Evaluating Combustion Characteristics of Impregnated Scots Pine Wood: Effects of Fire-retardant Coatings

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This study investigated the effects of fire-retardant coating treatments on the combustion resistance of Scots pine wood (*Pinus sylvestris* L.) impregnated with various chemicals. Borax, boric acid, zinc chloride, sodium silicate, and ammonium sulfate were used as impregnation materials. Fire-retardant paint and nano-enabled fire-retardant varnish were applied as coating materials. Combustion tests were conducted using a computer-controlled apparatus developed in accordance with the ASTM E69-22 (2022) standard. During combustion, mass loss and maximum temperature values were recorded every 30 s. As a result, impregnation applied to the Scots pine wood material was observed to reduce the mass loss, including 31% borax, 37% boric acid, 43% zinc chloride, 35% ammonium sulfate, and 31% sodium silicate. It was observed that coating formulation to the Scots pine wood decreased mass loss 59% with nano varnish and 71% with fire-retardant paint.

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Keywords: Fire retardant; Combustion; Scots pine wood; Coating

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INTRODUCTION

Wood is a natural material known as the oldest and unchanging building material. Research is carried out on a wide variety of properties of wood materials. Among the most important of these is fire safety. Combustion research provides important information for risk assessment. Most academic studies have examined wood species and reported significant differences between species (Budakçı *et al.* 2016; Seo *et al.* 2016; Özcan *et al.* 2016; Song *et al.* 2020; Kobelev *et al.* 2023; Mensah *et al.* 2023). Similarly, commercial fertilizers, such as diammonium phosphate and ammonium sulfate, improve the combustion characteristics of Calabrian pine, with higher concentrations giving better results (Baysal 2011).

Research on the combustion properties of varnished wood has shown that impregnation with boron compounds (boric acid and borax) generally decreases the combustion temperature, while varnishing tends to increase it (Atar and Keskin 2007; Atar 2008; Atar *et al.* 2012). The combination of polyurethane varnish and boric acid has been reported to result in the highest mass loss during combustion (Yapıcı *et al.* 2011).

There has been research focused on improving combustion protection for wood through special varnishes and coatings. Two-component intumescent varnishes show promise in reducing the flammability of wood by forming a heat-insulating coke layer when exposed to high temperatures (Tsapko *et al.* 2022). These coatings can significantly reduce the temperature of flue gases, limit surface damage, and reduce weight loss during combustion tests.

Fire retardant varnishes containing optimized inorganic components are effective in preventing ignition and flame spread (Tsapko *et al.* 2021). The studies also investigated the thermal conductivity of fire-retardant coking foam layers and found a coefficient of 0.36 W/(m·K), which contributes to the heat resistance of wood (Tsapko *et al.* 2020). Paint and varnish treatments are considered innovative methods for wood fire protection, creating thin protective layers that can achieve two levels of fire-retardant efficiency (Sethurajaperumal *et al.* 2021).

Synthetic varnish applied on chestnut wood resulted in lower combustion temperatures after outdoor exposure than water-based varnish. Impregnation with Wolmanit-CB resulted in shorter combustion and settling times than Tanalith-E (Fidan *et al.* 2016). For Eastern beech, the combination of boric acid and polyurethane varnish caused the highest mass loss during combustion (Yapıcı *et al.* 2011). Combustion values of some varnishes applied on oak wood decreased the total combustion time by 19 to 21% and increased the destruction time by 10 to 22%. Water-based varnish samples reached the highest combustion temperatures, while synthetic varnish samples had the lowest temperatures during combustion (Yaşar and Atar 2018). The effects of varnishes and impregnates applied to various wood materials on combustion resistance were investigated. The most critical factors in combustion resistance were CO_2 emission, weight loss, and temperature values (Özcan *et al.* 2012; Atar *et al* 2017; Tsapko *et al.* 2022).

Wood should be prioritized as a preferred building material over others. However, to ensure the safe transfer of wooden structures with historical and cultural significance to future generations as ‘cultural heritage,’ it is essential to implement effective fire prevention measures and enhance their fire resistance properties.

Academic studies have mostly focused on impregnating materials or coating processes (Bozkurt et al. 1993; Ayrılmış 2006). Studies focusing on both impregnating materials and coating formulation remain limited. One of the most important points of this study is the preference for coating formulation with fire retardant properties.

