DOI: 10.4067/s0718-221x2022000100428

# PROPERTIES OF GYPSUM PARTICLEBOARD WITH ADDED MINERAL DOLOMITE

**Omer Umit Yalçin**<sup>1,\*</sup>

https://orcid.org/0000-0003-2241-3677

Ali İhsan Kaya<sup>2</sup>

https://orcid.org/0000-0002-1860-9610

#### ABSTRACT

Red pine (*Pinus brutia*) wood particles and dolomite mineral were used in varying proportions to form mineral-added gypsum particleboards. Mechanical, physical, and chemical properties of the boards were tested. The increasing mineral content was found to improve the water absorption properties but the increased amount of gypsum in the mixture negatively affected the thickness swelling and water absorption properties. The usage of dolomite mineral in the board composition increased the internal bond properties and higher than the standard of 0,28 MPa. However, all types of boards had modulus of elastic, modulus of rupture and thermal conductivity results values below the standards. Moreover, the thermal conductivity values decreased in all board types because of the reduction of the mineral dolomite. Thermal gravimetric analysis, Fourier transform infrared spectrometry tests were applied to examine the thermal and flame retardancy properties of inorganic materials, wood-gypsum composites, which are used at different rates for synergistic effect. The gypsum and dolomite amount affected the thermal variation, whereas the increment in the weight of the wood particles also increased the thermal degradation. It was determined that stresses at 850-980 cm<sup>-1</sup> reveal Ca-O and Mg-O, reveal at 881cm<sup>-1</sup> C-OH, weak vibration at 1619 cm<sup>-1</sup> and a strong bond structure in the 1445-950-882 cm<sup>-1</sup> bands. These bands express the characteristic presence of the CaO and MgO belonging to dolomite. The study demonstrated the feasibility of producing mineral-based gypsum board products using wood chips.

Keywords: Dolomite, gypsum, Red pine wood, strength properties, thermal properties.

# **INTRODUCTION**

Gypsum is one of the oldest construction materials and its usage dates back several thousand years. Gypsum boards are extensively used worldwide in the building industry as an interior finishing material on walls and ceilings (Kang *et al.* 2018). Gypsum is an environmentally friendly and energy-saving building material with many advantages including constructability, sound and thermal insulation properties in addition to its decorative features, availability, low price, and ease of production (Martias *et al.* 2014, Han *et al.* 2017). Moreover, there is great interest in its fire safety properties. Therefore, gypsum boards are the most popular lightweight materials used in building construction (Wakili *et al.* 2007).

A number of studies have indicated that worldwide energy consumption has risen by more than 30 % in residential and commercial buildings. This rate is up to 40 % in the European Union and the United States. Therefore, reducing the energy consumption in structures by improving their thermal performance can decrease the above-mentioned rate (Sharifi *et al.* 2017).

<sup>1</sup>Isparta University of Applied Sciences. Forestry Faculty. Department of Forest Products Engineering. Isparta, Turkey. <sup>2</sup>Mehmet Akif Ersoy University. Vocational School of Technical Sciences. Department of Material Science. Burdur, Turkey. \*Corresponding author: omeryalcin@isparta.edu.tr

Received: 12.02.2021 Accepted: 16.02.2022

Lignocellulosic materials are used in composite manufacturing due to their low cost, nonabrasive and high natural properties, biodegradability, and abundance throughout the world (Ashori *et al.* 2011). Meanwhile, wood has long been used as a construction material for its strength and natural aesthetics. However, forests are being cut down to meet human demands, making wood composites a possible answer as an alternative solution. Wood composites include a range of different derivative wood products which involve binding wood fibers or chips together with plastics or minerals (Papadopoulos 2019).

Minerals can be combined with natural fibers for a natural, healthy, low-density, low-cost product with low thermal conductivity. Despite these advantages, studies carried out on the use of natural fibers with gypsum plaster as reinforcement are limited. Some studies have shown that the production of panels is possible using bamboo, wood fiber, bagasse, hemp hurds, pine sawdust, paper mill sludge, and wastepaper as raw materials (Amiandamhen *et. al* 2016, Sudin and Swamy 2006). In addition, coconut coir fiber, along with waste rice husk ash and slag have also been incorporated in gypsum (Sophia *et al.* 2016).

Since wood fibers are very sensitive to flame, it has become more and more important to improve the flame retardancy of composite board materials so that wood board products can become safer. Flame retardant can be obtained by interrupting the combustion process, which can cause the process to terminate before actual ignition can occur (Sain *et al.* 2004).

Dolomite mineral is abundant in nature and contains a sufficient amount of magnesium. This mineral is found in large proportions in dolomite rock. Ideal dolomite has a crystal lattice consisting of alternating layers of Ca and Mg, separated by layers of CO and is typically represented by the stoichiometric chemical composition  $CaMg(CO_3)_2$ , with calcium and magnesium present in equal proportions (Warren 2000). Large quantities of dolomite are used for various purposes worldwide; however, in Turkey, dolomite is mostly used for ornamental stonework, in concrete aggregate, for magnesium oxide in magnesium production, as a fire retardant, and as a component in iron and steel, ceramics, paints, fertilizer, glass, and cement (Ozdemir *et al.* 2017).

However, limited research has been conducted on the effects of mineral dolomite mixed with lignocellulosic material using gypsum as a binder. This study examined the production of composite boards using red pine wood and mineral dolomite with gypsum as the adhesive. Accordingly, tests were carried out for water absorption, thickness swelling, internal bond, bending strength, modulus of elasticity, thermal conductivity and flame combustion performance. The Fourier-transform infrared spectroscopy and thermogravimetric analysis properties of the boards were examined.

Some reseaches indicated that the addition of dolomite as filler to the structure of high-density fiber boards effects some physical and mechanical properties that water absorption (WA), thickness swelling (TS), modulus of elasticity (MOE) and modulus of rupture (MOE) lowered in some level while the fire resistance properties increased (Ozdemir 2016). Therefore, the addition of mineral fillers to the board gives the board its fire resistance properties by causing the combustible material (lignocellulosic particles in the board) to separate and reduce the thermal conductivity. Mineral binders cause fire retardation by coating and penetrating the lignocellulosic particles (Kozlowski *et al.* 1999).

