Effect of Mineral Materials Content as Filler in Medium Density Fiberboard

Ferhat Özdemir

The use of different mineral material types and contents in medium density fiberboard (MDF) production was investigated. Three different minerals (sepiolite, dolomite, and perlite) and five different ratios (3%, 6%, 9%, 12%, and 15%) were used according to the oven-dry wood fiber weight. These minerals were homogeneously added as powder between the wood fibers. Some physical, mechanical, limit oxygen index (LOI), and thermogravimetric analysis (TGA) tests of the boards were conducted. The mineral fillers negatively affected the physical properties such as water absorption (WA), thickness swelling (ThS), and mechanical properties such as modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond (IB). However, LOI and TGA test results showed a positive effect on combustion resistance depending on the type and rate of mineral fillers.

Keywords: Sepiolite; Dolomite; Perlite; LOI

Contact information: Department of Forest Products Chemistry and Technology, Faculty of Forestry, Kahramanmaraş Sütçü Imam University, Kahramanmaraş, Turkey; * Corresponding author: ferhatozd@hotmail.com

INTRODUCTION

The forest industry has been rapidly developing in recent years. For this reason, raw material supply is becoming more difficult and presents a large problem in terms of cost. Therefore, the use of wood-based boards such as OSB, MDF, and plywood becomes more attractive. Compared to the solid wood, the most important advantages of wood-based boards are the coating and shaping of their surfaces and the development of their structural properties during production. The biggest disadvantage of wood is its easy flammability. Fire retardants (FRs) are used for this purpose. Fire retardants are used in 60 to 65% of plastic, 20 to 25% of foam, 5 to 6% of textile, and 3 to 4% of forest industry sector products (Horrocks and Price 2001).

In order to improve the structural properties of wood-based boards, some applications are made to the wood fibers before and during production. Depending on the place of use such as inside and outside the building, a certain proportion of fillers are added to the fibers, and the physical, mechanical, and combustion resistance of the wood based boards can be adjusted. Mineral fillers between lignocellulosic fibers are used as fire retardant (Kozlowski *et al.* 1999). When boron compounds such as borax and boric acid have been used as fillers, the combustion resistance of wood-based boards has been improved (Özdemir and Tutuş 2013). Many fire retardant chemicals are used to prevent combustion in wood and cellulosic materials. The products based on boron compounds are used alone or in combination with their smoke suppressor properties. Due to their structural properties, inorganic salts such as boric acid and boron either reduce or prevent combustion (Baysal 1994). There are abundant mineral fillers in nature. Among these,

sepiolite, dolomite, and perlite minerals are suitable fillers for wood-based board production.

Sepiolite (Si₁₂Mg₉O₃₀(OH)(OH₂)₄·8H₂O) has a porous structure with SiO₂ (60.6%) and MgO (22.4%) (Sabah *et al.* 1997). It takes different colors including white, cream, gray, pink, and brown. Sepiolite is easy to process because it contains high amounts of water. The micropar diameter is 15 A°, the melting temperature is 1400 to 1450 °C, and the specific gravity is 2 to 2.3 g/cm³. Dolomite (CaMg (CO₃)₂) is a brittle mineral that contains plenty of calcium and magnesium carbonate (Adesakin *et al.* 2013). The specific weight is 2.8 g/cm³ and the hardness is 3.5 to 4. It is used in a wide area such as iron-steel, glass, ceramic, paint, fertilizer, brick, and cement construction. Perlite is a naturally occurring silica-based volcanic rock. Its porous structure gives perlite absorbency and surface cooling properties. It has SiO₂ (76%) and Al₂O₃ (17%). When heated at 900 to 1100 °C, its volume can be expanded by about 20 times. Its density is between 2.2 to 2.4 g/cm³, and its melting temperature is between 1260 and 1340 °C.

Almost no study has been conducted on the use of sepiolite, dolomite, and perlite as inorganic filler in the production of wood-based boards. The aim of this study was to determine the effect of sepiolite, dolomite, and perlite minerals on the physical, mechanical, and combustion resistance of the boards in the MDF board production.

