Effects of Heat-Treatment and Nano-Wollastonite Impregnation on Fire Properties of Solid Wood

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The effects of nano-wollastonite (NW) suspension impregnation on the fire-retarding properties of heat-treated solid wood of three species (beech, poplar, fir) were studied. Heat treatment was performed at two temperatures of 180 °C and 200 °C. Impregnation was carried out at a pressure of 3 bars for 30 min. The fire properties included ignition time, glowing time, back-darkening, back-splitting, back-firing, and length and width of the burnt area. Both impregnation with NW and heat-treatment generally improved all fire-retarding properties, although not always to a significant level. As a mineral material, NW acted like a physical shield against fire penetration into the texture of wood specimens, thus improving fire properties. Moreover, the high thermal conductivity coefficient of wollastonite increased the thermal conductivity of wood, therefore preventing the accumulation of heat at the point nearest to a piloted flame and contributing to the improvement of fire properties. The chemical degradation of wood cell components caused by heat-treatment further improved the fire properties. Cluster analysis indicated the significant effect of species on fire properties. Significant R-square values were found amongst fire properties related to the spread of fire on the surface of specimens. The combination of thermal modification and impregnation with NW provides suitable fire properties for solid wood.

Keywords: Fire-retarding properties; Mineral materials; Nano-fiber; Solid wood; thermal conductivity; Wollastonite

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INTRODUCTION

Fire safety is an important concern in all types of construction. All forms of wood, including solid wood and wood-composites, are very susceptible to flammability when exposed to fire or even high temperatures. The thermal degradation of wood occurs in stages. The degradation process and the exact products of the thermal degradation of cell wall polymers depends on the temperature and length of exposure (Goli *et al.* 2014). Several materials have been used to delay the spread of fire in wood (Singh and Singh 2012). The use of inorganic salt treatments designed to render wood fire-retardant is not a modern development. Recently, a pressure impregnation process was developed wherein these mineral salts, contained within suitable water-based mixtures, are forced deep into the wood (Özdemir and Tutuş 2016). This process results in a new building component known as fire-retardant-treated (FRT) lumber (Ayrilmis *et al.* 2007; Bueche 2013). Chemicals for FRT lumber are applied to wood and wood-based building materials such as plywood and structural lumber (including dimensional lumber used to fabricate roof trusses) in an attempt to reduce fires. The heat-transferring property of silver nanoparticles and wollastonite nanofibers (Taghiyari *et al.* 2013a,b) improves some fire-retarding properties, as it prevents the accumulation of heat in one location (Taghiyari 2012; Taghiyari *et al.* 2013a, b).

Fire-retardant treatments may negatively affect wood in various ways such as increased hygroscopicity, reduced strength, dimensional instability depending on the treatment, wood degradation, corrosion of metal fasteners, adhesion problems, increased abrasiveness, and treatment leaching (Winandy 1998; Winandy *et al.* 2002). As evidenced by structural problems in fire-retardant-treated plywood, fire-retardant chemicals and high temperature environments can reduce wood strength (Winandy *et al.* 2002). The combination of acidic fire-retardant chemicals and elevated temperatures may increase the rate of acid hydrolysis in the wood, thereby decreasing strength (LeVan and Winandy 1990). The effects of fire-retardant chemicals can be severe. Therefore, the initial effects of fire-retardant formulations on wood strength, stiffness, and fastener corrosion should be examined before fire-retardant-treated materials are used in areas with elevated temperatures, such as roof decks (Winandy 1998).

Except for flammability, fire-retardant chemicals are usually assessed according to the strength reduction of wood and corrosion on fasteners, increased hygroscopicity, and the amount of toxic and smoke gases produced (Winandy *et al.* 2002; Ayrilmis *et al.* 2007). Silicon compounds have recently gained attention for their ability to improve upon fire properties (Cai *et al.* 2016). However, the fire-retarding properties of nano-wollastonite (NW) as a silicate mineral ($CaSiO_3$) still needs to be studied in greater detail (Taghiyari *et al.* 2013b). As it does not have any acidic chemicals, and it is not expected to cause a reduction in strength or corrosion on fasteners. Its mineral nature can be considered a barrier or shield against fire or heat transfer. The heat-transferring property of wollastonite could improve the fire-retarding properties of composite materials (Taghiyari *et al.* 2013a,b; Zhu 2013). Concerning environmental aspects and health issues, wollastonite is categorized as a nontoxic mineral material that is not hazardous to humans or livestock. In fact, while reviewing the available epidemiological studies on wollastonite, no evidence was discovered to suggest that wollastonite presents any health hazard. However, further studies on workers with long term exposure to wollastonite dust are required before the health hazards of wollastonite can be evaluated in full (Huuskonen *et al.* 1983a). The long-term health effects caused by inhalation of wollastonite appear to be negligible, as no correlation between serum angiotensin-covering enzymes in wollastonite workers and slight pulmonary fibrosis has been reported (Huuskonen *et al.* 1983b). However, if wollastonite nanofibers penetrate into wood structures, they would not be inhaled, and there might be no hazardous effects.

NW has been reported to significantly improve biological resistance against wood deteriorating fungi in solid wood and wood-composite panels (Taghiyari *et al.* 2014a, b). NW also significantly improves fire-retarding properties in wood-composite panels (Taghiyari *et al.* 2013b). Heat treatment is the most commercially utilized wood modification method in recent decades (Hill 2006; Taghiyari and Moradi 2014), and it improves the dimensional stability and the biological resistance of wood (Schmidt 2006, 2007; Maresi *et al.* 2013; Hosseinpourpia and Mai 2016). However, there is little research on the effects of NW impregnation on the fire-retarding properties of heat-treated wood. The present study was therefore conducted to explore these effects following an NW-impregnation of heat-treated wood at 180 and 200 °C.

