

RESEARCH ARTICLE

Improving fire retardancy of medium density fiberboard by nano-wollastonite

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Summary

The improving effects of addition of nano-wollastonite on some fire properties of medium-density fiberboard (MDF) were studied in this work. Nano-wollastonite was added at four levels (2%, 4%, 6%, and 8%). The size range of at least 70% of nano-wollastonite particles were 30 to 110 nm. The results showed statistically significant improving effect of nano-wollastonite on time to onset of ignition. The improving effect was primarily attributed to an increase in thermal conductivity of board containing nano-wollastonite. This increase in turn resulted in better curing of resin, and a higher integrity of fibers thereof. Moreover, nano-particles provided a surface with reduced combustibility and therefore, penetration of fire to the inner layers of boards was delayed, thus improving fire properties. High and significant correlations were found between thermal conductivity coefficients of boards with different fire properties. It was concluded that for applications where fire properties are of prime importance, nano-wollastonite content of 8% can be recommended. Moreover, further studies are needed to compare and standardize the results obtained from the apparatus used here with those obtained from internationally recognized apparatuses like cone calorimeter.

KEYWORDS

medium-density fiberboard (MDF), minerals, nano-materials, thermal conductivity coefficient, Wollastonite

1 | INTRODUCTION

Wood is a versatile material with numerous applications and therefore, its plantation and harvesting is vastly studied all over the world.¹⁻⁶ The idea of protecting solid wood, and wood- and cellulose-based materials against different physical and chemical damages and attacks of living micro-organisms is as old as human civilizations.⁷⁻¹¹ Over time, numerous methods and materials have been examined and developed. Some methods involved changing the pathway of pyrolysis of wood.¹² This method is considered as one of the easiest and inexpensive ways to treat wood to reduce flammability. In another method, the surface of wood is protected with an isolating layer. Intumescent coatings are also categorized in this method. Alteration in the thermal properties of wood through changing its density, specific

heat, and thermal conductivity is another one that can also be used to improve fire retardancy in wood and wood-based composites. Other techniques can involve reduction in wood combustion by diluting pyrolysis gases or inhibiting the chain reactions of burning. Though several methods and techniques have been developed so far, research for more effective and non-toxic materials to improve fire retardancy is still in progress.^{11,13-17}

Improving effects of nanotechnology on different materials have been intensively and vastly studied.^{6,18-22} Wood-based materials and composites are no exception.^{23,24} Different nano-metals and nano-minerals were utilized to improve the heat-transfer property in solid wood species and wood composite mats; they were also used to improve biological resistance against different deteriorating fungi,^{7,9,25-28} to decrease hot-press time as a costly bottle-neck in

nearly all wood-composite manufacturing factories, and to increase thermal conductivity in solid wood and composite mats.²⁹

In this connection, wollastonite is a calcium inosilicate mineral (CaSiO_3), which contains small amounts of iron, magnesium, and manganese substituting for calcium, that has been proved as non-toxic and harmless for both human health and wildlife.³⁰⁻³⁴ Nano-wollastonite was reported to significantly improve the biological resistance of wood and wood-based composites against wood-deteriorating fungi,^{35,36} to improve thermal conductivity, physical and mechanical properties of wood-based composites,³⁶ and to give promising results in lowering ignitability of wood and composites at high retention levels.³⁷⁻⁴¹ However, the authors came across little or no research projects studying the outcome of nano-wollastonite (NW) at lower levels. Therefore, the present research work was carried out to find out if lower contents of NW can have improving effects on fire-properties of medium-density fiberboard (MDF) as a vastly used wood-based composite around the world.

2 | MATERIALS AND METHODS

2.1 | Board production

Wood fibers were purchased from the Sanaye Choobe Khazar Company in Iran (MDF Caspian Khazar). They were comprised a mixture of five species, namely beech (*Fagus orientalis*), alder (*Alnus subcordata*), maple (*Acer sp.*), and hornbeam (*Carpinus betulus*) from the neighboring forests of Amol city (Mazandaran Province), and poplar (*Populus nigra*) from private forest plantations. MDF boards were fabricated with 10 mm in thickness, and a target density of 750 kg/m^3 . A HT/MLM-170 hot press (Mehrabadi Mfg. Co., Tehran) was used with a nominal pressure of 160 bars. Hot-press temperature was 160°C , and duration was 7 minutes, for all treatments. Urea-formaldehyde resin (UF), a thermosetting resin from Sari Resin Manufacturing Company (Sari, Iran) was sprayed on fibers; it had a concentration of 10%, a viscosity of 200 to 400 cP, a gel time of 47 seconds, and a density of 1.277 g/cm^3 . Five replicate boards were produced in random order for each treatment. From each board, two fire specimens were cut to measure fire properties.

