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The optimum level of nano-wollastonite consumption as fire-retardant in poplar wood (*Populus nigra*)

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ABSTRACT

Fire-retarding properties of wollastonite nanofibers in poplar wood (*Populus nigra*) were studied here. Some physical properties such as water absorption, volumetric swelling and anti-swelling efficiency (ASE) were also measured. Specimens were prepared according to the ISO 11925 specifications for the fire-retarding properties and according to the ASTM D4446 -2002 specifications for the physical properties. Impregnation of wood specimens with nanowollastonite was carried out at consumption levels of 4, 6.3, 10 and 12%, using Rueping Method (empty-cell process) and compared with the control specimens. Five fire-retarding properties were measured, including weight loss (%), ignition point (s), fire endurance (s), glow endurance (s) and carbonization area (%). The obtained results indicated that fire-retarding properties were significantly improved in the NW-treated specimens. Furthermore, the NW-impregnated specimens gained higher dimensional stability. However, water absorption increased. Heat-conductivity of wollastonite limited accumulation of heat at one spot, furthermore, it acted as a physical barrier to deter heat and mass transfer between the gas and the condensed phase, consequently, fire-retarding properties were improved.

Keywords: *Poplar wood; Minerals; Nano-wollastonite; Rueping method; Fire-retarding properties; Water absorption; Anti-swelling efficiency.*

INTRODUCTION

Wood is a natural material produced by trees and uniquely applied for special purposes [1]; however, it suffers from some shortcomings, such as biological deterioration, water absorption and thickness swelling, etc. Although composite-boards offer the advantages of a homogeneous structure and the use of raw materials without restrictions as to the shape and size [2] and there are many studies to find methods for limitation of formaldehyde emission [3], however, there are many usages of solid woods than cannot be achieved by composite boards and therefore, wood is frequently modified to overcome these shortcomings [4].

One of its key disadvantages is its high flammability, which is determined by its composition [5]. The low-flammability of wood and wood based materials will contribute greatly to their applications; therefore the study on flame-retardant treatment of wood and wood based materials aiming to improve the flame-retardant properties has amused considerable industrial and scientific interests in recent years [5]. The materials commonly used as flame retardants in wood include bromine, chlorine, phosphorous, antimony, boron, nitrogen, silicon, zinc, metal hydroxides, melamine, or ammonium, or two or more of these elements. Some of these flame retardants are practically always used with another synergist, for example, antimony, boron, nitrogen, silicon, and zinc are often used with phosphorous or halogenated compounds based on chlorine and bromine, which are effective flame retardants, and ammonium polyphosphate that is known to intumesce [6-8]. They can improved fire resistance through the following mechanisms; 1) redirect decomposition and combustion reactions regarding the evolution of non-combustible gases, or produce heavy gases that interfere with the interchange of combustion gases and air, 2) redirect the combustion and decomposition reactions toward reducing the heat of combustion, 3) maintain the physical entirety of the material, and 4) increase the specific heat or thermal conductivity [6]. While there are environmental concerns toward the use of these materials, some of them are still widely used in industrial consumptions [6]. In addition, because of the water solubility, the treated wood with these materials are not suitable for exterior and underground construction, where the flame retardants are leached easily [5-6]. Furthermore, nanotechnology has been used in many sciences [9-11], the heat-transferring property of silver nanoparticles [12-26] showed some promising results to improve fire-retarding properties in solid woods [8].

In this connection, intumescent materials with commercial resin, such as styrene acrylic copolymer resin, have been demonstrated to be extremely effective in improving the fire performance of wooden materials used for indoor furnishing [27]. Also, in research for halogen-free retardant, intumescent flame retardant (IFR) has received considerable attention recently because it provides fire protection with minimum overall health hazards [28], and IFR is also the fastest and most convenient way to enhance the fire performance of wood-based materials. Under the heat of the earlier stage of a fire, intumescent paints achieve flame retardancy by forming a charred layer that acts as a physical barrier to deter heat and mass transfer between the gas and the condensed phases [29]. The charred layers, with low thermal conductivity, then form a carbonaceous layer by cyclization and cross-linking with phosphate ester [30]. The IFR system is composed of four components: (1) binder resin (BR), (2) a carbonizing substance (CS) to form the carbonized layer, such as polyols, (3) a foam-producing substance (FPS) to release inert gases NH_3, CO_2, and H_2O as well as to support intumescent action and char formation to further retard heat and mass transfer, and (4) a dehydrating agent (DA) to lower the decomposition temperature and accelerate the formation of a carbonized layer, such as ammonium phosphate, boric acid, and borax [31]. The impact of intumescent formulation has indicated that lower BR content increases the fire retardancy of coated plywood [32], and also it extends the survival duration of the phosphor-carbonaceous structure of chars.