EXPERIMENTAL

Specimen Preparation

Two commercial hardwoods, and one softwood, were chosen based on their importance in various industrial applications in Iran. These woods were beech (*Fagus orientalis* L.), poplar (*Populus nigra* L.), and silver fir (*Abies alba* Mill.). Sixty specimens, free from knots, checks, and physical or fungal damages were prepared for each type of wood, totaling 180 specimens. The number of replications for each treatment was 10 specimens. Dimensions of the specimens were 150 (length) × 130 (width) × 9 (thickness) mm^3. Specimens of each species were randomly divided into six main groups of control, NW-impregnated (NW), heat-treated at 180 °C (HT180), heat-treated at 200 °C (HT200), NW-impregnated and heat-treated at 180 °C (NW-HT180), and NW-impregnated and heat-treated at 200 °C (NW-HT200). To discover the effects of wollastonite on heat treatment, impregnation was conducted prior to heat treatment.

Nano-Wollastonite Impregnation

Aqueous NW gel was procured from the Mehrabadi Manufacturing Company of Mineral and Industrial Products (Tehran, Iran). The formulations of the NW and compound contents used are summarized in Table 1. The size range of wollastonite nano-fibers was 30 to 110 nm. NW impregnation was performed using a nano-suspension with a concentration of 10% according to the Lowry method (empty-cell process) at a pressure of 3 bars for 30 min (HaghighiPoshtiri *et al.* 2014). The pH of the suspension was between 9 and 11. Each specimen was weighed before and after the impregnation to measure the gain of NW suspension based on the volume of the specimens. Once impregnated, specimens were kept in a conditioning room (25 ± 2 °C, and 35 ± 3% relative humidity (RH)) for three months.

Table 1. Compounds and Formulation of the Nano-Wollastonite Gel Used in the Study (Taghiyari *et al.* 2013a; 2014a, b)

Nano-Wollastonite Compounds	Content (%)
CaO	39.77
SiO_2	46.96
Al_2O_3	3.95
Fe_2O_3	2.79
TiO_2	0.22
K_2O	0.04
MgO	1.39
Na_2O	0.16
SO_3	0.05
Water	4.67

Heat-Treatment Process

All HT and NW-HT specimens were randomly arranged in an oven before being heated for 5 h. Heat treatment was carried out at 180 °C for the HT180 and NW-HT180 specimens and at 200 °C for the HT200 and NW-HT200 specimens. The timing (5 h) started when the temperature inside the oven reached the final temperature for the first time. After 5 h of heat treatment, specimens were kept in the oven until they were cooled to avoid temperature shocking. The specimens were weighed using a digital scale before and after the heat-treatment process to calculate the weight loss.

Fire-Retardant Testing Device

Due to the unavailability of a cone calorimeter, the standard tests were conducted using a special device designed and built using piloted ignition as described by Taghiyari (2012). The device is shown in Fig. 1. The fuel used in the present study was natural gas comprised of mainly methane CH_4 (90 to 98%). However, other hydrocarbons were also reported by the supplier to accompany the methane (C_2H_6: 1 to 8%; C_3H_8: 2%; H_4H_{10} + C_5H_{12}: less than 1%; N_2 + H_2S + H_2O: less than 1.5%). The flow rate was 0.096 ± 2 L/s. In the device, a Bunsen-type burner was held vertically, onto which the specimen was mounted at a 45° angle (Taghiyari 2012). This device is referred to as a fixed fire test device (FFTD) for the remainder of the article. The internal diameter of the burner nozzle was 11 mm. The Bunsen-type burner provided a fairly mild and localized fire exposure to the testing specimens. Ignition and glowing times were measured from the moment the specimens were exposed to fire. As the burning continued, the back face of the specimen nearest to the flame of the burner started blackening, and soon after, a small hole or split appeared. These times were recorded and registered as back-blackening and back-splitting times. The test was terminated once the back-splitting occurred. The length and width of burning, as well as the weight loss of the specimens, were then measured. The device was placed in a three-wall-compartment in order to protect the burning flame from wind and air movements. Specimens were conditioned at 25 ± 2 °C and 35 ± 3% RH for eight weeks before fire testing (Figueroa *et al.* 2012).