For nano-wollastonite-treated (NW) boards, NW was mixed with resin and then sprayed on fibers. Four NW concentrations (2%, 4%, 6%, and 8%) were used based on dry weight of UF resin. NW gel was produced by Vard Manufacturing Company of Mineral and Industrial Products (Iran). At least 70% of particles ranged from 30 to 110 nm, with the remaining 30% bigger than this range. Formulation of NW gel has been reported by Taghiyari et al.⁷ Boards were conditioned in an environmental room ($25^\circ\text{C} \pm 2^\circ\text{C}$, $50\% \pm 3\%$ relative humidity) to reach an equilibrium moisture content of $7.5\% \pm 2\%$, before the fire property tests.

2.2 | Fire property tests

Studies on fire properties and heat release of newly developed composites and materials are usually reported based on cone calorimeter,

single item test, room corner test, or the Steiner tunnel test. However, not all material testing laboratories can afford these costly tests and apparatuses. Therefore, the idea of designing an easy apparatus and method was brought up so that every laboratory can test the surface flammability of new composites. Slide Fire Test Apparatus (SFTA) was designed and built, using piloted ignition, with the primary idea to test different surface flammability properties in low costs^{39,40} (Figure 1). Still, it is to be noted that SFTA does not provide some information such as heat release rate, as other internationally accepted apparatuses like cone calorimeter does, and therefore, further comparing studies should be carried out to come to a firm conclusion as to the functionality and effectiveness of new retardants found promising by SFTA. However, the ignitability of surfaces treated with newly developed retardants can easily and trustfully be measured by SFTA for comparison purposes with the results obtained from un-treated surfaces. Therefore, SFTA can give a primary idea with the lowest cost possible, and an easy way for comparison for new researchers, as to the impact of new fire retardants and additives. Natural gas was used as the fuel; it mainly comprised of methane CH_4 (90%-98%). The producer reported that other hydrocarbons accompanied methane (C_2H_6 : 1%-8%; C_3H_8 : 2%; $\text{H}_4\text{H}_{10} + \text{C}_5\text{H}_{12}$: less than 1%; and also $\text{N}_2 + \text{H}_2\text{S} + \text{H}_2\text{O}$: less than 1.5%). The gas flew steadily at 0.097 lt/s through a Bunsen-type burner held at 45° to burn specimens that were vertically positioned on a holder. The internal diameter of the burner was 11 mm. The burner was mounted on a slide, easily moving back and forth from the specimen. Once gas was ignited with the exact flow rate, and specimen was positioned firm in place, the slide was moved forward so that specimen was exposed to fire from the burner. The time it took for the specimen to develop a visible flame was measured as the "time to onset of ignition." This property shows ignitability of a surface. When the ignitability of a control surface is compared to that of a retardant-treated surface, comparison can be made as to the effectiveness of the retardant. Similarly, the time it took for the specimen to glow was registered as the "time to onset of glowing." Each specimen was exposed to piloted fire for 120 seconds in accordance with the standard ISO 11925-3. Once the fire-exposing time was up, a slide on which the burner was mounted, was pulled back to discontinue fire-exposure immediately. However, the time that evident fire continued on each specimen was registered as "duration of burning." This property indicates the potential of a surface to spread fire across; that is, how long a surface can keep fire on it when a source fire is extinguished. The shorter a surface keeps an evident fire, the better, as this reduces the potential of spreading fire across a panel. Once the flame on each specimen was extinguished, and it was cooled off, the length and width of the burnt area were measured.