In this study, the effects of fire-retardant paint and varnish with different impregnating materials on the fire resistance of Scots pine wood materials were investigated according to the principles outlined in the ASTM E69-22 (2022) standard.

EXPERIMENTAL

Wood Material

Scots pine wood used in the tests was provided using a “random selection” method from timber suppliers market of Karabuk city. Supplied wood material was dried until it was air-dry. Then, rough cutting was made of wood according to the future experiments. The collation process was made to represent the control group and the other groups. The authors ensured the wood material was knotless, resin free, free of growth defects, solid, straight grained, and to be part of the sapwood.

Impregnation Materials

In the experiment samples, borax, boric acid, ammonium sulfate, zinc chloride, and sodium silicate were used as fire retardants (inorganic chemicals). The chemical substances used were prepared as solutions in distilled water at room temperature (20 ± 3 °C). These chemicals properties and applications ratios are given table 1.

Table 1. Peculiarities of Impregnation Chemicals and Test Plan

Impregnation Chemicals	Solution Conc. (%)	Solvent	Purity (%)	pH		Density (g/mL)	
				BI	AI	BI	AI
Ammonium Sulfate	5	Pure water	97	6	5.5	1.05	1.06
Borax	5	Pure water	98	9.12	9.15	1.08	1.10
Boric Acid	5	Pure water	98	5.23	5.30	1.02	1.02
Zinc chloride	5	Pure water	99	6	5.5	1.07	1.07
Sodium silicate	10	Pure water	98	11.12	11.04	0.94	0.96

BI: Before impregnation AI: After impregnation

Coating Materials

This study used fire-retardant paint and nano-enabled fire-retardant varnish as coating materials. The fire-retardant paint was a polyurethane-based paint with fire retardant. This coating is a (INTUMES) type of material that is used to insulate or separate combustible material from heat and form a foam structure when heated. The nano-varnish was a polyurethane-based varnish with a fire retardant. It was a fire-retardant varnish system with no toxic smoke extraction that reduces the rate of progression by preventing the immediate ignition of fire. The Ecelak company of Izmir city supplied all coating materials used in experiments. The manufacturer's recommendations were considered when applying paint and varnish.

Preparation of Test Samples

Examples selected from the Scots pine wood in the preparation of test samples were prepared according to the TS ISO 3129 (2021) standard. Test specimen dimensions were cut properly in 9.5 x 19 x 1016 mm ± 0.8 mm in size according to ASTM E69-22 (2022). Impregnated and coating formulation Scots pine wood material and 6 test samples for control were prepared. Accordingly, 6 × 3 × 6 = 108 pieces of Scots pine wood material were prepared.

According to ASTM E69-22 (2022), the moisture of the material to be tested should be 12 ± 3% relative to its absolute dry weight. For this, samples were dried until 12% moisture balance occurred at 20 °C ± 2 °C and 65 ± 5% relative humidity in a conditioned cabinet.

Specific Weights

The moisture of the samples was determined in accordance with TS ISO 13061-1 (2021), and their densities in accordance with TS ISO 13061-2 (2021). Accordingly, test samples were weighed with a 0.001 g precision analytical scale after reaching fixed weight and dimensional stability by keeping in a conditioning cabinet at 20 °C ± 2 °C and 65 ± 3% relative humidity. After their volumes were determined by measuring the dimensions with a ± 0.01 mm precision caliper, the weight in air dry condition (M_{12}) and density value (ρ_{12}) according to volume (V_{12}) were calculated with the following equation:

$$\delta_{12} = M_{12} / V_{12} \text{ g/cm}^3 \quad (1)$$

Impregnation

The samples were dipped into the impregnation solution (having packing viscosity) for 48 h for a long-term period, provided the samples met the criteria of TS 344. Before the impregnation process, all samples were weighed and then kiln-dried at the temperature of 103 ± 2 °C until they reached constant weight. Then, the samples were weighed in an analytical balance with 0.01-g sensitivity. After impregnation, all impregnated samples were held for 15 days in circulating air for solvent evaporation. Impregnated test samples were kept at a temperature of 20 ± 2 °C and 65 ± 3% relative humidity until they reached constant weight.