The aim of this study to investigate the mechanical, physical, and chemical properties of composite boards and feasibility of producing mineral-based gypsum board products using wood chips.

# MATERIALS AND METHODS

The red pine (*Pinus brutia* Ten.) wood samples were provided by a particleboard company in Isparta, Turkey. After collection, the wood chips were cut into small pieces using a laboratory-type hammer mill, passed through a 5-8 mm screen, and then dried under atmospheric conditions until at least 10 % - 12 % moisture content was obtained. The dolomite was collected from Isparta-Aksu mining sites in Turkey. The great majority of the mineral dolomite used in the experimental samples has 18,72 % MgO and 31,50 % CaO compounds. The commercial gypsum with perlite was purchased from a company in Isparta, Turkey.

### Preparation of experimental samples

The sample preparation was conducted at room temperature. First, gypsum, mineral dolomite, and red pine wood chips were mixed to obtain a homogeneous mixture. The mixture was then added to water to reach a satisfactory level. Metal mold plates in dimensions of 40 cm  $\times$  40 cm  $\times$  10 mm were used to prepare the board paste. This was pre-pressed at 80 kg/cm<sup>2</sup> for 5 min and then at 1,5 N/mm<sup>2</sup> for 24 h using a laboratory-type press at 20 °C - 24 °C. The boards were kept between metal plates after the completion of the pressing process and acclimatized for three weeks.

## Characterization

The experimental boards were conditioned at 23 °C and 65 % relative humidity and samples were sawn to determine the internal bond (IB), modulus of elasticity (MOE) and rupture (MOR), and thickness swelling (TS, %)/ water absorption (WA, %) after 2 and 24 h immersion in water, in accordance with TS EN 310 (1999), TS EN 319 (1999), and TS EN 317 (1999) standards, respectively.

The thermal conductivity of the test samples was examined in accordance with the ASTM C 1113-90 (1990) standard by applying the hot wire method using the QTM 500 quick thermal conductivity meter. A standard flame combustion test system was applied according to TS EN-ISO 11925-2 (2002). The Shimadzu IR Prestige-21 Fourier-transform infrared (FTIR) spectrophotometer was used to analyze the compounds related to the surface. Thermogravimetric analysis (TGA) was carried out using a Perkin Elmer SII instrument in order to determine the changes in thermal degradation. The percentage of dolomite, wood, and gypsum used in the various board sample types according to their given codes is shown in Table 1. The purpose of increasing and decreasing the additive ratios of the reinforcement materials added to the composite board consisting of gypsum-wood mixture is to measure the effectiveness of the additional additive at each rate. In the first group of composites prepared in our article, 4 different composite materials were produced by keeping the 70 % gypsum amount constant and 4 different composite materials by keeping the 28 % gypsum amount constant. In this way, the synergistic activity of each of them was investigated in 8 different plates produced by keeping the amounts of red pine and dolomite variable.

Contents	Board Codes								
	Α	В	С	D	Е	F	G	Н	
Dolomite (%)	24	18	12	6	42	36	30	24	
Red pine wood (%)	6	12	18	24	30	36	42	48	
Gypsum (%)	70	70	70	70	28	28	28	28	

Table 1: Code numbers and mixture proportions (%) of board samples.

Measurements were conducted in the Forest Product Engineering Research and Application Laboratory at Isparta University of Applied Sciences. An analysis of variance (ANOVA) general linear model procedure was performed using the data to interpret the interaction of the manufactured boards. The Duncan test was used to make comparisons among board types for each property tested when the ANOVA was found to be significant.

# **RESULTS AND DISCUSSION**

Lignocellulosic material fibers are easily flammable. Fortunately, there are some fire retardant fillers that can be used. These are categorized as inorganic and organic flame-retardants depending on their chemical nature. Mineral dolomite has an ideal chemical composition of  $CaMg(CO_3)_2$  and belongs to a symmetry structure group in the trigonal subsystem of the hexagonal crystal system (Morrow 1982). Figure 1 shows the dolomite structure consisting of calcium and magnesium ions (Warren 2000). Fire retardant chemicals, especially inorganic retardants, improve the combustion properties of wood material (Kozlowski *et al.* 1995). The effective incorporation of MgO to wood materials has been shown to decrease combustibility and improve the flame retardancy and thermal stability of the cellulose fibers (Li *et al.* 2017). Dolomite mineral is rich in MgO and

CaO compounds and thus was able to provide the fire retardant characteristic to the test boards.



Figure 1: Calcium and magnesium ions in the structure of dolomite (Warren 2000).

The chemical composition of the mineral dolomite used in the board production is shown in Table 2, demonstrating that MgO and CaO compounds constitute the great majority of its composition.

Compound	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	$P_2O_5$	MnO	Water
											and
											Gases
Amount	1,89	0,10	0,02	18,72	31,50	0,01	0,01	<0,01	0,02	0,01	47,33
(%)											

 Table 2: Chemical composition of dolomite.

# Dimensional stability and mechanical properties

The water absorption (WA, %) and thickness swelling(TS, %) values of boards submerged in water for 2,0 h and 24 h are shown in Table 3. The results were observed for WA and TS properties of boards manufactured from red pine wood and gypsum with mineral dolomite. The composition of the dolomite and red pine wood content of these boards varied, while the gypsum, as binder, varied in proportions of 70 % and 28 %.

