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RESEARCH ARTICLE-CIVIL ENGINEERING

Change in Microstructure, Mechanical Strength, Fire Resistance, and Radiation Attenuation Properties of Gypsum Plaster with Boric Acid

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Abstract
In this study, the effect of boric acid addition on the microstructure of gypsum plaster was investigated to determine an environmentally friendly gypsum additive that may enhance mechanical strength, fire resistance, and X-ray radiation attenuation properties. The mechanical strengths of bare gypsum and boric acid-added (0–0.5% by weight with respect to gypsum amount) gypsum plasters were evaluated in terms of compressive and bending strengths. The effects of the different addition procedures of boric acid (0.1% by wt.) on the fire resistance of the gypsum plasterboard were also evaluated. X-ray radiation attenuation properties of boric acid-added (0.1% by wt.) gypsum plasterboard were investigated as well. XRD, ATR-FTIR, Helium pycnometer, N_2 adsorption–desorption, and SEM analysis were performed to determine the microstructural properties of gypsum plaster. XRD and ATR-FTIR analysis revealed that boric acid did not change the calcium sulfate dihydrate structure of gypsum plaster. Whereas, Helium pycnometer, N_2 adsorption–desorption, and SEM analysis showed that the physical properties of gypsum changed with an increase in pore volume, skeletal density, and particle size after boric acid addition. The increase in the pore volume and particle size decreased the mechanical strength of gypsum. However, boric acid addition on the gypsum plaster plate, especially using the spraying method, enhanced the fire resistance of gypsum. Additionally, boric acid slightly enhanced the X-ray radiation attenuation properties (0.7%) of the gypsum plasterboard.

Keywords Boric acid · Gypsum plaster additive · Microstructure · Mechanical strength · Fire resistance · X-ray radiation attenuation

1 Introduction

Gypsum plaster is one of the most widely used construction materials because it is energy efficient, healthy, environmentally friendly, and fire resistant, as well as being a low-cost building material [1]. Depending on the application, high-strength gypsum plaster may be required in the building industry. Fibrous materials are added to the structure of gypsum to increase its characteristics. Various gypsum additives (such as nano-structured cellulose fibers, retama monosperma, sand, rice husk, silica gel, sodium sulfate, calcium carbonate, and sodium tripolyphosphate) have been used as gypsum additives until now [2–7]. In industry, fiberglass is the most often used additive material. The addition of fiberglass to the plaster increases the viscosity of the plaster. Fiberglass is a costly reinforcing material that is also non-biodegradable [3].

Because of these characteristics, an ecologically friendly addition capable of improving the mechanical properties of gypsum plaster should be identified. Even though fiberglass is commonly used in industrial applications, the literature has studies on the use of various gypsum additives such as sand, rice husk, silica gel, sodium sulfate, calcium carbonate, and sodium tripolyphosphate. It is known that the additives used directly affect the mechanical properties of gypsum [4–7]. According to Moghadam and Mirzaei, sodium

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sulfate additions up to 0.4 wt% improved the compression and bending strengths of the gypsum [4]. On the other hand, Serna et al. investigated the effects of adding rubber bits made from recycled tires to gypsum plaster on its mechanical strength. According to the information they obtained from their research, it was determined that the compressive strength of gypsum plaster decreased by 18.3% and the bending strength by 16% [8]. In another study, carbon-based graphene powder addition enhanced the flexural strength of the gypsum plaster [9]. Doğan et al. investigated the effect of graphene-based additives on the mechanical strength and microstructure of gypsum plaster. They observed that the incorporation of graphene oxide (GO) into the structure of gypsum plaster reduced its mechanical strength. In addition, they observed that the reduced graphene oxide (rGO) additive improved both flexural and compressive strength, and boron-added rGO boosted the compressive strength while decreasing the flexural strength of gypsum plaster [10].