Based on the above mentioned literature review, fire-retardant chemicals may be assessed from three main perspectives beyond flammability: reduced strength on wood and corrosion on fasteners, increased hygroscopicity, and the amount of toxic and smoke gases produced. Although all these perspectives are considered over the years and in the new fire-retardant formulations nearly all requirement are met, but fire-retarding properties of nano-wollastonite, as a silicate mineral ($CaSiO_3$), should also be studied as it does not have any acidic chemicals. In the meantime, its mineral nature may be considered as a barrier towards fire or heat transfer. Furthermore, the possibility to use it as a fire retardant could also be considered a privilege. In the meantime, wollastonite mines are plenty in Iran and could decrease the preservation costs. Therefore, the idea occurred to study the possibility of using nano-wollastonite as a fire retardant in poplar wood. Furthermore, as the standard fire testing apparatuses such as the cone calorimeter are not readily available in all laboratories, one innovative fire-testing apparatus was designed and built that nearly all laboratories can afford. In the meantime, apart from the ignitability and weight

loss properties that are always measured for reporting purposes of fire-retarding chemicals, this apparatus can measure spread of flame over time which is important for safety purposes.

EXPERIMENTAL

Specimen preparation

Wood specimens were prepared according to the ISO 11925 standard in the size of 150 (length) × 100 (width) × 9 (thickness) mm^3. The length of the specimens was in longitudinal and the width in marginal direction, for the fire-retarding properties, and ASTM D4446 -2002 standard in the size of 2 × 2 × 2 mm^3, for the physical properties. Specimens were taken so that the proportion of sapwood/heartwood was the same as there are notable differences between their density and mechanical properties. Impregnation of wood specimens with nano-wollastonite was carried out at consumption levels of 4, 6.3, 10 and 12%, using Rueping method (empty-cell process) at a pressure of 3bars for 2 hours. Each specimen was weighted just before and after impregnation to measure the NW water solution absorption (Table 1). All tests were carried out also 2 months after impregnation process of the NW. Specimens were kept in conditioning chamber (20±5 °C, and 25% relative humidity) for 2 months before the tests were carried out on them as the drying conditions may affect dimensional stability, workability, finish, and adhesive wettability. The moisture content of the specimens at the time of testing was 6.5%. Seven wood specimens were made for each treatment for fire tests and ten for physical tests.

Table 1. The amount of nano-wollastonite suspension absorption for each of the four NW consumption levels

NW consumption level (%)	Nano-wollastonite absorption (g/cm3)
4	0.597 (0.013)
6.3	0.584 (0.008)
10	0.571 (0.058)
12	0.598 (0.033)

* Numbers in brackets are the standard deviation

Water absorption and volumetric swelling of each specimen were measured after dipping in water for 2 and 24 hours. Then the anti-swelling efficiency (ASE) of the specimens was also calculated for determine the effects of nano-wollastonite on dimensional stability of treated specimens.

From the preliminary weight of specimens before dipping in water (W_1) and second weight of specimens after dipping in water (W_2), the water absorption (WA) was calculated as follows:

$$WA\ (\%) = \{(W_2 - W_1) / W_1)\} \times 100$$

From the preliminary volume of specimens before dipping in water (V_1) and second volume of specimens after dipping in water (V_2), the volumetric swelling (VS) was calculated as follows:

$$VS\ (\%) = \{(V_2 - V_1) / V_1)\} \times 100$$

From the swelling of untreated specimens (S_u) and that of impregnated specimens (S_i), the anti swelling efficiency (ASE) was calculated as follows:

$$ASE\ (\%) = \{1 - (S_i / S_u)\} \times 100$$

Nano-wollastonite (NW) gel was procured from Vard Manufacturing Company of Mineral and Industrial Products, Iran. Wollastonite mines are plenty in Iran and therefore may decrease the preservation costs. The formulations of the nanowollastonite used in the present study are summarized in Table 2. The size range of wollastonite nano-fibers was 30 – 110 nm.

