Journal of Metals, Materials and Minerals, Vol. 33, No. 1, pp. 116-123, 2023

Thermal and mechanical evaluation of natural fibers reinforced gypsum plaster composite

Tugba MUTUK[1,*], Kerem ARPACIOĞLU[1], Sevim ALIŞIR[1], and Gökhan DEMiR[2]

[1] *Department of Metallurgy and Materials Engineering, Ondokuz Mayıs University, 55200, Samsun, Turkey*
[2] *Department of Civil Engineering, Ondokuz Mayıs University, 55200, Samsun, Turkey*

***Corresponding author e-mail:** tugba.isitan@omu.edu.tr

Received date:
1 October 2022
Revised date
2 March 2023
Accepted date:
3 March 2023

Keywords:
Natural fibers;
Gypsum;
Thermal conductivity;
Microstructure;
Compressive strength

Abstract

Many problems such as the increase in the world population, global drought, and greenhouse gases have caused materials used in industries to be reconsidered. This is how the concept of green composite material emerged. Using natural fibers to obtain such materials is important in terms of sustainability. This study is aimed to use hemp fiber and banana fiber as natural fiber additives in the gypsum composite to produce green bio-composite. Natural fibers are chemically modified with 5% NaOH solution. In this way, a good fiber/matrix interface interaction is provided. The composite hardness test result of 5 wt% HB fiber added sample is obtained as 50.4. Compared to the control plaster sample test result, which is 53.6, a slight decrease can be seen. However, it is observed that the fibers held the structure together and sopped the crack propagation. The increase in the porous structure with fiber addition caused a decrease in the thermal conductivity of the composite. Comparing thermal conductivity result of 5 wt% HB fiber reinforced gypsum composite (0.131 $W{\cdot}mK^{-1}$) with respect to pure gypsum result (0.237 $W{\cdot}mK^{-1}$), it gave a promising result as an insulation material.

1. Introduction

Calcium sulphate dihydrate ($CaSO_4.2H_2O$), which is called gypsum, is an economical building material used for many applications since ancient times. Gypsum can be also used in many systems such as drywall for interior building systems and decoration. It has low tensile strength and brittleness, as well as being low cost, easy shaping and lightness make gypsum favorable. Plasterboard walls and ceilings are widely used in buildings due to their many superior properties [1-3]. However, the use of such internal structural elements is limited in cases of damage, fire and insulation. To overcome such limitations and strengthen the structure, gypsum can be reinforced with fibers such as glass or a different kind of polymer fiber, etc. This kind of fiber addition especially stops crack propagation and improves mechanical properties [4-8]. Synthetic fibers (e.g. glass, carbon, etc.) are generally preferred as additive materials, but nowadays utilization of natural fibers is a major important topic in terms of sustainability and environmental preservation [9].

Energy consumption and environmental pollution in the construction industry are increasing due to the growing population. Therefore, the tendency for eco-friendly construction materials is increased [10]. Natural fiber can be used as an additive material in construction materials like cement, concrete [11], gypsum plaster [12], and lime [13]. Eco-friendly materials play an important role in the market of building materials. Among the natural fibers used, there are varieties such as hemp, palm and straw fibers [14,15], and short cellulose, etc [16-19]. Especially when used hemp fiber as a green additive material, it showed good performance in mechanical, thermal and physical properties [20,21]. The study of Charai *et al.* [22] showed that when hemp fibers were used in gypsum-based composites, an increase in the composites' bending behavior and thermal insulation was observed. Fabio *et al.* [23] examined the effects of different modified hemp fibers on composite impact strength. The results were promising for green composite production.

Another alternative that is used in this work is banana fiber since these fibers in the green composite have good interface interaction between natural fiber and matrix. Moreover, low density and low cost are important features of using these fibers [24-26]. Banana fiber is obtained by separating the stem after the separation of fruits and leaves. It is a waste and using it in the composite as recycling is important both in terms of sustainability and waste evaluation. In studies related to this, Akinyemi *et al.* [27] studied banana fiber and wood-bottom-modified cement mortars. This fiber was used as an additive and showed good results in tensile strength, especially in the microstructure of cement mortar. Other studies [28,29] similarly showed an improved compressive, flexural, or tensile strength of the cement and concrete composites. Moreover, it improved its microstructure treatment due to better bonding between the fibers and matrix.