Boron reserves are utilized more commonly in glass, detergent, metallurgy, and agriculture after being processed into boron compounds and unique boron products. They are also used in textiles, health, porcelain/ceramics, reflective requirements, fire retardant materials and additives, and nuclear energy [11]. In particular, some studies investigated the effect of boron addition on the mechanical strength of cement in the literature. Davraz observed that while boron trioxide (B_2O_3) had a negative effect on the flexural strength of the cementitious composite, it enhanced the compressive strength of samples [12]. Palta et al. found that adding 1.0% boric acid raised the compressive strength of self-compacting concrete, but a higher amount of boric acid addition resulted in a decrease in compressive strength. They also reported that bending strength was reduced as a consequence of the addition of boric acid [13]. Ozdemir et al. evaluated boron-containing clay wastes as cement additives, and they observed that the waste decreased the mechanical strength of the cement [14]. Boric acid addition resulted in a pronounced decrease in the compressive strength of glass ionomer cement, according to research by Prentice et al. [15]. Rai et al. found that the compressive strength of cement reduced as the boric acid ratio increased. They concluded that boric acid not only caused a delay in cement hydration but also increased the initial and final setting times [16]. Zayed et al. observed that the compressive and tensile strengths of concrete were adversely affected by boric acid [17]. In our previous study, the effect of boron-introduced reduced graphene oxide on the mechanical strength of gypsum plaster was investigated. The result showed that the boron-containing sample improved compressive strength while decreasing flexural strength [10]. Unfortunately, no research has been conducted to investigate the effects of boric acid addition on the microstructure of gypsum.

The gypsum plasters have a porous structure, which reduces the density and thermal conductivity value of the gypsum. When the fire was exposed to gypsum plaster, the free water in the porous and crystal water in gypsum plaster are gradually driven off at temperatures above 100 °C. Between the flame and the plaster, a vapor layer is formed by the water that decomposes and evaporates. [18, 19]. On the other hand, as discussed before, boron was also considered a fire retardant/preventing material as well. Using the different boron compounds, the flame-retardant property of polyester composite and the cotton fabric was improved [20, 21].

Protecting people from radiation is a critical issue in nuclear power plants, medical facilities, and dental clinics. Lead is one of the most well-known and common traditional radiation shielding materials [22]. In the study of Alsaif et al. the effectiveness of silicon dioxide, calcium oxide, phosphorus pentoxide and boron trioxide (SiO_2–CaO–P_2O_5–B_2O_3) bioactive glass systems containing different amounts of boron trioxide (B_2O_3) in protection against gamma rays was evaluated. As a result, the selected bioactive glasses were evaluated to have the potential to be used in protective masks for diagnostic radiation, as they offer good radiation protection properties in the tested energy range ($15 < E < 100$ keV) [23]. Nagaraja et al. investigated the X-ray, gamma, and neutron shielding properties in boron polymers. As a result, phenylethenylboronic acid was reported to be a good absorber of X-ray, gamma radiation, and neutron. According to the study of Orak and Baysoy [25], the addition of boron increases the effectiveness of neutron shielding in concrete [24].

However, building materials such as gypsum [26] and concrete [17, 27, 28] were used to shield X-rays, gamma-rays, and neutrons in the literature. Mann et al. investigated gamma-ray shielding properties of some building materials such as soil, gypsum, and limestone. They obtained that gypsum is a good shielding material at deep penetration in the energy region of 3–15 MeV [26]. Alabsy et al. observed that nano-lead oxide (PbO) incorporation improved the gamma-radiation-shielding ability of Gypsum–Lime–Waste Marble Mortars [22]. Hernandez-Murillo et al. investigated X-ray and gamma-ray shielding of concrete and gypsum. They concluded that concrete blocks and Portland concrete have better features than gypsum with respect to photon attenuation coefficients [27]. Sarkawi et al. [29] reported that due to the poor absorption of thermal neutrons in ordinary concrete, an additional compound was mixed into the original concrete to increase the neutron capture cross section of the concrete. In their study, they used ferro-boron compound to improve the radiation shielding properties of concrete. Zayed et al. observed that while boric acid addition enhanced the thermal and fast neutrons attenuation properties of serpentine concrete samples, the γ-rays attenuation properties

decreased significantly [17]. The effects of adding three different commercial boron compounds, boric acid, boric frit and borax, to two concrete materials consisting of carbonate and hematite aggregates, on their radiation shielding properties were investigated by Kharita et al. [28]. The results show that the addition of boric acid (H_3BO_3) and boric frit at 0.5–1% by weight has a detrimental effect on the setting of cement. It was evaluated that the addition of borax ($Na_2B_4O_7$) up to 1% by weight did not have a significant effect on the strength of concrete, but had significant effects on the shielding efficiency in 100-cm-thick concrete shields, as it reduced the trapping of gamma rays by up to 80% better than concrete without borate.

In the present study, the effect of boric acid addition on the microstructure, mechanical strength, fire resistance, and radiation attenuation properties of gypsum plaster was investigated. Mechanical tests (compressive and bending strength), fire resistance tests, and radiation attenuation tests were applied to the bare gypsum plaster and boric acid-added gypsum plasters.