Table 2. The compounds and formulation of the nano-wollastonite gel used in the present study

Nano-Wollastonite Compounds	Mixing ratio (%)
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

Fire-Retardant Testing Apparatus

Due to the unavailability of the standard tests by cone calorimeter, a special apparatus was designed by the second author (Figure 1). This apparatus was scientifically approved by The Iranian Research Organization for Science and Technology under license No. 3407. Piloted ignition was used in the design of the apparatuses [33]. The fuel in the present study was natural gas comprised of mainly methane CH_4 (90-98%); however, other hydrocarbons were also reported by the supplier to be accompanied (C_2H_6: 1-8%; C_3H_8: 2%; H_4H_{10}+C_5H_{12}: less than 1%; and also N_2 + H_2S + H_2O: less than 1.5%). The flow rate was 0.096 liters/s. The specimen was vertically mounted on a holder up-straight and exposed to a Bunsen-type burner (with the internal diameter of 11 mm) hold at 45 degrees to the surface of the specimen for 120 seconds in accordance with standard ISO 11925-3. The burner was fixed on a slide, moving back and forth, equipped with an adjustable stop to keep flame at a certain distance (5 mm) from the specimen. While the slide was back, the burner was turned on; the slide was then pulled forward abruptly to expose the flame to the specimen. The time it took for each specimen to catch an evident visible flame on the spot nearest to the Bunsen-type burner was registered as ignition point. After 120 seconds, the slide was pulled back to prevent over-exposure of the specimen to the flame. The times the specimen kept a visible fire, after the removal of the burner, as well as the time the specimen kept a visible glow, were also registered as fire and glow endurance time (the duration time of a visible flame and glow). Once the flame was extinguished or the specimen was no longer burning, the carbonization area was measured. The weight was also measured before and after the test to measure the weight loss. The whole structures of apparatus were put in a three-wall-compartment in order to protect the burning flame from wind and air movements.

