Using Wollastonite Nanomaterials in Gypsum-Wood Composites

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In this study, the effect of wollastonite on mechanical, physical, and morphological properties of composites made of sesame stem (*Sesamum indicum* L.) and gypsum was investigated. The mixing ratio of sesame stem as a lignocellulosic material with gypsum at three levels (10:90, 20:80, and 30:70) and wollastonite at three levels (0%, 5%, and 10%, based on dry weight of cement) were considered as the variables. Fire resistance (weight loss) was tested according to ISO 11925-3 and DIN EN 634-1 and two specifications for the mechanical and physical properties. Microstructural properties of composites were evaluated by scanning electron microscopic (SEM) imaging. The results showed that boards containing wollastonite increased the physical and mechanical properties compared with gypsum board without wollastonite. The modulus of rupture, modulus of elasticity, and internal bonding of the boards decreased with increased sesame stemcontent, and its maximum value was obtained when using 10% sesame stem. The addition of 10% wollastonite is recommended to significantly improve the fire retardancy of the boards. The results from SEM images showed that the wollastonite can fill in the gaps inside the boards and create a solid structure.

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Keywords: Sesame stem; Lignocellulosic material; Gypsum; Wollastonite

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INTRODUCTION

Gypsum panels are made from a combination of lignocellulosic or wood material and synthetic gypsum. The cost of producing these boards is low with the use of these natural materials. The cost of gypsum-wood composite board is about half the price of particle board made using urea formaldehyde resin. Therefore, this product has a high competitive power.

Advantages of gypsum board production include low energy consumption in the manufacturing process and low cost in production and consumption. A great deal of lignocellulosic materials from agricultural waste, such as wheat and sesame stems, in cultivation areas are produced annually. According to the Ministry of Agriculture Jihad of Iran, the area under sesame cultivation in Iran is 51,610 hectares per year, which produces 200 tons of sesame stems annually (Ahmadi *et al.* 2017).

These waste materials can be considered as an alternative primary substance for minerals of organic materials to produce mineral wood composites. The main problem of a gypsum composite is the incompatibility of lignocellulosic fibers with gypsum. The presence of extractive materials in wood and agricultural waste reduces the hydration acceleration of gypsum. Species containing more than 7% of the extractives can be considered as incompatible materials (Hachmi and Moslemi 1982). Compounds of the extractives can delay the hydration. Today, to reduce the incompatibility of wood chips and agricultural waste (lignocellulosic materials) with gypsum, it is suggested that extractives are removed from wood using hot water, followed by adding inorganic nanomaterials such as nano-silica and wollastonite, and other chemical additives (Moslemi *et al.* 1983; Zhengtian and Moslemi 1985). Wollastonite is typically high in purity because most ores must be beneficiated by wet processing, high-intensity magnetic separation, and/or heavy media separation to remove accessory minerals. The minerals most commonly found associated with wollastonite are calcite (calcium carbonate), diopside (calcium magnesium silicate), and garnet (calcium aluminum silicate). Wollastonite is hard, white, and alkaline (pH 9.8). It is exploited for its chemistry as a source of CaO and SiO_2, and its low ignition loss, low oil absorption, low moisture absorption, and fire-retardant properties (Ciullo *et al.* 1997). Tichi *et al.* (2020) found that wollastonite shows numerous positive effects on the physical, mechanical, and morphological properties of gypsum composites. The results from this study show that physical and mechanical properties of boards increased with increasing wollastonite content.

Properties of gypsum particleboard reinforced with polypropylene (PP) fibers showed that the addition of polypropylene fibers reduced the internal bonding (IB), modulus of rupture (MOR), modulus of elasticity (MOE); and increased the thickness swelling (TS) of gypsum board (Deng and Furuno 2001). The physical and mechanical properties of gypsum particleboard reinforced with Portland cement have been examined by Espinoza-Herrera and Cloutier (2011), who showed that Portland cement incorporation increased the mechanical resistance of the boards. The fire-retardant properties of wollastonite have been reported as promising when used in solid woods and wood-composite materials (Haghighi Poshtiri *et al.* 2014). Wollastonite increased the thermal conductivity coefficient in medium-density fiberboard (MDF) (Taghiyari *et al.* 2013). Wollastonite reportedly improves biological resistance against wood-deteriorating fungi (Karimi *et al.* 2012; Taghiyari *et al.* 2014). Therefore, the present study aimed to evaluate the effects of wollastonite on physical and mechanical properties of the gypsum board and also the effect of using sesame stem, as the replacement of the wood in gypsum board.