After this period, the principles specified in TS 345 (2022) were applied to determine the impregnant amount of retention. The impregnated samples were kiln-dried at 103 ± 2 °C until they reached a constant weight. After cooling, all dried samples in the desiccator were weighed on the scale. The dry weight of the samples was determined and recorded. The ratio of retention (R, %) were calculated as follows:

$$R(\%) = \frac{Mdi - Md}{Md} \times 100 \quad (2)$$

where M_{di} is the sample dry weight after impregnation (g) and M_d is the sample dry weight before impregnation (g).

The sapwood-derived wood samples were conditioned in a climate-controlled environment at 20 ± 2 °C and 65 ± 3% relative humidity. This process continued for two weeks, until the samples reached a constant weight under these standardized conditions.

Coating Formulation

Two different coating formulation materials, namely fire-retardant varnish and paint, were applied in accordance with ASTM D3023-98 (2024) standards. The preparation and application of the top coating formulation materials were carried out by considering the hardeners, thinners, or diluents and mixing ratios recommended by the manufacturers and were applied to the wood test samples with a roller. Again, in accordance with the manufacturer's recommendations, after waiting 24 hours, the samples were sanded with 180-grit sandpaper, the final top surface treatment was applied, and they were left to dry for three weeks at a temperature of 20 ± 2°C and a relative humidity of 65 ± 5% for 12% moisture content, and were then ready for the burning tests.

Layer Thickness of Varnish Dry Film

The thicker layer of varnish applied over wood is an essential factor in comparative testing. The layer thickness of the varnish put on the test samples and fully dried was measured by comparators with 5 mm (microns) precision.

Combustion Test

Combustion tests were made with a computer-controlled wood combustion apparatus prepared according to the principles of ASTM E69-22 (2022). Test specimens were prepared with dimensions of 9.5x19x1016mm ± 0.8mm. Butane gas was used to make an ignition flame. The gas flow was standardized to give a flame height of 25 cm, and the temperature must be 1000 C. The distance between the bottoms of the test samples, which were hung inside of the fire tube and the top of the gas pipe, were adjusted as 2.54 cm. During the test, mass loss and temperatures were determined each 30 seconds. Combustion testing continued for a total of 10 min for each sample, including 4 min with a flame source and 6 min without a flame source. The temperature limit was determined by using temperature sensors on the apparatus. The computer-controlled combustion system measures weight, heat, and humidity, transferring this data to a computer environment in real time for processing and storage. The computer-controlled combustion device used in the combustion test experiments and its principle are given in Fig. 1.

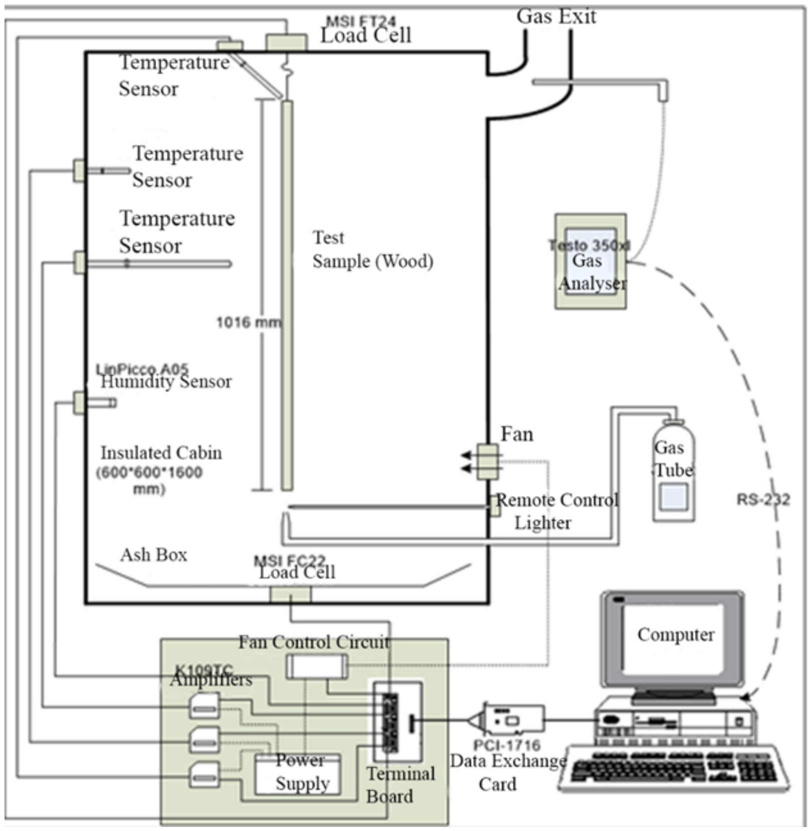

Fig. 1. Computer controlled combustion test setup (Özcan 2011)

