

Environment-Friendly Waterborne Fire Retardants for Protection of Wood and Bark against Fire Flames

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Flame retardancy was induced in maple (*Acer velutinum*) and ash (*Fraxinus excelsior*) wood and bark by means of mixtures consisting of bio-based materials such as starch (S) and glue (G) and water-based paint, MINWAX (M) in two different combined formulations with perlite (P) as the main fire retardant. The selected wood species as solid wood with two different treatable surfaces (with and without bark) were examined. The lowest and the highest mass loss occurred in the untreated-ash and untreated-maple wood samples with bark, while the lowest weight percent gain was related to PSGM-treated ash wood samples with bark, and the highest weight percent gain was related to PSGM-treated maple wood samples without bark. The lowest time to ignition and glowing point time were measured in the untreated-maple wood samples without bark, and the highest of them were measured in the PSGM-treated ash wood samples without bark. The effect of bark in the treated- and untreated-maple samples on the time to ignition and glowing point time was greater than the bark of treated- and untreated-ash samples, respectively. There was not any significant relationship between actual retention, weight percent gain, and mass loss for all treatments. However, there was significant difference between the individual and interaction agents on fire retardancy of treated and untreated samples.

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INTRODUCTION

Fire is one of the most important physically destructive factors of solid wood. Therefore, wood as a raw material must be protected against the fire. Wood as an eco-friendly and renewable natural material has been extensively protected through various fire retardants (FRs) and antiseptic fire resistance (Elvira-León *et al.* 2016; Gazizov *et al.* 2018a, b; Gazizov and Ivanchina 2018; Kmet'ová *et al.* 2022). Protection of solid wood against fire are performed with help of two methods, such as superficial and deep impregnation with special compositions containing FRs, as well as it is also common to use fire-resistant coatings (Östman *et al.* 2010).

Deep impregnation is more effective than surface impregnation, so that deeper penetration of fireproof solutions into the surface layers of wood is possible *via* hot and cold baths, as well as processing with help of industrial apparatus or autoclave in the

mode of successive increase in pressure or alternation of vacuum and increased pressure (vacuum impregnation) (Khvatov *et al.* 2019).

Currently, the use of natural mineral fillers, such as perlite, vermiculite, and expanded perlite and vermiculite (Wang *et al.* 2016; Szadkowski *et al.* 2020; Lanzón *et al.* 2022), and binders, such as gypsum, geopolymers, and starch, are also effective and have significant effects on flame retardancy of bio-based composite panels and wood (Manzello *et al.* 2007; Bumanis *et al.* 2020).

The characteristics of perlite are light weight, thermal insulation, and fire resistance. These characteristics prompt its use in applications such as heat storage (Zhang *et al.* 2016) and dye decolourisation (Pezzella *et al.* 2014). Therefore, perlite has been applied to prepare waterborne fire resistive coatings (Huang *et al.* 2020), and expanded perlite may decrease the plaster fire protection, especially if used in high doses (Lanzón *et al.* 2022).

Tsuyumoto *et al.* (2011) found a significant flame retardancy effect from starch and sodium polyborate (SPB) mixtures. They prepared the effective mixture as a flame retardant by simple coating with rigid polyurethane foam, nonwoven polyethylene terephthalate/ethylene vinyl alcohol copolymer, and nonwoven polypropylene.

One of the applications of FR paints is in combustible materials, such as wood, foam, and plastic, which are considered to reduce the rate of flame spread. These materials are based on silicone, casein, or vinyl resins. They are similar in paints, and their formulation is such that the tools used for them (brush, roller, or spray) should be the same as paints (Kusumgar *et al.* 2007).

The plant glue (Serish) is taken from the plants of the Asphodelaceae family. The root of *Eremurus persicus* plant (Serish) is a well-known glue of plant origin in Iran that has been used for centuries as natural glue in the textile, carpentry, binding, and book restoration industries. Thin layer chromatographic (TLC) analysis of ethanolic extract of *E. persicus* root showed that it contains polyphenols, flavonoids, and naphthoquinones (Beiranvand and Beiranvand 2021). People traditionally collected the roots, dried and powdered them, and mixed them with water to make glue (Koohkesh *et al.* 2020). Additionally, these materials are economical compared to chemical materials and can be easily purchased as raw materials in the market. The *E. persicus* essential oil is rich in terpenes and oxygenated terpene derivatives. Individually, limonene (16.2%), geranylgeraniol (15.2%), n-nonanal (9.5%), geranyl acetone (9.1%), benzene acetaldehyde (8.5%), linalool (7.9%), α -pinene (6.9%), and 1,8-cineol (5.2%) were the most abundant volatile compounds (Salehi *et al.* 2017). The roots of *Eremurus* species are rich of oligo and polysaccharides, including branched arabinogalactan, linear galactomannan, and short chains of fructose units with a single d-glucosyl unit at the nonreducing end that accumulate during their growth (Flamm *et al.* 2001; Karaman *et al.* 2011; Muhidinov *et al.* 2020; Pourfarzad *et al.* 2015; Smirnova *et al.* 2001).

Maple wood has a special place in the furniture and upholstery industry, and it is also suitable for flooring and making plywood and shoe molds, covering large surfaces of walls and tabletops, and sculpting (Golbabaei and Ebrahimi 2015; Naghdi *et al.* 2016). Common ash (*Fraxinus excelsior* L.) is a medium-sized deciduous hardwood tree and belongs to the Oleaceae family. This tree is mostly native to Europe and is distributed in northern Scandinavia and the southern Iberian Peninsula. Its bark is smooth and gray at first, then it has vertical cracks in the middle and its color becomes blackish gray. Ash wood has good strength and hardness and good elasticity (Azadi 2005; Beck *et al.* 2016).