EXPERIMENTAL

Materials

The wood fibers used as raw material in MDF production were obtained from the Kastamonu Entegre Factory (Adana, Turkey), which is a commercial company. Fiber mixed ratio (50% scotch and 50% maple) was determined and its moisture content was dried until it reached 3 to 4%. Sepiolite (İstanbul Maden, İstanbul, Turkey), dolomite (Tire Dolomit, Tire, İzmir, Turkey), and perlite (Erper Madencilik, Erzincan, Turkey) minerals were used as fillers (3, 6, 9, 12%, and 15%). The melting point of dolomite, sepiolite, and perlite was 2495 °C, 1550 °C, and 1260 °C, respectively. Mineral fillers have a particle size of 100 mesh. Urea formaldehyde glue (the molar ratio (U/F) 1.17, viscosity 65 cps, pH 8.5, density 1235 kg/m³, solids content 55%) was used as a binder at 12%. All mixing ratios were determined based on the oven-dried fiber weight.

Panel Manufacturing

The glued fibers, sepiolite, dolomite, and perlite minerals (3, 6, 9, 12%), and 15%) were homogeneously mixed in a resin blender. This mixture was cold pressed with a mold of 40 x 40x 50 cm dimensions and a mat cake was formed. The hot-pressing process was applied in Cemil Usta SSP 125 press machine, and 8 mm thick, 0.670 to 0.700 gr/cm³ density boards were produced. A total of 3 boards were produced for each production parameter. The density profiles (density: 0.70 g/cm³) of the boards were checked with the GreConTM device (Fagus, Nürnberg, Germany).

Parameters	MDF
Temperature (°C)	183
Press (MPa)	3.5
Time (s)	300

Table 1. N	MDF Produc	tion Hot Pressi	ing Parameter	Values
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The test samples were conditioned for two weeks at a temperature of 20 °C and a relative humidity of 65%. Hot press application parameters are shown in Table 1.

Test Methods

The thickness swelling (ThS) and water absorption properties (WA, 2-24h) were carried out according to the EN-317 (1993) standard. The modulus of rupture (MOR), modulus of elasticity (MOE) (EN-310, 1993), and internal bond (IB), (EN-319, 1993) were performed in accordance with the relevant standards. The ASTM 2863-09 (2006) standard was applied in the combustion resistance test (LOI). For each test group, 16 samples were tested for physical and mechanical properties, and 6 samples for the LOI tests.

Thermogravimetric Analysis

The test samples were separately ground in the Wiley mill. The milled test sample flours were screened using a 100 mesh size. For each test group, 8 to 10 mg the test sample flour was tested on Perkin Elmer thermogravimetric analyzer. The test samples were heated with dry nitrogen gas stream in the range of 20 to 700 °C. The nitrogen gas flow rate was set to 100 mL min⁻¹, and the heating rate to 10 °C min⁻¹. Thermal characteristics of test samples were analyzed and weight losses were determined by a computer program. For each group, tests were performed in 3 replicates.

Interfacial Morphologic Analysis

To determine the distribution of mineral fillers among wood fibers, the surfaces of the samples were exposed to the vacuum and coated with gold. In the KSU-USKIM Laboratory, using the Jeann-5600 Scanning Electron Microscope (SEM) device with an acceleration voltage of 20 kV, the interfaces of the test specimens were observed.

Statistical analysis

Simple variance analysis was performed at a 95% confidence level to determine the effect on board properties of mineral materials used in board production. The Duncan discrimination test was used to determine the differences between the board groups.