EXPERIMENTAL

Materials and Methods

Panel production and specimen preparation

The gypsum (calcium sulfate hemihydrate powder) used in this research was procured from Omid company (Semnan, Iran). The sesame material used in the study was a type of sesame stem obtained from the northern regions of Iran (Fig. 1a). The sesame stem is straight and its height ranges from 50 to 200 cm, with most of it measuring 70 cm. The sesame grains were separated from the stem and then dried in a dryer to a moisture content of 12%. The sesame stem used for this study had no grains or ears. Nano-wollastonite was produced in cooperation with Vard Manufacturing Company of Mineral and Industrial Products (Tehran, Iran). Dimensions of the wollastonite are given in Fig. 1b.

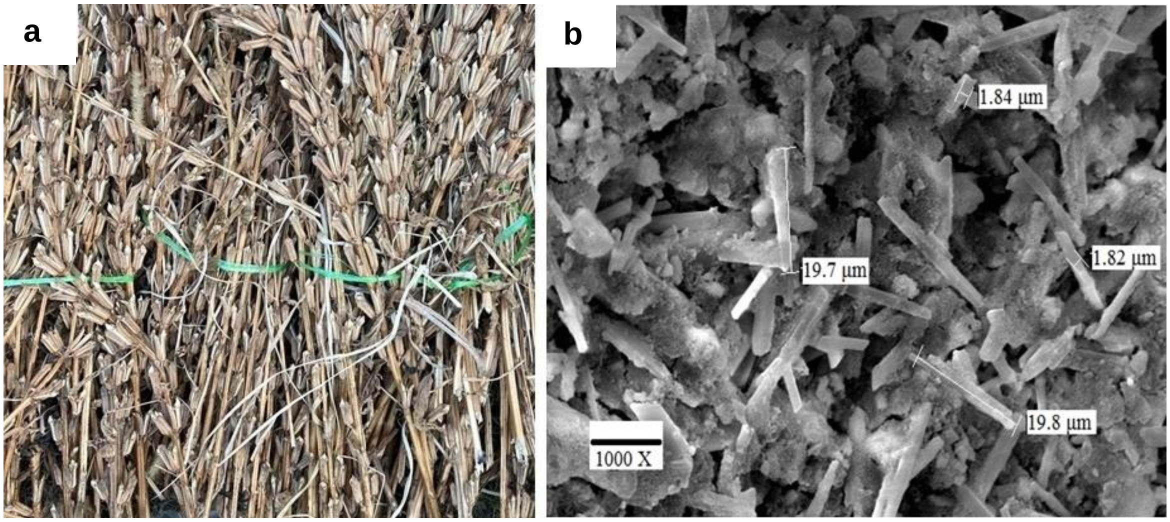

Fig. 1. Sesame stem (a) and SEM micrograph of wollastonite (b)

First, to reduce the extraction material, the sesame stem was washed with warm water (60 °C) and then milled into chips (Fig. 2a). The dimensions of the sesame stem are shown in Table 2. The cake density of all boards prepared was 1.1 g/cm^3, and their dimensions of 550 mm × 270 mm × 16 mm (board volume) were used as fixed factors. Treatment codes and the percentage of mixing are shown in Table 3. Therefore, for the production of boards, the density of raw materials in each treatment was calculated first using the density formula ($D = M/V$, where D (g/cm^3) is board density, M (g) is board mass, and V (cm^3) is board volume). Then, the weight ratios (board mass) of each board were determined. Next, using a digital scale, the ratios of water and wollastonite were determined, water containing wollastonite was sprayed on the sesame stem chips (the length, width, and thickness of sesame stem chips were 3 to 4 cm, 4 to 6 mm, and 1 mm, respectively).

After moistening the chips in a laboratory mixer for 5 min, gypsum was sprinkled on them and they were remixed in a high-speed mixer until a smooth homogeneous mixture of gypsum and sesame stem was obtained. Subsequently, the cake from the mixing machine was poured uniformly into a 55 × 27 × 6 cm^3 metal mold and pressed under pressure of 30 kg/cm^2 (Burkle-LA-160, Stuttgart, Germany) under cold conditions (30 °C) retarding for 24 h to reach the final thickness (16 mm). After this stage, the boards were removed from the mold. To minimize the drying speed of the cement and prevent the formation of small cracks due to drying, as well as the complete setting of the cement boards, the boards were held at room temperature for 28 days. Then, the authors used a circular saw to convert the boards to their final dimensions 45 cm × 17 cm × 1/6 cm (Fig. 2b). According to the variable factors, nine treatments were created. Three repetitions were prepared from each treatment, and a total of 27 laboratory boards were made.