The bark protects the tree from weather conditions, insect pests, and browsing, and it also has a crucial role in wood fires. Much research has been conducted on this subject. Schafer *et al.* (2015), Catry *et al.* (2010), Lawes *et al.* (2011), Wang and Wangen (2011), Do Vale and Elias (2014), Dickinson and Johnson (2001), and Dickinson (2002) established that the relative thickness of the bark significantly influences the survival of trees in a fire. Bauer *et al.* (2010) and Hengst and Dawson (1993) examined the probability of survival for a tree if its surface is exposed to fire. They found that the fire resistance of the bark depends on thickness and moisture content, and that the different physical qualities of tree species have a negligible influencing role. Bauer *et al.* (2010) mentioned that the transfer potential provided for biomimetic heat insulation and fire-stopping behavior is found in many species of tree bark.

This study aims to evaluate the effect of perlite (P), starch (S), glue (G), and plastic paint, MINWAX (M) in combined formulations on the fire behavior of wood that are compared to control samples. This matter has received little attention in previous studies. It is not clear whether the use of two different formulations of natural compounds: 1) mixture of P, S, and G in water solution; 2) mixture of P, S, G, and M in soluble to water, are beneficial for the protection of wood and cellulosic materials against fire. The authors' hypothesis is that in order to delay the fire, the fluid slurry resulting from the potential formulation of these materials can be easily applied and sprayed on standing trees in the forest and close to the house, pastures, and grasslands before and during exposure to fire.

In this study, the fire retardancy properties, such as mass loss (ML), flame point or time to ignition (TTI), glowing point time (GPT), weight percent gain (WPG), actual retention (AR) of velvet maple- and common ash-treated wood with environment-friendly waterborne FRs in two treatable surfaces with bark and without bark, were evaluated and compared with control samples.

EXPERIMENTAL

Materials

Perlite, starch, glue, and MINWAX

Raw perlite (3 kg) as the one of fire retardants was supplied and prepared from the Pars Chemical Company, Tehran, Iran (Fig. 1a). The material was air-dried for several days and ground into small pieces (powder) and sieved between 60- and 80-mesh screens. Then, it was weighed and bagged, so that the moisture content of perlite powder was around $10 \pm 2\%$ when used. The ingredients of perlite are: 70 to 75% silicon dioxide (SiO_2), 12 to 15% aluminium oxide (Al_2O_3), 3 to 4% sodium oxide (Na_2O), 3 to 5% potassium oxide (K_2O), 0.5 to 2% iron oxide (Fe_2O_3), 0.2 to 0.7% magnesium oxide (MgO), 0.5 to 1.5% calcium oxide (CaO), and 3 to 5% loss on ignition (chemical/combined water) (Arifuzzaman and Kim 2017).

In this research, corn starch polymer produced by Mehshad Company, Yazd, Iran, with a melt flow index of 3 g/10 min and a density of 1.3 g/cm^3 was used (Fig. 1b). Glue is also a natural binder that is extracted from plant and animal elements. The plant glue (Serish powder) used in this study has also been used by gluing industries (Fig. 1c). The water-based paint with MINWAX trademark was used in this study (Fig. 1d).

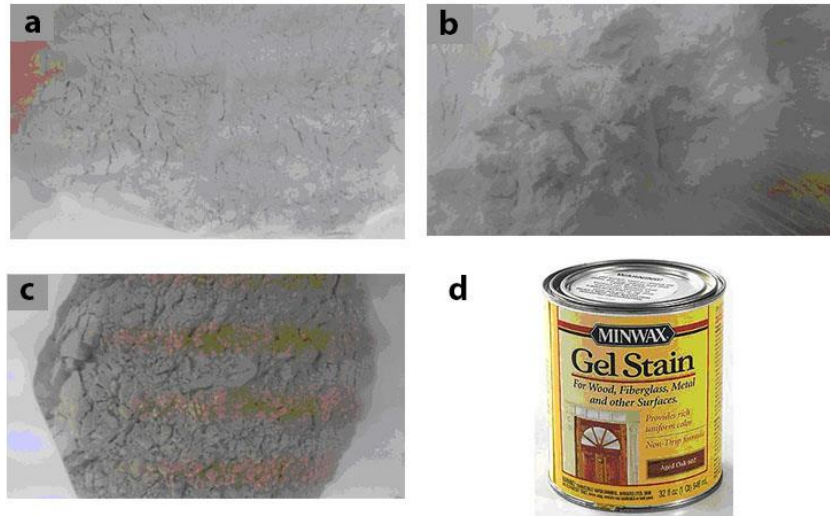


Fig. 1. The materials used in this study: a: perlite; b: starch; c: glue (Serish); d: MINWAX paint

Preparation of Test Specimens

Defect-free maple (*Acer velutinum* Boiss.) (AV) and ash (*Fraxinus excelsior* L.) (FE) woods with and without bark were first cut into $20 \times 15 \times 1 \text{ cm}^3$ (L \times T \times R) blocks and prepared according to the EN ISO 11925-2 (2010) standard (Fig. 2). All specimens were oven-dried at $103 \pm 2 \text{ }^\circ\text{C}$ for 48 h before and after treatment.