Data Analyses

A total of 108 wood samples were systematically prepared to investigate the effects of the coating formulation on fire retardancy. The samples were derived from the combination of five distinct impregnation chemicals and two types of coating materials. Each treatment group included six replicates, resulting in a complete factorial design (5 impregnation material × 2 coating material × 6 replicates = 108 samples). Analysis of variance (ANOVA) was employed to evaluate the statistical differences among the experimental groups, followed by applying Duncan's multiple range test to determine the significance levels of the observed variations. Statistical analyses were performed using Minitab 17.3.1.0 (Stat Ease, State College, PA, USA).

RESULTS AND DISCUSSION

Result on Wood Material, Varnishes, and Impregnations

The results of the measurements taken for Scots pine used as wood material, borax, boric acid, zinc chloride, ammonium sulfate, sodium silicate used as impregnating compounds, and fire-retardant paint and varnish used as coating formulation are given below.

Scots pine wood's absolute dry specific weight was 0.48 g/cm³, and the air-dry specific weight was 0.52 g/cm³. The layer thickness of fire-retardant paint applied over Scots pine wood was 104 µm, and the layer thickness of nano varnish was 110 µm. The retention values obtained because of impregnation of test samples prepared from Scots pine wood material with the prolonged immersion method were borax 1,76% boric acid 2.49%, zinc chloride 1.34%, ammonium sulfate 1.62%, and sodium silicate 2.72%.

Combustion Test Results

Mass loss values (%)

The average values of % mass loss measurements obtained in combustion test results of Scots pine wood material with control, nano-varnished, and fire-retardant painted samples are given in Table 2, and multiple variance analysis results are shown in Table 3.

In Table 2, based on the results of the % mass loss of the control (coating formulation not applied) samples, maximum mass loss after combustion with flame source was detected in control (not impregnated) samples with 48.3%, whereas minimum mass loss was detected in samples impregnated with zinc chloride with 20.2%. Maximum mass loss from combustion was detected in the control samples with 98.2% and minimum mass loss was measured in samples impregnated with zinc chloride with 28.4%.

Table 2. Average Values of Mass loss (%)

Coating Formulation	Impregnation Materials	End of Combustion with Flame Source (8th measurement)	End of Combustion (20th measurement)
Control	Control	48.26	98.19
	Boric Acid	28.53	38.12
	Zinc Chloride	20.19	28.46
	Ammonium Sulfate	25.53	37.19
	Sodium Silicate	24.16	33.49
	Borax	27.53	39.16
Nano varnish	Control	18.23	40.11
	Boric Acid	17.03	29.36
	Zinc Chloride	23.56	28.92
	Ammonium Sulfate	18.26	30.43
	Sodium Silicate	26.18	36.39
	Borax	22.48	31.06
Paint	Control	13.99	28.82
	Boric Acid	13.46	21.83
	Zinc Chloride	12.46	20.11
	Ammonium Sulfate	16.45	23.41
	Sodium Silicate	19.86	23.94
	Borax	17.23	26.49

8th : As a result of flame-induced combustion 20th : As a result of combustion

According to the % mass loss results of nano-varnished samples, maximum mass loss after combustion with flame source was detected in samples impregnated with sodium silicate (26.2%), whereas minimum mass loss was detected in samples impregnated with boric acid (17.0%). The maximum mass loss as a result of combustion was detected in control (not impregnated) samples with 40.1%, and the minimum mass loss was detected in samples impregnated with zinc chloride (28.9%).

Based on the % mass loss results of the samples with fire-retardant paint, maximum mass loss after combustion with flame source was detected in samples impregnated with sodium silicate (19.8%), and minimum mass loss was detected in samples impregnated with zinc chloride (12.5%). The maximum mass loss as a result of combustion was detected in control samples (28.8%), and the minimum mass loss was detected in samples impregnated with zinc chloride with 20.11% (Table 2).