2.3 | Thermal conductivity measurement

Thermal conductivity was measured using a KD2-Pro Thermal Properties Analyzer device, produced by Decagon Devices Inc. (USA). Heat was applied to a single needle (TR-1) for a time (th). The temperature was monitored in the needle of the device all the way during heating

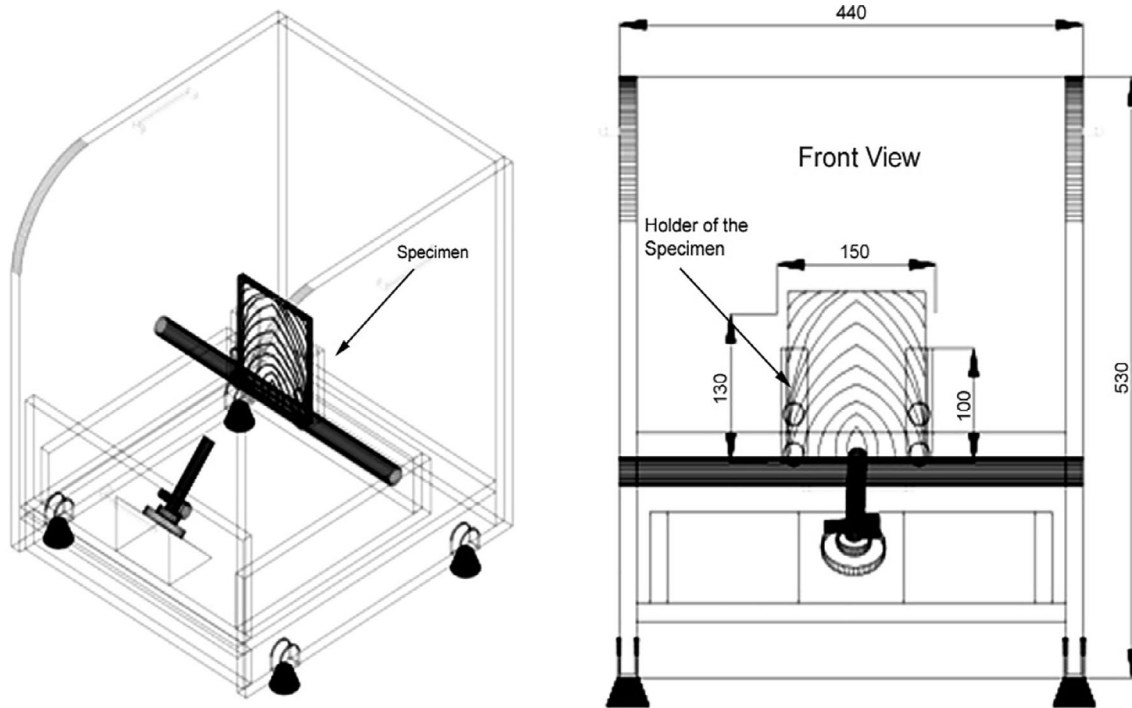


FIGURE 1 Schematic picture of the slide fire testing apparatus (SFTA) (invented under Iranian Patent No. 67232; approved by Iranian Research Organization for Science and Technology under license No. 3407)³⁹

and for an additional time equal to th after heating. The temperature during heating was then calculated by Equation (1).³⁵

$$T = m_0 + m_2t + m_3 \ln t \quad (1)$$

where m_0 is the ambient temperature during heating, m_2 is the rate of background temperature drift, and m_3 is the slope of a line relating temperature rise to logarithm of temperature.³⁵

The thermal conductivity was then calculated by Equation (2).

$$k = \frac{q}{4\pi m_3} \quad (2)$$

where k is the thermal conductivity ($\frac{W}{m \cdot K}$), q is the heat input (W), and m_3 is the slope of a line relating temperature.³⁵

2.4 | SEM imaging

A scanning electron microscope (SEM; Hitachi SU8010, Japan) was used at the Thin-Film Laboratory, FE (field emission)-SEM lab, School of Electrical and Computer Engineering, The University of Tehran.^{35,41,42} The SEM apparatus was equipped with a field-emission cathode in the electron gun, providing a narrow probe beam to improve spatial resolution and to minimize sample charging and damage. Preparation of specimens included mounting them on an aluminum stub with double-sided tape and sputter coated with a gold alloy.^{35,41,42}