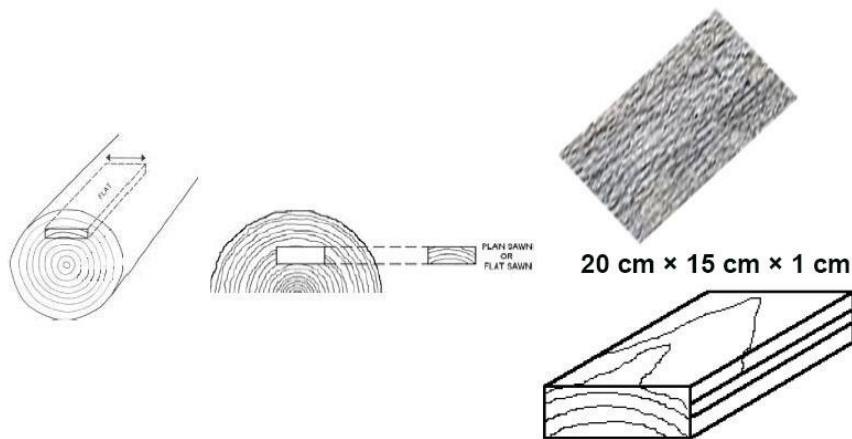


Fig. 2. Preparation of wood specimens with and without bark from the log

Independent variables and their levels were as follows:

- Species: maple (*A. velutinum* Boiss.) and ash (*F. excelsior* L.)
- Treatable surfaces: wood with bark and wood without bark
- Fire retardants: 1) Formulation PSG with a concentration of 22.5%; 2) formulation PSGM with a concentration of 42.5% as the treating materials and untreated (UT) as the control samples were used (Table 1).

The prepared specimens were impregnated with two different solutions having the abovementioned compositions. Preparation of specimens and measurement of the fire retardancy parameters of wood were completed according to JISA-1322 (1982) and BS 476-6 (1997) standards, respectively. The fire test was a single flame source test.

Manufacturing Process

The initial experiments were done by trial and error to achieve an effective natural formulation that has the best efficiency in delaying the TTI point (flaming time) and GPT on wood. In this way, a new combination of perlite with bio-based binder materials such as starch and a natural adhesive material such as glue (Serish powder) was obtained by dissolving in a certain volume of water. This natural compound is easily dissolved in water and a simple formulation with flame retardant properties was obtained.

Changes were made in the amount of P, S, G, and M in the formulation of flame retardant. In experiment number 1, the untreated samples were tested. In experiment number 2, the samples were treated with a solution of P (as the main flame retardant) and starch (as the first adhesive). In experiment number 3, the samples were treated with a solution of P and glue (as the second adhesive). In experiment number 4, the samples were treated with a solution of perlite, starch, and glue. In the final experiment, perlite, starch, glue, and MINWAX plastic paint were used in the treatment of the samples. After giving the result of each experiment, in the next experiment, attention was paid to the favorable effect of the previous experiment along with the adjustment in the compositions and the experiment of the effect of another additive.

Preparation of Treatment Solutions and Impregnation

Formulation with a concentration of 22.5%, including a mixture of perlite (100 g), starch (10 g), and glue (15 g) in 1000 mL water; and formulation with a concentration of 42.5%, including a mixture of perlite (100 g), starch (10 g), glue (15 g), and water-based paint, MINWAX (200 g) in 1000 mL water, were prepared.

The FRs test specimens were treated (T) with solution at a concentration of 22.5% and 42.5%. Four sets of 10 specimens were immersed in the PSG solution, and four sets of 10 specimens were also immersed in the PSGM solution for 24 h according to Table 1. Four sets of 10 specimens were not treated with treatment solutions; these were the control. In total, 120 specimens were tested for fire retardancy parameters according to the JISA-1322 (1982) standard.

Table 1. FRs Treatment for Protection of Maple and Ash Wood Samples with and without Bark against Fire

Species	Treatable Surfaces (TS)	FRs Treatment	Concentration (%)	Replicate	Total Samples
Maple (AV)	Wood without Bark	UT	-	10	30
		PSG	22.5	10	
		PSGM	42.5	10	
	Wood with Bark	UT	-	10	30
		PSG	22.5	10	
		PSGM	42.5	10	
Ash (FE)	Wood without Bark	UT	-	10	30
		PSG	22.5	10	
		PSGM	42.5	10	
	Wood with Bark	UT	-	10	30
		PSG	22.5	10	
		PSGM	42.5	10	

After the impregnation operations, each group of samples was placed in the laboratory environment conditions for 2 weeks to reach the equilibrium moisture content

($10 \pm 2\%$), so that the FR solutions underwent the diffusion, penetration, and fixation processes well, and then the samples were weighed to determine the amount of treating solution absorbed. All treatments used in the present study are summarized in Table 1.

After impregnation, actual retention (AR) and weight percent gain (WPG) of the treated samples was determined according to the literature (Tascioglu *et al.* 2012; Simsek *et al.* 2013; Mohammadnia Afrouzi *et al.* 2015; Ahmet *et al.* 2017; Nayeri *et al.* 2017; Gupta *et al.* 2021) and by the following Eqs. 1 and 2,

$$AR = ((G \times C)/V) \times 10 \quad (1)$$

where G is the difference between sample weight after impregnation and sample weight before impregnation (kg), C is the concentration (%), and V is the sample volume (m^3).

$$WPG (\%) = ((M_2 - M_1)/M_1) \times 100 \quad (2)$$

In Eq. 2, M_2 is the mass (g) after treatment, and M_1 is the mass (g) before treatment.

Fire Test

The treated and untreated samples were fixed in the device clamp according to Fig. 3, and the flame was placed on the end of the sample at 45° . The distance between wood and device was 10 cm. The time to ignition (TTI) was recorded with a timer in seconds (s). After the sample reached the flame point, the flame was set aside, and the glowing point time (GPT) after removing the fire nozzle was recorded in s.