This study aims to produce green bio-composite by adding such natural fibers to gypsum composite to investigate the effect of banana and hemp fibers on the mechanical, microstructural and thermal properties of gypsum plaster-based composite. Nowadays, synthetic fibers are used in construction materials. Furthermore using natural fiber as an additive material enables low cost, low density, low thermal

conductibility and sustainability making them a good potential using a replacement for synthetic fibers in composite materials. The suitability of such bio-based fibers is being investigated and in light of the results obtained in this study, natural fibers can be used to replace synthetic fibers in the construction industry. Chemical treatment processes were applied to all fibers. After this modification process, hemp and banana fibers were analyzed by Fourier Transform Infrared Spectroscopy (FT-IR). Hemp and banana fibers were reinforced with gypsum plaster. Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDX), Thermal conductivity, hardness and compressive strength tests were used to examine the composites.

2. Experimental study

2.1 Materials

In this study, gypsum plaster maintained by BMT company from Sivas, Turkey was used under TS EN 13279-1 standard [30]. Banana fibers were purchased from a local company and hemp fibers were produced by the Hemp Research Institute of Ondokuz Mayıs University. Since fibers are seen as quite untidy, hemp fiber and banana fiber were cut nearly the size of 10 mm to 15 mm (Figure 1).

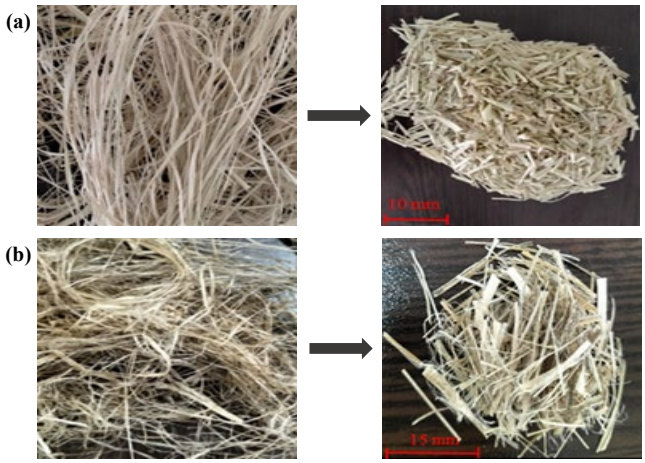

Figure 1. Fibers cutted to specific sizes (a) hemp fibers, and (b) banana fibers.

2.2 Pre-treatment of hemp and banana fibers

Natural fibers are cellulose-based materials by their nature, structures and essential components of these fibers are cellulose, hemicellulose and lignin [9,31,32]. In order to use natural fibers as additives in the composite, they should be subjected to a pre-treatment process. This is because impurities such as pectin, lignin, wax and high amounts of hydroxyl groups in the fibers prevent the bonding of these fibers to the matrix, weaken the interface and adversely affect the mechanical properties of the composite [9,33-35]. With the alkali treatment, some of these substances on the surface of the cellulose fibers are removed and a large number of open cellulose ends that can interact with the matrix are formed on the fiber surface. The alkali treatment increases the free energy of the fiber on the surface. At the same time, it improves the mechanical bonding at the fiber/matrix interface by making the fiber surface rough [36-38]. Sodium hydroxide (NaOH) is the most commonly used surface treatment for natural fiber to enable bond between fiber and gypsum [39,40]. In this study, fibers were modified with NaOH solution (5%w/w) for three hours. They were then washed with distilled water. Following this step, fibers were dried in the oven for 8 h at 90°C. After modification, hemp and banana fibers were analyzed by FT-IR. The FTIR spectra were recorded in the range of 400 cm^{-1} to 4000 cm^{-1}. The peak at 1237 cm^{-1} in the untreated hemp fiber was also completely removed in the chemical treated (Figure 2(a)). This peak represents the C=O stretching of the acetyl group of lignin [41]. After chemical treatment, the peak at 1629 cm^{-1} which belongs to the carbonyl group on hemicelluloses is reduced in the untreated hemp fiber. This reduction illustrates that hemicelluloses partially remove from the fiber surface [35]. The band at 1733 cm^{-1} represents the C=O stretching in untreated hemp fiber removed after NaOH treatment [42]. When these structures were separated from the hemp fiber surface, a rougher and better bonding surface was obtained.

