



Epsomite as flame retardant treatment for wood: Preliminary study



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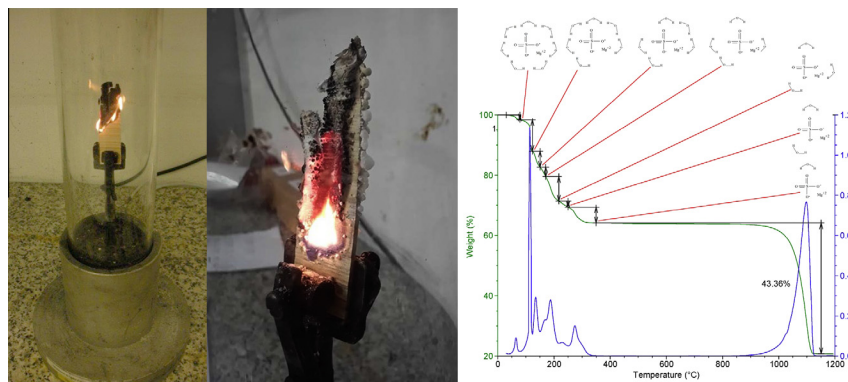
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HIGHLIGHTS

- Epsomite as flame retardant for wood has been investigated.
- Epsomite is highly soluble in water at room temperature (731 g L^{-1}).
- A high concentration solution of Epsomite can be used to impregnate wood.
- Epsomite decomposes endothermically in a wide range of temperatures.
- Epsomite acts as a heat sink lowering the temperature and delaying the ignition.

GRAPHICAL ABSTRACT



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ABSTRACT

The effect of epsomite as flame retardant for wood has been investigated and compared with a commercial borax 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.

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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 CO_2 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 CO_2 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

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a thermal degradation that can be divided in several temperature ranges: (i) 100–200 °C: release of water and non-combustible gases, such as CO₂; (ii) 200–300 °C: pyrolysis of hemicellulose and lignin and production of charred wood; (iii) 300–455 °C: depolymerisation of cellulose (300–350 °C) and lignin (370–400 °C) and release of flammable gases; and (iv) >450 °C: 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 (MgSO₄·nH₂O) 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 × 64 mm × 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 °C 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 °C·min⁻¹ and a sample mass of 35.0 ± 0.5 mg. The sample was previously stabilised at 30 °C and heated up to 1300 °C. 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 °C). The remaining dehydration steps occur gradually between 60 and 303 °C, with a total water mass loss around 51.3%wt. In the range of 896 and 1148 °C 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 and 303 °C. 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

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