2.5 | Statistical analysis

Statistical analysis was carried out by a SAS software, version 9.2 (Cary, NC USA). One-way analysis of variance (ANOVA) was performed on the average values to ascertain significant differences at the 95% level of confidence. Hierarchical cluster analysis, including dendrograms and Ward methods (using squared Euclidean distance intervals), were carried out using SPSS/18, version 18 (IBM, USA). The scaled indicator above the cluster shows similarities and differences on a number-value basis between treatments. Smaller numbers on the scale indicator mean more similarity, while larger numbers indicate that there are differences to a greater extent. When different treatments are connected by a vertical line on smaller numbers on the scaled indicator, it means that these treatments are overall considered similar, and when they are connected on larger numbers, they may be considered quite different. Fitted-line, contour, and surface plots were designed in Minitab software, version 16.2.2 (Minitab Inc., USA).

3 | RESULTS AND DISCUSSION

Results of the present study clearly showed that there was a statistically significant difference at 95% level of confidence in the time to onset of ignition among the treatments (Figure 2A). The highest and lowest times to onset of ignition were observed in NW-8% (25 seconds) and control (11.7 seconds) boards, respectively. That is, the ignitability of the surface was decreased to less than half by addition of 8% nano-wollastonite to UF resin.

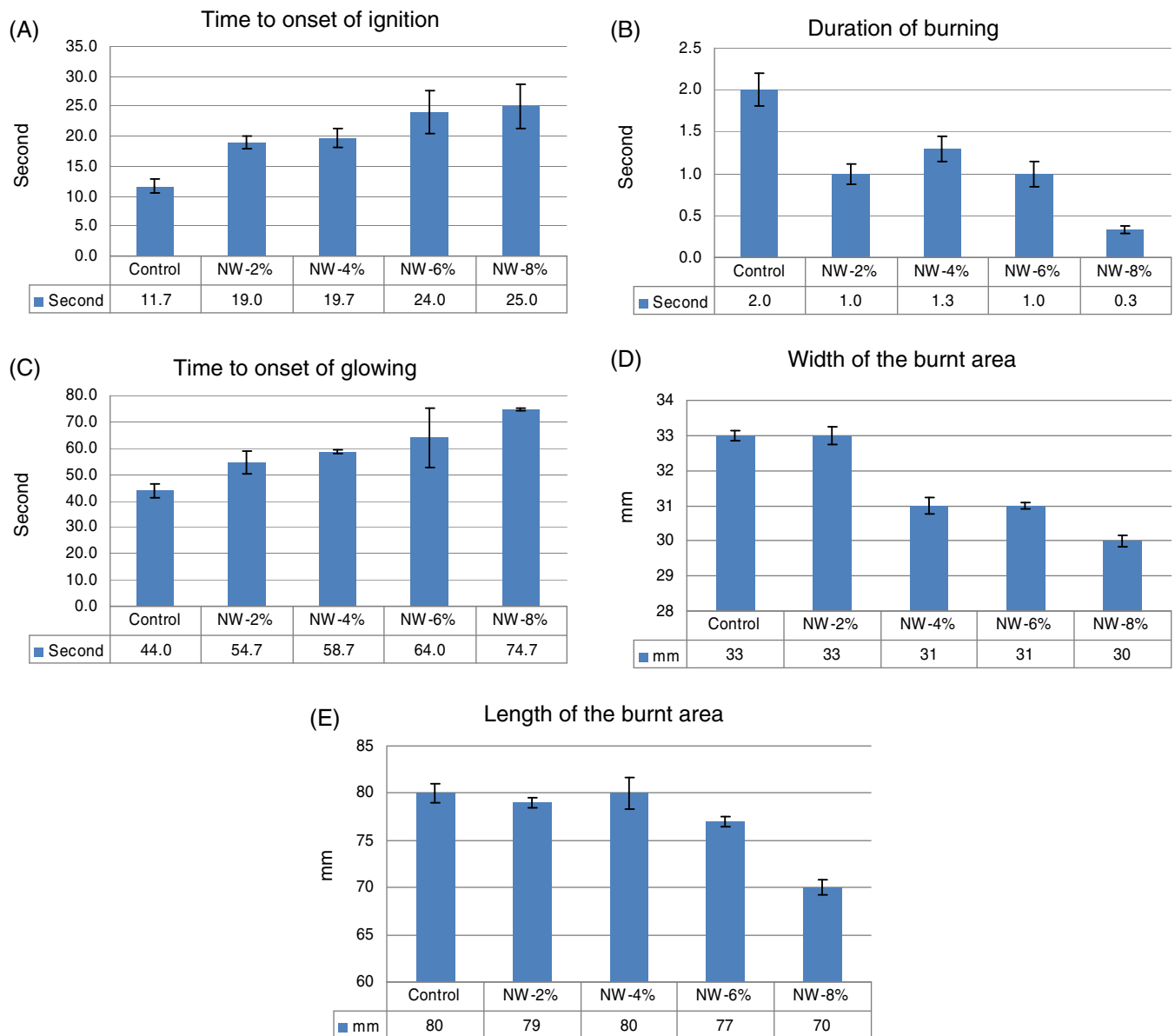


FIGURE 2 Fire properties of times to onset of ignition A, and glowing B, duration of burning C, width D, and length E of the burnt area in the five treatment boards (NW, nanowollastonite; 2, 4, 6, and 8 = nanowollastonite contents) [Colour figure can be viewed at wileyonlinelibrary.com]

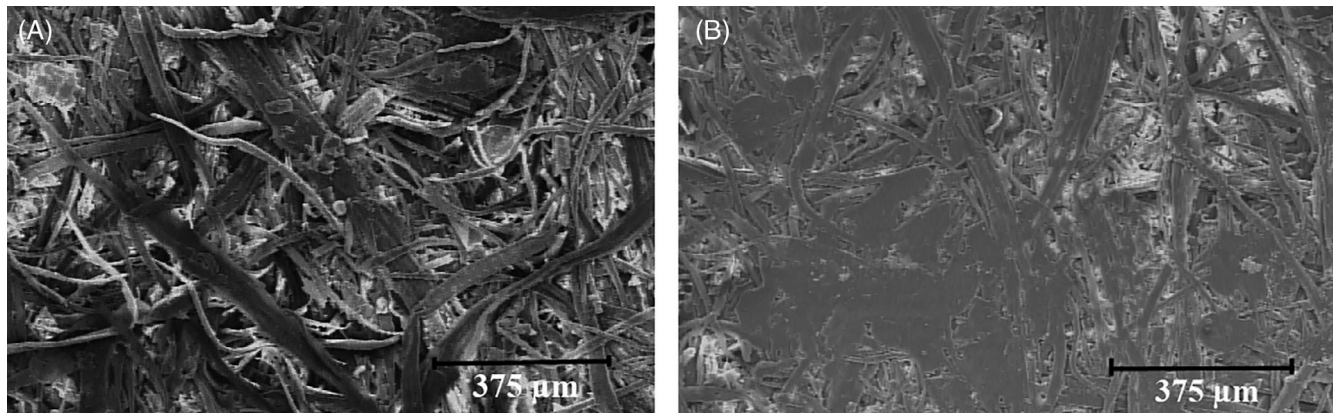
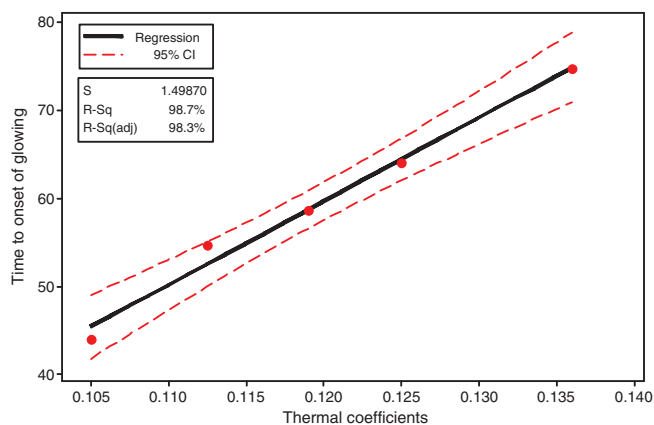
Time to onset of ignition was increased as NW-content increased, though in a few cases the increase was not statistically significant. This increase was attributed to some reasons. Firstly, addition of NW to boards improved thermal conductivity coefficient (Table 1).^{35,43} This improvement made the fibers on the surface of MDF boards to better stuck together and kept firmly in their position, consequently a higher integrity of fibers in composite was achieved, and eventually an improvement in mechanical properties was reported.^{35,44} This better integrity of fibers delayed the process of catching fire on the surface layer of the boards containing NW; in other words, loose fibers in control boards tend to catch on fire more easily in comparison with more integrated fibers in NW-treated boards. Scanning electron microscopy (SEM) showed loose fibers on the surface of control MDF boards (Figure 3A), while NW-treated board showed higher integrity