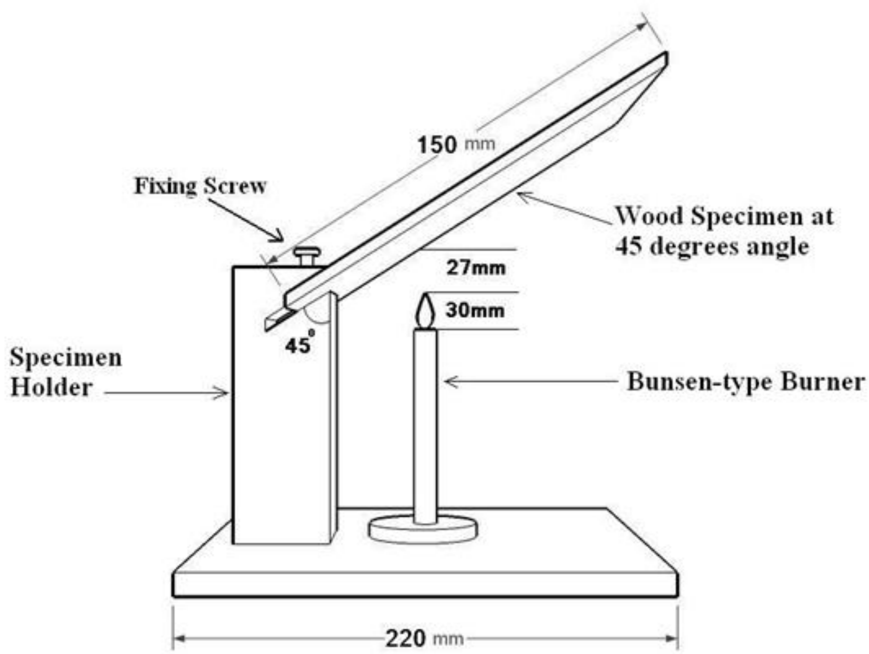


Fig. 1. Schematic picture of fixed fire testing device (Iranian patent No. 67232 approved by The Iranian Research Organization for Scientific and Technology under license No. 3704) (Taghiyari 2012)

Scanning Electron Microscopy (SEM)

SEM imaging was performed in the thin-film laboratory FE-SEM lab (Field Emission) at the School of Electrical and Computer Engineering, The University of Tehran. A field-emission cathode in the electron gun of a scanning electron microscope (S-4700, Hitachi, Tokyo, Japan) provided narrower probing beams at low, as well as high, electron energy, resulting in both improved spatial resolution and minimized sample charging and damage.

Statistical Analysis

Statistical analysis was conducted using SAS software, version 9.1 (2003; Cary, NC, USA). Analysis of variance (ANOVA) was performed on the data to conclude significant differences at a 95% confidence level. Regression and hierarchical cluster analyses, including dendrogram and using the Ward method with squared Euclidean distance intervals, were carried out using SPSS/20 (2011; IBM Corp., New York, USA). A cluster analysis was performed to find similarities and dissimilarities between treatments according to more than one property (Ada 2013). The scaled indicator in each cluster analysis showed how similar or different the treatments were. Lower scale numbers showed more similarities, and higher numbers showed dissimilarities. Fitted-line plots were made using Minitab software, version 16.2.2 (2010; State College, PA, USA).

RESULTS AND DISCUSSION

The uptake of NW suspension was higher in beech wood (1.68% in control specimens) compared to poplar and fir wood (Fig. 2). This was expected, as beech is known to be an impregnable wood species, whereas fir is refractory (Siau 1995; Taghiyari *et al.* 2010). Poplar is difficult to treat, but it is inaccurate to call it a refractory species. Previous research has demonstrated the irregular impregnability of poplar wood (Van Acker *et al.* 1990). This could explain the very low NW uptake of poplar wood in comparison to the NW uptake of fir wood (Fig. 2).

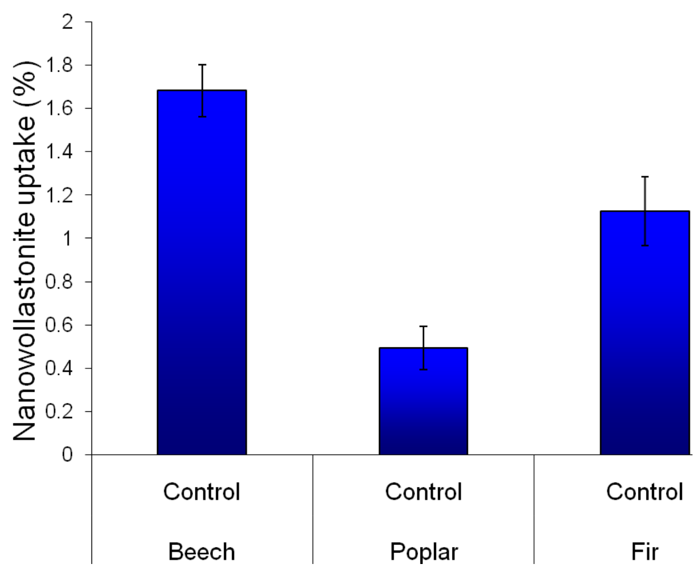


Fig. 2. NW suspension uptake (%) in heat-treated beech, poplar, and fir

NW-impregnated samples showed increased weight loss compared with control samples at both treating temperatures (Fig. 3). This was true for all species and was attributed to the high thermal conductivity of wollastonite, transferring heat to the inner parts of the NW impregnated specimens (Taghiyari *et al.* 2013a, b). Therefore, the process of wood polymer degradation caused by the heat-treatment should have also occurred deeper in the mass of the NW impregnated specimens. The weight loss was positively related to the increase in temperature, as a result of the higher degradation of wood polymers, especially hemicelluloses (Mahnert *et al.* 2013). SEM micrographs showed thinned cell walls in beech caused by heat treatment (Fig. 4).

