Epsomite as flame retardant treatment for wood: preliminary study

J.C. Elvira-León, J.M. Chimenos*, C. Isábal, J. Monton, J. Formosa, L. Haurie

a Departament de Ciència dels Materials i Enginyeria Metal·lúrgica, Universitat de Barcelona, Martí i Franquès, 1, E-08028 Barcelona, Spain

b Departament de Construccions Arquitectòniques II, Universitat Politècnica de Catalunya, Av. Dr. Marañón 44-50, 08028 Barcelona, Spain

* Corresponding author. Tel: +34-934 021298; fax: +34-934 035438. E-mail address: chimenos@ub.edu (J.M. Chimenos)
Abstract

The effect of epsomite as flame retardant for wood has been investigated and compared with a commercial boron salt. Both flame retardants have been introduced into wood samples by vacuum impregnation. Epsomite is a hydrated sulphate salt with a water solubility of 731 g·L⁻¹ at room temperature. Thanks to this high solubility it was possible to obtain elevated epsomite loadings in comparison with the borax salt. Flame retardancy was evaluated by means of the limiting oxygen index, the dripping test and the exposition to a direct flame (Bunsen test). The results showed that the addition of epsomite increases the limiting oxygen index, delays the time to ignition and the evolution of the temperatures through the wood.

Keywords: magnesium sulphate; vacuum impregnation; endothermic decomposition; fire protection; timber.
1. Introduction

Preservation of natural resources and carbon footprint are crucial issues in the present and future of the construction sector. Reports from the EU and the UK indicate that the construction industry is responsible of 40-50% of the CO2 emissions [1, 2]. Wood from sustainably managed forests is an environmentally friendly alternative to other construction materials such as concrete or steel [3]. It is a renewable, sustainable and easily workable material that widely used in the building industry for thousands of years, not only as an integral part of structures but also as the main source of furnishings. The British Woodworking Federation affirms that the use of one tonne of timber, instead of other materials, reduces the CO2 emissions in one tonne. Despite this advantages, one of the limitations when using wood as building material is its performance in case of fire. As it is described by White and Dietenberger [4], wood exposed to high temperatures undergoes a thermal degradation that can be divided in several temperature ranges: (i) 100 - 200ºC: release of water and non-combustible gases, such as CO2; (ii) 200 - 300ºC: pyrolysis of hemicellulose and lignin and production of charred wood; (iii) 300 - 455ºC: depolymerisation of cellulose (300-350ºC) and lignin (370-400ºC) and release of flammable gases; and (iv) > 450ºC: oxidation of the charred wood residue.

Treatment of wood with flame retardants is one of the strategies to increase fire protection. The aim of flame retardants is to prevent or delay the ignition and to diminish the effects of combustion. Improvement of the behaviour of wood in front of fire can extend the use of timber products in the building sector.

Preservative treatments can be applied to wood by different methods. The nature of the protective agent and the final requirements of the wood product will determine the treatment method. The UNE-EN 351-1 [5] standard describes the classification of preservative penetration and retention of wood and wood-based products. The Spanish Technical Building Code (CTE), in the chapter Structural Safety: Timber structures, describes the usage classes and relates them with the required
level of protection in front of biotic attacks. In the case of fire protection treatments wood products are classified depending on their fire reaction using the UNE EN 13501 standard [6]. Although this standard does not require the evaluation of the penetration depth, the achievement of deeper penetrations are related with higher levels of protection. The protective agent is usually added in liquid form, dispersed or solubilized in a solvent through an impregnation process that involves vacuum and/or pressure cycles to ensure maximum penetration of the chemical agent [7]. Dipping, spray or vapour boron processes are other methods to apply protective treatments to wood, but they usually lead to lower protection depths [8,9].

Boron and phosphorous/nitrogen compounds are among the most common flame retardants used in wood [10-12]. Boron salts create a protective vitreous barrier, which hinders the release of flammable gases and the access of oxygen to wood, while phosphorous-based compounds promote charring and some of them also inhibit the radical combustion reaction [13, 14].