Table 2. Sesame Stem Chip Dimensions

	Length (cm)	Width (mm)	Thickness (mm)
Average dimensions	5 to 7	15 to 20	2 to 3

Table 3. Treatments for Determination of Mechanical and Physical Tests of Gypsum Boards

Treatment	Treatment Code	Weight Ratios of Each Board			
		Weight Nano	Weight Sesame Stem Chips	Weight Gypsum	Total Weight
A	WW+10% S + 90% G		261.36 g	2352.24 g	2613.6 g
B	WW+20% S + 80% G		522.72 g	2090.88 g	2613.6 g
C	WW+30% S + 70% G		784.08 g	1829.52 g	2613.6 g
D	5% W+10% S + 90% G	117.612 g	261.36 g	2234.628 g	2613.6 g
E	5% W+20% S + 80% G	104.544 g	522.72 g	1986.336 g	2613.6 g
F	5% W+30% S + 70% G	91.476 g	784.08 g	1738.044 g	2613.6 g
G	10% W+10% S + 90% G	235.224 g	261.36 g	2117.016 g	2613.6 g
H	10% W+20% S + 80% G	209.088 g	522.72 g	1881.792 g	2613.6 g
I	10% W+30% S + 70% G	182.952 g	784.08 g	1646.57 g	2613.6 g

Note: WW: Without Wollastonite; S: Sesame stem; G: Gypsum

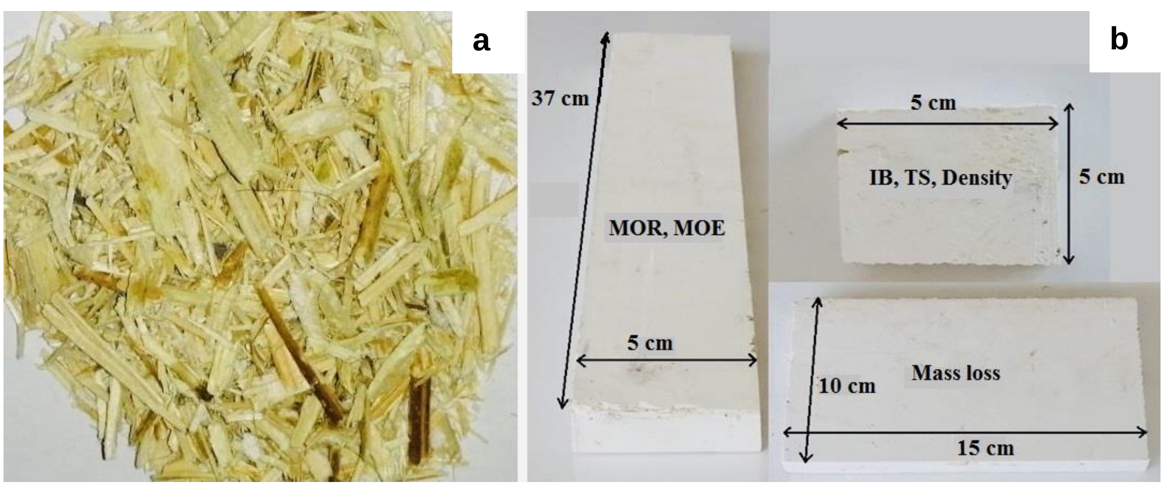

Fig. 2. Sesame stem chip (a) and boards dimension for tests (b)

Mechanical and physical tests

Boards were cut from the panels for static bending testing including MOR, MOE, and IB in accordance with the dimensions and procedures described in DIN EN 634-1 and DIN EN 634-2 (DIN 1995) using a universal testing machine (model GT-TCS-2000; Taichung Industry Park, Taichung, Taiwan) at the speed of 10 mm/min (three-point bending test). Three samples were evaluated for each treatment.

Physical test

The effect of composite formulation on the apparent density and thickness swelling (TS) after 2 h and 24 h immersion in water (15 °C) was determined in samples with the dimensions of 50 mm × 50 mm × 16 mm.

Fire retardant testing device

Measurement of fire resistance (percentage of weight loss) was tested according to the ISO 11925-3 (2020) standard with dimensions of 15 cm × 10 cm. From the preliminary weight of specimens before fire exposure (W_1, g) and second weight of specimens after fire exposure (W_2, g), the mass loss (*ML*) was calculated as Eq. 1: ML (%) = $\{(W_2 - W_1) / W_1\} \times 100$

Scanning electron microscopy

The scanning electron microscopy (SEM) analysis was performed using a Philips model XL 30 FEG (Seoul, North Korea) at the School of Electrical and Computer Engineering, Noshirvani University of Technology, Babol, Iran.

Statistical analyses

To perform statistical analysis, SPSS software and factorial (Dell Inc., Landolock, TX, USA) testing in a completely randomized design were utilized. To completely randomize design (CRD) the data, the techniques of analysis of variance and grouping of means based on Duncan's multi-domain test (DMRT) were performed.

RESULTS AND DISCUSSION

Density Boards

Wollastonite addition to gypsum board increases the density and decreases the air content in these mortars.