2 Experimental

2.1 Preparation of Gypsum Plaster Materials and Mechanical Tests

In this study, the desired amount of boric acid (H_3BO_3, 99.8%) was dissolved in 720 ml of deionized water. Then, the boric acid (0–0.5% by weight with respect to gypsum amount) dissolved water solution and 1 kg of gypsum were stirred in a mortar using a mechanical mixer at a mixing speed of 215 rpm for 1 min. Gypsum plaster was produced from ABS Gypsum from Turkiye. The prepared gypsum mixture was poured into a mold (4 × 4 × 20 cm) after stirring. After the first freezing of the plasters, the excess amount of gypsum was removed with a spatula. After it had fully frozen, air curing was applied for 7 days at 40 °C in an oven. The bare gypsum sample was labeled as GP. In addition, boric acid-added gypsum samples were labeled as their boric acid content. For instance, gypsum plaster prepared using 0.1% weight boric acid based on the amount of gypsum is labeled as GP-0.1B. Finally, the mechanical tests (compressive and bending strength) were performed following the TS EN 13279-2 standard using three different samples.

2.2 Fire Resistance Test of Gypsum Plasterboard

The same synthesis procedure described above was used for GP and GP-0.1B samples. However, the dimension of the mold used in the synthesis of the gypsum plaster plate was 300 × 75 × 10 mm. Furthermore, to observe the effect of the boric acid-adding procedure on the fire resistance of gypsum, boric acid (0.1% by wt) was dissolved in the required amount of water and the dissolved boric acid was sprayed on the two surfaces of the bare gypsum plaster plate at the end of the air curing step of the 7th day. Then the sample was dried at 40 °C for 3 days. The sample was labeled as GP-0.1B/S.

During the fire resistance test, a bending moment was applied to the gypsum plaster plate, which was positioned between two burners and heated by the burner flame. The test specimen was placed between two burners, the long side (300 mm) horizontal, and the short side (75 mm) vertical. The bottom long edge of the specimen and the lower rim of the burner should be parallel. The test specimen, which was 300 mm long and 75 mm wide, was placed in the sample holder from its one edge with the short side vertically. A 300 g mass was placed on the other side of the test specimen to apply the bending moment. The load was applied from a point 260 mm away from the specimen holder. A platform was placed under the loaded mass with a distance of 10 mm. The burner was ignited, and the gas flow was adjusted such that the temperature from each thermocouple (1000 ± 50) °C can be read. The time to gypsum plaster plate breakage was measured. Fire resistance tests of bare and boron gypsum samples were performed depending on the TS EN 12664 standard.

2.3 X-ray Radiation Attenuation Test of Gypsum Plasterboard

The same synthesis procedure described above was used for GP and GP-0.1B samples. However, the dimension of the mold used in the synthesis of the gypsum plasterboard was 110 × 150 mm rectangular shape with 13 mm thickness. The test was performed using narrow beam geometry described in Standard TS EN 61331-1:2014 [30]. 100 kV X-ray calibration system tube voltage value was used for the test. The attenuation ratio value was calculated according to TS EN 61331-1. The ratio of the decrease in the measured dose rate when the test object is inserted into the radiation field. The attenuation ratio value of gypsum materials was compared with a thickness of reference lead having the same attenuation ratio as well.

2.4 Characterization of the Samples

X-ray diffraction (XRD) analysis (Rigaku Ultima-IV diffractometer) using Cu K radiation (= 0.15406 nm, 40 kV, and 30 mA) with a scanning rate of 2°/min was performed to determine the crystallinity of gypsum plasters. The structural bonds and functional groups of the samples were determined using a Jasco FTIR/4700 spectrometer in the medium infrared region of 4000–400 cm^{-1} with a high resolution (4 cm^{-1}). Physical properties such as average pore