Another important group of flame retardants, widely used in the polymer industry, are the substances that decompose endothermically releasing water vapour or carbon dioxide. These compounds absorb part of the combustion heat and therefore, reduce the temperature of the wood, and the inert gases released dilute the flammable gases in the condensed phase. Among the compounds that decompose endothermically, the magnesium compounds are effective flame retardants and are widely used to increase flame retardancy of polymers [15]. For instance, magnesium hydroxide (Mg(OH)$_2$) and basic magnesium carbonates have been used by the authors as flame retardant fillers in poly(ethylene-co-vinyl acetate) (EVA) and polyethylene (PE) [16-19]. However, the disadvantage of some of these flame retardants is their limited solubility in water, which is the preferred solvent for the wood impregnation process due to economical and environmental reasons.

As magnesium hydroxide and magnesium carbonates, the hydrated magnesium sulphate (MgSO$_4$·nH$_2$O) decomposes endothermically in a wide range of temperatures, until its complete
dehydration. The available commercial forms of magnesium sulphate usually occur as hydrates. Specifically the heptahydrate sulphate, also referred to as epsomite or Epsom salt, is the most stable hydrate stage and the only naturally occurring member of the MgSO₄·nH₂O series. However, unlike the magnesium hydroxide or magnesium carbonates, epsomite is highly soluble in water at room temperature (731 g·L⁻¹) [20]. This elevated solubility makes possible to obtain a concentrated saturated solution. As far as authors know, the use of epsomite as flame retardant for wood seems uncommon and has been hardly referred in literature [21] and experimental results describing its flame retardancy have not been reported. Therefore this research work is a preliminary study intended to assess the flame retardant effects of this heptahydrate magnesium sulphate (MgSO₄·7H₂O) on wood. The results obtained are compared with those resulting from a commercial hydrated sodium borate, widely used as flame retardant preservative on wood.

2. Materials and methods

2.1. Materials

Two different species of structural softwoods were chosen from a Spanish timber supplier. The presence of sapwood and heartwood has great influence on the results, since heartwood has low impregnability than sapwood. For this reason, even if the specimens used contain a mixture of sapwood and heartwood, wood specimens with a higher proportion of heartwood with regard with the other specimens have been discarded. Specimens with knots or other relevant characteristics that could affect significantly the results have also been discarded. They were Scots pine (Pinus Sylvestris L.) with a density of 461 kg·m⁻³, and two sets of Black pine (Pinus Nigra) with different densities: Pinus Nigra-A (653 kg·m⁻³) and Pinus Nigra-B (574 kg·m⁻³). Densities have been measured on wood samples with 12% of moisture content. The dimensions of the wood specimens were 77 mm x 64 mm x 21 mm with the higher surface in the transverse section. Previous to the impregnation the specimens were dried to constant weight at 103 ± 2 °C in an oven.
The magnesium sulphate used in this research work was a heptahydrate salt (MgSO₄·7H₂O; epsomite), technical grade, which is commonly used as a fertilizer to correct a magnesium or sulphur deficiency in the soil. Chemical characterization of epsomite was carried out by X-ray fluorescence (XRF) using a Philips PW2400 X-ray sequential spectrophotometer. FRX revealed a content of 29.9%wt SO₃ and 15.7%wt MgO and a loss on ignition (LOI) at 1000 ºC of 54.3%wt. A small content of CaO lower than 0.05%wt, probably from a calcium sulphate, was also determined. Thermogravimetric analysis (TGA) of the epsomite (Fig. 1) was performed on a TA Instruments SDT Q600 Simultaneous TGA-DSC equipment, in air atmosphere (flow rate of 100 mL·min⁻¹) with a continuous heating rate of 10 ºC·min⁻¹ and a sample mass of 35.0 ± 0.5 mg. The sample was previously stabilised at 30 ºC and heated up to 1300 ºC. The shape of the TGA curve shows that the mass loss occurs in several steps. This is clearly showed by the derivative of the TGA curve (DTG), dashed line in Fig. 1. One or more water molecules were released in several steps during the dehydration process, as it is shown in Fig. 1. Thus, the first step corresponds to the loss of one water molecule to give MgSO₄·6H₂O at a low temperature (36-59 ºC). The remaining dehydration steps occur gradually between 60 and 303 ºC, with a total water mass loss around 51.3%wt. In the range of 896 and 1148 ºC anhydrous magnesium sulphate decomposes to MgO (17.0%wt) and releases SO₃ (31.7%wt), corroborating the results previously obtained by XRF. Moreover, differential scanning calorimetry (DSC) was simultaneously determined by the same thermal decomposition experiments to measure the heat flow associated with the thermal decomposition (Fig. 2). In this case, the heat flow signal was corrected by taking into account the sample’s mass at each temperature. The dehydration process requires 1977 J·g⁻¹ as it is shown in Fig. 2 by the integration of endothermic peaks between 36-303ºC. This value of heat of dehydration is much higher than other hydrated inorganic salts commonly used as fire retardants, such as aluminium hydroxide (1170 J·g⁻¹) or magnesium hydroxide (1244 J·g⁻¹). Moreover, the water content per mass unit is also much higher in epsomite (51.2%wt vs. 34.6%wt and 31.0%wt, respectively) [13].
Commercial sodium tetraborate pentahydrate (Na$_2$B$_4$O$_7$·5H$_2$O), technical grade Neobor® from Riotinto Minerals, also known as borax 5 mol, widely used as fire retardant preservative, was used in this study for comparative purposes. It starts to dehydrate at about 65ºC and loses all water of hydration when heated above 320ºC. Notice that this dehydration temperature range is very similar to epsomite dehydration range.