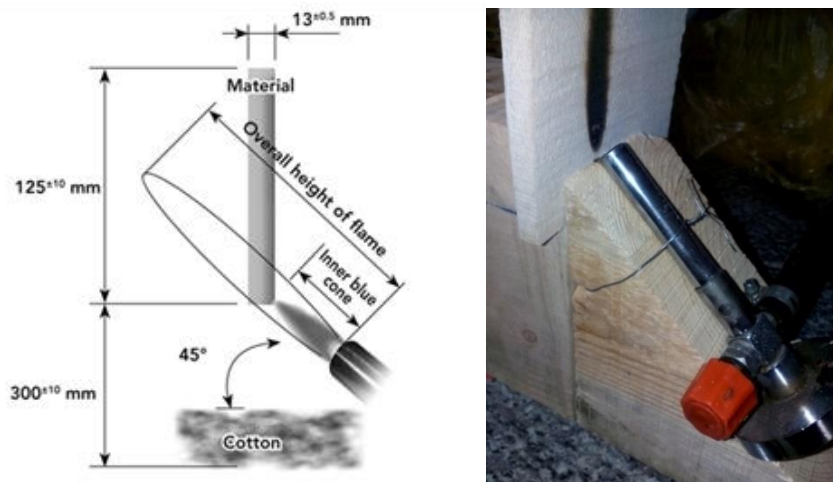


Fig. 3. Measuring the flammability of building materials by JISA-1322 (1982) standard

Mass Loss Test

Mass loss test of samples was performed according to KS F ISO 5660-1 (2003) standard guidelines. In this method, the mass of each test sample was measured with a digital scale with an accuracy of 0.001 g before and after the fire test. After testing the average value of 10 tested specimens have to be defined. The mass loss (P , %) was calculated according to the literature (Ozcifci *et al.* 2007; Sled 2012) and following Eq. 3,

$$P (\%) = (P_1 - P_2/P_1) \times 100 \quad (3)$$

where P_1 , mass of specimen before testing, in g; P_2 , mass of specimen after testing, in g.

Statistical Analysis

A univariate analysis of variance was conducted using the IBM SPSS statistics for Windows, version 24.0, software package (SPSS Inc., IBM Corp., Armonk, NY, USA) ($p < 0.05$ and $p < 0.01$) to evaluate the effect of the different species, treatable surfaces, and FRs treatment on ML, TTI, GPT, AR, and WPG, as the fire retardancy parameters. Significant difference among the average values of the FR-treated and untreated specimens were determined using Duncan's multiple range test (DMRT).

RESULTS AND DISCUSSION

Mass Loss of Untreated and FR-Treated Samples Duo to Combustion

Statistically, results showed that the interaction between the variables wood species, treatable surfaces, and FRs was negative and had a significant effect on the mass loss (ML) of samples.

According to a statistical analysis, the individual effect of variables wood species, treatable surfaces, and FRs showed significant effects on the ML measured. The interaction between wood species and treatable surfaces was negative and indicated a significant effect on the ML factor within the range of 95% and 99% confidence for the experimental FRs investigated. But the interactions between wood species and FRs and treatable surfaces and FRs were positive and did not have any significant effect on the ML factor.

Results indicated that the lowest ML value (0.39%) due to combustion was obtained in the UT-FE wood samples with bark and the highest ML (2.06%) was found in the UT-AV wood samples with bark (Fig. 4). Because the FE bark is thicker than the AV bark, it can be said that the bark of FE showed more resistance to ML than the AV bark. Thus, it was apparent that the fire resistance of the bark depends on thickness, density, and moisture content and that the different physical qualities of tree species have a negligible influencing role (Hengst and Dawson 1993; Bauer *et al.* 2010). Researchers have stated that the bark thickness is a better predictor of resistance to cambial injury from fires than either bark moisture or density (Lawes *et al.* 2011).

Compared to the samples that are treated with FRs, the best material regarding mass reduction is a mixture of perlite, starch, glue, and water-based paint.

The average ML values of untreated and FR-treated samples showed that ML in the AV (1.45%) was higher than the FE (0.95%) as the species; ML in the samples without bark (1.30%) was higher than the samples with bark (1.09%) as the treatable surfaces; and ML in the untreated samples (1.35%) was higher than the PSG-treated samples with a concentration of 22.5% (1.20%), and ML in the PSG-treated samples was higher than the PSGM-treated samples with a concentration of 42.5% (1.04%) as the FRs.

Lublóy *et al.* (2023) examined the fire performance of Norway spruce- and Scots pine-treated wood with 7 different precautions such as 1.09 g IPBC + 0.35 g propiconazole, 1.09 g IPBC + 0.35 g propiconazole, 80 g tebuconazole + 0.50 g IPBC + 0.15 g cypermethrin, 80 g tebuconazole + 0.50 g IPBC + 0.15 g cypermethrin, 0.8 g IPBC + 0.8 g propiconazole + 0.15 g cypermethrin, 15 g boric acid + 1.53 g borax, and 44.0 g boric acid + 0.8 g Alcyl-dimethyl, benzyl ammonium chloride. The ML percentages were 7.54, 7.49, 7.50, 7.65, 7.65, 5.94, and 6.69% for Norway spruce-treated wood; 8.14 and 8.07% for untreated wood, respectively, while the ML percentages were

10.53, 9.76, 10.95, 9.78, 10.54, 7.00, and 9.14% for Scots pine-treated wood; 11.43 and 10.70% for untreated wood, respectively.