among surface fibers (Figure 3B). In this connection, high and significant correlation was calculated between thermal conductivity coefficients of different treatment boards with the fire properties measured in the present project. The *R*-square between thermal conductivity coefficients and time to onset of glowing was as high as 98.7% (Figure 4). Furthermore, it has been reported that NW makes bonds with the wood cell-wall polymers.⁴² In that work, a strong adsorption of NW on cellulose polymers was calculated. As reported, these new chemical bonds largely contribute to improvements in the physical and mechanical properties of composite panels.^{35,43-46} The ultimate outcome of the formation of new bonds would be a better integrity among fibers in the composite matrix, resulting in an improvement in fire properties. In addition, NW had a second impact on increasing the time to onset of ignition by transferring the heat at the point nearest

TABLE 1 Thermal conductivity coefficient ($\text{W m}^{-1} \text{K}^{-1}$) in the five treatments of the medium-density fiberboards (NW = nanowollastonite content)³⁵

MDF board treatments	Control	NW-2%	NW-4%	NW-6%	NW-8%
Thermal conductivity coefficient ($\text{W m}^{-1} \text{K}^{-1}$)	0.105 (C)*	0.113 (B)	0.119 (B)	0.125 (A)	0.136 (A)

Note: *A, B, and C denote statistically significant differences ($P < .05$).

**FIGURE 3** SEM images of untreated A, and NW-treated B, MDF boards: loose wood fibers on the surface of untreated (control) board are characteristically shown⁴²**FIGURE 4** Fitted-line plot of the five treatment boards calculating R-square of 98.7% between thermal coefficient values vs times to onset of glowing in the five treatment boards [Colour figure can be viewed at wileyonlinelibrary.com]

to the piloted ignition throughout the body of specimens. That is, accumulation of heat to reach the ignition level was significantly delayed.¹²

Similarly, a statistically significant improvement in time to onset of glowing was observed by an increase in NW-content of boards (Figure 2B). The highest and lowest times to onset of glowing occurred in NW-8% (74.7 seconds) and control (44 seconds) boards, significantly. This increase in time to onset of glowing was attributed to the same reason that was discussed above for the time to onset of ignition. Contour and surface plots clearly demonstrated an

increasing trend in both times to onset of ignition and glowing versus thermal conductivity coefficients (ie, NW-content) increased (Figure 5A,B).

With regard to fire property of duration of burning once the piloted fire was removed from specimens, the highest and lowest durations were in agreement with the improvements achieved in the two properties discussed above as a result of addition of NW (Figure 2C). It is to be noted that for the two properties of times to onset of ignition and glowing, the improvement would be in form of an increase in the time values, while improvement in duration of burning would be in form of a decrease. The highest duration of burning was in control boards (2 seconds). The lowest duration was found in boards with the highest NW-content (0.3 second). This indicated that the time needed for flame to spread across the surface of NW8%-treated MDF panels would be improved to more than 500% in comparison to control panels. This indicated the effect of the addition of NW in shortening the time to the extinguishment of a visible fire on specimens. In this regard, the high duration of burning in the control boards is attributed to their lower mechanical properties.³⁵ That is, fibers on surface of boards were not as strongly integrated to each other as in boards with NW-content, resulting in an easier catching on fire and higher duration of burning in the control boards. Moreover, the mineral nature of NW particles provided a surface with reduced combustibility against penetration of fire to inner parts of boards, still decreasing duration of burning. It is also hypothesized that inclusion of NW may have increased the density and consequently created a blockage to volatile emissions from the subsurface of the pyrolysis zones, delaying pyrolysis. In this connection, former studies reported a significant decrease in both gas and liquid permeability of nano-treated composites,⁴⁵