2.2. Impregnation method

Just before vacuum impregnation, saturated solutions of MgSO$_4$·7H$_2$O (1.23 g·mL$^{-1}$) or Na$_2$B$_4$O$_7$·5H$_2$O (1.03 g·mL$^{-1}$) were prepared at room temperature. Saturated salt solutions were prepared using deionized water and filtered to eliminate any non-dissolved salt crystals.

Impregnation was performed in a vacuum-autoclave of a diameter of 45 cm and a height of 150 cm. For each experimental conditions and fire resistant test method proposed, wood specimens were completely immersed in triplicate in the saturated solution and afterwards vacuum was applied (20 – 10 mbar; 15 – 7 mmHg) during 22h. Although impregnation of only three specimens not allows to determine the variability of a wood species regarding the impregnability, for a preliminary study it is enough to give indications about effectiveness of the process. The impregnation treatment consisted of two vacuum cycles in order to extract the maximum air from wood cavities, and permeate and distribute the maximum amount of flame retardant solution through the interior of the wood.

After impregnation process, specimens were initially drained and then dried in an oven at 35 ± 2 ºC to constant weight. Low drying temperature was selected to avoid losing the first hydration water molecule of epsomite (see Fig. 1). Subsequently, and before to evaluate the fire retardant performance of wood treated, the magnesium sulphate (or sodium borate) precipitated on the surface of specimens was removed using a soft brush. In order to determine the remaining moisture of the wood samples dried at 35ºC, there were used blank wood samples impregnated only with water. In
all cases moisture was between 5 to 6%, which was taken into consideration to calculate the amount of flame retardant retained.

2.3. Test methods

Scanning electron microscopy (SEM) has been used to verify the presence and distribution of the flame retardant in the wood cells. The fire behaviour of the treated samples was evaluated using three different fire tests: dripping test, limiting oxygen index (LOI) and Bunsen burner test.

2.3.1. SEM

Samples of 20 mm x 20 mm x 3 mm were cut from each wood sample and they were observed in a high resolution low-vacuum FEI Quanta 200 SEM equipment. In order to obtain these micrographs the specimens were previously chipped by means of a blow with a hammer and a chisel. In this chipping process part of the impregnated epsomite could fell out of the cell lumen. Therefore, it is not intended to determine the overall distribution by means of SEM observation.

2.3.2. Dripping test

A radiator device described in the Spanish UNE 23.725-90 standard [22] was employed to measure the degree of extinguish ability of combustion. Impregnated specimens (77 mm x 64 mm x 21 mm) are placed on a metallic grid 300 mm below a heat source of 500 W, which is taken away and put back after each ignition and extinction. Three specimens for each flame retardant formulation were tested and the parameters determined were the number of ignitions (No), the time at which the first ignition occurs (t₀) and the average time of flame persistence during the first 5 min of combustion (tₘ).