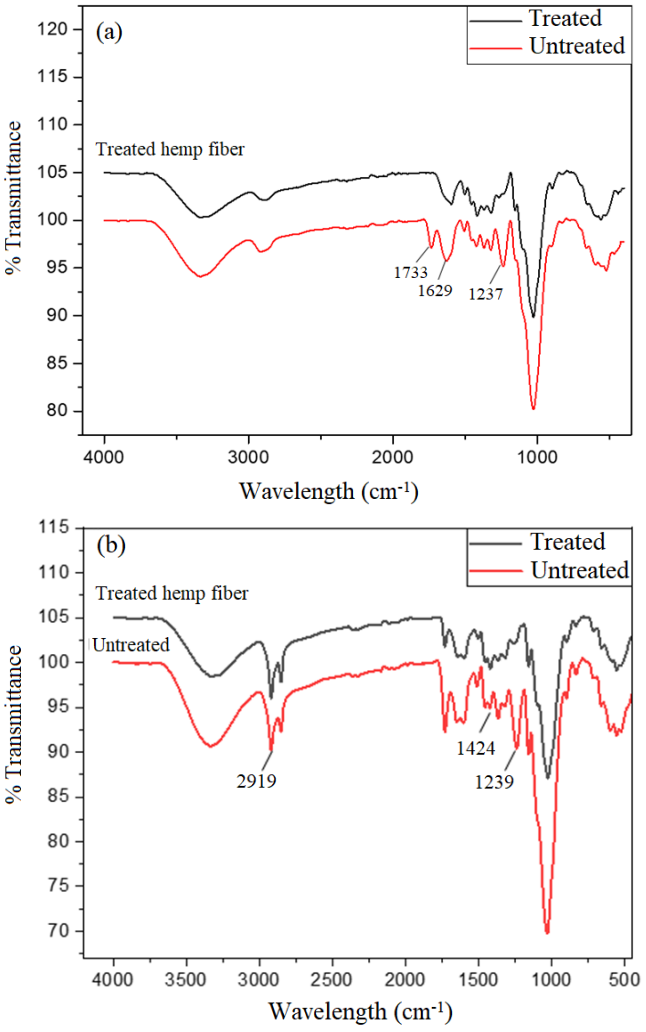

Figure 2. FT-IR spectra of the untreated and treated fibers (a) hemp fiber (b) banana fiber.

In Figure 2(b), the peak at 2919 cm^{-1} represents the C-H stretching from the $–CH_2$ group of cellulose in the untreated banana fiber [43]. The band at 1239 cm^{-1} belongs to hemicellulose and lignin after 5% NaOH chemical treatment, which is a characteristic peak that is removed as clearly seen in Figure 2(b) [44]. The peak at 1424 cm^{-1} shows that the C-H bond of lignin in untreated fiber is reduced in the treated banana

fiber [45]. The decrease or absence of these characteristic peaks indicates removing of lignin and wax content on the banana fiber surface.

SEM evaluated the microstructure of the treated and untreated hemp and banana fibers. SEM micrographs are given in Figure 3. While untreated fiber surfaces are smooth (Figure 3(a-c)), chemical treatment surfaces are seen as rough in Figure 3(b-d). After the NaOH-modified process, obtaining rough surface enabled good interface between fiber-matrix.

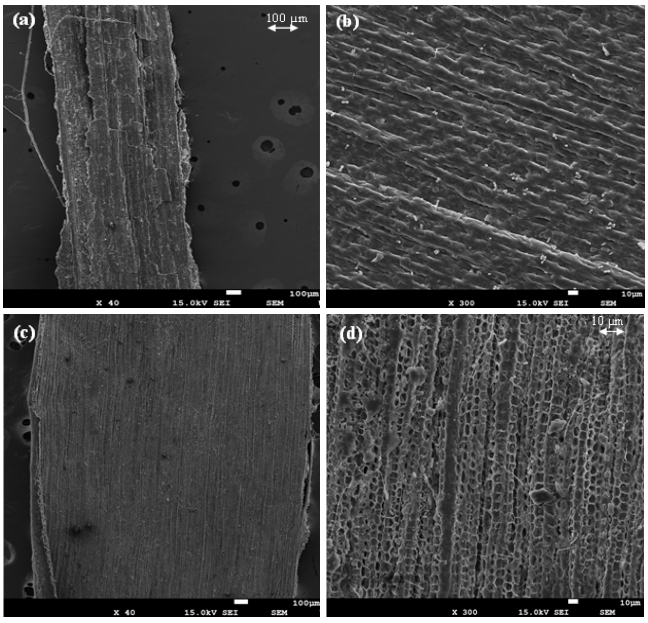

Figure 3. SEM micrograph of untreated (a-c) hemp fibers, banana fibers (b-d) chemical treated of hemp fibers, banana fibers.