According to the results of multivariate analysis of variance in Table 3, the impact of coating, impregnation, and measurement time on % mass loss values was significant, and the effect of all other interactions was simultaneously substantial.

Table 3. Multiple Variance Analysis of the Average Values of Mass Loss

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Factor A	75615.531	2	37807.766	73291.956	0.000
Factor B	50190.167	5	10038.033	19459.153	0.000
Factor C	237601.884	19	12505.362	24242.175	0.000
A*B	90330.011	10	9033.001	17510.856	0.000
A*C	24237.137	38	637.819	1236.440	0.000
B*C	23826.178	95	250.802	486.190	0.000
A*B*C	41337.208	190	217.564	421.758	0.000

Factor A = Coating type, Factor B = Impregnate materials, Factor C = Time of measurement

Duncan test results performed to determine the significance level of the value exchange of % mass loss depending on the coating formulation and impregnation are given in Table 4. While the values in the table evaluate the measurement time in general, they show the variation of coating formulation and impregnation on % mass loss.

Table 4. Duncan Test Results of Mass Loss ($p \leq 0.05$) *

Impregnation Materials	Coating Formulation					
	Control		Nano varnish		Paint	
	Average	HG	Average	HG	Average	HG
Borax	39.16	kl	31.06	g	26.49	d
Boric acid	38.12	jk	29.36	ef	21.83	b
Zinc chloride	28.46	e	28.93	e	20.11	a
Ammonium sulfate	37.19	ij	30.43	fg	23.41	c
Sodium silicate	33.49	h	36.39	i	23.94	c
Control	98.19	m	40.11	l	28.82	e

HG: Homogeneity group

Statistical comparison of impregnation materials and surface processes showed significant differences in mean values of % mass loss of control, Scots pine wood material group coating formulation with fire-retardant paint, and nano varnish. As a result of the statistical comparison of impregnation materials in interactions, there were significant differences between the control groups of all impregnation materials. Zinc chloride impregnation material prevented an increase in mass loss of test samples; similar results were obtained from academic research (Uysal and Kurt 2006; Kurt and Uysal 2010; Foppiano *et al.* 2018; He *et al.* 2019; Silva *et al.* 2020). Zinc chloride impregnation may have slowed the degradation of the wood material by combustion. Mass loss values were lower because samples impregnated with zinc chloride had higher ignition temperatures.

Fire-retardant nano-varnished samples generally showed lower mass loss values than the control (without coating formulation). However, fire-retardant painted samples showed much lower mass loss values than fire-retardant nano-varnished samples. The charred layer formed by the fire-retardant paint limited the direct contact of oxygen with the wood material. This layer may have created resistance to burning by preventing the spread of flames. Academic studies have shown that coating treatments positively affect combustion resistance (Mariappan 2017; Olimat *et al.* 2020).

Temperature Values

Average values obtained from the combustion tests of the control and nano-varnished fire-retardant paint samples of Scots pine wood materials are given in Table 5, and the multiple variance analysis results are shown in Table 6.

Table 5. Average Values of Temperature (ºC)

Coating Formulation	Impregnation Materials	End of Combustion with Flame Source (8th measurement)	End of Combustion (20th measurement)
Control	Borax	226	151
	Boric Acid	230	155
	Zinc Chloride	161	85
	Ammonium Sulfate	163	102
	Sodium Silicate	170	95
	Control	229	230
Nano varnish	Borax	168	98
	Boric Acid	153	94
	Zinc Chloride	173	87
	Ammonium Sulfate	176	91
	Sodium Silicate	172	110
	Control	185	135
Paint	Borax	173	92
	Boric Acid	150	92
	Zinc Chloride	142	84
	Ammonium Sulfate	151	87
	Sodium Silicate	152	81
	Control	196	91

8th : As a result of flame-induced combustion; 20th : As a result of combustion

According to the results obtained from Table 5, while the control (not impregnated) samples (13 measurements) had the highest temperature limit with 542 °C, the lowest temperature was detected in the samples (8 measurements) impregnated with zinc chloride with 161 °C.

According to the temperature results obtained from combustion tests of nano varnish samples, maximum temperature was detected in control and impregnated samples (8th measurements) with 185 °C, and the minimum temperature was detected in samples (8th measurements) impregnated with boric acid with 153 ° C.