Board Code	WA	WA	TS	TS	IB	MOR	MOE
	(2 h)	(24 h)	(2 h)	(24 h)	(MPa)	(MPa)	(MPa)
А	3,2	5,3(a)	0,2	0,7(a)	0,43(c)	1,82(c)	14,27(a)
В	9,9	12,7(a)	0,4	0,8(b)	0,39(c)	1,92(a)	20,46(bc)
С	16,1	19,9(a)	0,9	6,6(c)	0,21(b)	2,63(b)	32,55(ab)
D	23,1	35,2(a)	11	12,3(d)	0,11(a)	4,53(a)	35,74(c)
F value		1,629 <sup>ns</sup>		1763,724***	86,087***	35,060***	4,320*
Е	52,8	57,2 (b)	9,3	10,1(a)	0,3(a)	3,79(a)	47,14(a)
F	49,3	59,9(a)	19,9	20,5(a)	0,28(a)	2,97(a)	41,87(a)
G	77,3	80,3(b)	22,3	22,5(a)	0,28(a)	1,73(a)	36,49(a)
Н	89,4	94,5(b)	24,3	24,4(a)	0,24(a)	1,27(a)	31,27(a)
F value		8,940**		0,676 <sup>ns</sup>	0,682 <sup>ns</sup>	0,664 <sup>ns</sup>	1,909 <sup>ns</sup>

Table 3: The water absorption (WA %) and thickness swelling (TS %) values of the boards.

(\*\*\*) denotes 99,9 % confidence level, (\*\*) denotes 99 % confidence level, (\*) denotes 95 % confidence level, (ns) denotes statistically insignificant, and (a, b, c, d, e) denote homogeneous groups.

The lowest WA values (5,3 %) were observed on board type A. However, the minimum WA value was found on board type E among the boards with 28 % gypsum. It is important to note that increasing the mineral content was found to improve the WA properties in both board groups.

The thickness swelling (TS) values of the boards were found to be higher than the standard value of 12,5 % under some conditions in the F,G, and H type boards. This may have been a result of not using a hydrophobic substance in the production of the boards (Nemli 2003). However, TS properties were seen to be below the standard value in all boards bonded with 70 % gypsum. Therefore, after 24 h, with gypsum used at 70 % as adhesive, the lowest TS value (0,7 %) was found with board A and the highest (12,3 %) with board D. Likewise, in the 28 % gypsum-bonded boards, the highest TS value (24,4 %) was found in board H, produced using 24 % dolomite/48 % red pine wood/28 % gypsum, and the lowest (10,1 %) in board E,with a combination of 42 % dolomite/30 % red pine wood/28 % gypsum. The presence of mineral dolomite seemed to have a negative effect on TS properties. However, the increased amount of gypsum in the mixture negatively affected the TS and WA properties. A clearly significant difference was found between the 28 % and 70 % gypsum presence.

The statistical data indicated that the F values of the boards coded A-D and E-H were 1,629 (p= 0,258) and 8,940 (p= 0,004) for WA properties, respectively. Likewise, the F values of these boards were found as 1763,724 (p=0,000) and 0,676 (p=0,586) for TS properties. According to these results, significant differences were found among the E-F boards on WA properties and among the A-D boards on TS values. However, the WA values of the A-D boards and the TS values of the E-H boards were found to be statistically insignificant.

According to the ANOVA results for internal bond (IB) and modulus of rupture (MOR) properties of the boards, significant differences were found among boards A-D at a 99,9 % confidence level, while the range of modulus of elasticity (MOE) values of the boards was found at a 95 % confidence level. Moreover, the IB,MOE, and MOR results were found to be statistically insignificant in the E-F board samples.

The IB, MOR, and MOE values of the boards produced from dolomite, red pine wood, and gypsum mixtures are shown in Table 3. The highest IB value (0,43 MPa) was seen in the A coded board (24 % dolomite/6 % red pine wood/70 % gypsum) and the lowest IB (0,11 MPa) in board D (6 % dolomite/24 % red pine wood/70 % gypsum).

Table 3 shows that approximately all boards exhibited IB values higher than the standard of 0,28 MPa. This can be explained by the fact that the mineral substances in the board structure had a hydrophobic effect on the binder that increased the adhesive absorption of the particles. For this reason, the usage of dolomite mineral

in the board composition increased the IB properties.

The MOR values ranged from 1,82 MPa to 4,53 MPa. However, the EN 312 (2012) requirement for general-purpose particleboards is a MOR value of 12,5 MPa. All experimental types of boards had MOR values below the standard. Moreover, the MOE values of all boards were found to be below the standard value of 1600 MPa. The lowest MOE value (14,27 MPa) was observed in board type A and the highest value (47,14 MPa) was in board D.

Table 3 shows an increasing tendency from A to D and from E to H for MOR and MOE properties. The reduction in MOR and MOE values may have been due to the presence of large dolomite particles in the board, which consequently lessened the compatibility of the wood particles. On the other hand, the MOR of gypsum has been reported to be in the range of 1,0 Mpa to 4,0 Mpa, depending on the specimen and other factors (Cramer *et al.* 2003). Compared to literature studies mentioned above, this indicates that the effect of the gypsum on the boards was negligible.

# Thermal conductivity and density values

The thermal conductivity and the density values obtained from the various types of boards prepared in the presence of gypsum as a binder are given in Table 4. The highest thermal conductivity value (0,8068 W/ mK) was found for board A. The lowest thermal conductivity value (0,2398 W/mK) was obtained for board H, which was a mixture of 30 % dolomite/42 % red pine wood/28 % gypsum. These thermal conductivity results show that all board samples had values below the 0,065 w/mK standard determined for building and thermal insulation material (Yalçın 2018). It seemed that when the amount of gypsum in the mixture was increased, the thermal insulation property of the material decreased. In addition, the thermal conductivity values decreased in all board types because of the reduction of the mineral dolomite.

Board Code	Thermal Conductivity (W/mK)	Density (kg/m³)	Board Code	Thermal Conductivity (W/mK)	Density (kg/m³)
А	0,8068 (0,035)	1,637 (0,024)	E	0,2966 (0,009)	0,900 (0,026)
В	0,5702 (0,024)	1,194 (0,008)	F	0,2788 (0,007)	0,887 (0,012)
С	0,3910 (0,030)	1,073 (0,011)	G	0,2792 (0,004)	0,881 (0,014)
D	0,2985 (0,018)	0,932 (0,010)	Н	0,2398 (0,006)	0,875 (0,022)

**Table 4:** Thermal conductivity and density values of boards.

Standard deviation in brackets

Table 4 shows the density values of the board samples compared to the thermal conductivity data. The density increased depending on the mineral material increment, and the density differences of the samples affected the thermal performance of the board. The highest density (1,637 kg/m<sup>3</sup>) was found in board A and the highest thermal conductivity was also observed in the same sample. However, the lowest density (0,875 kg/m<sup>3</sup>) was found in board H. This was due to fact that large mineral particles can cause a reduction in thermal conductivity in wood-gypsum boards. Therefore, the increment of particle content and amount of space between particles led to lower thermal conductivity (Bekhta and Dobrowolska 2006).