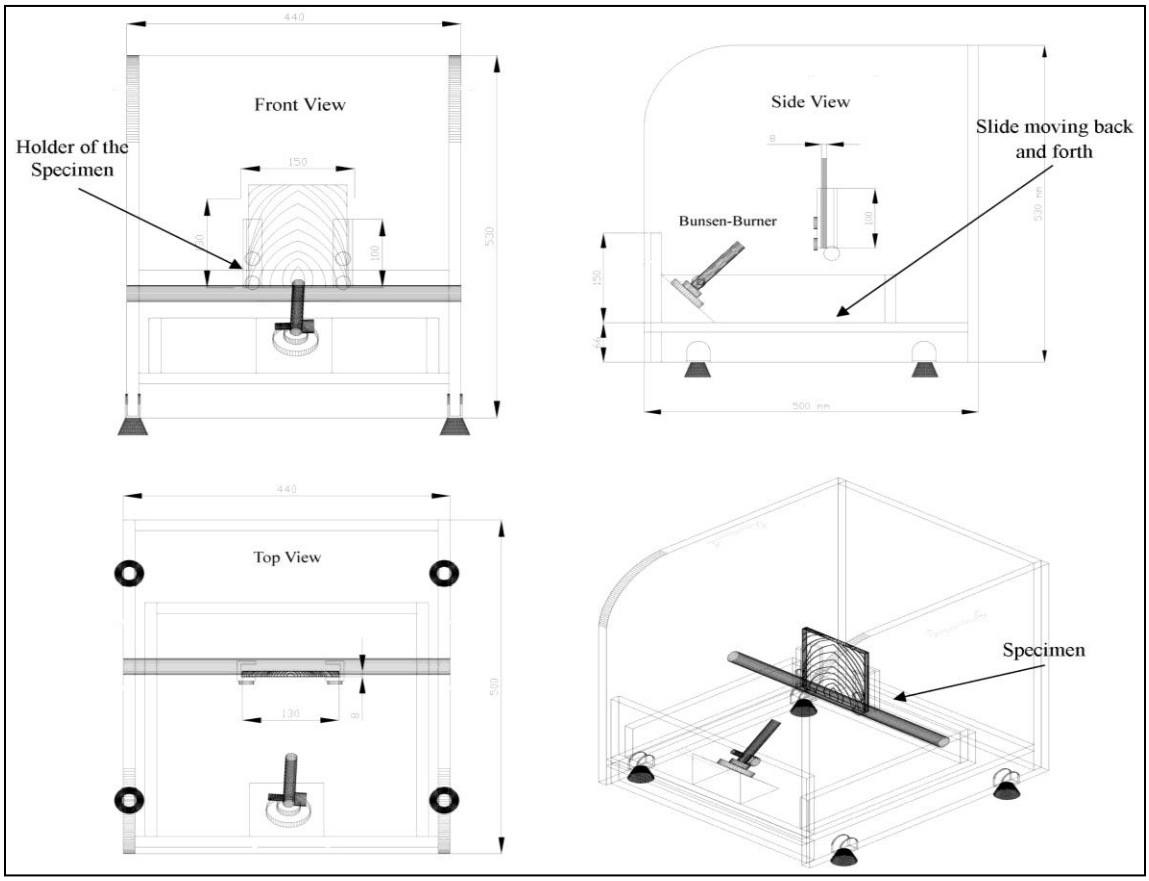

Fig. 1. Schematic drawing of the slide fire testing apparatus (SFTA) (Iranian patent No. 67232; approved by The Iranian Research Organization for Scientific and Technology under license No. 3704)

Statistical Analysis

Statistical analysis was conducted using SPSS software program, version 16. One-way analysis of variance (ANOVA) was performed on the data to conclude significant differences at the 95% level of confidence. Hierarchical cluster analysis, including dendrogram and using Ward methods with squared Euclidean distance intervals, was also carried out by this software.

RESULTS AND DISCUSSION

Weight Loss

NW-treatments showed decreasing affects on the weight loss in comparison to the control specimens (Figure 2). The lowest of weight loss was observed in NW10% treated specimens (48.22% in comparison with control specimens). NW4% treated specimens did not show significant difference with the control specimens.

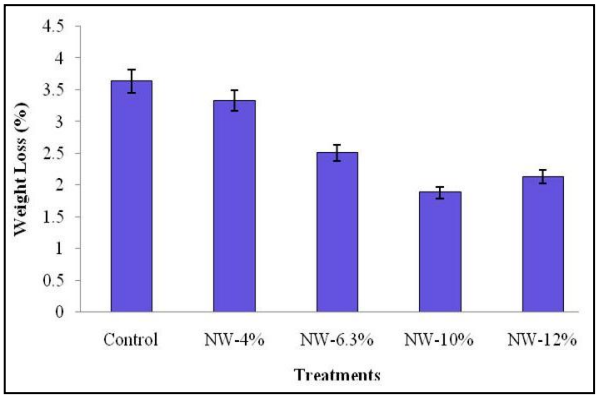

Fig. 2. Weight loss (%) for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (%)

Ignition Point

NW-treatments had also increasing effects on the ignition point (Figure 3). This increasing effect is outstanding in NW12% treated specimens (90.99% in comparison with control specimens). NW4% treated specimens did not show significant difference with untreated specimens.

Fire Endurance

The results showed high significant difference between the untreated and NW-treated specimens (Figure 4). Fire endurance decreased in all NW-treated specimens. Only NW4%-impregnated specimens do not seem much different to normal untreated wood. In NW6.3%-treated specimens, reduction of 95.45%, as well as NW10 and 12%-treated specimens was observed.

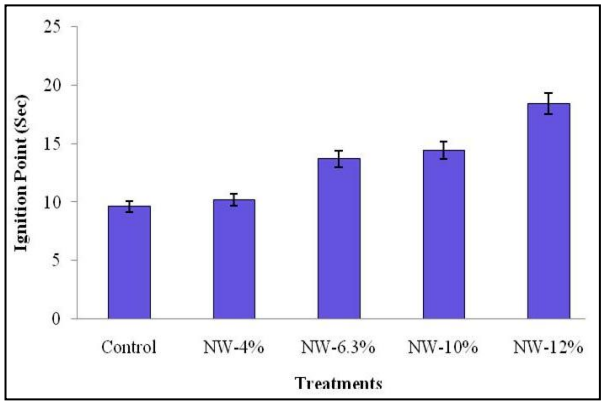

Fig. 3. Ignition point for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (Sec)