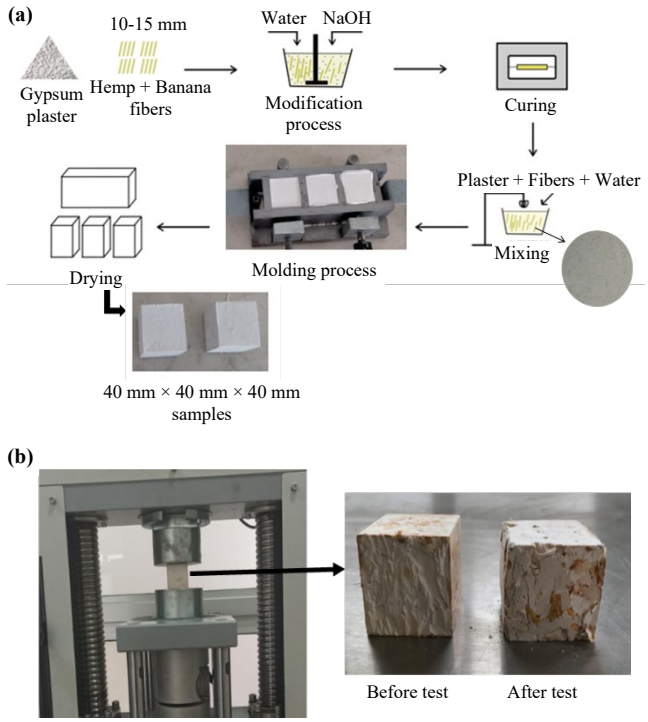

Figure 4. (a) Schematic diagram of fiber-gypsum plaster composite preparing process (b) 40 mm × 40 mm × 40 mm composite mold compressive strength unit and samples before and after test.

2.3 Preparation of fiber-gypsum composites

The water/binder ratio was determined as 0.6 according to TS EN 13279-1 [30] and all fiber-gypsum composites were prepared as seen in Figure 4(a). In the first step in order to ensure that the fibers are spread evenly in the mortar, the cutting process of the fibers was carried out. Therefore, hemp and banana fibers (HB fibers) were cut from 10 mm to 15 mm and modified with NaOH solution. After the chemical treatment process, HB fibers were added to a water/gypsum mixture with different ratios (1, 3 and 5 wt%). The mixing process continued until a homogeneous mixture was obtained with the mortar mixing device. After this step, a mortar was molded with different sizes for different analyzes. The casting samples were cured in the oven at 40°C for 48 h. Mechanical treatment was analyzed by using a compressive strength test cubes of 40 mm × 40 mm × 40 mm mold size and 40 mm × 40 mm × 160 mm of them were used for thermal conductivity measurement tests.

2.4 Mechanical testing method

The sizes of the compressive test specimens were 40 mm × 40 mm × 40 mm. After all the molds were cleaned and oiled properly, compressive tests were conducted with a compressive strength test manufactured by 5982-100 kN tension-compression test unit. The compressive strength of a material is expressed as the ratio of the load applied to the surface area of the material exposed to pressure. The data obtained from the digital display after the experiment are calculated with the following Equation (1) [46]:

$$\sigma_c = \frac{F}{A} \tag{1}$$

where σ_c is compressive strength, F is force and A is the cross-sectional area.

The Shore-D test unit is used for hardness tests of rigid materials. In this study, the sizes of the hardness test specimens were taken as 40 mm × 40 mm × 160 mm. Manual digital hardness tester equipment was used for the hardness test and the hardness values of the samples were determined by applying a constant load to the surface at four different points.