# **Combustion behavior**

Figure 2 shows the results of combustion experiments carried out with a single flame source. The temperature values were measured every 30 s for up to 300 s using a heat measuring device from the back side of the board surface, according to the DIN 4102-1 (1998) standard. As seen in Figure 2, the highest surface temperature passed to the opposite side of the flame source (105,6 °C) was found in board Cafter 300 s. However, in all samples bonded with 70% gypsum, the lowest value (89,3 °C) was observed in board D after 300 s. The highest surface temperature (104,1 °C) was found in board F after 300 s. At the same time, in all samples bonded with 28 % gypsum, the lowest value (99 °C) was observed in board H after 300 s (Figure 2). Generally, all types of boards exhibited similar test results.



Figure 2: Surface temperature values of combustion experiment.

#### Thermal degradation through thermogravimetric analysis (TGA)

The thermogravimetric (TG) curves of boards A, B, C, and D are shown in Figure 3. Thermogravimetric analysis (TGA) is used to determine the thermal stability and thermal conductivity of a material (Chen *et al.* 2013). The mass losses occurring in material heated to high temperatures are measured, and the thermal stability and burning behavior of the material are characterized by calculating the mass loss according to the temperature ranges. The TGA is the most commonly used method for determining the temperature-time deterioration of the components of composite materials (Kaya and Sahin 2016, Kaya 2015). The study examined the TGA-DTG thermograms of composite materials with a dolomite mineral additive and gypsum binder produced in the form of board samples. The composite boards produced with dolomite additive were examined in two groups according to the amount of gypsum as the binding material and comparisons were made. The derivative thermogravimetry (DTG) curves of boards A,B,C, and D are shown in Figure 4. Degradation reactions took place in four phases in the first group of gypsum boards (A, B, C, D coded boards with 70 % gypsum). The initial temperature ( $T_{onset}$ ), maximum temperatures ( $T_{max1-2}$ ), final temperature ( $T_{endset}$ ), and mass loss are shown in Table 5 for each board type.



Figure 3: Thermogravimetric (TG) curves of board types A, B, C, D.



Figure 4: Derivative thermogravimetry (DTG) curves of board types A, B, C, D.

**Table 5:** Initial temperature  $(T_{onset})$ , maximum temperatures  $(T_{max1-2})$ , final temperature  $(T_{endset})$ , and massloss values of board types A, B, C, D.

Board	T <sup>c</sup> <sub>onset</sub>	Mass	T <sup>d</sup> <sub>max1</sub>	Mass	T <sup>e</sup> <sub>max2</sub>	Mass	T <sup>f</sup> <sub>endset</sub>	Mass Loss	Residue
Code		Loss (%)		Loss (%)		Loss (%)		(%)	
A	85	0,27	120	4,16	364	8,38	884	27,15	71
В	84	2,50	119	5,67	362	24,24	881	48,46	49
C	82	3,67	118	7,85	351	29,80	873	65,10	28
D	80	4,21	117	10,17	350	39,62	870	64,34	18

 $T^c$  initial temperature at the first phase of degradation (°C);  $T^d$  temperature of the maximum peak at the first phase of degradation (°C);  $T^c$  temperature of the maximum peak at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C); T

The chemically bound water was removed (via dehydration) with the increase of temperature ( $T_{onsel}$ ) in the initial phase of the board structure. Thus, up to 120 °C, the mass loss was around 9 % in the board. In this first phase, physical and chemical changes occur, especially in the structure of gypsum (CaSO<sub>4</sub>2H<sub>2</sub>O) (Guerrero and Hernandez 2000, Chang *et al.* 1999, Espinoza-Herrera and Cloutier 2009). The calcium sulphate structure loses water and crystallizes (Shahraki *et al.* 2009, Kurugol and Tekin 2011).

Phase I 
$$CaSO_4 2H_2O \rightarrow CaSO_4 \frac{1}{2}H_2O + \frac{3}{2}H_2O$$

Phase II: 
$$CaSO_4 \frac{1}{2} H_2O \rightarrow CaSO_4 + \frac{1}{2} H_2O$$

In the second phase (from 180 °C to 350 °C), the dolomite kept mass loss at the minimum level in board A due to its resistance to thermal degradation. Although the mineral additives delayed the degradation of hemicellulose and cellulose at these temperatures, degradation had begun.

The thermogravimetric (TG) curves of boards E, F, G and H are shown in Figure 5. In the third phase, the addition of the dolomite to the mixture provided an effective protection against the thermal deterioration of the wood particles. The inorganic material additive increased the hemicellulose and cellulose degradation temperature from 240 °C to 350 °C (Biermann 1993, Todor 1976). At these temperature values, a decrement was observed in mass loss, which was positively affected by the mineral addition in this phase.

The fourth phase started from 650 °C because of the decomposition in the structure of the dolomite and gypsum. The calcite and magnesium oxide in the dolomite structure decomposed and released carbon dioxide, and mass loss increased, with the peaks formed after 750 °C and 880 °C (Guerrero and Hernandez 2000, Chang *et al.* 1999, Shahraki *et al.* 2009, Todor 1976). The maximum mass loss was 64,34 % and the minimum was 27,15 %. The residue was determined as 71 % at the least loss and 18 % at the most loss. As a result, thermal stability had increased with the addition of dolomite to the composite structure consisting of wood particles with 70 % gypsum.



Figure 5: Thermogravimetric (TG) curves of board types E, F, G, H.