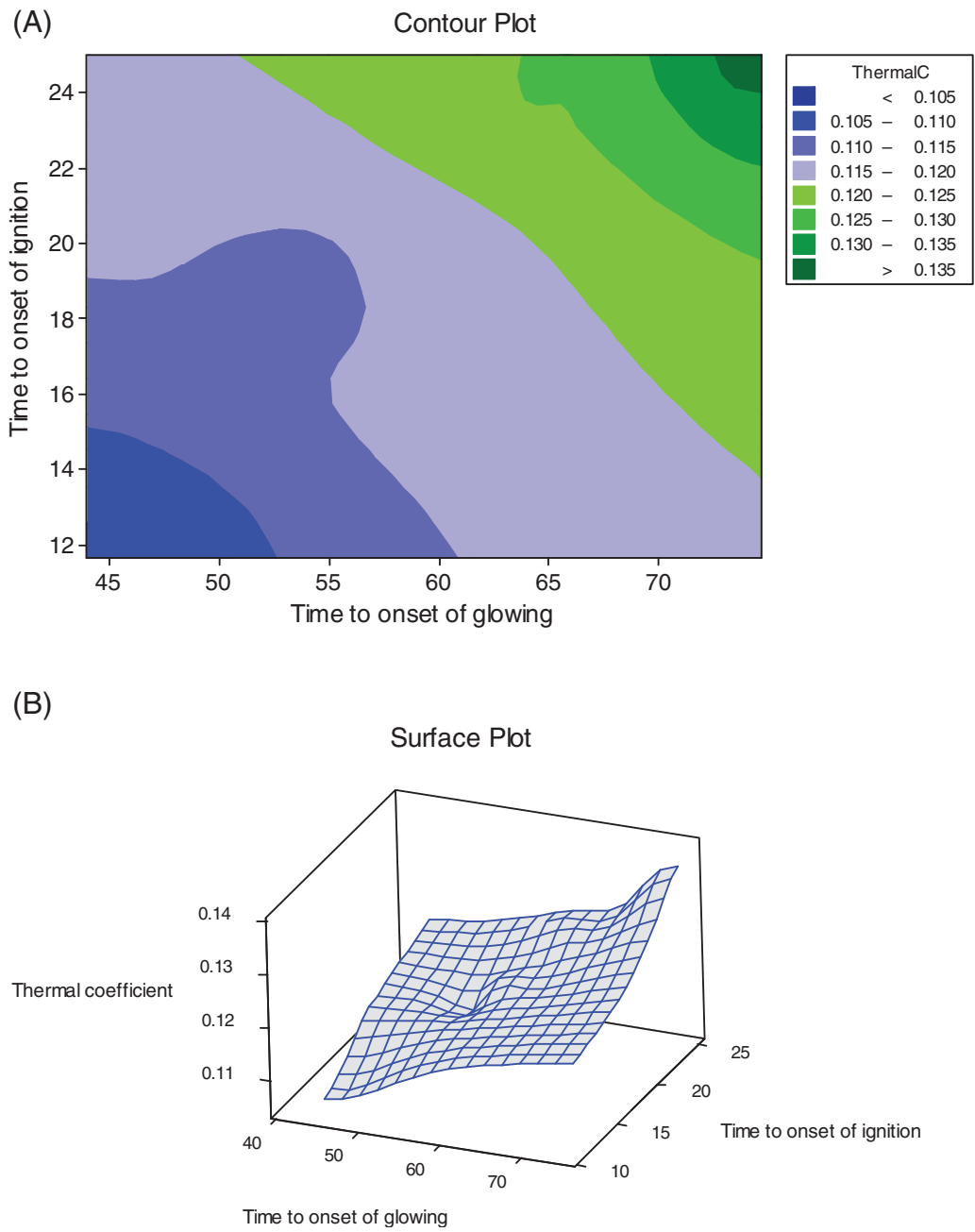


FIGURE 5 Contour A, and surface B, plots of the five treatment boards based on times to onset of ignition and glowing vs thermal coefficient values in the five treatment boards [Colour figure can be viewed at wileyonlinelibrary.com]