3. Results and discussion

3.1 Mechanical characterization of composites

Figure 5(a-b) shows the effects of the composite material produced from HB fibers on the compressive strength. According to the graph, the increase in the ratio of fibers used in the mixture caused the compressive strength of the gypsum composite to decrease compared to the reference sample. As a consequence of this increase in the fiber ratio, the material strength was adversely affected. If we compare the ultimate compressive strength result of 1 wt% HB (HB 1) fiber added composite (6.8 MPa) with the result of the control gypsum plaster sample (10.9 MPa), it can be observed that the compressive strength results decrease as the HB fiber additive ratio (1, 3, and 5 wt%) increase in the composite. This can be explained as follows: as the

fiber additive increase, so do porosity of the composite. This caused a reduction in the compressive strength of the composite which is in line with the previous studies. Compressive strength results of other studies about hemp fiber-reinforced gypsum plaster composite change from 5 MPa to 10 MPa which agree with previous results same as our study [12,47-49]. However, the fiber-reinforced composite did not break into pieces during the test as it was compressed. Since the fiber interface interaction is good, there is not much fragmentation in the composite structure under deformation. Figure 4(b) shows that the fibers held the structure together. In addition, this situation created a stopping effect of crack propagation.

As for the hardness test results, the composite hardness test result of 5 wt% HB fiber added sample is obtained as 50.4. Compared to the control plaster sample test result, which is 53.6, a slight decrease can be seen (Figure 5(c)). This porous structure caused a decrease in mechanical strength. The lowest hardness value given in the TS EN 13279-1 [30] standard for plaster is 10 (shore hardness value). It is seen that all gypsum composite samples provide the desired hardness value and such a decrease is also observed in some studies [50,51].

3.2 Thermal conductivity test of plaster composites

The thermal conductivity of HB fiber-gypsum plaster composites was measured according to the procedure described in TS EN 13279-2 [52]. The sizes of the thermal conductivity test specimens were 40 mm × 40 mm × 160 mm and this analysis was made by using KD2 PRO thermal conductivity device. Thermal properties give important information about soil, lime, cementitious, gypsum, or other porous materials. Thermal conductivity is the ability of a material to transfer heat. Thermal property measurements are needed in various industries and research fields. The heat transfer coefficient of materials maintains information about the heat conduction of the related material. The heat conductivity coefficient is especially important for construction materials. In this study, in the thermal conductivity measuring device, where the transient line heat source method is used, a KS-1 coded needle immersed in the desired fluid is used to make the measurement (Figure 6(a)). The needle is given instantaneous power for a certain period of time and then this tip is cooled in the fluid for a certain period of time. The heat transfer coefficient is calculated by using Equation (2) with the temperature/time change that the device shows instantly and the calculated heat transfer coefficient is read from the device screen [53].