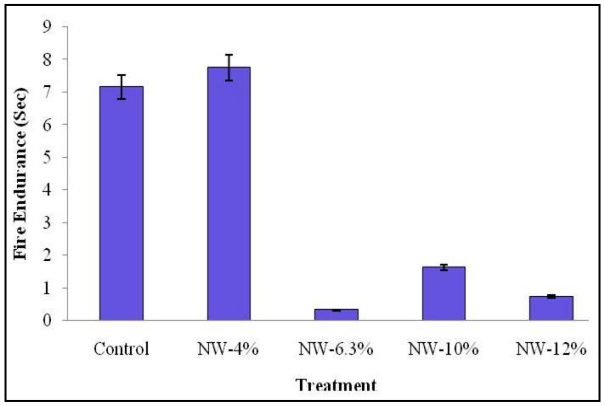

Fig. .4 Fire Endurance for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (Sec)

Glow Endurance

All NW-treated specimens significantly tended to decrease the glow endurance in comparison to untreated specimens (Figure 5).

Carbonization area

Nano-wollastonite impregnation in four consumption levels has made carbonization area to decrease (Figure 6). For example in NW12%-treated specimens, reduction of 52.76% observed as well as NW6.3 and 10%-treated specimens.

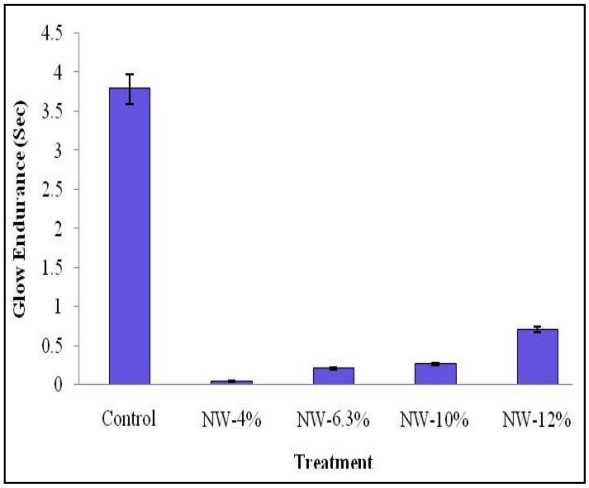

Fig. 5. Glow Endurance for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (Sec)

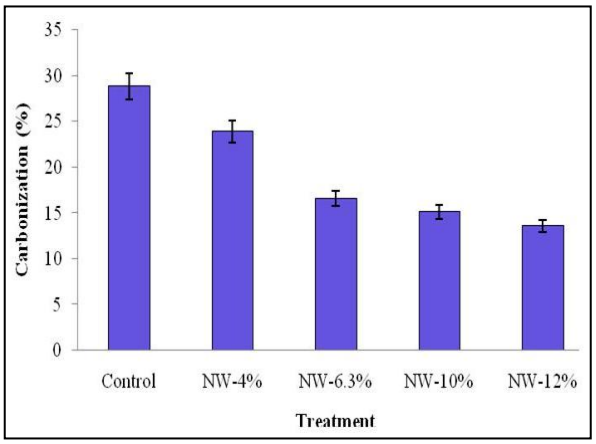

Fig. 6. Carbonization area for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (%)

Water absorption

The measures of water absorption after dipping in water were increased and this increase was evident after 24 hours (Figure 7). There was no significant difference between water absorption in variety NW-treated specimens after 2 hours. The highest and lowest water absorption in impregnated specimens was seen in NW12 and 4%-treated specimens (an increase of 32.77% and 25.59% after 2 hours, 40.38% and 21.46% after 24 hours in comparison with the untreated specimens respectively).

Volumetric swelling

Volumetric swelling after dipping in water was decreased (Figure 8). The lowest of this property was seen in NW12%-treated specimens (a decrease of 39/52% after 2 hours, 41.06% after 24 hours in comparison with control specimens). The NW10%-treated specimens almost improved this property as well as NW4 and 6.3%-treated specimens.