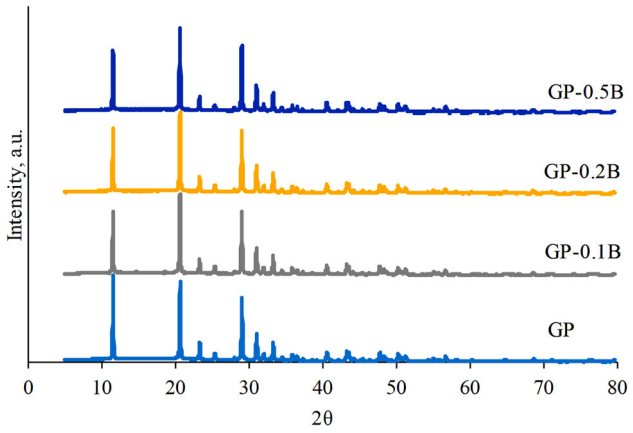


Fig. 1 XRD patterns of bare gypsum and boric acid-added gypsum

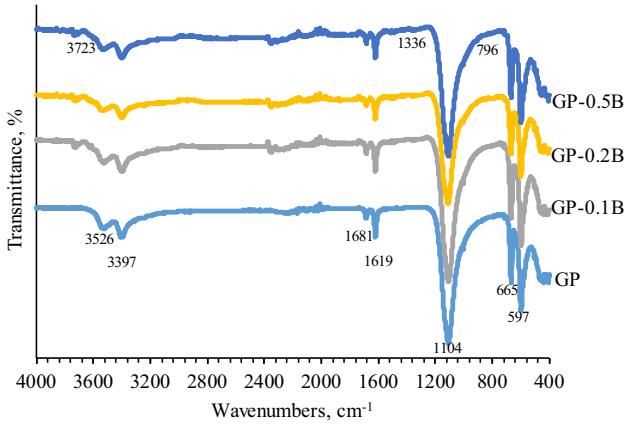


Fig. 2 ATR/FTIR spectra of the bare gypsum and boric acid-added gypsum

diameter and total pore volume were obtained using a Quantachrome Autosorb-1C device. The samples were degassed for 3 h at 120 °C and the N_2 adsorption–desorption isotherms were obtained at 77 K. The pore size distributions were determined using the Barrett–Joyner–Halenda (BJH) technique. Scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM–EDS) analyses of the materials' morphological structure and chemical composition were performed using a QUANTA 400 F FE-SEM, 30 kV device.

3 Results and Discussion

3.1 Characterization Results of Gypsum Plasters

In the study, the effect of boric acid addition on the microstructure of the gypsum plaster was investigated. To determine the crystallinity of the bare gypsum plaster and H_3BO_3-added gypsum plasters, XRD analysis was performed (Fig. 1). XRD patterns of GP samples represented five characteristic diffraction peaks at 2θ of 11.6°, 20.70°, 23.36°, 29.06°, 31.08°, and 33.34°, indicating the formation of that calcium sulfate dihydrate ($CaSO_4.2H_2O$) [31–34]. No additional new diffraction peak was observed for the H_3BO_3-added gypsum samples. The results indicated that gypsum plaster kept its calcium sulfate dehydrate structure after boric acid addition. However, the intensity of the diffraction peaks decreased from 1 to around 0.75 with respect to gypsum plaster after boric acid addition. The results indicated that boric acid addition decreased the crystallinity of gypsum plaster due to the increase in the curing time of the gypsum with boric acid addition. In our previous study, boron-added reduced graphene oxide decreased the crystallinity of bare gypsum plaster as well. However, boron-added reduced graphene oxide decreased the curing time of the gypsum and the reduced graphene oxide composite [10]. Davraz reported that boric acid addition increased of setting times of fresh mortars [12].

To determine functional groups and structural bonds of the bare gypsum and H_3BO_3-added gypsum, ATR-FTIR analysis was performed in the medium infrared region of 4000–400 cm^{-1}. Small vibrations between 2000 cm^{-1} and 2400 cm^{-1} were related to the characteristics of ATR measurement. The bands at 3526 and 3397 cm^{-1} were attributed to the stretching vibrations of the H_2O molecules in the gypsum samples, indicating the presence of hydration water in gypsum (Fig. 2) [35–37]. Similarly, the band at 1681 and 1619 cm^{-1} were attributed to the bending vibrational modes of water. The strong band at 1100 cm^{-1} and the bands at 665 and 598 cm^{-1} were assigned to sulfate groups SO_4^{2-} [38–40]. For the case of H_3BO_3-added gypsum samples, a new small band at 3722 cm^{-1}, which was attributed to H_2O molecules, was observed. Ceylan and Cicek observed the 3720 cm^{-1} confirms the OH group in FTIR analysis of the boron waste sample which includes large amounts of B_2O_3, CaO, and MgO in its structure [37]. Small bands at 796 and 1336 cm^{-1} were assigned to BO_3^{3-} groups. Albayrak observed the band at 783 cm^{-1} to the symmetric bending and the band at 1304 cm^{-1} to the antisymmetric stretching of BO_3^{3-} groups, respectively [41].