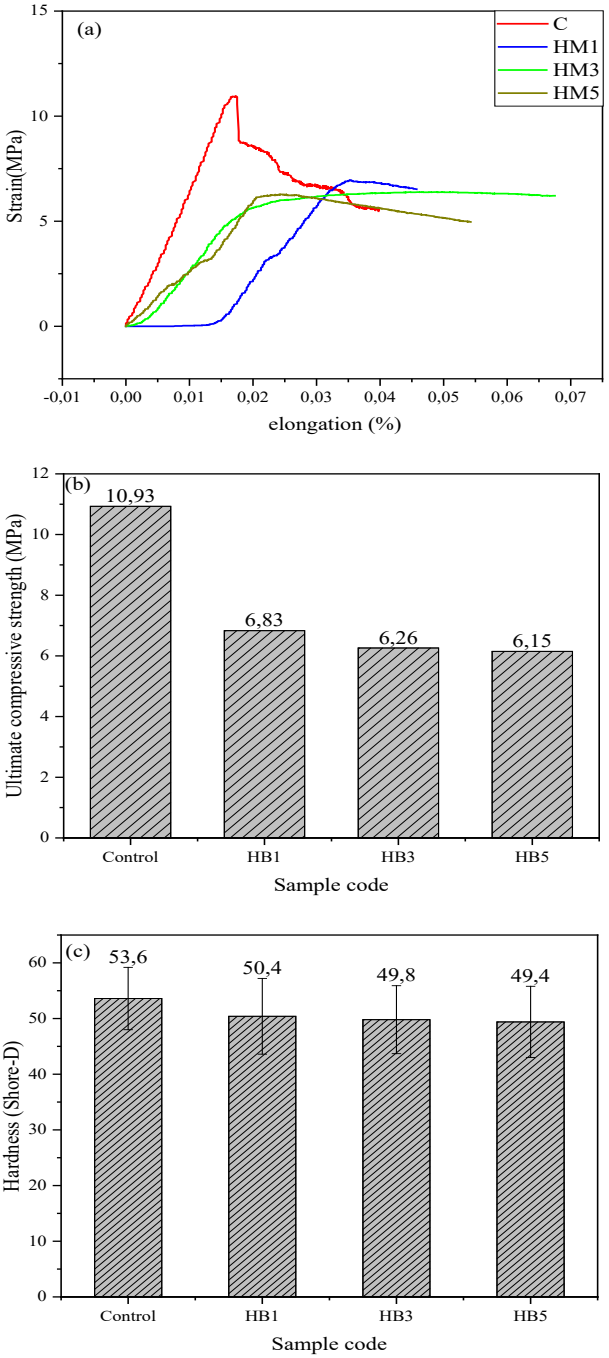

Figure 5. Test results of composites (a) stress-elongation graph (b) ultimate compressive strength result (c) hardness (Shore-D) test results of composites.

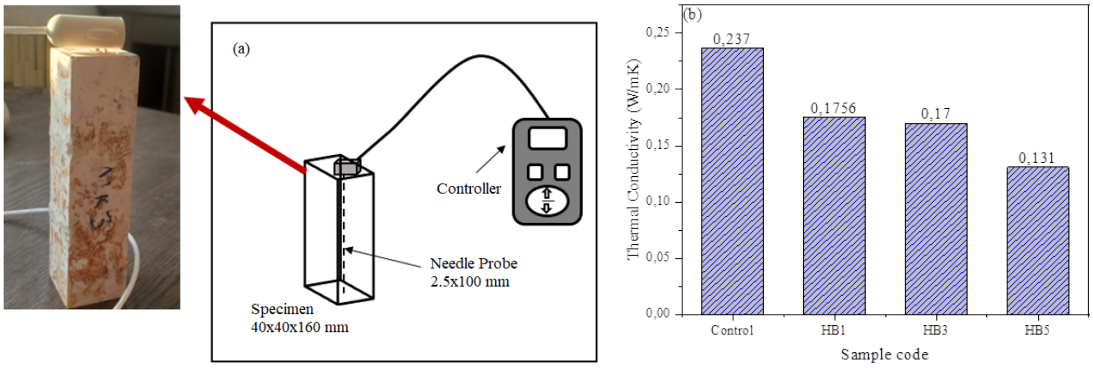

Figure 6. Thermal conductivity (a) test unit (b) test results.

$$k = \frac{q(lnt_2 - lnt_1)}{4\pi(\Delta T_2 - \Delta T_1)} \quad (2)$$

where q is the constant heat flux value applied from the source, ΔT_1 and ΔT_2 are the varying temperature differences, t_1 and t_2 are the time difference, and k is the heat transfer coefficient ($W \cdot mK^{-1}$).

HB 5 wt% fiber additive composite thermal conductivity test result is 0.131 $W \cdot mK^{-1}$, which is lower than the control specimen of plaster value, 0.237 $W \cdot mK^{-1}$ (Figure 6(b)). This is due to low conductivity values of hemp fibers (~0.05 $W \cdot mK^{-1}$) and banana fibers (~0.06 $W \cdot mK^{-1}$) [54]. These kinds of natural fibers decreased the plaster composite thermal conductivity which was observed by the studies in the literature. This shows that it can be used as a thermal insulator in the building to reduce building energy consumption [55,56]. In addition, the porous structure worked in thermal conductivity. Heat can be trapped in the porous structure and the thermal conductivity value decreases in this way. It was aimed to produce gypsum plasterboard composite with natural fibers additive to use inside or outside the construction for thermal insulation. Since the thermal conductivity has a low value (0.131 $W \cdot mK^{-1}$), it seems to be promising for the insulation material.