Figure 6: Thermogravimetry (DTG) curves of board types E, F, G, H.

Degradation reactions took place in two phases in the second group consisting of 28 % gypsum (boards E, F, G, H). Initial temperature  $(T_{onset})$ , maximum temperatures  $(T_{max1-2})$ , final temperature  $(T_{endset})$ , and mass loss

#### Maderas. Ciencia y tecnología 2022 (24): 28, 1-16

are shown in Table 6. The DTG curves of boards E, F, G, and H are shown in Figure 6. The moisture content of the composite material decreased in the first phase that started at 97 °C, and the mass loss increased due to the presence of the wood chips. The first maximum temperature peak was observed at 124 °C. The cellulose, hemicellulose, and lignin were degraded in the second phase when the temperature reached 250 °C and the mass loss was found to be 10 % on average.

**Table 6:** Initial temperature  $(T_{onset})$ , maximum temperatures  $(T_{max1-2})$ , final temperature  $(T_{endset})$ , and mass<br/>loss values of board types E, F, G, H.

Board	T <sup>c</sup> <sub>onset</sub>	Mass	T <sup>d</sup> <sub>max1</sub>	Mass	T <sup>e</sup> <sub>max2</sub>	Mass	T <sup>f</sup> <sub>endset</sub>	Mass	Residue
Code		Loss (%)		Loss (%)		Loss (%)		Loss (%)	
E	97	1,00	124	5	734	32,95	826	47	37
F	98	6,25	125	10,6	752	70,11	825	68	34
G	98	1,55	123	6,25	675	21,58	825	45	12
Н	99	4,46	123	12,61	674	58,8	826	60	5

 $T^c$  initial temperature at the first phase of degradation (°C);  $T^d$  temperature of the maximum peak at the first phase of degradation (°C);  $T^c$  temperature of the maximum peak at the second phase of degradation (°C);  $T^r$  final temperature at the second phase of degradation (°C).

However, with the degradation of dolomite at 700 °C, mass loss reached the highest level. The degradation of dolomite occurs in two phases: calcium oxide decomposition and carbon dioxide release (Shahraki *et al.* 2009).

Phase I:  $MgCa(CO_3)2 \rightarrow MgO + CaCO_3 + CO_2$ 

Phase II:  $CaCO_3 \rightarrow CaO+CO_2$ 

The thermal degradation of lignin, hemicellulose, and cellulose was delayed by the reduction of oxygen in the environment. The degradation of dolomite occurred in peaks at 750 °C and 850 °C. The mass loss was around 50 % - 60 % at these temperatures. This decomposition of dolomite is characterized in the DTG curves (Kristóf-Makó and Juhász 1999, Maitra *et al.* 2005, L'vov and Ugolkov 2003).

The amount of gypsum and dolomite by weight affected the thermal variation in the gypsum boards with dolomite additive, whereas the increment in the weight of the wood particles also increased the thermal degradation. The oxygen increment in the environment accelerated combustion and caused the wood particles to decay (Espinoza-Herrera and Cloutier 2009). As a result of the tests, for a successful thermal assessment, it is recommended that the wood chip content be kept below 20 % - 30 % by weight for thermal stability.

#### Functional groups assessment

A FTIR analysis was made to characterize the structure of the composite material and the use of functional groups in the main structure. The FTIR spectrum of boards A, B and C is shown in Figure 7. The transition to a crystal structure was accelerated. According to the FTIR spectrum the 3526-3400 cm<sup>-1</sup> absorption bands show strong bonds and -OH vibration stresses (Kondo and Sawatari 1996, Schwanninger *et al.* 2004). The 2525 cm<sup>-1</sup> and 2250 cm<sup>-1</sup> band refers to the vibrations of the presence of C-H (Kondo and Sawatari 1996, Schwanninger *et al.* 2004, Pasquali and Herrera 1997) and the1446 cm<sup>-1</sup> absorption band indicates O-C = O and CH<sub>2</sub> stress titers and shows they have a strong bond structure.



Figure 7: FTIR spectrum of board types A, B, C and D.

The tensile vibrations at 881cm<sup>-1</sup> show –C-OH as moderate bonds characteristic of dolomite. These characteristic bands are prominently detected in the FTIR signals (Gunasekaran and Anbalagan 2008). Stresses at 850-980 cm<sup>-1</sup> reveal Ca-O and Mg-O (Shahraki *et al.* 2009), at 1040 cm<sup>-1</sup>, 884,525 cm<sup>-1</sup>, and 480 cm<sup>-1</sup>, a weak bond and SiO<sub>2</sub> symmetrical and asymmetrical bond stresses (Tiwari *et al.* 1996, Agan *et al.* 2006), at 2929 cm<sup>-1</sup>, symmetrical C-H stresses and -OH stresses belonging to the methyl and methylene group (Kondo and Sawatari 1996, Schwanninger *et al.* 2004, Pasquali and Herrera 1997), at 1642 cm<sup>-1</sup>, C=O strain vibrations and stretching vibrations, C=C aromatic ring, C-C strain hemicellulose, lignin, carboxyl, and acetyl group stresses; at 1445 cm<sup>-1</sup>, C-H bending (Fengel 1991), at 1071 cm<sup>-1</sup>, C-C, C-O-H, C-H vibration stresses expressing the bonds in the hemicellulose and cellulose glucose ring, (Faix 1991, Fengel 1991, Marechal and Chanzy 2000) and at 884 cm<sup>-1</sup>, a C-O asymmetric stretching band (Kondo and Sawatari 1996, Falcao and Araujo 2013).

When the FTIR spectrum of the E, F and G board samples (Figure 8) are examined, the 3538-3455 cm<sup>-1</sup> band -OH group shows strong vibrations caused by water in the structure (Pandey 1999). The increase in the amount of red pine wood accounts for the increase in cellulose, hemicellulose, and lignin in the structure and more OH groups (Kim *et al.* 2007). In this band range, the transmitted value is higher in the H group than in the others. In the range of 2930-3000 cm<sup>-1</sup>the bands show C-H. The presence of the methyl and methylene group in the cellulose and hemicellulose expresses weak tensile vibrations (Esteves *et al.* 2013).