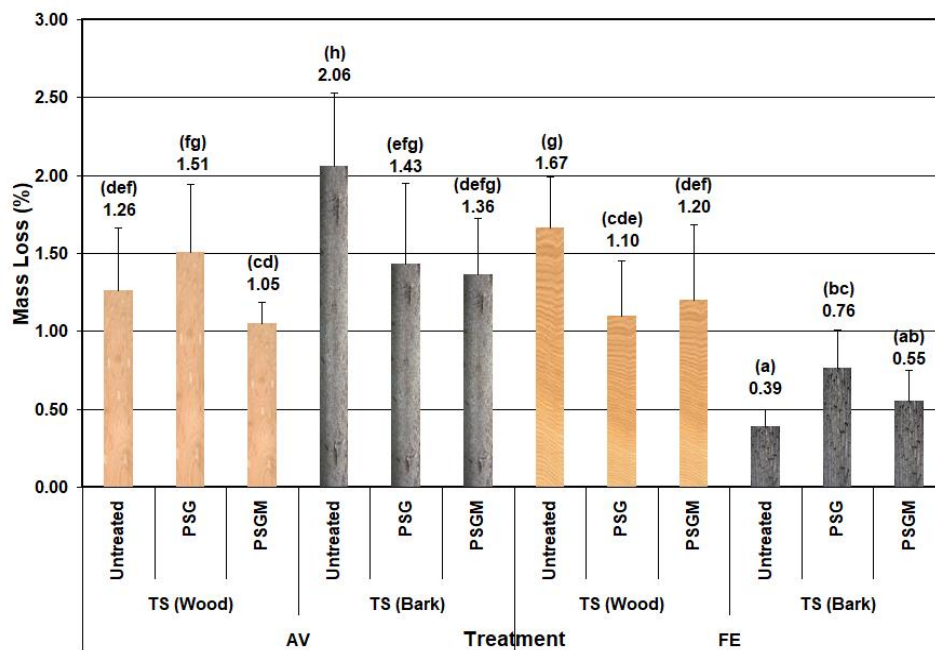


Fig. 4. Mean values \pm standard deviation of the mass loss of untreated and FR-treated samples. Different letters in each column indicate a statistical difference ($p < 0.05$) among the treatment groups

Free-extractives *Pinus taeda* wood exhibits a more significant weight loss at approximately 20 °C higher than when the extractives are present in wood, whilst for the *Eucalyptus grandis* a more pronounced degradation process occurs only at approximately 10 °C higher when the extractives are present in wood (Poletto 2016). This occurs because extractives are compounds with low molecular weight and may promote wood degradation at relative low temperatures (Guo *et al.* 2010; Kilulya *et al.* 2014), reducing wood thermal stability.

The standard defines a flame retardant as effective if mass loss does not exceed 1.5 g for surface-treated wood and 2.5 g for impregnated wood (Lublóy *et al.* 2023). The ML of untreated and FR-treated samples were classified according to Sled (2012): $ML \leq 9\%$ - The class I of fire retardant efficiency is given; $9\% \leq ML \leq 25\%$ - The class II of fire retardant efficiency is given; and $ML > 25\%$ - this treatment is not providing the fireproofing effect and is not the fire retardant. According to the classification of ML by Sled (2012), all untreated and FR-treated samples in this research were placed in class I of $ML \leq 9\%$ - thus, the I class of fire retardant efficiency is given.

Time to Ignition of Untreated and FR-Treated Samples

Statistically, it was shown that the interaction between the variables wood species, treatable surfaces, and FRs was positive and exhibited no significant effect on the time to ignition (TTI) of samples. According to a statistical analysis, the individual effect of variables wood species, treatable surfaces, and FRs showed significant effects on the TTI measured. The interaction between wood species and treatable surfaces, wood species

and FRs, as well as treatable surfaces and FRs were negative and had a significant effect on the TTI factor within the range of 95% and 99% confidence for the experimental FR-treated samples investigated.

Results indicated that the PSGM-treated FE wood samples without bark and UT-AV wood samples without bark had the highest and lowest TTI, so that the formulation with a mixture of P, S, G, and M with a concentration of 42.5% delays the fire point (TTI) by 337 s (Fig. 5). This time in the UT-samples is low; especially regarding UT-AV, the reduction is very considerable, so that the TTI reach to 22 s (Fig. 5).

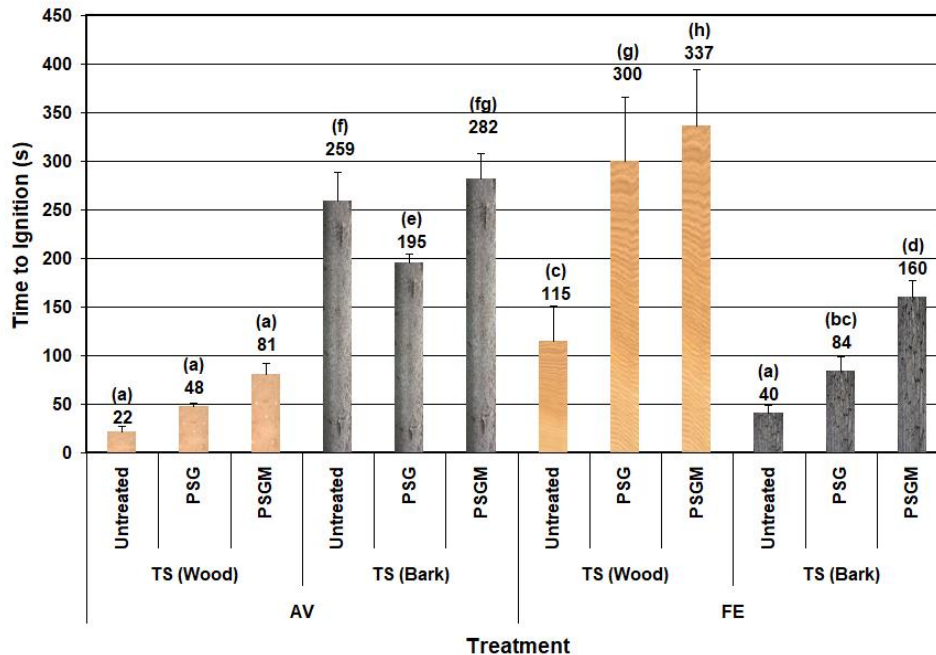


Fig. 5. Mean values \pm standard deviation of the time to ignition of untreated and FR-treated samples. Different letters in each column indicate a statistical difference ($p < 0.05$) among the treatment groups