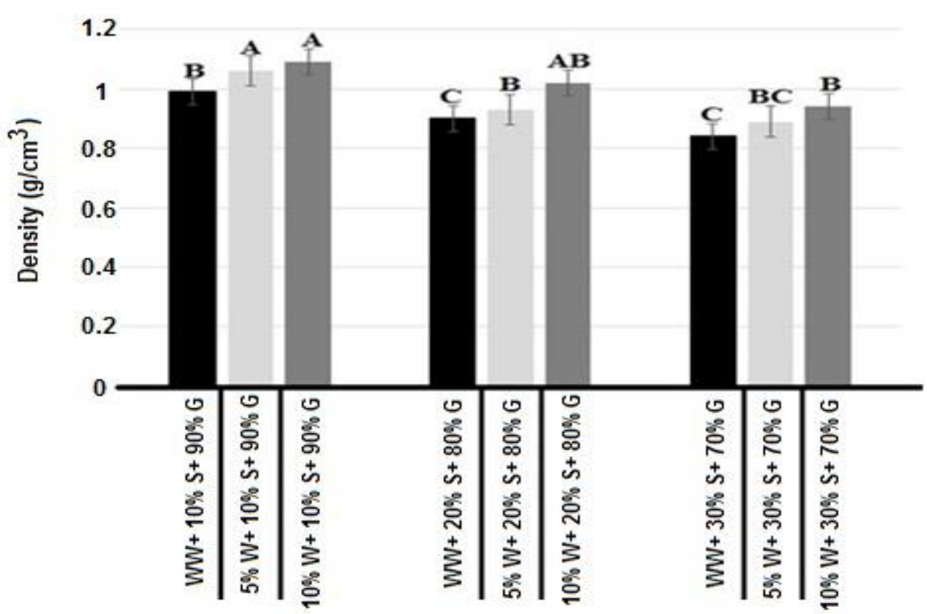

Fig. 3. Interaction of sesame stem, gypsum, and wollastonite mixing ratio on density of composites with Duncan's grouping (A, B, and C)

The highest density was when 10% wollastonite and 10% sesame stem were used (Fig. 3). Nano-wollastonite can fill the porosity inside the boards and increase the density of the board, due to its nano-dimensions. Also, the results of the physical and mechanical properties of the boards have stated that by increasing the density of the boards, the resistance properties of the gypsum boards have improved.

Mechanical Properties

The average values of the mechanical properties in terms of MOR, MOE, and IB are presented in Figs. 4 to 6. Mechanical properties generally increased in the gypsum boards that contained wollastonite compared to the gypsum boards without wollastonite. The highest MOR, MOE, and IB values were in the 10% wollastonite and 10% sesame stem, and its lowest was obtained in the without wollastonite and 30% sesame stem chip treatments (Figs. 4 to 6).

An increase in the level of agricultural waste (sesame stem) extractives—especially phenolic and sugary material contents—reduces gypsum hydration heat, slows the hydration reaction time, and increases sesame stem retention time (Rangavar *et al.* 2016). The possible reason for strength increase with increasing wollastonite could be related to the hydration temperature. Gypsum boards prepared with wollastonite, due to high activity and large specific surface of nano-wollastonite, exhibit higher maximum hydration temperatures, which resulted in faster and better curing of sesame stem with gypsum and thus increased board strength. These results are consistent with the studies conducted by Tichi and colleagues (Tichi *et al.* 2020).