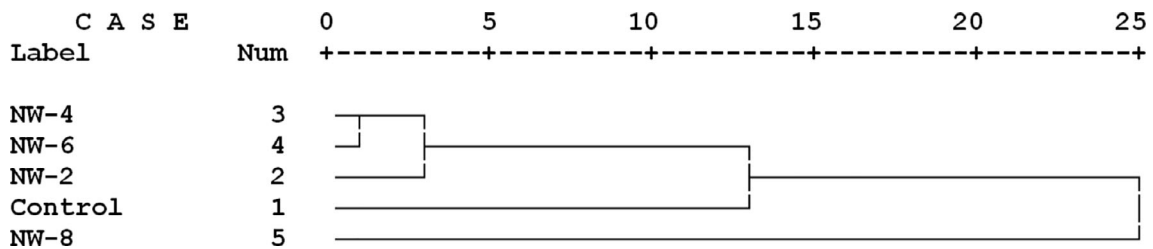


FIGURE 6 Cluster analysis of the five treatment boards based on fire properties measured in the present research project (NW, nanowollastonite; 2, 4, 6, and 8 = nanowollastonite contents)

indicating the potential of the above mentioned hypothesis. However, as density profile of panels was not measured in the present study, further studies should be carried out to come to a final conclusion in this regard.

Width and length of the burnt areas followed the same overall trend as the three properties were already discussed (Figures 2D,E); that is, increase in NW-content had a significant improving effect on length and width of the burnt area, though the improvement at lower NW-content (2%) was not statistically significant.

Cluster analysis based on all fire properties measured in the present work illustrated significantly different clustering of NW-8% treatment in comparison to other treatment boards (Figure 6) because the vertical connecting line is on the number "25" based on the scaled indicator. Other boards with NW-contents (2%, 4%, and 6%) showed similar clustering, as the vertical connecting lines among all these three treatments are less than the number "5" based on the scaled indicator. It can therefore be concluded that the overall fire properties of these three treatments can be considered rather similar, though some differences can be observed when each fire property is considered separately and individually. Control boards were clustered significantly different with all NW-treated boards. As MDF panels produced with UF resin are susceptible to vapor and water and therefore they are mainly used for interior design. Though there are not much difference between NW6% and NW8% in some of the fire properties measured here, in order to provide more protection against fire, the highest NW-content of 8% is recommended. Previously, Taghiyari et al³⁵ recommended NW-content of 6% to the industry for improving the mechanical properties. Comparison of the clustering analysis based on fire properties with that based on physical and mechanical properties in the previous study³⁷ revealed that the optimum level of NW-content would be different, if fire or mechanical properties are of major importance.

4 | CONCLUSION

Nano-wollastonite (NW) was added to medium density fiberboard (MDF) at four levels of 2%, 4%, 6%, and 8%, based on the dry weight of urea-formaldehyde resin, to investigate its effects on fire properties in comparison to untreated MDF boards. Three properties of times to onset of ignition and glowing, as well as duration of burning, were significantly and steadily improved as NW-content was increased in MDF boards. The two properties, width and length, of the burnt area showed a slight improvement only at higher NW-contents of 6% and 8%, respectively. The improvement in fire properties was partially attributed to the improvement in thermal conductivity of boards, achieved as a result of the addition of NW. The improved thermal conductivity in turn resulted in a better integration of fibers, and ultimately fire properties were significantly improved. Moreover, nano-particles provided a surface with reduced ignitability, partially adding to the improving effect. High and significant R-square values were found between thermal conductivity coefficients of the five treatments with most of the fire properties. It was concluded that, increasing time to onset of ignition, along with decreasing duration of burning when the piloted fire is removed, can be considered

as two important fire properties that were improved. In this connection, where fire properties are of major importance, an 8% content of NW should be recommended to provide the highest improvement. However, as SFTA is a newly-developed apparatus for primary studies and screening tests on new materials, further studies on large-size specimens with internationally recognized test methods and apparatuses like cone calorimeter should be carried out to come to a final conclusion in to the effectiveness and the best loading of NW in MDF panels.

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