



## A novel lightweight gypsum composite with diatomite and polypropylene fibers



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### ABSTRACT

Plaster and composites made up of gypsum have been widely used in building for different purposes. In this work, a new gypsum composite containing diatomite and polypropylene fibers was produced. Diatomite, fossilized of microscopic plants, was used up to 20% while fiber up to 1%. Samples with  $120 \times 120 \times 20$  mm and  $40 \times 40 \times 160$  mm size were produced to do tests. In addition to XRF, XRD, TGA and SEM investigations of diatomite, physical, mechanical and thermal test were done on produced samples. Also, Analysis of Variance (ANOVA) test were done in order to see the effect of diatomite and fibers studied on the quadratic model. It was observed that the usage of diatomite increases porosity that results in decreasing unit weight and thermal conductivity coefficient. Fibers increase mechanical properties.

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### 1. Introduction

Gypsum has been used as a construction material since ancient times. The oldest applications of gypsum plaster or gypsum binder were dated by archeologists to 7000 years BCE [1]. Gypsum has been an important subject among many researchers due to abundant availability of gypsum in nature, its ecological and technological properties, low energy consumption of manufacturing and many other positive properties [2]. Today, gypsum and gypsum derived materials such as plaster, board and composite have been increasingly used for many purposes in building construction [3–17] since they presents a good fire resistance, thermal and sound insulation properties etc. Gypsum products as construction materials can be improved in point of physical, mechanical, thermal and sound insulation properties. In order to improve physical, thermal and sound insulation properties of gypsum composites, their porosity can be used by using pore making agents such as expanded silica gel granules and vermiculite [10,17]. To improve their mechanical properties such as bending and compressive

strengths, fibers can be introduced into the gypsum composites since fibers such as glass fiber, carbon fiber, polypropylene fiber, steel fiber, polyamide fiber, polyester fiber have been extensively used in construction technology for their specific advantages [3–7,18–20]. Thus, usage fiber reinforcement in gypsum composites can increase the materials strength.

Diatomite is a siliceous sedimentary rock, which consists principally of fossilized skeletal remains of diatoms, unicellular aquatic plants related to algae. These microscopic plants have the unique capability of extracting silica from water to produce their skeletal structure. When diatoms die, their skeletons settle to form a diatomite deposit. It is a chalk-like, soft, friable, earthy, very fine grained rock, usually light in color. It is very finely porous and very low in density. As the diatoms are composed of an amorphous form of silica containing a small amount of microcrystalline material, diatomite is chemically stable and inert. They are generally amorphous, hydrated or opaline silica ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ) and sometimes clay minerals, calcite and organic components [21]. Due to the open structure of diatom skeletons, it is a lightweight rock with enough hardness. Diatomite is commonly found in volcanic environments, with the deposits forming in lakes in volcanically active areas. Turkey has abundant of diatomaceous earth deposits in Afyon region [22]. Thus, diatomite composed of amorphous opaline silica

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can be transformed into other mineral phases such as disordered cristobalite/tridymite or quartz [21]. Diatomite has a low density and porous structure which is desired for thermal performance, fire resistance and sound absorption, that makes it attractive for use as a lightweight construction material. Due to its low density, diatomite can be used as a constituent of concretes and plasters. The use of diatomite in gypsum mix should allow a reduction of the density and thermal conductivity values of the gypsum composites. Existing studies use different fiber type, while no results have been reported for using both diatomite and polymeric fibers in gypsum composites. Thus, in this study, gypsum composites containing diatomite and polypropylene fibers were produced to investigate their thermal conductivities, physical and mechanical properties.

## 2. Experimental studies

### 2.1. Gypsum

A commercially available gypsum plaster mix characterized according to TS EN 13279-1 [23] was used in this study. The properties of gypsum used were given in Table 1.

### 2.2. Diatomite

In this study, diatomite for gypsum composite production obtained from Selko A.S. manufacturer in Bartın, Turkey was used. It was characterized for chemical (X-ray fluorescence analysis), mineralogical (X-ray diffraction analysis), thermal (thermogravimetric analysis) and microstructural (scanning electron microscopy analysis) properties.

The diatomite particles smaller than sieving intervals of 100-mesh (149  $\mu$ m) were used for mixes. The chemical and physical properties of diatomite are presented in Table 2. Diatomite was added to the mixtures at ratios of 10% and 20%. The amount of diatomite listed is the ratio of the diatomite mass to the total dry mass.

The microstructure of diatomaceous earth is shown in Fig. 1. They have different morphological structures such as cylindrical and others. Their particle sizes are smaller than 25  $\mu$ m. The individual particles commonly include submicron sized pores. The surface area of diatomite was measured as 13.925 m<sup>2</sup>/g by a B.E.T. surface area analyzer. Semi-quantitative chemical analysis of diatomite grains were determined by the energy-dispersive X-ray spectroscopy (EDS). According to EDS analysis, the chemical composition of diatomite is 92.7  $\pm$  0.5% SiO<sub>2</sub>, 6.09  $\pm$  0.4% Al<sub>2</sub>O<sub>3</sub> and 1.2  $\pm$  0.1% CaO. Also, X-ray fluorescence (XRF) analysis of diatomite was performed. The analysis result is given in Table 2. The diatomite includes mainly silicon oxide, also the oxides of aluminum, calcium, iron and others. Loss on ignition of diatomite is about 10.5% by weight.

