according to varnish type, the highest value was observed in synthetic varnish coated specimens of combustion with flame source 13%. Due to high oxygen content in synthetic varnish applied specimens, fire-retardant properties were observed. Furthermore, water-based varnish coated specimens had a reducing effect in oxygen (O_2) amount. Depending on the oxygen amount, water-based varnish is thought to have an effect of accelerating combustion (Table 5).

As determined in Table 5, average CO_2 amounts based on combustion with flame source, without flame source and glowing according to impregnative were found to be the highest in Tanalith-E specimens of combustion without flame source 19% and lowest in Wolmanit-CB impregnated specimens of combustion with flame source 6.43%; according to varnish type, the highest

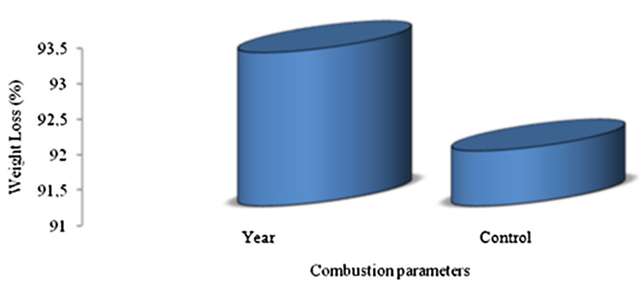

Fig. 2. Weight loss of annual and control samples.

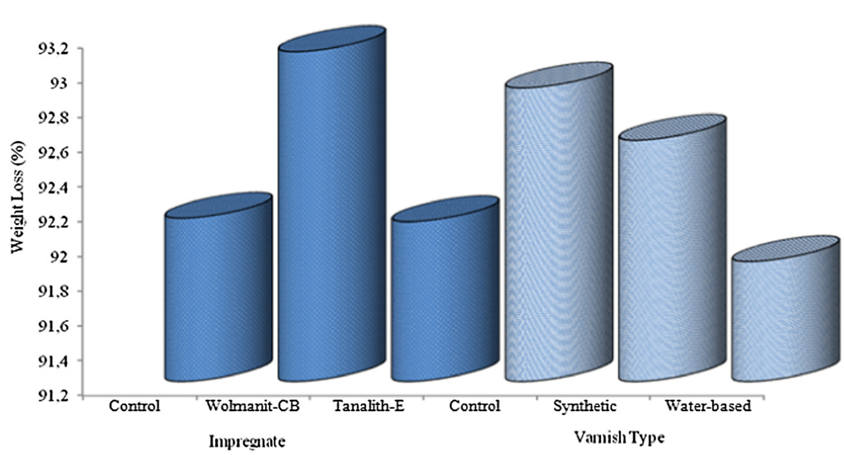

Fig. 3. Weight loss of impregnated and varnished spruce wood after combustion test.

Table 5
Groups formed according to average values and least significant difference (LSD) test of gas analysis amounts after combustion test of spruce wood.