The average TTI values of untreated and FR-treated samples showed that TTI in the FE (173 s) was higher than the AV (148 s) as the species; TTI in the samples with bark (170 s) was higher than the samples without bark (151 s) as the treatable surfaces; TTI in the PSGM-treated samples with a concentration of 42.5% (215 s) was higher than the PSG-treated samples with a concentration of 22.5% (157 s) as the FRs; and TTI in the PSG-treated samples was higher than the untreated samples (109 s).

The TTI of untreated- and treated-AV wood sample with bark was higher than the untreated- and treated-AV wood sample without bark. According to the Fengel and Wegener (2003) theory, tree barks contain relatively high amounts of extractives (20% to 30%). Additionally, Kain *et al.* (2013) found that tree bark has interesting properties for use as an insulation material, namely relatively low thermal conductivity and high heat storage capacity.

The flame-retardant mechanism of the bark cement-boards was investigated by researchers. According to their findings, a mineral-enriched matrix in the composites was the main reason for their flame retardancy. Cement as a non-combustible building material and bark as a natural barrier of tree against fire plays an important role in flame

retardancy (Pacher *et al.* 2022). Tree bark contains the phenolic compounds that provide fire-retardant properties to such composites, together with the protective role of bark as fire-stopping, is found in many species (Bauer *et al.* 2010). Starch is a bio-based polymeric component derived from renewable and widely available biomass resources. In addition, starch was chosen as the main component of FR systems in the other studies because of its good adhesion to wooden surfaces (Tretsiakova-McNally *et al.* 2021).

The investigation results of Seo *et al.* (2017) on fire properties of *Pinus densiflora* utilizing fire-retardant chemicals based on borated and phosphorus showed that for time to ignition (TTI), there was no noticeable difference between the untreated and fire-retardant treated wood (FRTW)-vacuum specimens. On the other hand, the FRTW-pressure specimen was ignited on the surface of the material after a testing time of 688 s. The mass loss rate (MLR) curves of the untreated and FRTW-vacuum specimens showed an initial rapid mass loss, which continued after 300 s. The MLR in the FRTW-pressure specimen remained similar at the 400 s mark of the test time. Also, after 700 s the MLR of the FRTW-pressure specimen decreased. To modify the flammability of wood-plastic composites (WPC), Umemura *et al.* (2014) were added various fire retardants, such as ammonium polyphosphate (APP), melamine polyphosphate (MPP), and aluminum hydroxide (Al(OH)₃) to WPCs. The results illustrated that the ignition time of PP, WPC with 50 wt% wood flour, WPC + APP, WPC + MPP, and WPC + Al(OH)₃ was 31.6, 21.4, 19.6, 20.4, and 24.8 s, respectively.

Final values of mass loss and time to ignition of untreated and HR-Prof-retardant-treated spruce wood samples by painting, spraying, immersion, or vacuum method was determined by Mitterová (2022). He measured the mass loss of samples in interior, protected exterior, and exterior environments about 68, 85, and 90% for untreated samples, while for treated samples was 48, 71, and 89%. The ignition time for untreated samples was 95, 61, and 37 s, whilst for treated specimens was 155, 92, and 37 s.

Glowing Point Time of Untreated and FR-Treated Samples

Glowing time can be defined as the time in seconds that a specimen continues to glow under the conditions of these test methods after it has ceased to flame. Statistically, results showed that the interaction between the variables wood species, treatable surfaces, and FRs was positive and had no significant effect on the glowing point time (GPT) of samples.

According to a statistical analysis, the individual effect of variables wood species, treatable surfaces, and FRs showed significant effects on the GPT measured. The interaction between wood species and treatable surfaces, wood species, and FRs, as well as treatable surfaces and FRs were negative and had a significant effect on the GPT factor within the range of 95% and 99% confidence for the experimental FR-treated samples investigated.

Results indicated that the PSGM-treated FE wood samples without bark and UT-AV wood samples without bark had the highest and lowest GPT, respectively, so that the formulation with a mixture of P, S, G, and M with a concentration of 42.5% delayed the glowing point time (GPT) by 529 s (Fig. 6). This time in the UT-samples was low; especially regarding UT-AV, the reduction was very considerable, so that the GPT reach to 49 s (Fig. 6) with the exception of UT-AV wood samples with bark (500 s).

The average GPT values of untreated and FR-treated samples showed that GPT in the FE (319 s) was higher than the AV (282 s) as the species; GPT in the samples with bark (330 s) was higher than the samples without bark (271 s) as the treatable surfaces;

GPT in the PSGM-treated samples with a concentration of 42.5% (365 s) was higher than the PSG-treated samples with a concentration of 22.5% (310 s) as the FRs; and GPT in the PSG-treated samples was higher than the untreated samples (227 s).