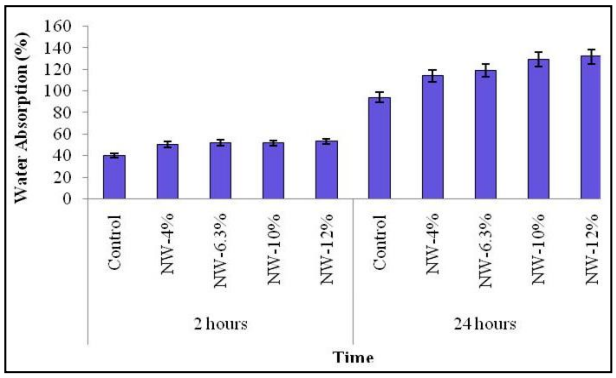

Fig. 7. Water Absorption after 2 and 24 hours dipping in water for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (%)

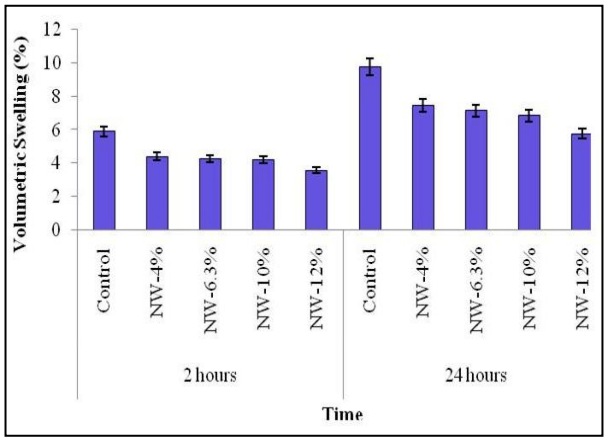

Fig. 8. Volumetric swelling after 2 and 24 hours dipping in water for 5 treatments of control specimens, nano-wollastonite-impregnated specimens in four levels of consumption (%)

ASE

The results showed that NW-impregnation significantly increase dimensional stability of the treated specimens (Figure 9). The highest ASE was seen in NW12%-treated specimens (39.52% and 41.07% after 2 and 24 hours dipping in water, respectively).

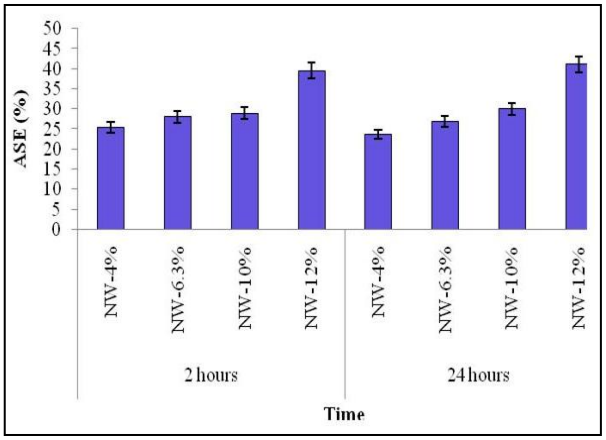

Fig. 9. ASE after 2 and 24 hours dipping in water for 4 treatments of nano-wollastonite-impregnated specimens (%)

As to the weight loss, NW treatments showed significant difference with the control specimens (Figure 2). Only NW4%-impregnated specimens did not seem much different to normal untreated wood and are therefore clustered with the control specimens. There was no significant difference between NW10 and 12%-treated specimens. Overall, these two NW content can be considered an optimum levels of NW-consumption for the weight loss. This property exactly shows fire-retarding property of Nano-wollastonite [34].

All NW-treatments increased the ignition point significantly, except NW4% treated specimens; this can imply that NW-consumption level of 4% is not enough to significantly increase the ignition point. The amount of increase is outstanding in NW12% treated specimens (90.99%). An increase in ignition point shows NW's heat-transfer property that is more than wood's but less than metallic material's (Table 3) [35]; that is, due to the transfer of heat from the spot nearest to the burning flame of the Bunsen-type burner, accumulation of heat is delayed at the spot, and therefore, the ignition point is increased. Wollastonite nano-fibers were also reported to increased thermal conductivity coefficient of medium density fiberboards (MDF) [36]. Heat-transferring property of silver nanoparticles [12-21] was also reported to improve some of the fire-retarding properties in solid woods [8].