2.3.3. Limiting Oxygen of Index (LOI)

The Limiting Oxygen Index (LOI) was determined following UNE-EN ISO 4589 [23]. Although LOI test primarily was developed for plastics, this method has been successfully used to
determine fire performance of wood [24]. The ISO 4589 standard establishes the test procedure to determine the LOI on vertically oriented small specimens, in this case of 70 mm x 10 mm x 1 mm, under a mixture of nitrogen and oxygen flow. LOI is the minimum concentration of oxygen at which a specimen will support combustion under the criteria defined in the standard.

2.3.4. Bunsen burner test.

This relatively simple test for evaluating the effectiveness of flame retardants involves exposing the specimens of treated wood to a Bunsen flame for a certain period of time. It is based upon a flammability test for thermoplastic elastomers intumescent products described by Abu-Isa in the bibliography [25]. A scheme of the device is shown in Fig. 3. The apparatus includes a three-wall steel to form a square-shaped chamber with an open side (front). The wood sample is placed on top of the walls and the burner is adjusted during each test so that the tip of the blue cone of the flame touches the lower surface of the test specimens. Three thermocouples were affixed in the wood specimen to monitor temperatures in the exposed and non-exposed surface during the test: a thermocouple was placed at the central position of the lower surface and other two at the central and external position of the upper surface (see Fig. 3). This test records the evolution of temperature in front of time on the non-exposed surface of the samples. Although it is not related to the EN 13501 standard [6], the Bunsen burner test gives information about the effectiveness regarding fire reaction.

3. Results and discussion

The flame retardant loading was determined by weight differences between the dry weight of specimens after impregnation and before impregnation. As it was aforementioned, the moisture content of wood samples after drying at 35°C was 5 - 6%. Therefore, this residual moisture content has been subtracted to determine the retained percentage of flame retardant. The mean flame retardant loading (FRL; % wt) is shown in Table 1 (average). Although the three replicates showed similar FRL more replicates would be necessary to perform an extensive study on the impregnation
process. As can be observed, the amount of both flame retardant chemicals follows the same trend, i.e. both Pinus Nigra retain higher amounts of preservative than Pinus Sylvestris. Between both Pinus Nigra the sample with lower density (Pinus Nigra-B) exhibits higher retentions than Pinus Nigra-A. These differences in the impregnation results can only be related to the internal structure of the tested specimens. Besides, in all cases it can be observed a great difference in the degree of impregnation obtained with epsomite and borax. As expected, the large difference in solubility between both products allows obtaining saturated solutions with higher amount of epsomite than borax 5 mol. Therefore, for the same impregnation process, the amount of epsomite permeated is higher than the amount of borax 5 mol.

Fig. 4 shows the micrograph of a radial section of the Pinus Nigra-B impregnated with a saturated solution of epsomite, once the specimen has been dried at 35 °C. As it can be seen, the wood cells (tracheids and rays) are fully loaded with an inorganic salt. The characterization of this solid (yellow square in Fig. 5a) by energy-dispersive X-ray spectroscopy (EDS) shows the presence of O, Mg and S (Fig. 5b) as major elements, corresponding to magnesium sulphate contained in the cells of wood. A micrograph of the same type of wood impregnated with borax 5 mol can be seen in Fig. 6a. The EDS analysis of one of the particles that can be distinguished in Fig. 6a is depicted in Fig. 6b. The presence of sodium in the EDS analysis confirms the permeation of borax 5mol. However, as it can be observed, in this case the cells are not saturated with flame retardant due to the lower amount permeated into the sample.

However, this amount of epsomite may be too large for certain application (i.e. beams, joists, studs, roofing, etc.) where the weight of the treated wood is essential to determine their mechanical properties and the criteria selection. In these cases, the amount of epsomite loaded can be adjusted diminishing the concentration of magnesium sulphate in the impregnation solution. Moreover, because epsomite has a high solubility and diffusivity, this fire retardant preservative can move out
of a piece of wood under severe wetting. Hence, epsomite–treated wood should only be used where it is protected from major water exposure.

3.1. Dripping test

Table 2 shows the average results of the main parameters obtained with this test: the first time of ignition, the number of ignitions and the average time of flame persistence. The goal in this test is to develop materials that do not exhibit any ignition when exposed to the heating source. If ignition occurs, it is preferred that the flames appear at longer times and that they do not persist onto the sample.