Figure 8: FTIR spectrum of board types E, F, G and H.

The vibrations in this band are the highest in group H. It shows a weak vibration at 1619 cm<sup>-1</sup> and a strong bond structure in the 1445-950-882 cm<sup>-1</sup> bands. These bands express the characteristic presence of the CaO and MgO belonging to dolomite (Ji *et al.* 2009). The vibrations in these bands are higher in the E group than in the others. This is caused by the strong SiO<sub>2</sub> stresses at 890 cm<sup>-1</sup> and 1151 cm<sup>-1</sup> and medium stresses at 472 cm<sup>-1</sup> (Tiwari *et al.* 1996, Agan *et al.* 2006). The presence of lignin carboxyl and acetyl groups at 1151 cm<sup>-1</sup> and the C-H group from the cellulose structure at 890 cm<sup>-1</sup> can be explained by the excess amount of wood in this group of composites (Faix 1991, Fengel 1991, Marechal and Chanzy 2000). Strong bonds at 890 cm<sup>-1</sup>,730 cm<sup>-1</sup>, and 671 cm<sup>-1</sup>, and medium bonds at 605 cm<sup>-1</sup>and 1472 cm<sup>-1</sup> result from the mineral additive gypsum (Prasad *et al.* 2005).

#### CONCLUSIONS

This study examined some properties of gypsum particleboard composed of gypsum and red pine wood chips admixed with mineral dolomite. It is important to note that increasing the mineral content has been found to improve WA properties. However, the presence of mineral dolomite had a negative effect on TS properties. All board types had higher IB values than the standard value of 0,28 MPa. This can be explained by the fact that the mineral substance in the board structure produced a hydrophobic effect against the binder, thus increasing the adhesive absorption of the particles. As a result, the usage of mineral dolomite in the board composition enhanced the IB properties.

The bending strength (MOR) and Modulus of Elasticity (MOE) values in all experimental types of boards were below the standard value requirement for general-purpose particleboards. This may have been caused by the presence of large dolomite particles in the board that decreased the compatibility of the wood particles.

In addition, the density was increased depending on the mineral increment, and the density differences of the board samples affected their thermal performance. This was attributed to the fact that large mineral particles can cause a decrease in thermal conductivity of wood-gypsum boards and consequently, the increment of particle content and amount of space between particles can lead to lower thermal conductivity. Meanwhile, after 300 s, the surface temperature values of the samples ranged from 89,3 °C to 104,1°C.

Thermal stability increased with the addition of dolomite to the composite structure of the group consisting of 70 % gypsum and wood particles. Therefore, for a successful thermal assessment, it is recommended that the wood chip content be kept below 20 % - 30 % by weight for thermal stability.

Overall, the results of this work show that the combination between wood particles, gypsum, and dolomite provides a good alternative to improve the thermal characteristic of the boards.

However, the mechanical properties of boards could not meet the standarts of composite boards such as construction boards. Therefore, it can be usable some places where is not strength neccesity such as insulation applications, indoor panels and siding. Besides, further reseaches should conduct to improve mechanical, physical and technological properties of mineral based particlebords.

#### REFERENCES

Agan, S.; Dana, A.; Aydınlı, A. 2006. TEM studies of Ge nanocrystal formation inPECVD grown SiO2: Ge / SiO2 multilayers. *J Phys Condens Matter* 18: 5037-5045. https://doi.org/10.1088/0953-8984/18/22/004

Amiandamhen, S.O.; Meincken, M.; Tyhoda, L. 2016. Magnesium based phosphate cement binder for composite panels: A response surface methodology for optimisation of processing variables in boards produced from agricultural and wood processing industrial residues. *Ind Crops Prod* 94: 746–754. https://doi.org/10.1016/j.indcrop.2016.09.051 Ashori, A.; Nourbakhsh, A. 2011. Preparation and characterization of polypropylene/wood flour/nanoclay composites. *European Journal of Wood and Wood Products* 69(4): 663-666. https://doi.org/10.1007/ s00107-010-0488-9

**ASTM. 1990.** Standard Test Method for Thermal Conductivity of Refractories by *Hot Wire*. ASTM. C1113-90. 1990. Annual Book of ASTM Standards.

Bekhta, P.; Dobrowolska, E. 2006. Thermal properties of wood-gypsum boards. *Holz Roh Werk* 64: 427-428. https://doi.org/10.1007/s00107-005-0074-8.

Biermann, C.J. 1993. Essentials of Pulping and Papermaking. Academic Press: New York. https://www.cabdirect.org/cabdirect/abstract/19940603802

**Chang, H.; Huang, P.J.; Hou, S.C. 1999.** Application of thermo-Raman spectroscopy to study dehydration of CaSO<sub>4</sub>-2 H<sub>2</sub>O and CaSO<sub>4</sub> - 0.5 H<sub>2</sub>O. *Mater Chem Phys* 58(1):12-19. https://doi.org/10.1016/S0254-0584(98)00239-9

Chen, X.L.; Huo, L.L.; Jiao, C.M.; Li, S.X. 2013. TG-FTIR characterization of volatile compounds from flame retardant polyurethane foams materials. *Journal of Analytical and Applied Pyrolysis* 100: 186-191. https://doi.org/10.1016/j.jaap.2012.12.017

**Cramer, S.M.; Friday, O.M.; White, R.H.; Sriprutkiat, G. 2003.** Mechanical Properties of Gypsum Board at Elevated Temperatures. In Fire and materials 2003: 8th International Conference, January 2003, San Francisco, CA, USA. London: Interscience Communications Limited, c2003: 33-42.

**DIN. 1998.** Fire behaviour of buildings materials and buildings components-part 1: buildings materials, concepts, requirements and tests. DIN. 4102-1. 1998. German Standards.