According to the temperature results obtained from combustion test of samples with fire-retardant paint, maximum temperature was detected in control samples (8th measurements) with 196 °C, and minimum temperature is detected in the samples (8th measurements) impregnated with zinc chloride with 142 C (Table 5).

Table 6. Multiple Variance Analysis of the Average Values of Temperature

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Factor A	893318.926	2	446659.463	8509.657	0.000
Factor B	1378371.275	5	275674.255	5252.085	0.000
Factor C	1403642.149	19	73875.903	1407.467	0.000
A*B	1103419.222	10	110341.922	2102.210	0.000
A*C	445692.499	38	11728.750	223.454	0.000
B*C	698859.311	95	7356.414	140.153	0.000
A*B*C	1269594.326	190	6682.075	127.305	0.000

Factor A = Coating type, Factor B = Impregnate materials, Factor C = Time of measurement

According to Table 6, the impact of coating formulation, impregnation, and measurement time on % mass loss values was significant, and the effect of all other interactions was significant.

Duncan's test was performed to determine the significance level of the value exchange of temperature depending on the coating formulation and impregnation, and the results are given in Table 7. While the values in the table evaluate the measurement time in general, they show the variation of coating formulation and impregnation with temperature.

Table 7. Duncan Test Results of Temperature ($p \leq 0.05$)*

Impregnation Materials	Coating Formulation					
	Control		Nano varnish		Paint	
	Average	HG	Average	HG	Average	HG
Borax	181	g	149	f	128	cde
Boric acid	172	g	125	bcde	118	abcd
Zinc chloride	117	abc	125	bcde	107	a
Ammonium sulfate	129	cde	136	e	114	ab
Sodium silicate	127	bcde	130	de	109	a
Control	283	h	154	f	136	e

HG: Homogeneity group

When examining the results of the statistical comparison of the impregnation type and coating formulation of Scots pine wood material, it was observed that there were statistical differences in the average temperature values of coating formulation with fire-retardant paint and nano varnish. As a result of the statistical comparison of impregnation materials in interactions, it was observed that there were significant differences between the control groups of all impregnation materials. While there was a difference between nano varnish groups, no significant difference was detected in fire-retardant paint groups.

Zinc chloride-impregnated samples were found to give the best values in both control and coating formulation samples. Non-impregnated samples were found to give negative results in all three groups. These findings show that using impregnation resulted in lower temperature values in both coating formulation and control samples. Below the combustion temperature, zinc chloride may have formed a protective layer on the surface of the wood material, preventing the heat from penetrating inside. These results are in parallel with previous studies (Özcan 2019; Garrido *et al.* 2022; Zhengbin *et al.* 2019).

CONCLUSIONS

Impregnation applied to Scots pine wood resulted in mass loss rates of 31% with borax, 37% with boric acid, 43% with zinc chloride, 35% with ammonium sulfate, and 31% with sodium silicate. It was observed that coating formulation to Scots pine wood decreased mass loss by 59% with nano varnish and 71% with fire-retardant paint.

1. When the mass loss values were compared according to coating materials, fire retardant paint samples gave the lowest mass loss values.
2. When the mass loss values were compared according to impregnation materials, in general, zinc chloride samples gave the lowest mass loss values. However, zinc chloride resulted in the lowest mass loss in control samples with no coating formulation.
3. Nano varnished control (not impregnated) samples yielded better results. These results showed that it is unnecessary to apply impregnation to the wood surface that will be nano-varnished. However, this cannot be considered for fire-retardant paint.
4. Temperature values generally gave the highest results in the 8th measurement. The transition from combustion with a flame source to combustion without a flame source after the 8th measurement is considered to be the reason.
5. Temperature values gave the highest results in control samples (not impregnated and coating) in the 13th measurement, which was the reason for the continuation of the combustion.
6. Temperature values were in parallel with mass loss values in general. Temperature values increased as mass loss values increased.
7. It is recommended that zinc chloride be used for the impregnation of Scots pine wood material.
8. Fire-retardant paint is recommended to coating formulation Scots pine wood material.
9. For structural elements that will be exposed to outdoor conditions, it is recommended that Scots pine material used in the construction of garden and city furniture be impregnated with zinc chloride and that fire-retardant paint be used on the upper surface.

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