Table 3. Thermal conductivity of wood, silver and wollastonite

Material	wood	silver	wollastonite
Thermal conductivity (w/m k)	0/19 - 0/28*	4.29	2.16

* Thermal conductivity of wood in linear dimension

As to the fire endurance, all NW treatments showed significant difference with the control specimens it is only in NW4% treated specimens (Figure 4). This property exactly shows fire-retarding property of Nano-wollastonite. There was no significant difference between 6.3, 10 and 12% NW-treated specimens. Overall, these three NW content can be considered an optimum level for fire endurance. This proves that Nano-wollastonite particles tend to alter the nature of wood and may only transfer the heat and therefore delay the process of heat accumulation. Once the surface layer of wood has reached the temperature to be ignited, the further process of fire endurance seem much different to normal untreated woods; Nano-wollastonite impregnation decrease fire endurance to a rather noticeable extent (Figure 4).

The highest glow endurance was observed in untreated specimens, and all NW-treatments show the same effect on this property. All NW contents (4, 6.3, 10 and 12%) generally resulted in a decreasing trend in glow endurance and there was no significant difference between these NW contents.

The fire-retarding property of Nano-wollastonite to some extent is obvious from studying the carbonization area (Figure 6). Nano-wollastonite 6.3, 10 and 12%-impregnated specimens, on the other hand, do not show any significant difference on this property although these made it to be decreased. And NW content of 4% also significantly decreases the carbonization area.

The lowest water absorption was observed in untreated specimens, and all NW-treatments show the same effect on this property after 2 hours dipping in water. All NW contents (4, 6.3, 10 and 12%) generally resulted in an increasing trend in water absorption and there was

no significant difference between these NW contents after 2 hours. The amount of this property was same in both NW10 and 12%, also NW4-6.3% treated specimens after 24 hours. This result may be due to high pressure (3 atm) application method and strike Nanoparticles that led to create tiny crack within pore on cell wall membrane and this result more relevance (collectively) porosity, therefore more water absorption was seen.

In discuss to decrease in volumetric swelling after 2 and 24 hours, the lowest amount of this property was observed in NW12% and after that in NW10% as well as NW6.3% treatments. To be noted to increase of dimensional stability, it can be concluded that NW particles could have a good coverage on cell wall and prevented hydroxyl groups' action with water molecules.

Clustering the five treatments, based on all the fire (weight loss, ignition point, fire and glow endurance, carbonization area) and physical (water absorption and volumetric swelling) measured properties, again shows that control specimens are clustered quite separately, it is only in NW 4% treatments of fire retarding properties that clustered with untreated specimens together (Figures 10, 11, 12).

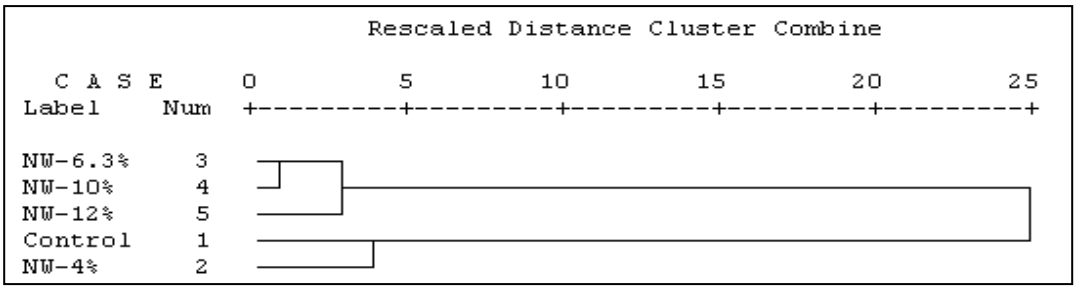

Fig. 10. Cluster analysis of the five treatments on all fire measured properties (weight loss, ignition point, fire and glow endurance, carbonization area)