Espinoza-Herrera, R.; Cloutier, A. 2009. Thermal degradation and thermal conductivity of gypsum-cement particleboard. *Wood Fiber Sci* 41(1): 13-21. https://wfs.swst.org/index.php/wfs/article/view/231

Esteves, B.; Velez Marques, A.; Domingos, I.; Pereira, H. 2013. Chemical changes of heat treated pine and *Eucalyptus* wood monitored by FTIR. *Maderas-Cienc Tecnol* 15(2): 245-258. http://dx.doi.org/10.4067/S0718-221X2013005000020

Faix, O. 1991. Classification of lignins from differ rent botanical origins by FT-IR spectroscopy. *Holzforschung* 45: 21–28. https://doi.org/10.1515/hfsg.1991.45.s1.21

Falcao, L.; Araujo, M.EM. 2013. Tannins characterization in historic leathers by complementary analytical techniques ATR-FTIR, UV–vis and chemical tests. *J Cult Herit* 14: 499–508. https://doi.org/10.1016/j. culher.2012.11.003

**Fengel, D. 1991.** Moglichkeiten und Grenzen der FTIR-Spektroskopie bei der Charakterisierung von cellulose. *Das Papier* 46: 7–11.

**Guerrero**, A.S.; Hernandez, M.S. 2000. Thermodynamic solubility of Ca(OH)<sub>2</sub> in simulated radioactive sulphate liquid waste. *J Am Ceram Soc* 83(4): 882-888. https://doi.org/10.1111/j.1151-2916.2000.tb01289.x

Gunasekaran, S.; Anbalagan, G. 2008. Spectroscopic study of phase transitions in natural calcite mineral. *Spectrochim Acta Part* A 69: 1246-1251. https://doi.org/10.1016/j.saa.2007.06.036

Han, F.Q.; Tan, X.; Zhao, F.Q. 2017. Modification of Wood Fiber for Use in Cement Board. In: IOP Conference Series: Materials Science and Engineering. *IOP Publishing* 281(1): 012020. https://doi.org/10.1088/1757-899X/281/1/012020

Ji, J.; Ge, Y.; Balsam, W.; Damuth, J.E.; Chen, J. 2009. Rapid identification of dolomite using a Fourier Transform Infrared Spectrophotometer (FTIR): A fast method for identifying Heinrich events in IODP Site U1308. *Marine Geology* 258(1-4): 60-68. https://doi.org/10.1016/j.margeo.2008.11.007

Kang, Y.; Chang, S.J.; Kim, S. 2018. Hygrothermal behavior evaluation of walls improving heat and moisture performance on gypsum boards by adding porous materials. *Energ Build* 165: 431-439. https://doi.org/10.1016/j.enbuild.2017.12.052

**Kaya, A.I. 2015.** A study of composite materials that produced from recovered fibers of recycled waste papers. Ph.D Thesis, Suleyman Demirel University, Graduate School of Applied and Natural Sciences: Isparta, Turkey. http://tez.sdu.edu.tr/Tezler/TF02868.pdf

Kaya, A.I.; Sahin, H.T. 2016. The Effects of Boric Acid on Fiberboard Made from Wood/Secondary Fiber Mixtures: Part 3. Utilization of Recycled Waste Office Paper Fibers. Composites. *Chem Sci Inter J* 1-8. https://doi.org/10.9734/ACSJ/2016/27758

Kim, S.Y.; Chang, H.M.; Kadla, J.F. 2007. Polyoxometalate (POM) oxidation of milled wood lignin (MWL). *J Wood Chem Technol* 27(3-4): 225-241. https://doi.org/10.1080/02773810701702188

Kurugol, S.; Tekin, Ç. 2011. Evaluation of Sivas Traditional Sweet Plasters. *Gazi University Journal of Science* 24(1):161-173. https://dergipark.org.tr/tr/pub/gujs/issue/7418/96920

Kondo, T.; Sawatari, C. 1996. A Fourier transform infra-red spectroscopic analysis of the character of hydrogen bonds in amorphous cellulose. *Polymer* 37: 393–399. https://doi.org/10.1016/0032-3861(96)82908-9

Kozlowski, R.; Helwig, M.; Przepiera, A. 1995. Light-weight, environmentally friendly, fire retardant composite boards for paneling and construction. *Inorganic Bond Wood Fiber Compos Mater* 4(1): 6-11.

Kozlowski, R.; Mieleniak, B.; Helwig, M.; Przepiera, A. 1999. Flame resistant lignocellulosic-mineral composite particleboards. *Polym Degrad Stab* 64: 523-528. https://doi.org/10.1016/S0141-3910(98)00145-1

Kristóf-Makó, É.; Juhász, A.Z. 1999. The effect of mechanical treatment on the crystal structure and thermal decomposition of dolomite. *Thermochim Acta* 342: 105-114. https://doi.org/10.1016/S0040-6031(99)00290-7

L'vov, B.V.; Ugolkov, V.L. 2003. Kinetics of free-surface decomposition of dolomite single crystals and powders analyzed thermogravimetrically by the third-law method. *Thermochim Acta* 401: 139-147. https://doi.org/10.1016/S0040-6031(02)00506-3

Li, X.; Zhang, K.; Shi, R.; Ma, X.; Tana, L.; Ji, Q.; Xia, Y. 2017. Enhanced flame-retardant properties of cellulose fibers by incorporation of acid-resistant magnesium-oxide microcapsules. *Carbohydr Polym* 176: 246-256. https://doi.org/10.1016/j.carbpol.2017.08.096

Maitra, S.; Choudhury, A.; Das, H.S.; Pramanik, J. 2005. Effect of compaction on the kinetics of thermal decomposition of dolomite under non-isothermal condition. *J Mater Sci* 40(18): 4749-4751. https://doi.org/10.1007/s10853-005-0843-0

**Marechal, Y.; Chanzy, H. 2000.** The hydrogen bond network in I<sub> $\beta$ </sub> cellulose as observed by infrared spectrometry. *J Mol Struct* 523(1-3): 183-196. https://doi.org/10.1016/S0022-2860(99)00389-0

**Martias, C.; Joliff, Y.; Favotto, C. 2014.** Effects of the addition of glass fibers, mica and vermiculite on the mechanical properties of a gypsum-based composite at room temperature and during a fire test. *Compos: Part B* 62: 37-53. https://doi.org/10.1016/j.compositesb.2014.02.019