	Factor	Combustion with flame source				Combustion without flame source				Glowing			
		CO_2 (%)	NO (ppm)	O_2 (%)	CO (ppm)	CO_2 (%)	NO (ppm)	O_2 (%)	CO (ppm)	CO_2 (%)	NO (ppm)	O_2 (%)	CO (ppm)
Year	Control	7.21 s	7.24 a	15.84 a	11,816 b	17.6 b	20.37 a	2.46 a	29,642 b	9.59 a	39.31 b	10.51 a	17,213 a
	Year	7.63 a	0.51 b	10.98 b	19,866 a	19.92 a	7 b	0.36 b	31,787 a	9.84 a	54. 65 a	10.74 a	15,017 b
	Avg.	6.43 a	3.88	13.41	15,841	18.76	13.69	1.40	30,715	9.72	46.98	10.63	16,115
	S_x	7.08	4.76	3.44	5692.21	1.64	9.45	1.48	1516.74	0.18	27.8	0.16	1552.81
	LSD	0.45	0.48	1.06	1926	0.62	1.94	0.63	1126	1.15	5.27	1.15	2193
Impregnate	Control	7.21 s	7.24 a	15.84 a	11,816 b	17.6 b	20.37 a	2.46 a	29,642 b	9.59 a	39.31 b	10.51 a	17,213 a
	Tanalith-E	7.63 a	0.51 b	10.98 b	19,866 a	19.92 a	7 b	0.36 b	31,787 a	9.84 a	54. 65 a	10.74 a	15,017 b
	Wolmanit-CB	6.43 a	3.88 a	13.41 a	15,841 b	18.76 a	13.69 b	1.40 b	30,715 b	9.72 a	46.98 a	10.63 a	16,115 a
	Avg.	7.08	4.76	3.44	5692.21	1.64	9.45	1.48	29,642 b	0.18	27.8	0.16	17,213 a
	S_x	0.45	0.48	1.06	1926	0.62	1.94	0.63	31,787 a	1.15	5.27	1.15	15,017 b
	LSD	1.23	3.44 b	13.25 a	15,235 a	19.11 a	12.37 b	0.96 b	30,715	10.18 a	41.08 b	10.19 a	16,115
Varnish Type	Control	7.21 s	7.24 a	15.84 a	11,816 b	17.6 b	20.37 a	2.46 a	29,642 b	9.59 a	39.31 b	10.51 a	17,213 a
	Water-based	7.63 a	0.51 b	10.98 b	19,866 a	19.92 a	7 b	0.36 b	31,787 a	9.84 a	54. 65 a	10.74 a	15,017 b
	Synthetic	6.43 a	3.88	13.41	15,841	18.76	13.69	1.40	30,715	9.72	46.98	10.63	16,115
	Avg.	7.08	4.76	3.44	5692.21	1.64	9.45	1.48	1516.74	0.18	27.8	0.16	17,213 a
	S_x	0.45	0.48	1.06	1926	0.62	1.94	0.63	1126	1.15	5.27	1.15	15,017 b
	LSD	1.23	3.44 b	13.25 a	15,235 a	19.11 a	12.37 b	0.96 b	31,207 a	10.18 a	41.08 b	10.19 a	16,115

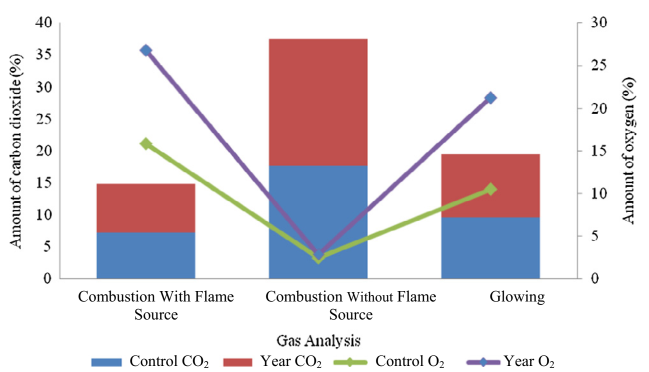

Fig. 4. Average values of O_2 and CO_2 amounts of year and control groups in combustion with flame source, without flame source and glowing.

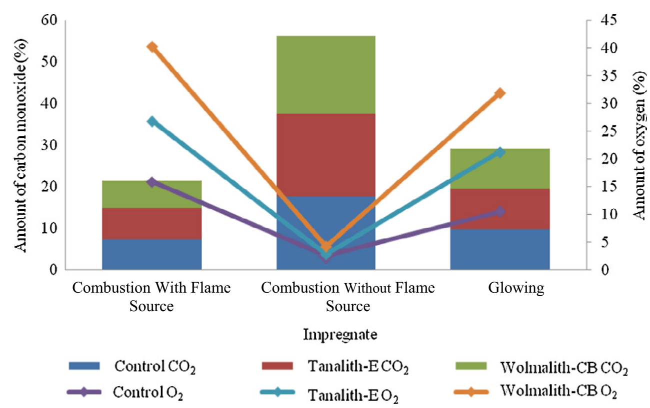

Fig. 5. Gas analysis amounts of impregnites in combustion with flame source, without flame source and glowing.