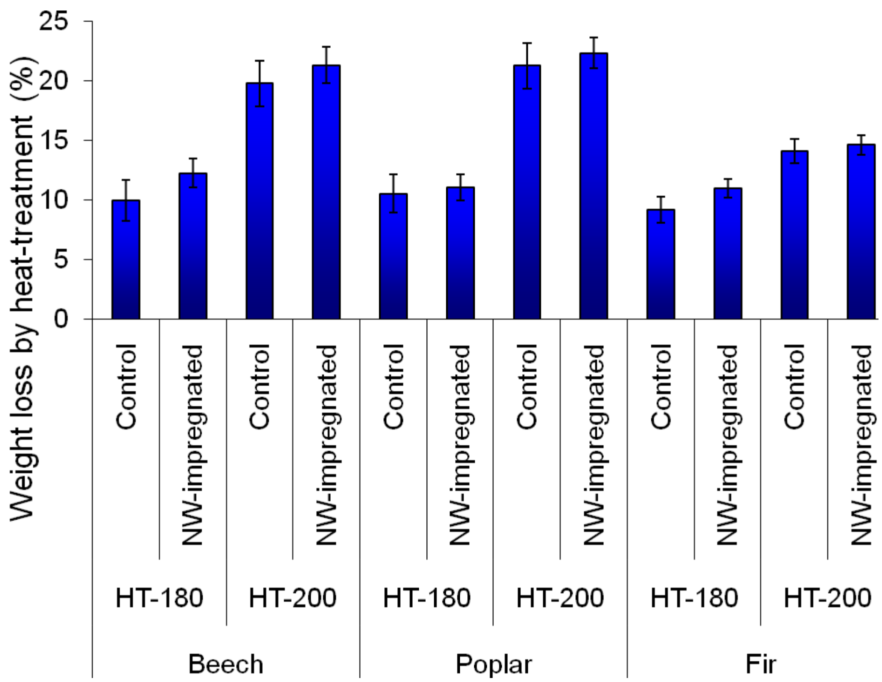


Fig. 3. Weight loss (%) in NW-impregnated and heat-treated beech, poplar, and fir at 180 and 200 °C (NW = nano-wollastonite; HT = heat-treated)

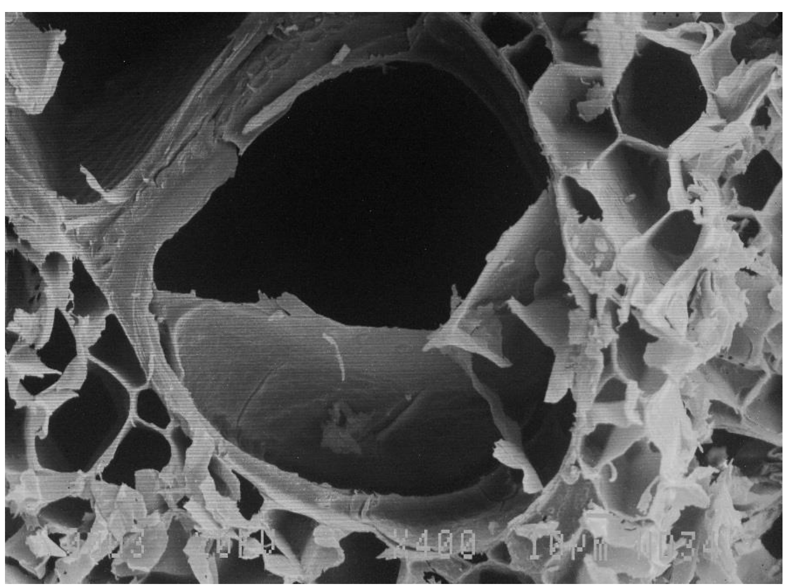


Fig. 4. Micrograph showing thinned cell walls in beech wood caused by heat-treatment at 200 ºC

(400x magnification)

NW impregnation significantly increased the ignition time in the untreated control samples of the three species (Fig. 5). The maximum increase was observed in beech specimens (more than 150% in comparison to untreated control), which was attributed to the higher uptake of NW (Fig. 2). Wollastonite nano-fibers improved the ignition time in two ways: a) their mineral nature acted as a physical shield against the piloted fire, and b) their high thermal conductivity coefficient transferred the heat to the inner-structure of the specimens (Taghiyari *et al.* 2013a, b), thus postponing the accumulation of heat and eventual ignition of specimens. Heat treatment also increased the ignition time for nearly all of the species and treatments. Only poplar-HT180 did not show improvement in ignition time. This can be attributed to the mild decomposition of poplar cell wall compounds during heat treatment at 180 °C (Ling *et al.* 2016), which resulted in the presence of a high amount of intact polysaccharide that may act as thermal conductor. Increases in ignition time of thermally modified wood might have been related to the degradation of the wood cell wall polymers brought about by the pre-pyrolysis process. This process reduces the amount of volatile pyrolysis product, thereby decreasing the thermal conductivity of wood (Hirata and Kawamoto 1991; Kol and Sefil 2011).

NW-impregnated beech exhibited an increased glowing time, although this increase was not significant in NW impregnated poplar and fir (Fig. 6). Heat treatment increased the glowing time of beech and fir, while poplar did not show a clear trend. The increase in glowing time caused by heat treatment can be attributed to the degradation of many cell wall components, which prevented the remaining wood from glowing as fast as in un-heated specimens (Hill 2006).