Morrow, D.W. 1982. Diagenesis 1. Dolomite - Part 1: The Chemistry of Dolomitization and Dolomite Precipitation. *Geosci Canada* 9(1). https://journals.lib.unb.ca/index.php/GC/article/view/3279

Nemli, G. 2003. Effects of Some Manufacturing Factors on the Properties of Particleboard Manufactured from Alder (*Alnus glutinosa* subsp. *barbata*). *Turk J Agric For* 27: 99-104. https://dergipark.org.tr/tr/download/article-file/120048

**Ozdemir, F. 2016**. Investigate on effect of dolomite mineral on some properties of high density fiberboard (HDF). *KSU. Journal of Engineering Science* 19: 93-98. http://jes.ksu.edu.tr/tr/download/article-file/227198

**Ozdemir, F.; Odabaş-Serin, Z.; Tutuş, A. 2017.** Investigation of effect of some fire reterdant chemicals and mineral materials used in surface coating on combustion performance of particleboard. *BioResources* 12(4): 8862-8869. https://bioresources.cnr.ncsu.edu/resources/investigation-of-the-effect-of-some-fire-retar-dant-chemicals-and-mineral-materials-used-in-surface-coating-on-combustion-performance-of-particleboard/

Pandey, K.K. 1999. A study of chemical structure of soft and hardwood and wood polymers by FTIR spectroscopy. J Appl Polym Sci 71(12): 1969-1975. https://doi.org/10.1002/(SICI)1097-4628(19990321)71:12%3C1969::AID-APP6%3E3.0.CO;2-D

Papadopoulos, A.N. 2019. Advances in Wood Composites. *Polym J* 48. https://doi.org/10.3390/polym12010048

Pasquali, C.E.L.; Herrera, H. 1997. Pyrolysis of lignin and IR analysis of residues. *Thermochim Acta* 293: 39-46. https://doi.org/10.1016/S0040-6031(97)00059-2

**Prasad, P.S.R.; Chaitanya, V.K.; Prasad, K.S.; Rao, D.N. 2005.** Direct formation of the γ-CaSO4 phase in dehydration process of gypsum: In situ FTIR study. *Am Min* 90(4): 672-678. https://doi.org/10.2138/am.2005.1742

Sain, M.; Park, S.H.; Suhara, F.; Law, S. 2004. Flame retardant and mechanical properties of natural fibre–PP composites containing magnesium hydroxide. *Polymer Degradation and Stability* 83(2): 363–367. https://doi.org/10.1016/S0141-3910(03)00280-5

Schwanninger, M.; Rodrigues, J.C.; Pereira, H.; Hinterstoisser, B. 2004. Effects of short-time vibratory ball milling on the shape of FT-IR spectra of wood and cellulose. *Vib Spectrosc* 36: 23–40. https://doi.org/10.1016/j.vibspec.2004.02.003

Shahraki, B.K.; Mehrabi, B.; Dabiri, R. 2009. Thermal behavior of Zefreh dolomite mine (Central Iran). *J Mining Metall B: Metall* 45(1): 35-44. https://doi.org/10.2298/JMMB0901035S

Sharifi, N.P.; Shaikh, A.A.N.; Sakulich, A.R. 2017. Application of phase change materials in gypsum boards to meetbuilding energy conservation goals. *Energ Build* 138:455-467. https://doi.org/10.1016/j.en-build.2016.12.046

Sophia, M.; Sakthieswaran, N.; Babu, G. O. 2016. Gypsum as a construction material-a review of recent developments. *Inter J Inno Res Sci Technol* 2(12): 1-9. https://www.academia.edu/26952348/Gypsum\_as\_a\_Construction\_Material\_A\_Review\_of\_Recent\_Developments

Sudin, R.; Swamy, N. 2006. Bamboo and wood fibre cement composites for sustainable infrastructure regeneration. *J Mater Sci Technol* 41: 6917-6924. https://doi.org/10.1007/s10853-006-0224-3

Tiwari, S.; Rana, F.; Hanafi, H.; Hartstein A.; Crabbe, E.F.; Chan, K. 1996. A silicon nanocrystals based memory. *Appl Phys Lett* 68: 1377. https://doi.org/10.1063/1.116085

Todor, D.N. 1976. Thermal Analysis of Minerals. 1st Edition Abacus Press. https://doi.org/10.1180/claymin.1978.013.1.11

**TS EN. 1999.** Wood Based Panels Determination of modulus of elasticity in bending and of bending strength. TS EN 310. 1999. Turkish Standards.

TS EN. 2012. Particleboards - Specification. TS EN 312. 2012. Turkish Standards.

TS EN. 1999. Particleboards and fibreboards- Determination of swelling in thickness after immersion in water. TS EN 317. 1999. Turkish Standards.

**TS EN. 1999.** Particleboards and fibreboards- Determination of tensile strength perpendicular to the plane of the board. EN 319:1999. Turkish Standards.

**TS EN ISO. 2002.** Reaction to fire tests - Ignitability of building products subjected to direct impingement of flame - Part 2: Single-flame source test. TS EN ISO 11925-2. 2002. Turkish Standards.

Wakili, K.G.; Hugi, E.; Wullschleger, L.; Frank, T.H. 2007. Gypsum board in fire-modeling and experimental validation. *J fire Sci* 25(3): 267-282. https://doi.org/10.1177/0734904107072883

Warren, J. 2000. Dolomite: Occurrence, evolution and economically important associations. *Earth-Sci Rev* 52: 1-81. https://doi.org/10.1016/S0012-8252(00)00022-2

Yalçın, Ö.Ü. 2018. Investigation of performance properties of plates produced from some lignocellulosic sources with mineral (dolomite and olivine) additives. Ph.D Thesis, Isparta University of Applied Sciences, The Institute of Graduate Education. Isparta, Turkey. https://tez.yok.gov.tr/UlusalTezMerkezi/tezSorguSonu-cYeni.jsp