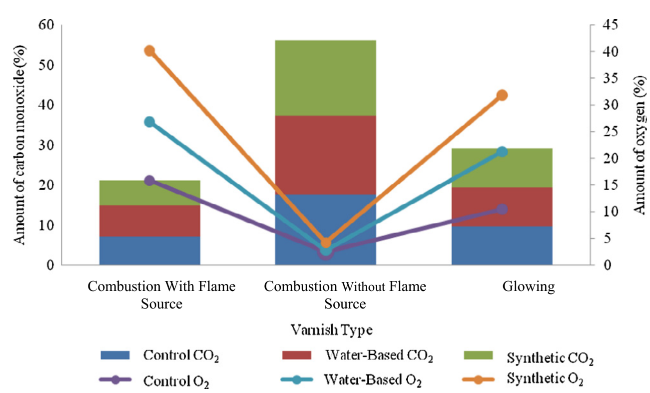

Fig. 6. Average values of gas analysis results of varnished spruce wood based on combustion with flame source, without flame source and glowing.

value was observed in synthetic varnish coated specimens of combustion without flame source 19% and lowest in control specimens of combustion with flame source 6%.

Regarding NO amounts based on combustion with flame source, without flame source and glowing, the highest value was observed in Wolmanit-CB impregnated specimens of glowing (51 ppm) and the lowest in Tanalith-E impregnated specimens of combustion with flame source (4 ppm); according to varnish type, the highest average value was observed in synthetic varnish coated specimens of glowing (49 ppm) and the lowest in synthetic varnish coated specimens of combustion with flame source (4 ppm) (Table 5).

In the literature, according to gas analysis data of the specimens obtained after combustion tests of surface treatments applied on sapele tree material, regarding average O_2 values obtained as a result of combustion test, the highest value 18.6% was observed in Boric acid impregnated and polyurethane varnish applied sapele wood and the lowest value 14.07% was observed in Tanalith E impregnated and water-based varnish applied sapele wood [13]. According to gas analysis results obtained from LVL specimens of laminated wood materials produced from yellow pine wood, since self combustion continues after the removal of flame source from the flame chimney, CO amount was observed to increase [15]. Our study shows similarities with these articles.

Average values of the gas analysis in terms of oxygen (O_2) and carbon dioxide (CO_2) in combustion with flame source, without flame source and glowing of year, control, impregnative and varnish types are given in Figs. 4–6.

According to this; the average values of the interaction between CO_2 amounts due to combustion with flame source, without flame source, glowing and year, impregnative, varnish type was found to be the exact opposite of the average values of interaction between O_2 amounts and varnish type, impregnative (Figs. 4–6).

4. Conclusions

According to average weight loss values measured as a result of combustion tests, the highest weight loss of spruce according to impregnative interaction was obtained in Tanalith-E impregnated specimens and the highest in Wolmanit-CB impregnated specimens. The reason for this is thought to be boron compounds in Wolmanit-CB impregnated specimens. According to varnish type interaction, highest weight loss was observed in control specimens and the lowest in synthetic varnish coated specimens. This situation may be interpreted as Wolmanith-CB impregnated and synthetic varnish coated specimens' having a fire-retardant property.

In combustion with flame source, the highest O_2 amount was observed in control specimen and the lowest in synthetic varnish coated specimens. In combustion with flame source, since varnished specimens burned less than the control specimen, decrease in O_2 amount was less compared to control specimen. In combustion without flame source and glowing, the highest O_2 amount was observed in Wolmanith-CB impregnated and water-based varnish coated specimens and the lowest in control specimens.

The average values of the interaction between CO_2 amounts due to combustion with flame source, without flame source, glowing and year, impregnative, varnish type was found to be the exact opposite of the average values of interaction between O_2 amounts and varnish type and impregnative.

The increase in CO amount, a sign of combustion, was found to be the highest in Tanalith-E treated specimens and lowest in Wolmanith-CB treated specimens. In combustion with flame source, the highest CO amount was observed in Tanalith-E treated specimens. In the control specimen, since self combustion continues after the removal of flame source from the flame chimney, CO amount was observed to increase. Other impregnatives showed a reducing effect in CO increase.

As a result, wooden materials to be used in places with fire hazard should better not be coated with varnish after impregnation. Use of Wolmanith-CB for impregnative and synthetic varnish for the varnish can be advised.

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