RESULTS AND DISCUSSION

Mechanical Properties

The mechanical properties, LOI test, and statistical analysis data of the test boards produced by adding different mineral fillers in certain ratios are given in Table 2. The control sample bending strength value was 27.5 N/mm². This value was 27.3 and 23.4 N/mm² for B and C coded samples with sepiolite. The values obtained were in accordance with the standards (\geq 23 N/mm²). In samples with dolomite added, only 3% B-coded samples meet the required standard for bending resistance, whereas in other sample groups, the standard values were not reached. In the samples with perlite added, the desired standard values could not be reached in any group according to the obtained data. The use of minerals as fillers adversely affected the mechanical properties. Significant decreases in bending strength values were determined due to the increase in mineral filler use. This negative effect increased with the increase in mineral filler

content. In the use of 15% of filler material, the most negative effect on bending resistance was the dolomite (72.2%), perlite (54.4%), and sepiolite (37.1%). The other groups did not carry standard values, except for the samples containing sepiolite at 3 to 6% and dolomite at 3%.

Code	Mineral Ratio (%)	MOR (N/mm ²)	MOE (N/mm ²)	IB (N/mm ²)	LOI (%)
А	Control	27.5 (3.03) ^a	2783 (244) ^a	0.65 (0.02) ^a	24.7 ^h
В	3% Sep	27.3 (3.02) ^a	2745 (388) ^a	0.59 (0.04) ^b	25.5 ^{efgh}
С	6% Sep	23.4 (1.62) ^{bc}	2148 (181) ^{bc}	0.51 (0.03) ^{de}	26.0 ^{cdefg}
D	9% Sep	18.8 (2.35) ^{def}	2089(186) ^{cd}	0.45 (0.04) ^g	26.3 ^{bcdef}
E	12% Sep	17.9 (2.02) ^{def}	2080 (211) ^{cd}	0.39 (0.03) ^h	27.2 ^{ab}
F	15% Sep	17.3 (1.66) ^ŕ	1719 (96) ^{ef}	0.31 (0.04) [,]	27.7ª
G	3% Dol	24.6 (2.62) ^b	2397 (225) ^b	0.53-(0.03) ^{cd}	25.3 ^{fgh}
Н	6% Dol	20.6 (2.46) ^{cde}	2001 (153) ^{cd}	0.45 (0.02) ^{fg}	25.7 ^{efgh}
J	9% Dol	12.9 (0.85) ^h	1617 (55) ^{ef}	0.36 (0.03) ^h	26.2 ^{bcdefg}
K	12% Dol	11.9 (0.84) ^h	1505 (179) ^f	0.29 (0.03) [,]	26.5 ^{bcde}
L	15% Dol	7.65 (0.46) ⁱ	1111 (72) ^g	0.22 (0.04) ^j	26.8 ^{abcd}
М	3% Per	21.0 (0.58) ^{cd}	2093 (106) ^{cd}	0.56 (0.02) ^{bc}	25.2 ^{gh}
N	6% Per	19.7 (0.56) ^{def}	2087 (57) ^{cd}	0.49 (0.03) ^e	25.5 ^{efgh}
Р	9% Per	19.6 (0.47) ^{def}	2009 (63) ^{cd}	0.44 (0.02) ^{fg}	25.8 ^{defg}
0	12% Per	14.8 (0.87) ^{gh}	1808 (194) ^{de}	0.36 (0.04) ^h	26.3 ^{bcdef}
R	15% Per	12.5 (0.84) ^{gh}	1550 (48) ^{ef}	0.29 (0.04)	27.0 ^{abc}

Table 2. Average Values of the Mechanical Properties of the Boards

*The values in parentheses are the standard deviation; different letters in the same column indicate statistical differences at the 95% confidence level (According to EN 622-5 standard MOR is ≥23 N/mm² and IB is ≥0.60 N/mm²)

According to the EN 622-5 (2006) standard, IB values were ≥ 0.6 N/mm². In the boards produced, only the control sample met this requirement. The boards produced by adding mineral fillers did not meet the requirement for the IB standard. The highest IB values were determined in the control sample while the lowest IB values were determined in the samples produced by adding 15% for each mineral filler. These values were determined as sepiolite (F group), dolomite (L group), and perlite (R group) as 0.31, 0.21, and 0.20 N/mm², respectively. The mineral filler type and the usage rate negatively affected IB values as it prevented the fibers from sticking together. As the mineral use rate for all minerals increased, the decrease in the IB values continued. This decrease was similar to the trend of the MOR and MOE. Mineral fillers increased the contact surface between the fibers and the glue and also created a barrier effect. It prevented the glue from being placed in the gaps and reduced the mechanical locking of the fiber and glue (Ayrılmış *et al.* 2017). As the use of mineral fillers increased, the surfaces of the fibers were coated with more mineral filler.