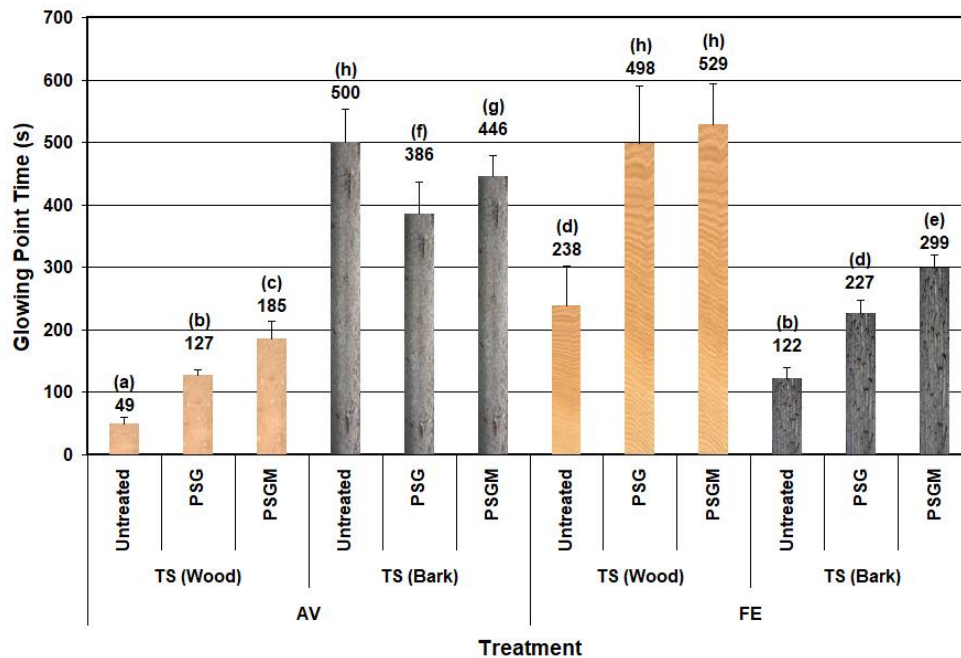


Fig. 6. Mean values \pm standard deviation of the glowing point time of untreated and FR-treated samples. Different letters in each column indicate a statistical difference ($p < 0.05$) among the treatment groups

It is probable that the main components of AV bark in UT- and treated wood samples that inhibit fire are graphite, inserted and distorted graphite-graphene aggregates (Tributsch and Fiechter 2008), and polyphenols (tannins), because the tannins contribute to the generation of graphite during charring. Furthermore, tannins are high molecular weight (up to 20,000) compounds that form complexes with proteins and alkaloids, which precipitate. The likely mechanism of action is as follows: The tannins are able to neutralize radicals due to their endowment to donate electrons (Hemingway and Lacks 1992) and *via* electron donation, as well as through forming a protective layer against the heat and against diffusion to the flame of combustible volatile compounds (Pacher *et al.* 2022). Tannins determine reduction of heat power, which could retard and slow down the fire.

One of the most abundant chemical compounds in the AV wood that was identified by Vaysi *et al.* (2019) is benzaldehyde (48%). The degree of hydrophobicity contributed by the phenyl group from benzaldehyde is low, so the hydroxyl group plays the main role in the solubility of benzaldehyde-based Novolac (Nemoto *et al.* 2009).

Fengel and Wegener (1989) reported that organic solvents usually extract lipophilic extractives, such as sterols, terpenoids, fatty acids, resin acids, and waxes, which play a role in influencing the ignitability of biomass because of their volatility.

Homovanillic acid, palmitic acid, and stigmasta-3,5-dien-7-one were identified in the bark extracts from sugar maple (SM) trees in addition to the compounds identified in the wood extracts, with the exception of xanthene-9-carboxylic acid. Palmitic acid has

been previously observed in SM extractives (Miller *et al.* 1990; Goundalkar *et al.* 2010). Sinapaldehyde was also found in the SM wood and bark extracts, but quantification was not possible because of poor peak separation in the bark.

The fact is that the starch acted as an appropriate binder substance. Tsuyumoto *et al.* (2011) has used it along with SPB fire retardant. A high flame-retardant effect of the SPB on starch can be caused by the carbonized layer formation due to the dehydration of cellulose-based material such as wood, paper, and cotton cooperating with the flame retardancy, and this matter should apply to other general carbohydrates. Thus, it is expected that the flame retardancy is caused by the synergistic effect of carbonized layer formation along with the binding effect of starch.

Commercial corn starch was added for the adhesive formulations with poplar bark for enhanced resistance to fire the bark-based panel bonded with clay (Tudor *et al.* 2020). Kebke *et al.* (2020) found that the smoldering time is shorter for all starch FR samples than for untreated wood fiber and also the application of well-soluble additives has a positive effect on fire and smoldering protection.

Paint systems contribute considerably to weather protection are usually needed to maintain the fire performance properties of fire retardant-treated (FRT) wood products for exterior applications (Östman and Tsantaridis 2016).

Actual Retention of FR-Treated Samples

Statistically, results showed that the interaction between the variables wood species, treatable surfaces, and FRs is positive and did not have any significant effect on the actual retention (AR) of samples. The individual effect of variables wood species, treatable surfaces, and FRs showed significant effects on the measured AR. The interaction between wood species and treatable surfaces, wood species and FRs, as well as treatable surfaces and FRs were negative and exhibited a significant effect on the AR factor within the range of 95% and 99% confidence for the experimental FR-treated samples investigated.

Table 2 shows that the lowest AR values of FR-treated samples observed by the treatment of 22.5% PSG-treated AV wood samples without bark (33.2 kg/m^3) and the highest AR values of FR-treated samples showed by the treatment of 42.5% PSGM-treated AV wood samples without bark (144.7 kg/m^3).