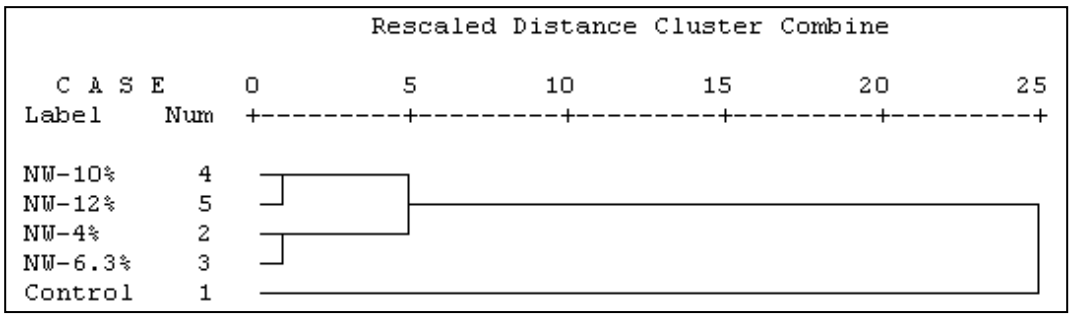

Fig. 11. Cluster analysis of the five treatments on all physical measured properties (water absorption and volumetric swelling)

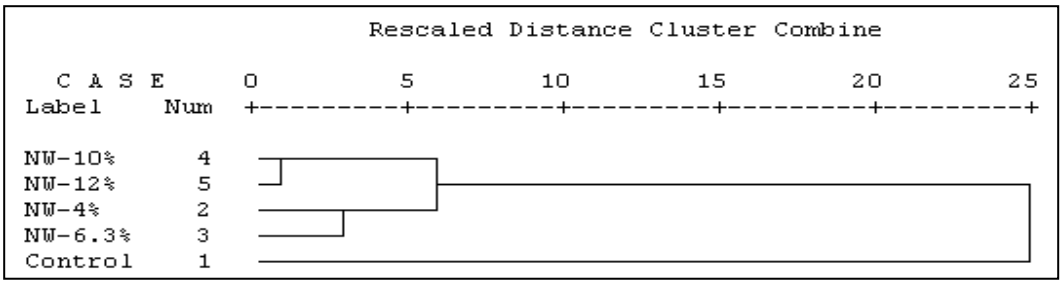

Fig. 12. Cluster analysis of the five treatments on all measured properties (weight loss, ignition point, fire and glow endurance, carbonization area, water absorption and volumetric swelling)

The heat-transfer property of the wollastonite nanoparticles delayed accumulation of the heat needed for the burning process in the surrounding parts of flame and therefore, the carbonized area was naturally decreased in all cases of nano-wollastonite-impregnated specimens. Furthermore, wollastonite nanofibers formed a layer that acts as a physical barrier to deter heat and mass transfer between the gas and the condensed phases [29]. It may be concluded that nano-wollastonite clearly shows some potentials in improving some of the fire-retarding properties in poplar wood. It is to be noted that it uses its heat-transferring property in this regard. Therefore, its other aspect as fire retardants should still be studied in detail, such as toxic gases and smoke as well as hygroscopicity.

In the meantime, cluster analysis of the five treatments, based on all properties measured (weight loss, ignition point, fire and glow endurance, carbonization area, water absorption and volumetric swelling), showed that two pairs of the treatments were clustered quite closely, namely the NW12% and NW10% as well as NW6.3% and 4% treatments (Figure 12). It may then be concluded that improvement in fire-retarding and physical properties of NW-4% and NW-10% are not significantly different from NW-6.3 and -12%, respectively. NW4% treatment had a tendency to be clustered with control treatments; it may imply that 4% of NW is not enough to improve the fire-retarding properties. However, NW10%, and 12% are clustered closely which approves their similarity in fire-retarding and dimensional stability properties. In this connection, the improved fire-retarding and dimensional stability values of NW10 prove that the optimum treatment.

CONCLUSIONS

1- Nano-wollastonite significantly improved fire-retarding properties in poplar wood; heat-conductivity of wollastonite limited accumulation of heat at one spot, furthermore, it acted as a physical barrier to deter heat and mass transfer between the gas and the condensed phase, consequently, fire-retarding properties were improved;

2- Nano-wollastonite increased dimensional stability in poplar wood; however, water absorption increased due to more water absorbed by wollastonite nanofibers;

3- NW-consumption level of 4% is not enough to significantly improve fire-retarding properties; also, NW-12% was closely clustered to NW-10% in all analysis, therefore, NW-10% is recommended to improve fire-retarding properties as well as increase dimensional stability in poplar wood.

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