The increase in the mineral filler content reduced the effect of interconnecting the fibers, causing MOR, MOE, and IB resistance to be adversely affected. In addition, the high hydrophilic properties of the mineral fillers were effective in reducing the interfacial adhesion. Due to its large surface area, siloxane, and silanol groups, sepiolite can exhibit a certain activity in its use with plastic materials having elastic properties and acts as a semi-reinforcing filler (Alvarez 1984). However, a similar reinforcing effect was not observed in wood fiber use. Mineral fillers have a positive effect by reducing the burning potential of wood, but they may have negative effects on properties such as

hygroscopicity, strength, stability, toxicity, adhesiveness and mechanical properties and attractiveness to paint coatings (Holmes 1974).

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Code	Mineral Ratio (%)	2 ThS (%)	24 ThS (%)	2 WA (%)	24 WA (%)
A	Control	15,4 (1,5) ^a	28,2 (4,0) ^a	28,8 (2,2) ^a	47,6 (8,4) ^a
В	3% Sep	23,3 (2,3) ^c	32,1 (4,0) ^{bc}	49,5 (4,1) ^{cd}	86,7 (5,4) ^c
С	6% Sep	34,5 (1,9) ^e	37,4 (2,1) ^{ef}	49,5 (4,1) ^{hı}	97,5 (6,7) ^d
D	9% Sep	37,3 (2,8) ^{fg}	40,2 (2,6) ^g	94,9 (7,3) ^{ıj}	107,2 (8,0) ^e
E	12% Sep	40,6 (1,7) ^h	44,5 (2,5) ^{hı}	104,6 (7,0) ^k	111,7 (11,0) ^{efg}
F	15% Sep	41,2 (3,8) ^h	46,9 (2,0)	109,7 (8,4) ^k	114,9 (9,1) ^{fg}
G	3% Dol	17,2 (1,2) ^{ab}	30,6 (2,7) ^{ab}	29,7 (2,3) ^a	52,4 (4,8) ^a
Н	6% Dol	36,6 (3,4) ^{ef}	39,1 (3,3) ^{fg}	54,7 (5,5) ^d	98,8 (6,8) ^d
J	9% Dol	39,3 (3,7) ^{gh}	43,9 (2,5) ^h	66,8 (6,5) ^e	127,6 (8,4) ^h
K	12% Dol	45,2 (3,8)	49,6 (3,9) ^j	76,1 (7,7) ^f	132,4 (9,1) ^{hı}
L	15% Dol	49,0 (2,6) ^j	52,7 (3,8) ^k	81,9 (4,5) ^g	135,4 (7,1) [,]
М	3% Per	18,2 (2,4) ^b	29,3 (2,7) ^a	36,2 (4,4) ^b	51,1 (7,9) ^a
N	6% Per	23,3(2,1) ^c	34,2 (2,1) ^{cd}	48,6 (4,0) ^c	69,2 (5,3) ^b
0	9% Per	29,6 (1,6) ^d	35,8 (2,5) ^{de}	88,8 (8,6) ^h	110,3 (10,3) ^{ef}
P	12% Per	31,5 (1,5) ^d	38,5 (3,7) ^{fg}	97,2 (6,4) ^j	112,3 (9,6) ^{efg}
R	15% Per	34,6 (2,0) ^e	40,1 (2,4) ^g	108,0 (7,8) ^k	118,4 (6,7) ^g

Table 3. Average Values of the Physical Properties of the Board Groups

 Produced

* WA rate according to EN 622-5 standard is \leq 35%.