3.3 Flame test of composites

The flame test analyses were done with the TS EN ISO 11925-2 standard [57]. In the vertical position, the burner flame is lit and the flame is stabilized. The burner fire is fed by 30% propane and 70% butane gas. Then, it is adjusted to give a 16 mm to 20 mm high flame. The burner is tilted 45^0 relatives to the vertical axis and the flame is advanced horizontally until it reaches the preset contact point with the test sample. Flame is applied for 15 s from the moment it contacts the test sample (Figure 7(a)). The temperature was reached on the composite surface from room temperature to 700°C to 800°C. This range of temperature was measured on the exposed surface by means of a thermocouple located in the action center. Furthermore, according to the standard, the length of the effect on the area subjected to fire should not exceed 150 mm on the composite surface. Therefore, after the test was checked whether the flame tip reaches 150 mm above the application point of the flame and the duration of this. Actually, gypsum is A1 class non-combustible material. However, in this study, when 1, 3, and 5 wt% HB fibers were added to the gypsum composite, their behavior against fire had been observed and analyzed. As seen in Figure 7(b), the reaction of the material when exposed to heat is within the standards and none of them could not reach 150 mm. These test results suggest that if natural fiber composite has fire resistance, it shows that this kind of naturally-added material is a promising building material that sheds light on further studies.

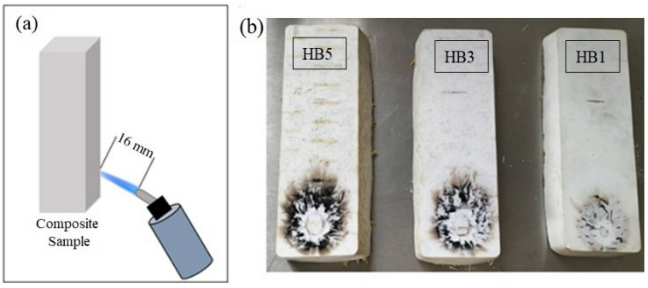

Figure 7. (a) Flame test set-up (b) Flame test results of HB-gypsum composite.

3.4 Microstructural analyzes

All composite microstructures are analyzed with SEM-EDX. Small and large pore structures can be seen in Figure 8(a). Mainly, pore structures were observed around the fiber structure in Figure 8(b-c). Because HB fiber additives increased pores in the structure, these pores enable low thermal conductivity results. It provides thermal insulation by trapping the heat in these pores. It was found that these small and large pores structures are responsible for low compressive strength results, which is supported by previous studies [27,58]. The fiber surface was obtained more roughly after the chemical treatment process. Therefore, this situation enables better interface interaction between fiber-matrix as shown in Figure 8(d-e). Gypsum-HB fiber composite microstructure was analyzed by SEM-EDX elemental mapping. EDX mapping analyzes are seen in Figure 9(c-g) in which different elements rich regions with different colors. The fiber SEM image is shown clearly in Figure 9(a-b) that the fiber is embedded in the gypsum plaster. This image also shows how the fibers bond with the matrix well. This is evidenced supported by elemental mapping images which is the fiber is clearly visible with elemental mapping sodium (Na), Oxygen (O) and Carbon (C) rich region. In addition, the presence of fiber in that region is seen with the black color in other elements.