Table 2. Mean Values \pm Standard Deviation of the Actual Retention (AR) of FR-treated Samples at Different Concentration Levels

Species	Treatable Surfaces	FRs Treatment	Conc. (%)	AR (kg/m^3)	Subset for Alpha	
					0.05	0.01
Maple (AV)	Wood without Bark	PSG	22.5	33.18 ± 5.65	a	A
		PSGM	42.5	144.69 ± 24.19	d	C
	Wood with Bark	PSG	22.5	40.20 ± 7.92	ab	A
		PSGM	42.5	73.53 ± 15.75	c	B
Ash (FE)	Wood without Bark	PSG	22.5	34.08 ± 8.33	a	A
		PSGM	42.5	65.55 ± 9.68	c	B
	Wood with Bark	PSG	22.5	72.15 ± 17.18	c	B
		PSGM	42.5	48.87 ± 12.45	b	A

Different letters in each column indicate a statistical difference ($p < 0.05$ and $p < 0.01$) among the treatment groups

The average AR values of FR-treated samples showed that AR in the AV (72.9 kg/m³) was higher than the FE (55.2 kg/m³) as the species, AR in the samples without bark (69.4 kg/m³) was higher than the samples with bark (58.7 kg/m³) as the treatable surfaces, and AR in the PSGM (83.2 kg/m³) with a concentration of 42.5% was higher than the PSG (44.9 kg/m³) with a concentration of 22.5% as the FRs.

Weight Percent Gain of FR-Treated Samples

Statistically, results showed that the interaction between the variables wood species, treatable surfaces, and FRs was negative and had significant effect on the weight percent gain (WPG) of samples. According to a statistical analysis, the individual effect of the variables wood species, treatable surfaces, and FRs showed significant effects on the WPG measured. The interaction between wood species and treatable surfaces, the interaction between wood species and FRs, as well as the interaction between wood species and FRs were negative and had a significant effect on the WPG factor within the range of 95% and 99% confidence for the experimental FRs investigated.

Table 3 shows that the lowest WPG values of FR-treated samples observed by the treatment of 42.5% PSGM-treated FE wood samples with bark (0.87%) and the highest WPG values of FR-treated samples shown by the treatment of 42.5% PSGM-treated AV wood samples without bark (10.32%).

The average WPG values of FR-treated samples showed that WPG in the AV (4.8%) was higher than the FE (2.5%) as the species; WPG in the samples without bark (5.3%) was higher than the samples with bark (2.0%) as the treatable surfaces; and WPG in the PSGM (4.2%) with a concentration of 42.5% was higher than the PSG (3.1%) with a concentration of 22.5% as the FRs.

Table 3. Mean Values \pm Standard Deviation of the WPG of FR-treated Samples at Different Concentration Levels

Species	Treatable Surfaces	FRs Treatment	Conc. (%)	WPG (%)	Subset for Alpha	
					0.05	0.01
Maple (AV)	Wood without Bark	PSG	22.5	4.41 \pm 0.71	d	C
		PSGM	42.5	10.32 \pm 1.88	e	D
	Wood with Bark	PSG	22.5	2.31 \pm 0.76	b	B
		PSGM	42.5	2.23 \pm 0.37	b	B
Ash (FE)	Wood without Bark	PSG	22.5	2.96 \pm 0.77	bc	B
		PSGM	42.5	3.35 \pm 0.61	c	B
	Wood with Bark	PSG	22.5	2.67 \pm 0.73	bc	B
		PSGM	42.5	0.87 \pm 0.26	a	A

Different letters in each column indicate a statistical difference ($p < 0.05$ and $p < 0.01$) among the treatment groups

Overall, the type of species, the treatable surfaces, the presence of treating materials, and its concentration can affect the ML, TTI, GPT, AR, and WPG of FR-treated samples compared to the untreated samples against fire flame. According to the findings in the previous literatures and the current results, apparently whole properties of wood and bark, such as anatomical, morphological, physical, and thermal properties, the content and the type of extractives, as well as the type of different species, could be affected the fire retardancy parameters.

CONCLUSIONS

1. According to the findings of this study, fire-retardant (FR) chemicals had a favorable effect on the measured parameters of fire retardancy and provided a certain amount of protection against combustion.
2. As a result of this study, the type of species, the treatable surfaces, the type of treating materials, and its concentration can be affected in the retention of FRs. The average actual retention (AR) values in the *Acer velutinum* Boiss. (AV) were higher than the *Fraxinus excelsior* L. (FE) as the species, AR in the samples without bark was higher than the samples with bark as the treatable surfaces, as well as AR in the perlite-starch-glue-MINWAX (PSGM) formulation with a concentration of 42.5% was higher than the PSG with a concentration of 22.5% as the FR.
3. The weight percentage gain (WPG) in the AV was higher than the FE as the species; WPG in the samples without bark was higher than in the samples with bark as the treatable surfaces, as well as WPG in the PSGM with a concentration of 42.5% was higher than the PSG with a concentration of 22.5% as the FR.
4. Compared to the samples that were treated with FRs, the best material regarding mass reduction is a mixture of perlite, starch, glue, and water-based paint, MINWAX. The average mass loss (ML) values in the AV were higher than the FE as the species, and the ML in the samples without bark was higher than in the samples with bark as the treatable surfaces.
5. Results indicated that the formulation with a mixture of P, S, G, and M with a concentration of 42.5% delayed the time to ignition (TTI) and glowing point time (GPT). These times in the UT-samples were very low; especially regarding UT-AV, the reductions were very considerable.
6. According to the authors' observations, the findings were consistent with the findings of other researchers. This means a high flame-retardant effect of the perlite on starch, glue, and paint can be caused by the strong carbonized layer formation due to the dehydration of surface layers of wood and bark cooperating with the flame retardancy. It is probable that the high flame retardancy is caused by the synergistic effect of carbonized layer formation along with the binding effect of starch, glue, and paint.

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