Physical Properties

The test results and statistical analysis data of thickness swelling and water absorption characteristics are given in Table 3. For 2 and 24 h, the control sample thickness swelling was 15.4 to 28.2%, while the water absorption amount was 28.8% and 47.6, respectively. The thickness swelling and water absorption properties of the test panels increased as the amount of mineral filler usage was increased.

The highest values of these properties were obtained in 15% mineral applications. According to this mineral utilization rate, thickness swelling for 2 and 24 h were determined as 41.2% to 46.9% in sepiolite mineral (F group), 49.0 to 52.7% in the dolomite mineral (L group), and 34.6 to 40.1% in the perlite mineral (R group). Besides, the amount of water absorption was determined as 109.7 to 119.9% in the F group, 81.9 to 135.4% in the L group and 108.0 to 118.4% in R group.

Compared with the control sample, the amounts of increased thickness for 2 to 24 h in the 15% mineral content were in the F group 267.5% to 166.8%, L group 318.1% to 186.8%, and R group 224.6 to 142.1%, respectively. The amount of water absorption in the F group was 380.2% to 241.3%, in the L group 284.3% to 284.4%, and in the R group 375.0 to 248.7%. This increase in the thickness swelling and water absorption properties of the boards was due to the material properties of the mineral materials such as hydrophilic properties. Sepiolite has a fibrous structure and has continuous channel gaps throughout the fiber (Rodriguez *et al.* 1994). Due to this feature, it has an extremely high sorption characteristic and can hold water up to 200 to 250 times its own weight (Sabah *et al.* 1997). For dolomite and perlite minerals, due to the magnesium and calcium content of the structure, the ThS and WA features have been found to have a negative impact (Sabah and Çelik 1999). The results were consistent with similar studies using FR chemicals. The ThS and WA properties of the MDF boards can be reduced by using

optimum mineral material content, covering the surfaces and edges of the boards with laminate etc. coating materials.

Limiting Oxygen Index (LOI)

The LOI value of the control sample was 24.7. The lowest (3%) and highest (15%) addition rates of sepiolite mineral were 25.5 and 27.7, respectively. These values in the dolomite were 25.3 and 26.8, respectively, and 25.2 and 27.0 in the perlite, respectively. The values of all mineral usage rates were different compared to the control sample. All mineral materials and usage rates had a positive effect on the LOI values. Figure 1 shows that they have a slightly positive effect depending on the usage quantities. The most positive effect was found in the samples of sepiolite 15% (F group).

The LOI test is the principle of determining the minimum oxygen content in the oxygen-nitrogen mixture that is required to continue the combustion process. The LOI values of easy burning materials are low ($\leq 28\%$) while slow and difficult burning materials are high ($\geq 28\%$). Heat conductivity is one of the important factors affecting combustion (Le Van 1984). Due to the woody porous structure, the heat conductivity of the wood material varies according to the tree species and the direction of the fibers in the same tree, depending on the type of binders and additives in the wood boards produced by various binders and the added fillers and additives (Kamke and Zylkowoski 1989). Fire retardants reduce the pyrolysis temperature of wood in a combustion environment. Fire retardant chemicals reduce the combustion temperature by absorbing the temperature of the combustion environment and increase the carbonization and reduce the pyrolysis temperature of the test samples are shown in Fig. 1. Oxygen, heat and combustible material are required for combustion. Mineral fillers reduce the ambient temperature by absorbing.



Fig. 1. Average values of the LOI test results of the test samples

Thermogravimetric Analysis

Depending on the mineral substance type and utilization rates, the weight loss caused by temperature increase in MDF boards was analyzed by the TGA method. In the measurements up to 700 degrees, it was found that all mineral fillers had a positive effect on the combustion resistance compared to the control sample. The amount of weight loss in mineral filled MDF boards was lower than the control sample. The thermal losses (initial and end) temperature values and weight losses, which are measured depending on the type and use of the mineral material, are given in Table 4.

As shown in Table 4, each mineral substance has a different effect on the combustion resistance. However, as the usage rate of all mineral fillers increased, the

combustion resistance was positively affected. Sun *et al.* (2012), in the production of MDF boards, the use of fire retardant (FR) chemical substances were shown to increase fire resistance.