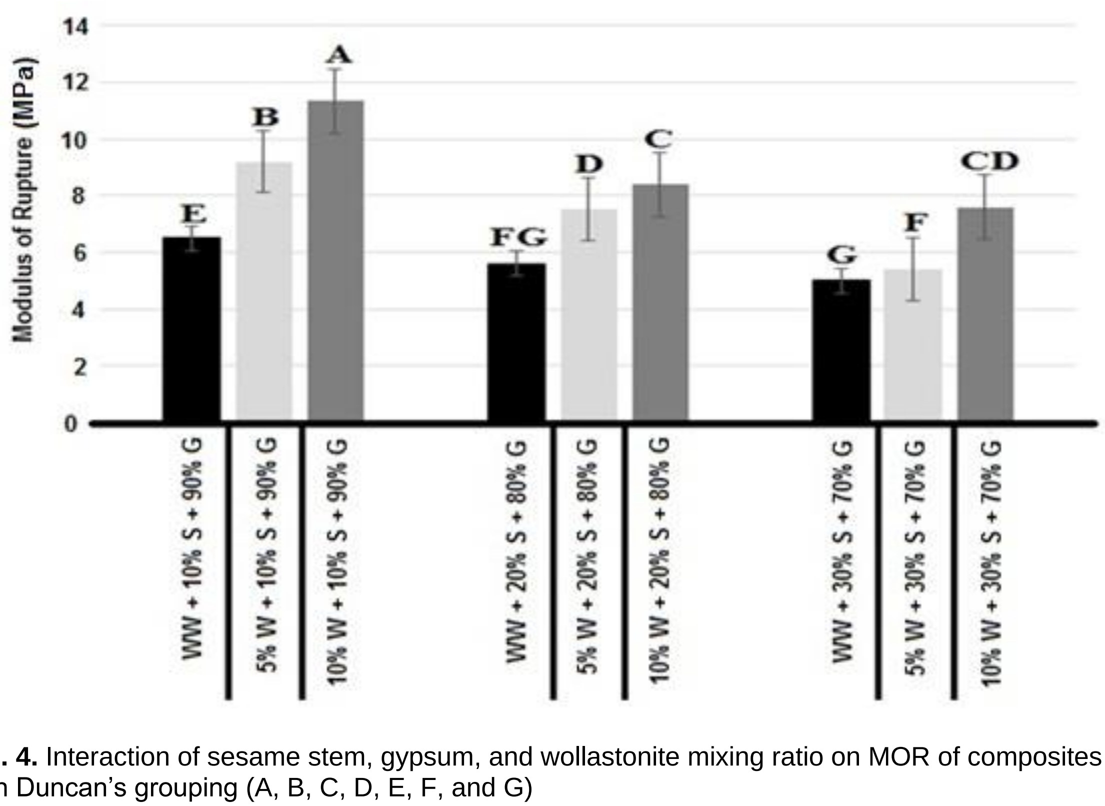

Fig. 4. Interaction of sesame stem, gypsum, and wollastonite mixing ratio on MOR of composites with Duncan's grouping (A, B, C, D, E, F, and G)

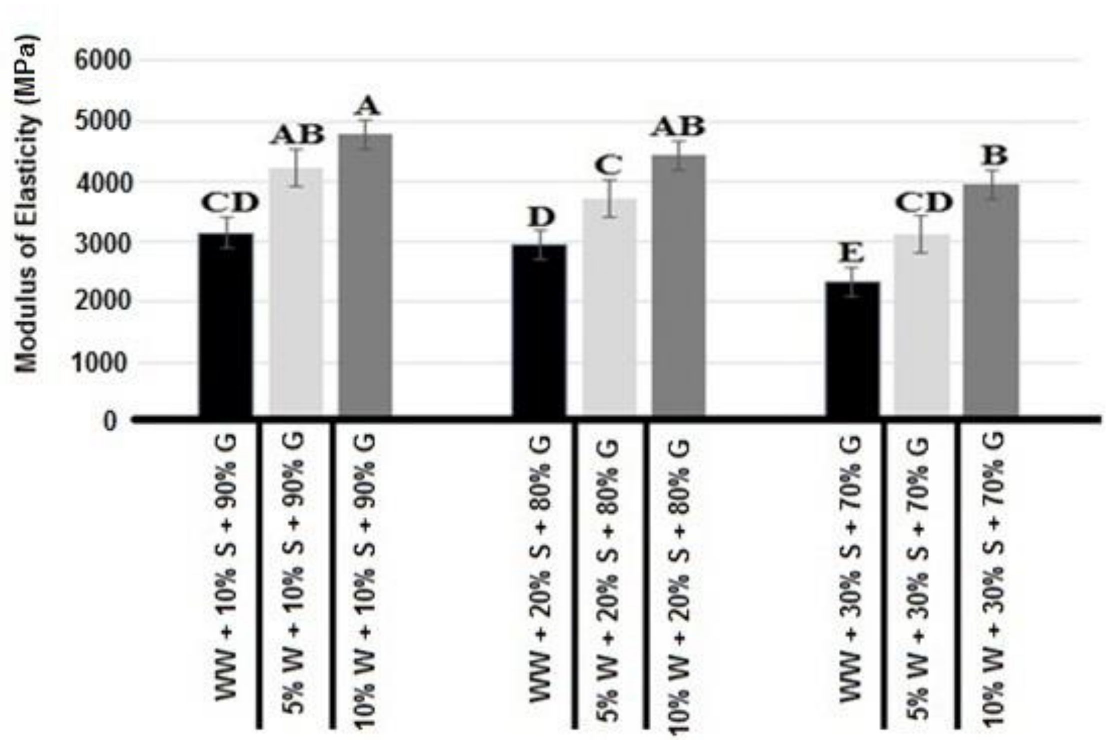

Fig. 5. Interaction of sesame stem, gypsum, and wollastonite mixing ratio on MOE of composites with Duncan's grouping (A, B, C, D, and E)