As it can be seen in Table 2 the untreated wood samples exhibit lower times to ignition and longer average combustion times compared with the wood samples containing flame retardants. Among the different wood types it should be noticed that the Pinus Nigra-A shows a remarkably longer length of flame persistence, both for the untreated sample and the treated ones. This behaviour could be related with the presence of duramen, which is hardly impregnable, and resin in the Pinus Nigra-A samples.

Regarding the results obtained with the treated wood, both fire retardant chemicals significantly improve the fire behaviour of all specimens. Noteworthy, epsomite increased the time to ignition ($t_0$) by 56% to 200% in comparison with the untreated wood. Epsomite acts through an endothermic effect, as other hydrated compounds, lowering the temperature of the material near the flame during their endothermic decomposition and diluting the flammable gases with the release of water [13]. As a result, the wood is cooled and the time to ignition is increased. According to that, the increase in the time required for the first ignition ($t_0$) is related to the amount of epsomite loaded and therefore, the heat involved the dehydration process of the seven molecules of water of crystallization.
However, depending of the type of wood, the auto-extinguishing ability ($t_m$) was not so different between both used fire retardant chemicals. Several mechanisms can be involved in the auto-extinguish process depending on the fire retardant chemical used, and probably two or more of them may be feasible in a given case [13, 26]. While the water vapour provided by hydrated inorganic salts dilutes the flammable gases produced by wood to prevent the formation of a flammable mixture, borax form glassy films on exposure to high temperature, which serves as an insulating blanket and reduces the volatile products.

3.2. Limiting Oxygen of Index (LOI)

The average results of limiting oxygen indices (LOI) are shown in Table 3. As it is expected the LOIs values of wood specimens increase after being treated with flame retardants. High loaded epsomite samples experience a remarkable LOI enhancement up to 27%, while the impregnation with borax 5 mol causes only a moderate increase with regard to untreated wood. Probably the differences in the amount of flame retardant loading highly affect the LOI values.

3.3. Bunsen burner test

Non-treated wood specimens showed very similar evolution of the temperatures on the non-exposed surface of the specimens tested. Therefore, and in order to make easier the visualization of the results of the treated samples in the graphs only appears the curve of the untreated Pinus Nigra-B. Fig. 7 shows the evolution of temperatures in the non-exposed surface in front of time for the different wood samples treated with epsomite. Since results were very similar among replicates, only the temperature profile of one of the replicas is shown in the figure. In all cases, around 70 ºC it can be observed a plateau where the temperatures remain constant for a period of time, after which the temperatures rise with a stronger slope. The wood without flame retardant treatment has a shorter plateau and the thermocouple reaches 150 ºC in approximately 15 mintues. The presence of epsomite results in a remarkable enhancement of the length of the plateau and therefore there is a delay in the
increase of temperatures on the non-exposed surface. Epsomite treated samples did not reach 150 °C after 30 minutes of test, in the case of the Pinus Nigra-A the temperature at 30 minutes was still below 110 °C.

This phenomenon was also observed in other fireproofing materials containing hydrated minerals that decompose endothermically [27, 28]. This mechanism of evaporation and condensation of water takes place in the hot area and the cold area, respectively, and it progresses from the heat focus of the surface exposed to the flame to the non-exposed surface. As the temperature was recorded on the non-exposed surface, the temperature of this plateau was under 100°C as a consequence of the thermal equilibrium (convection and radiation), which is produced between the non-exposed surface and the ambient [27].

The specimens treated with borax 5 mol did not show an improvement in this test compared to the non-treated wood samples. Fig. 8 shows the evolution of temperatures on the non-exposed surface for the different samples of Pinus Nigra-A. As it can be observed the sample treated with borax 5 mol undergoes a similar behaviour as the untreated sample. Probably at low flame retardant loading, 7%wt in this case, the formation of a protective glassy film is not to delay the combustion under the conditions of the Bunsen test.

4. Conclusions

The high solubility of epsomite in water at room temperature makes possible to treat wood samples without heating and without using organic solvents. Using a vacuum impregnation process it is possible to obtain wood samples permeated with large amounts of this heptahydrate salt into the internal structure of wood, up to 412 kg·m⁻³ of epsomite in a Pinus Nigra wood sample. The epsomite loading could be probably increased using a vacuum-pressure impregnation system, or decreased by adjusting the concentration of epsomite in the solution impregnated. Given its high
solubility and non-toxicity, epsomite could be an interesting alternative to improve the fire behaviour of indoor wood panelling and structural timber elements.