The amount of weight loss in MDF boards with mineral filler was lower than the control sample. Table 4 shows the thermal degradation (first and end) temperature values of the test samples as well as the weight loss due to the type of mineral material and its utilization rate.

Codo	Mineral	Thermal	Thermal	Thermal	Total weight
Code	ratio	degradation initial	degradation end	degradation	loss
	(%)	temperature (°C)	temperature (°C)	weight loss (%)	(%)
А	Control	282.1	360.3	69.1	78.8
В	3% Sep	283.2	359.7	60.3	72.7
С	6% Sep	284.1	357.4	58.4	70.2
D	9% Sep	285.7	355.5	56.7	67.9
E	12% Sep	289.8	353.8	53.8	67.5
F	15% Sep	292.3	354.8	54.4	66.8
G	3% Dol	283.0	360.6	63.8	76.6
Н	6% Dol	283.8	359.8	62.6	75.2
J	9% Dol	284.4	359.6	62.3	74.4
K	12% Dol	285.3	358.3	60.9	73.8
L	15% Dol	288.9	358.1	60.2	71.4
М	3% Per	282.4	358.9	62.7	74.7
Ν	6% Per	283.1	356.7	61.5	74.1
Р	9% Per	285.1	354.4	60.1	72.3
0	12% Per	287.7	353.9	57.8	69.0
R	15% Per	290.3	353.8	56.3	68.9

Table 4. Average Weight Loss Values of MDF Test Samples

As can be seen in Table 4, each mineral substance had a different effect on the combustion resistance. However, as the usage rate of all mineral fillers increased, the combustion resistance was positive. Sun *et al.* (2012) stated that combustion resistance increased with the use of Fire Retardant (FR). The highest mass loss was determined in control samples (78%) at 700 °C. The most positive effect was obtained in 15% of all mineral fillers, while the least effect was obtained in 3% utilization rates. The most effective fillers were sepiolite 15% (66.8%), perlite 15% (68.9%), and dolomite 15% (71.4%), respectively.

As the mineral filler usage rate increased, the weight loss decreased. The reason for this loss of weight is that it improves the thermal isolation by increasing the amount of carbonization of the mineral substances during combustion, especially at low temperatures (Kollman and Cote 1968). These mineral fillers also positively affected and improved thermal decomposition temperatures. The fillers reduce the combustion temperature by absorbing the temperature of the combustion environment and preventing the release of flammable gases. TGA and LOI test results were consistent and similar results were obtained.

Morphological Observation

Homogeneous distributions of the 100 mesh size fillers between the wood fibers were obtained using the scanning electron microscope and are shown in Fig. 2.

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Fig. 2. SEM micrographs of the distribution of sepiolite (a), dolomite (b) and perlite (c) among wood fibers and control (d)

CONCLUSIONS

- 1. The mineral fillers had a negative effect on the mechanical properties of the boards such as modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond (IB) and increased the negative effect as the utilization rate increased.
- 2. The rate of water absorption and thickness swelling increased significantly as the mineral use amount increased. The maximum thickness swelling ratio for 2 and 24 h was determined in sepiolite 15% (F group) samples. The water absorption for 2 h was obtained in the sepiolite 15% (F group) samples while the water absorption values for 24 h were determined in the dolomite 15% (L group) samples.
- 3. The mineral fillers had a positive effect on the limit oxygen index (LOI) values. The most positive effect was on the sepiolite 15% (F group) samples. Mineral fillers can be used to increase the combustion resistance of medium density fiberboard.
- 4. TGA results revealed that mineral fillers had a positive effect on the combustion resistance. The most effective use rate for all fillers is 15% and the filler is sepiolite.

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Article submitted: October 9, 2018; Peer review completed: December 31, 2018; Revised version received and accepted: January 7, 2019; Published: February 1, 2019. DOI: 10.15376/biores.14.1.2277-2286