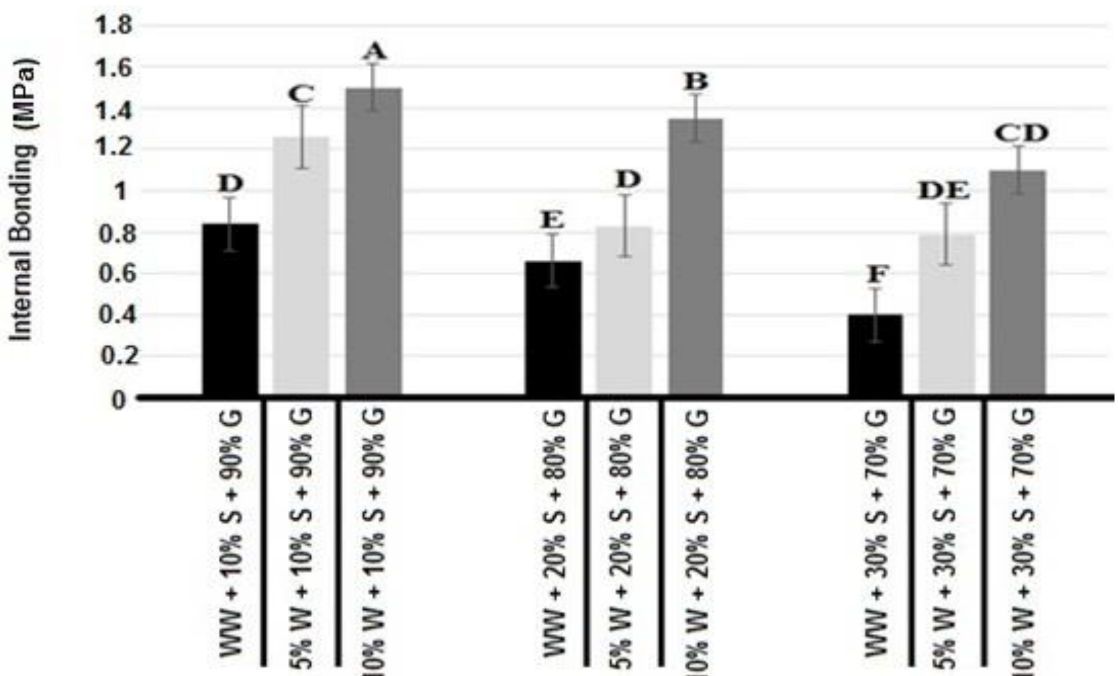

Fig. 6. Interaction of sesame stem, gypsum, and wollastonite mixing ratio on IB of composites with Duncan's grouping (A, B, C, D, E, and F)

Physical Properties

The physical properties of the boards that were measured included thickness swelling (TS) after 24 h immersion in water, density, and fire resistance. The results of these tests are presented in Figs. 7 and 8. Figure 7 indicates that any increase in wollastonite content decreased the TS of boards. The lowest TS was observed for 10% wollastonite and 10% sesame stem, and the highest was observed for the sample without wollastonite and 30% sesame stem. The specimen incorporating 10% wollastonite had the highest density.

A reduction in density occurs due to both the substitution of denser gypsum particles by lighter wollastonite particles and air-entrapment in the boards. It was also observed that with increasing amounts of wollastonite, the dimensional stability of the boards improved. This can be attributed to the hydrophobicity of wollastonite (Deng and Furuno 2001). Wollastonite, because of its high surface area and small size, fills the possible pores inside the board and blocks the water entering the board, thereby reducing the thickness swelling of the board and increasing the density of the board (Tichi *et al.* 2019). Wollastonite significantly (99%) improved the fire-retardant properties in the gypsum boards (Fig. 8). The lowest mass loss was noted with the sample containing 10% wollastonite and 10% sesame stem chip (Fig. 8). This is because wollastonite has a high heat transfer and its heat is not kept at a single point and is rapidly transferred to a point where it has less heat, and this results in increased fire resistance. Secondly, wollastonite is intrinsically fire resistant due to the presence of minerals such as silica, titanium, magnesium, calcium, and iron. As a result, boards made with higher amounts of wollastonite have higher relative fire resistance. These results are consistent with the studies conducted by Taghiyari *et al.* (2014).