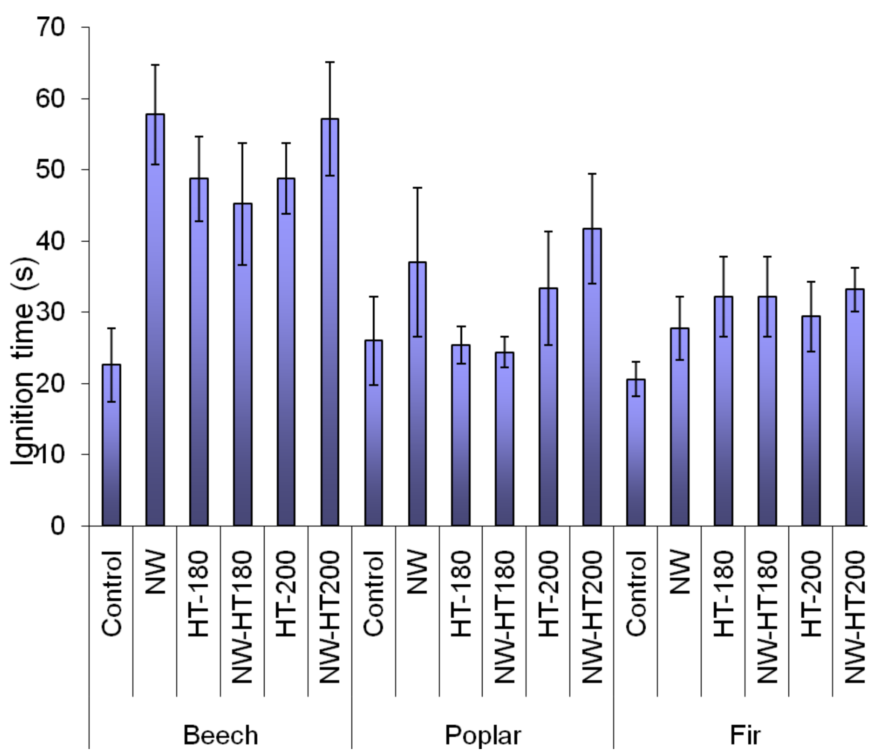


Fig. 5. Ignition time (s) in NW-impregnated and heat-treated beech, poplar, and fir at 180 and 200 °C (NW = nano-wollastonite-impregnated; HT = heat-treated)

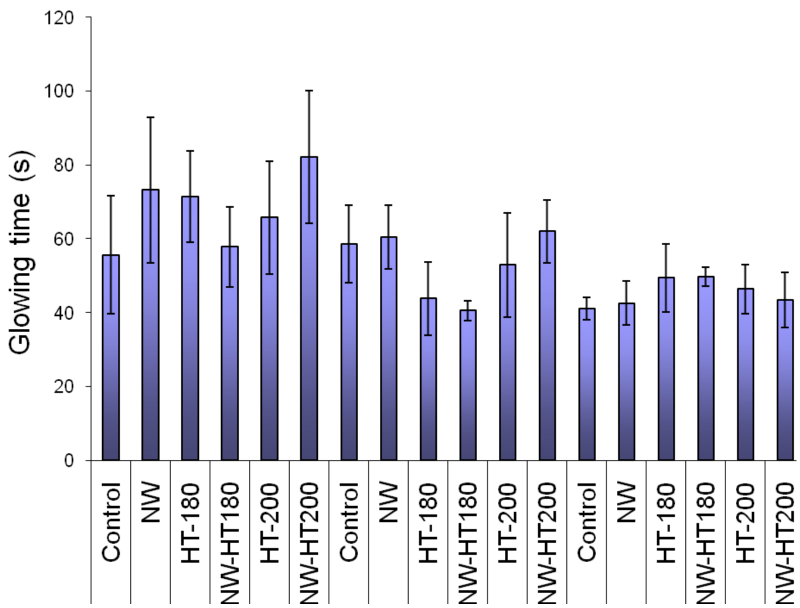


Fig. 6. Glowing time (s) in NW-impregnated and heat-treated beech, poplar, and fir at 180 and 200 °C (NW = nano-wollastonite-impregnated; HT = heat-treated)

As shown in Fig. 7A, the back-darkening time was longer in untreated fir compared to untreated beech and poplar. This might be due to the faster decomposition of cell wall polysaccharides and higher releases of volatile product in hardwood than softwood (Esteves and Pereira 2009). Treatment of NW impregnated wood at higher temperatures (200 °C) resulted in a considerable increase in back-darkening times in nearly all of the wood species. However, it was more obvious in modified fir. This may have been caused by a lack of fire penetration into the inner-structure of fir solid wood brought about by its lower density. Both back-splitting and back-firing times showed similar back-darkening trends (Fig. 7B and C). Since they were both hardwoods, the back-splitting index in beech was expected to be higher, because of its higher density compared to poplar. However, the results showed no great difference, as was the case with fir wood. This can be attributed to beech woods' vulnerability to radial cracks that form near the rays (Boonstra *et al.* 2006). This may also demonstrate the significant effect of species, with regards to the density and different chemical compositions of the cell walls in hardwood and softwood, on the penetration of fire and heat transfer into the inner-structure of solid wood. Improvements to the indexes related to the backside of specimens illustrated the capability of fire penetration into cell wall tissue. Indexes concerning safety measures for the life and property of human beings are quite vital to improve upon. In fact, when considering safety measures, every second counts.

Impregnation with NW tended to increase the length and width of the burnt area (Fig. 8A and B). The mineral nature of wollastonite nano-fibers may act as a physical shield against fire penetration into the inner-structure of wood, instead spreading the flame over a larger area. Heat treatment did not show a regular trend in all of the treatments and species, although the length and width of the burnt area in heat-treated specimens tended to be smaller.