Fig. 2 shows X-ray diffraction analysis of the diatomite. The broad hump in the vicinity of the cristobalite and quartz peaks (from 20° to 40°) is typical for diatomite. This case indicates an amorphous phase (opaline silica) as well as crystalline silica phases.

The TGA curve of diatomite is given in the Fig. 3 where a total weight loss of about 11% for 1100 °C. In the first weight loss, the dehydration of the diatomite takes place between 20 and 200 °C which corresponds to the loss of free water physically absorbed on the surfaces and in the pores of diatomite particles. The mass losses in the range of 200–500 °C can be attributed to the removal of chemically absorbed water molecules of amorphous opaline silica (SiO<sub>2</sub>·nH<sub>2</sub>O).

### 2.3. Polypropylene fibers

Polypropylene fibers with 22  $\mu$ m diameter and 12 mm length were used, their properties listed in Table 3. The fibers were added into the mixtures in the ratios of 0.5 wt.% and 1 wt.%. The amount of fibers is counted as a ratio to the total dry mass.

**Table 1**  
The properties of gypsum used.

Workability time (min)	60–90
Final setting time (min)	150
Compressive strength (MPa)	2.5
Flexural strength (MPa)	1
Dry density (kg/m <sup>3</sup> )	650–1000
1000 $\mu$ m (% passing)	100
150 $\mu$ m (% passing)	60

**Table 2**  
Chemical composition of the diatomite (XRF analysis, wt.%).

Compounds	Diatomite
SiO <sub>2</sub>	75.1
Al <sub>2</sub> O <sub>3</sub>	8.37
CaO	2.35
MgO	0.55
Fe <sub>2</sub> O <sub>3</sub>	1.93
K <sub>2</sub> O	0.58
Na <sub>2</sub> O	0.03
TiO <sub>2</sub>	0.32
Loss on ignition	10.5

### 2.4. Preparation and testing of the gypsum composites

Experimental set and mix proportions of the gypsum composite are presented in Table 4. The gypsum mixtures containing with diatomite and PP fibers were prepared in a mixer. The gypsum and additives were first put in the mixer and mixed for 5 min in order to achieve a homogeneous dry mixture. Water was then added to the dry mixture and mixed for 3 min so as to obtain the plaster slurry. The ratio of plaster mix/water used was 900 g/585 ml. The water used during these experiments was the room temperature of about 20 °C. Two different shaped molds with dimensions of 120  $\times$  120  $\times$  20 mm and 40  $\times$  40  $\times$  160 mm were prepared to produce test samples. Rectangular (40  $\times$  40  $\times$  160 mm) and plate shape (120  $\times$  120  $\times$  20 mm) samples were formed by slurry casting for the mechanical tests and thermal conductivity determination, respectively. For each test, three series of specimens were produced. In the meantime, the slurry was compacted by a shaker for 10 s in order to ensure complete filling of the mold. All the specimens were kept in molds for 24 h at room temperature, and then removed from molds. The specimens were left to dry in ambient conditions for 28 days, further dried in an oven maintained at 40 °C for 48 h. Dry unit weight values of the samples were measured after drying steps. Then porosity and water absorption values, mechanical properties like compressive and bending strengths and thermal conductivity were determined (Table 5).

Compressive and bending strengths, of the gypsum composite samples were determined according to TS EN 13279-2 [24]. The dry unit weight, porosity and water absorption values were determined by Archimedes method (soaking period of 24 h in water at room temperature). In order to determine the thermal conductivity of the samples a TCI Thermal Conductivity Analyzer has been used. This instrument has been developed by C-Therm Technologies and it is based on the Modified Transient Plane Source (MTPS) method, a non-destructive technique allowing us to obtain the thermal conductivity and effusivity of the samples tested [25,26].

## 3. Results and discussion

### 3.1. Unit weight

The unit weights of produced gypsum composites were presented in Fig. 4. As seen from Fig. 4, the unit weight values of the composites varied between 1007.3 and 881.5 kg/m<sup>3</sup> by depending on composition. The highest value was observed in reference gypsum mixture (A1) as 1007.3 kg/m<sup>3</sup>. The lowest value belongs to the mixture C3 including 20% diatomite and 1% fiber. As clearly understood from Fig. 4, unit weight decreases in the increment of diatomite and fiber concentration in the mixture. The effect of diatomite is dominant than that of fibers. The effect of fibers is only around 1%. The reason behind decreasing unit weight is that diatomite has a lower density due to its porous structure and its concentration in the mixture. When diatomite and fibers are used in the ratio of 20% and 1%, respectively, unit weight decreases 12.5%. The relation between unit weight and porosity is presented in Fig. 5.

The ANOVA tests also were done for the unit weight data based on the quadratic model. The design referred to quadratic model with R-squared of 0.9983 and standard deviation of 2.94. The ANOVA test results for the dry unit weight data are given in Table 6. The sum of squares is used as a measure of overall variability in the data. Mean square values are obtained by dividing the sum of squares by the degrees of freedom. The model F-value of 357.07 implies the model is significant. There is only a 0.02% chance that a Model F-Value this large could occur due to noise. Values of

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