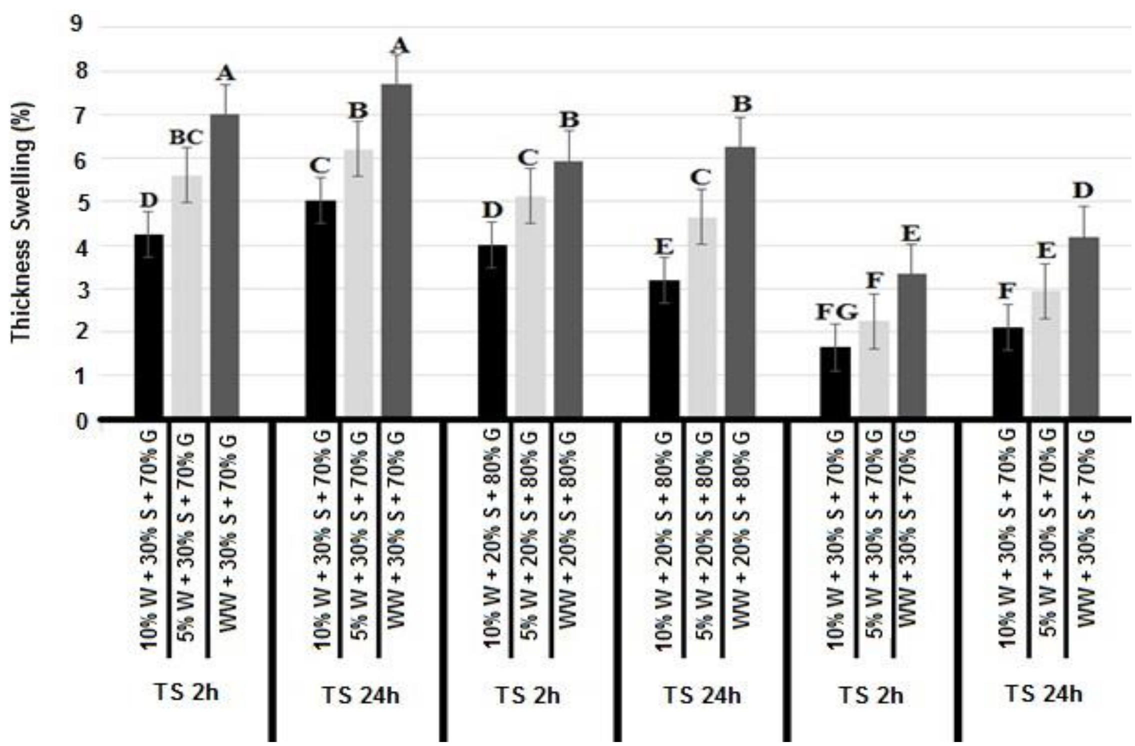

Fig. 7. Interaction of sesame stem, gypsum, and wollastonite mixing ratio on the TS of composites with Duncan's grouping (A, B, C, D, E, F, and G)

Effect of varying levels of wollastonite on the TS of composites composed of different percentages of sesame stem is shown in Fig. 7.

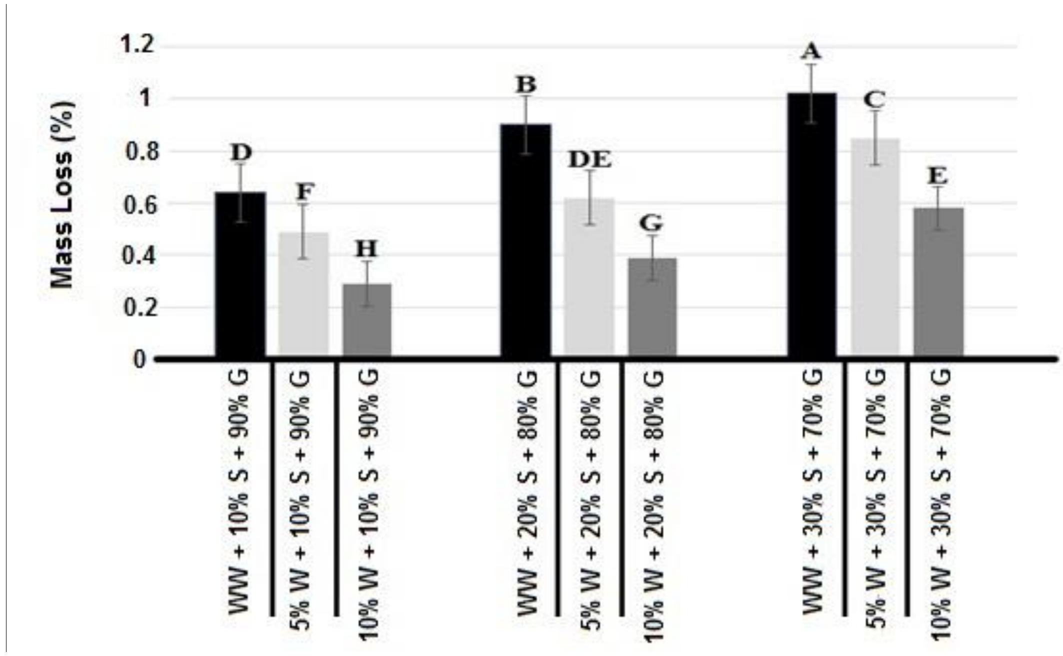

Fig. 8. Interaction of sesame stem, gypsum, and wollastonite mixing ratio on mass loss of composites with Duncan's grouping (A, B, C, D, E, F, G, and H)

Scanning Electron Microscopy

The SEM micrographs were taken from the central part of the samples. As depicted in Fig. 9a, increasing wollastonite content from 0% to 10% improved the connectivity and compatibility between sesame stem and gypsum, which can lead to higher mechanical and physical strengths of boards.