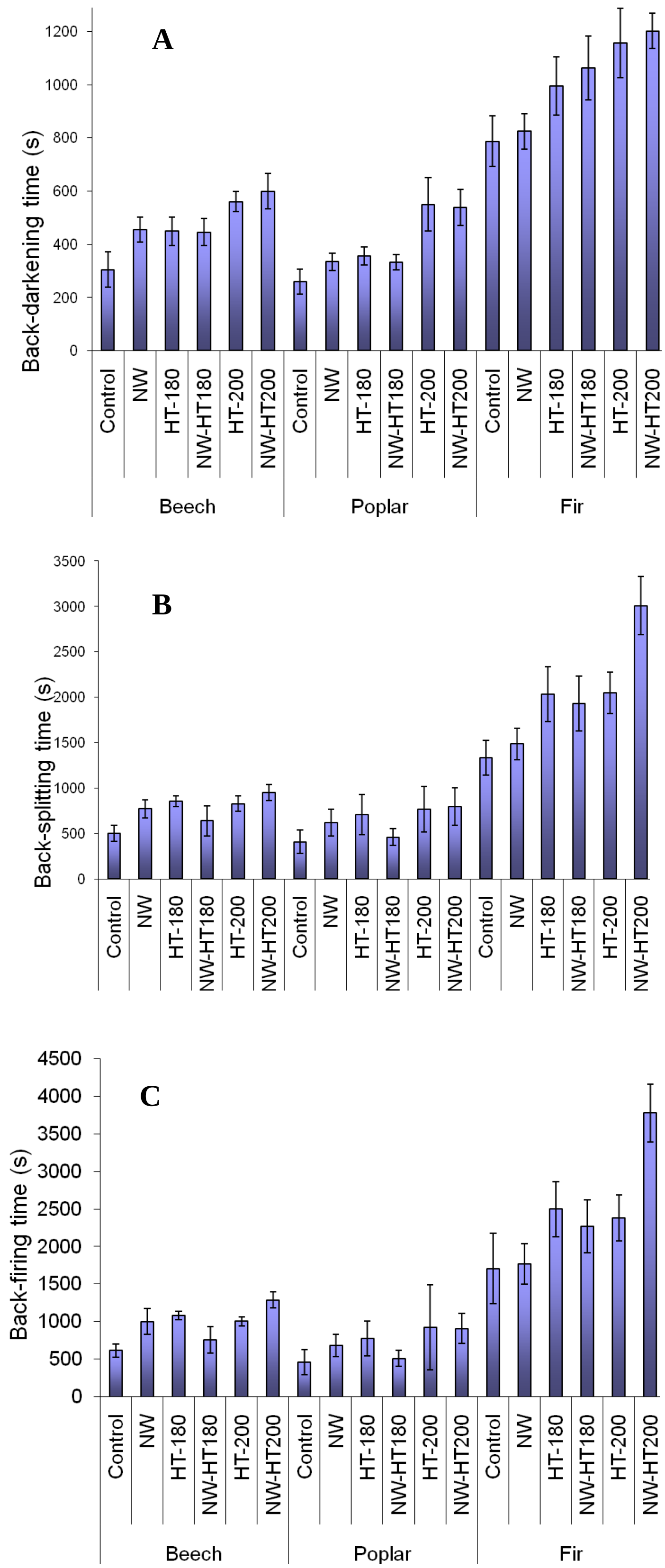
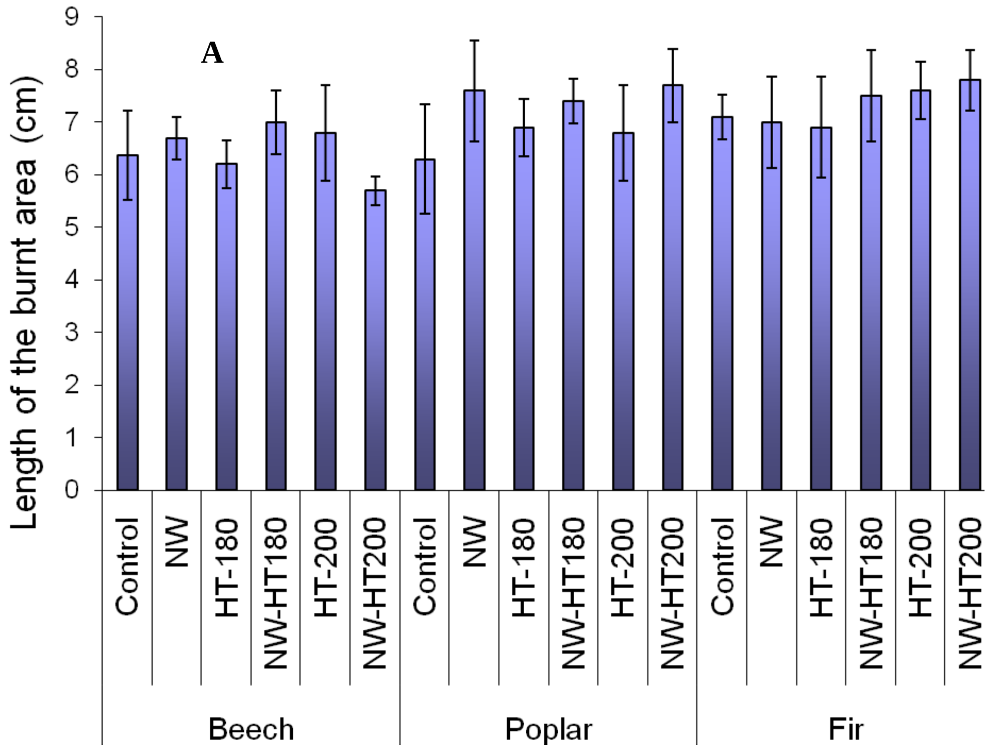


Fig. 7. Back-darkening (A), back-splitting (B), and back-firing (C) times (s) in NW-impregnated and heat-treated beech, poplar, and fir at 180 and 200 °C (NW = nano-wollastonite-impregnated; HT = heat-treated)