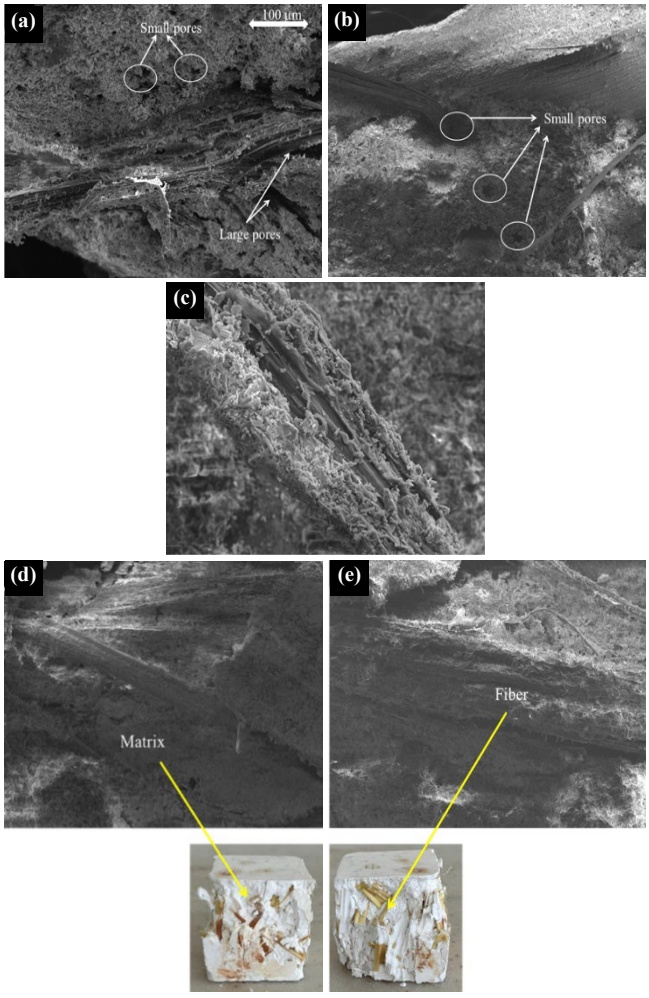

Figure 8. SEM image of (a-b) plaster-HB5 (c) HB3 (d-e) plaster-HB1 fiber composite.

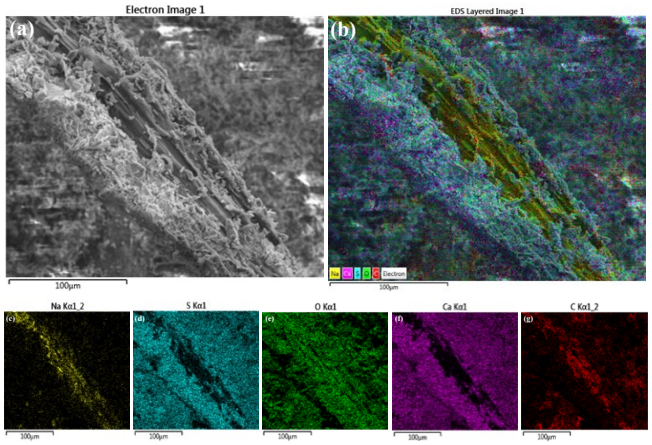

Figure 9. SEM-EDX image of HB3 fiber – gypsum composites (a-g).

4. Conclusion

In this study, we investigated the effect of natural (hemp and banana) fibers on the mechanical, thermal conductivity and micro-structure of gypsum-based composite. Thermal conductivity analyses were performed to examine whether the fibers in the gypsum plaster composite could provide thermal insulation. The aim of the compressive strength test is to observe the result of the increase of the porous structure with the effect of the fiber additive. These analyzes are important for the use of natural fibers in the construction industry as green composites in terms of sustainability.

Chemical treatment was applied to the fibers with a 5% NaOH solution. Better fiber/matrix interface interaction was observed in the microstructure after chemical treatment. Comparing thermal conductivity result of 5 wt% HB fiber reinforced gypsum composite (0.131 $W{\cdot}mK^{-1}$) with pure gypsum result (0.237 $W{\cdot}mK^{-1}$) it gave a promising result as an insulation material. Furthermore, gypsum is A1 class non-combustible material. However, it is aimed to investigate the combustion behavior of gypsum composite as HB fiber is added at 1, 3 and 5 wt% various ratios. When examined according to the TS EN standards, no sample reaching 150 mm in 15 s was observed in the exposed flame zone. This is a promising result for the use of natural fibers in building materials.

As the fiber additive increased, the porous structures formed in the composite material gave good results in thermal conductivity and combustion tests. However, decreases were observed in mechanical test results such as hardness test (Shore-D-surface hardness test) and compressive strength test. Moreover, while the hardness test is 50.4 in the 5 wt% HB fiber added sample, it is 53.6 in the pure gypsum sample. There was not much decrease in strength. In addition, while the expected lowest pure gypsum hardness is 10 which is acceptable in the TS EN standard. The mechanical results in this study are preliminary and promising for other studies.

Natural fibers are both sustainable and recyclable. In line with the positive results and comments obtained in this study, it may be a guide for natural fibers to replace synthetic fibers in building materials that can be used in composite structures. Thus, in light of these results, other results can lead to green composite production using natural fibers such as hemp and banana fibers and to produce recyclable environmentally friendly construction and insulation materials.

Acknowledgements

The authors pleased to acknowledge the financial support for this study from Ondokuz Mayıs University, Scientific Research Project Department under the grants (PYO.MUH.1908.21.002).

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