According to the results of this preliminary study, the effectiveness of epsomite as a flame retardant preservative has been demonstrated using three fire tests, which have allowed assessing the improvement on the fire behavior of the specimens impregnated with epsomite. Epsomite undergoes an endothermic dehydration with an associated heat of 1977 J·g$^{-1}$ and a release of water of 51.2%wt. Therefore, epsomite acts as a heat sink lowering the temperature of the material near the flame and delaying the ignition of treated wood. Moreover, the great amount of water generated from the dehydration process, which is involved in the vaporization-condensation mechanism, keeps the temperature of the area immediately around of flame below 100ºC, increasing the insulation capacity of the impregnated wood.

In this study the amounts of borax permeated into the wood using the same impregnation process where significantly lower than for the epsomite. However, since boron salts act trough a different flame retardant mechanism, mainly forming a protective glassy film that hinders the penetration of oxygen into the condensed phase, it could be interesting to evaluate possible synergistic effects with epsomite.

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REFERENCES


Table 1. Flame retardant loading for each wood sample.

<table>
<thead>
<tr>
<th></th>
<th>Epsomite (MgSO$_4$·7H$_2$O)</th>
<th>Borax 5 mol (Na$_2$B$_4$O$_7$·5H$_2$O)</th>
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<tr>
<td>FRL (wt%)</td>
<td>FRL (wt%)</td>
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<td>Pinus Sylvestris</td>
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</tr>
<tr>
<td>Pinus Nigra-A</td>
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<td>7</td>
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<td>Pinus Nigra-B</td>
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Table 2. Dripping test results. Number of ignitions (No); time at which the first ignition occurs ($t_0$); average time of flame persistence ($t_m$).

<table>
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<th>Untreated specimens</th>
<th>Borax 5 mol (Na$_2$B$_4$O$_7$·5H$_2$O)</th>
<th>Epsomite (MgSO$_4$·7H$_2$O)</th>
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</thead>
<tbody>
<tr>
<td>No</td>
<td>$t_0$ (s)</td>
<td>$t_m$ (s)</td>
</tr>
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</tr>
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<td>29</td>
</tr>
<tr>
<td>Pinus Nigra-B</td>
<td>#15</td>
<td>46</td>
</tr>
<tr>
<td>Species</td>
<td>LOI (%)</td>
<td>Borax 5 mol (Na$_2$B$_4$O$_7$·5H$_2$O)</td>
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<td>-----------------</td>
<td>---------</td>
<td>------------------------------------</td>
</tr>
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<td>Pinus Sylvestris</td>
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<tr>
<td>Pinus Nigra-B</td>
<td>26.0</td>
<td>30.5</td>
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</table>
Figure Captions.

Figure 1. Thermogravimetric analysis (TGA) and derivative of the TGA curve (DTG) of the epsomite (MgSO₄·7H₂O) up to 1200ºC.

Figure 2. Differential scanning calorimetry (DSC) of the epsomite (MgSO₄·7H₂O) in air atmosphere (100 mL·min⁻¹) with a heating rate of 10ºC·min⁻¹.

Figure 3. Bunsen burner test apparatus.

Figure 4. Environmental scanning electron microscopy (ESEM) micrograph of the radial section of the Pinus Nigra-B impregnated with a saturated solution of epsomite (MgSO₄·7H₂O).

Figure 5. a) ESEM micrograph of the radial section of Pinus Nigra-B impregnated with epsomite and b) Energy-dispersive X-ray spectroscopy (EDS) of flame retardant chemical loaded into the treated wood.

Figure 6. a) ESEM micrograph of the radial section of Pinus Nigra-B impregnated with borax 5 mol and b) Energy-dispersive X-ray spectroscopy (EDS) of the flame retardant chemical loaded into the treated wood.

Figure 7. Temperatures recorded in the central position of the non-exposed surface for the untreated specimen and for the specimens impregnated with epsomite.

Figure 8. Temperatures recorded in the central position of the non-exposed surface for the untreated Pinus Nigra-A and impregnated with epsomite and borax 5 mol.
Figure 1.

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