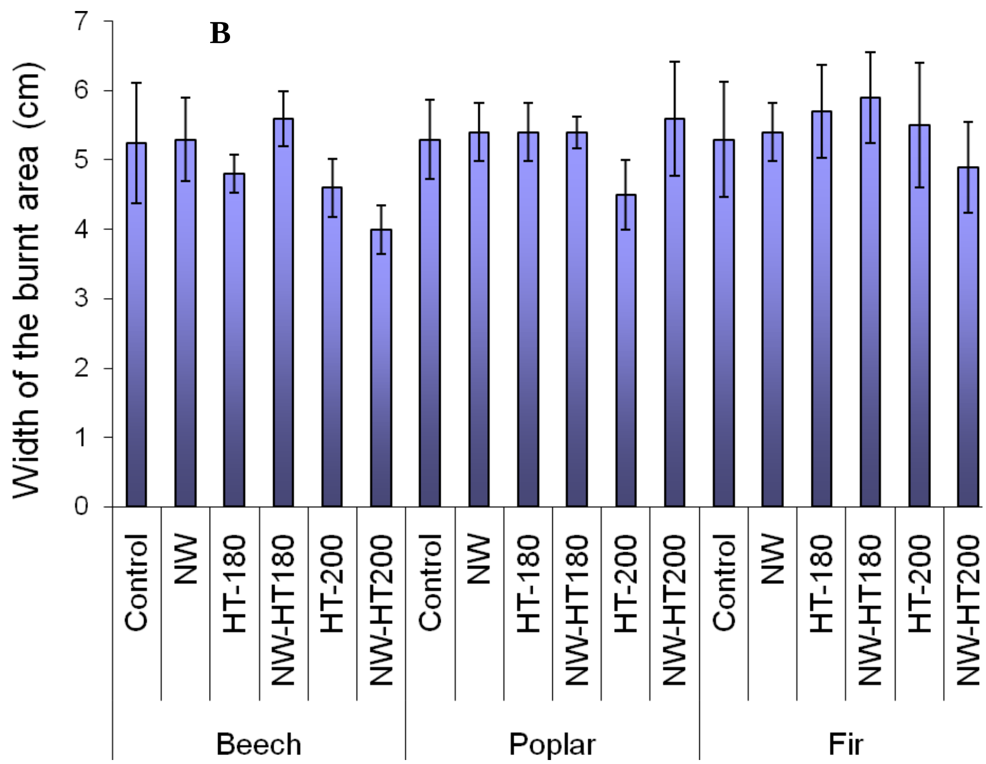


Fig. 8. Length (A) and width (B) of the burnt area (cm) in NW-impregnated and heat-treated beech, poplar, and fir at 180 and 200 °C (NW = nano-wollastonite-impregnated; HT = heat-treated)

A cluster analysis performed according to the seven fire properties explored in the present study revealed that all of the treatments relating to fir wood were clustered significantly different than the other two species (Fig. 9). This demonstrated the considerable effect of species on the fire properties of solid wood. The control specimens of both beech and poplar species were clustered very similarly.

However, the NW impregnated and heat-treated specimens were clustered quite differently. This indicated that the effect of both types of treatments (NW impregnation and heat treatment) have high, and significant, effects on fire properties.

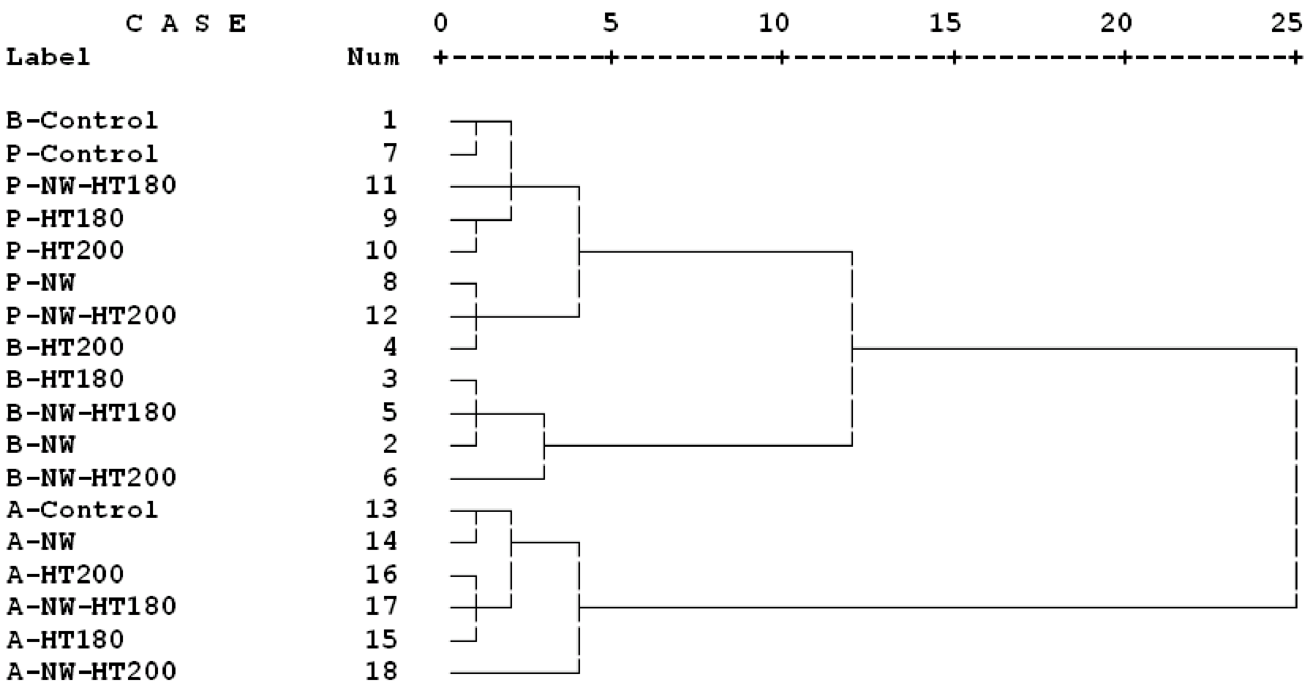


Fig. 9. Cluster analysis of the 18 treatments studied, based on seven fire properties (B = beech; P = poplar; A = fir wood; NW = nano-wollastonite-impregnated; HT = heat-treated)

An R^2 value of 76% was calculated (Fig. 10A). Moreover, high and significant correlations were calculated among the three properties related to the backside of specimens (back-darkening, back-splitting, and back-firing) (Fig. 10 B and C).