Furthermore, higher percentages of wollastonite in the boards and reaction of this material with calcium sulfate resulted in high production of hydrated calcium silicate gel (Fig. 9a). This gel prevents the penetration of chlorine ions, sulfates, and other harmful chemicals into the boards and seals, protects, and reduces permeability; while also improving durability and self-healing between gypsum and sesame stem (Tichi *et al.* 2019). According to Fig. 9a, the presence of wollastonite (10%) with gypsum (90%) is also effective in the microstructural improvement of gypsum boards. Furthermore, it was observed that improving the adhesion of chips and gypsum leads to the higher strength of the composites. In contrast, the samples contain some pores and large cavities (Fig. 9c). These points indicate the reduced strength of boards without wollastonite particles compared with that of other boards.

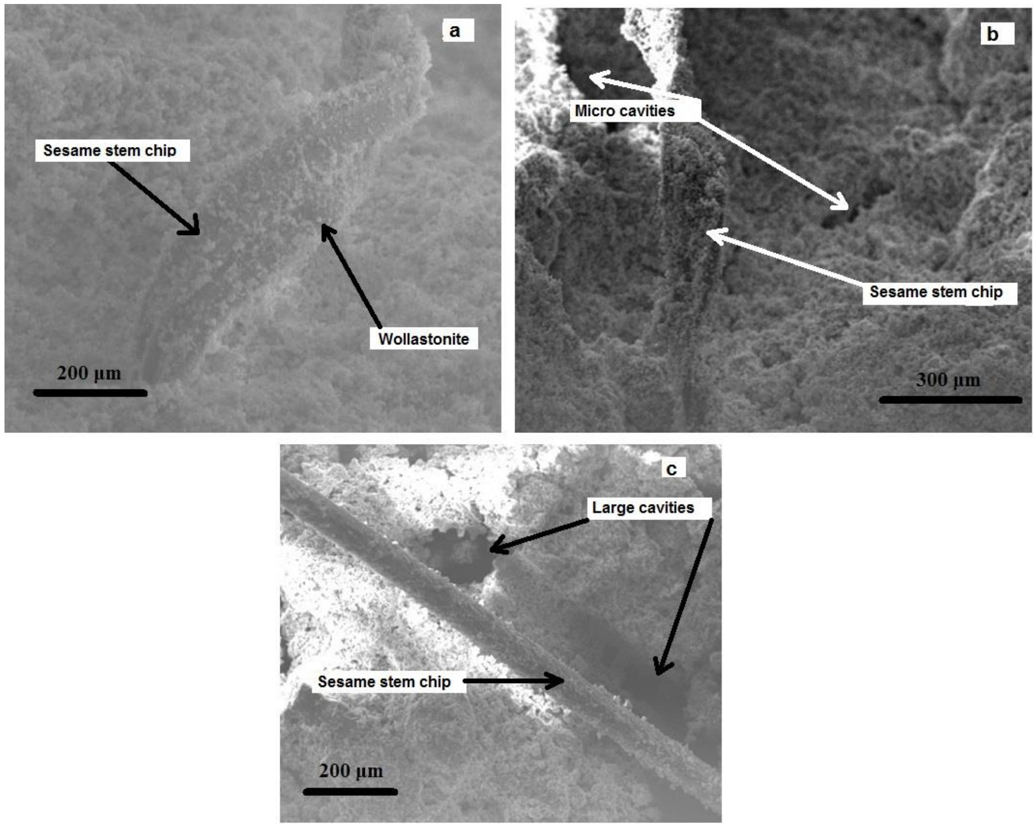

Fig. 9. Scanning electron microscopy of gypsum- sesame stem composite in the presence of 10% wollastonite (a); 5% wollastonite (b); and without- wollastonite (c)

CONCLUSIONS

1. Flexural strength, modulus of elasticity (MOE), and internal bonding (IB) increased with addition of nano-wollastonite content. This was attributed to better compaction and distribution of particles and higher hydration temperature of boards.
2. The thickness swelling (TS) of the gypsum composite boards decreased with the addition of nano-particles (5% and 10%). This could have been related to the hydrophobic character of wollastonite. Wollastonite's density is greater than that of gypsum; as a result, by increasing wollastonite percentage, board density increases.
3. Wollastonite significantly improved fire-retardant properties in gypsum board. The high thermal conductivity of wollastonite increased heat transfer, which improved the fire resistance of the composite boards. The optimum treatment was found with boards manufactured with 10% nano-wollastonite and 10% sesame stem.

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