Amongst the other properties, however, no high or significant correlations were found. This may indicate that fire properties related to the spread of fire on the surface of specimens, or those related to the penetration of fire into the inner-structure of solid wood, are integrated closely together. However, no clear or meaningful relations were found between the surface and penetration properties.

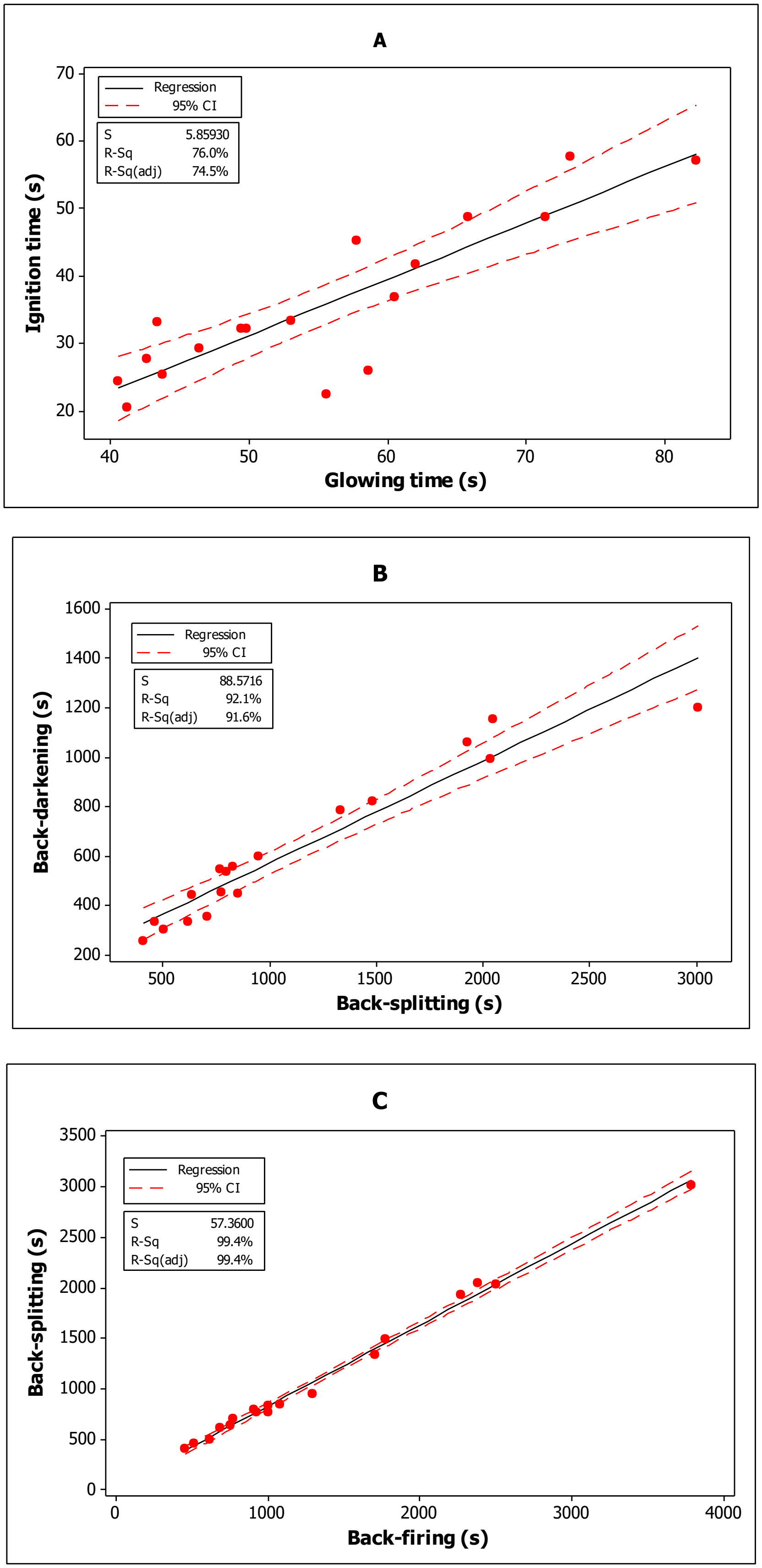


Fig. 10. Fitted-line plot between the different fire properties in the 18 treatments assessed

CONCLUSIONS

1. Treatments of nano-wollastonite (NW) impregnated solid wood at high temperatures (180 °C and 200 °C) improved the properties related to fire resistance, although not always to a significant level.
2. The mineral nature of nano-wollastonite acted as a physical shield against fire penetration. Its high thermal conductivity also improved the fire properties.
3. The degradation of cell wall components caused by the heat treatment resulted in an improvement of fire properties.
4. Fire properties were significantly dependent on the wood species. Moreover, high R-square values of the properties related to the penetration and spread of fire over and into the specimens indicated the importance of selection of proper species for particular applications.

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