To determine the physical properties of the gypsum samples, a helium pycnometer and N_2 adsorption–desorption analysis were used. The structural properties of the samples were determined using N_2 adsorption–desorption analyses. Besides that, the skeletal densities of gypsum samples were determined using a helium pycnometer. Table 1 represents the total pore volume and skeletal density of gypsum as well as the average pore size. As seen in Table 1, mainly as the amount of H_3BO_3 incorporation increased, the total pore volume and skeletal density of the gypsum plaster increased. Furthermore, the average pore diameter increased as well.

Table 1 Physical properties of gypsum plasters

Sample	Average BJH adsorption pore diameter (nm)	Total pore volume (cc/g)	Skeletal density (g/cm^3)
GP	2.5	0.043	2.54
GP-0.1B	2.2	0.047	2.81
GP-0.2B	3.1	0.067	2.96
GP-0.5B	3.1	0.053	2.91

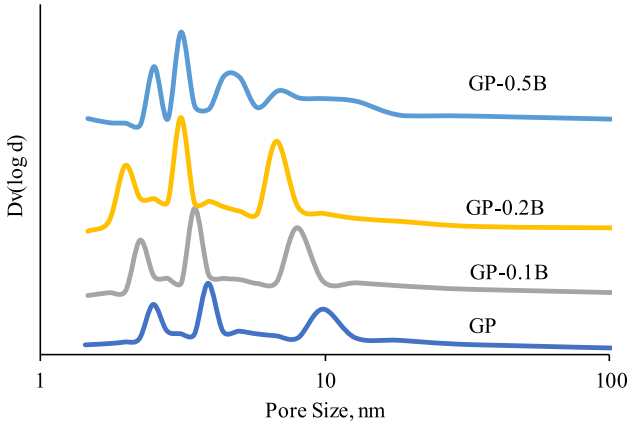


Fig. 3 Pore size distributions of bare gypsum plaster and H_3BO_3-added gypsum plaster

The pore size distribution of the gypsum plaster represented that gypsum plaster possesses different pore structures (Fig. 3). Boric acid addition did not change the pore size distribution much for the low concentration of boric acid. However, the higher amount of boric acid addition significantly changed the pore size distribution. Palta et al. reported that boric acid caused the porosity of the material to decrease, while Zayed et al. observed that boric acid had a negligible effect on the density of the serpentine concrete and the porosity increased [13, 17]. The effect of structural change with boric acid addition on the compressive and bending strength of gypsum plasters will be discussed further.

3.2 Mechanical Strength Results of Gypsum Materials

In the present study, the effect of boric acid addition on the mechanical strength of gypsum plaster was investigated. The bending and compressive strength of the gypsum plaster and different amount of H_3BO_3-added gypsum plaster were given in Fig. 4. As seen in Fig. 4., the highest compressive and bending strengths were obtained with the bare gypsum plaster. Boric acid addition decreased both the compressive and bending strength of the gypsum plaster. The

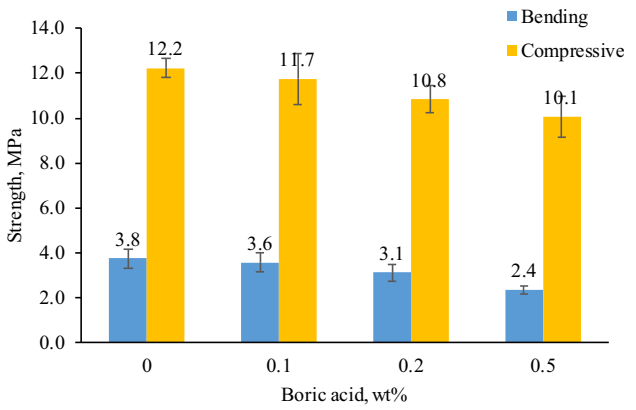


Fig. 4 The effect of boric acid addition on the compressive and bending strength of the gypsum plaster

decrease in bending strength and compressive strength indicated some physical changes in the microstructure of the gypsum after boric acid addition. XRD and FTIR analysis showed that no significant change was observed in the chemical properties of the gypsum structure and it kept the calcium sulfate dihydrate structure. However, N_2 adsorption–desorption analysis and Helium pycnometer showed that boric acid addition increased the skeletal density and pore volume (the porosity) of the gypsum plaster. The results indicated that boric acid addition led to a change in the gypsum matrix by creating new open pores and interconnecting pores. Furthermore, the average pore size of gypsum was increased as well. The increase in pore volume and pore size was considered the reason for the lower bending strength and compressive strength. In particular, the pore size distribution change in the GP-0.5B sample was obvious and the lowest mechanical strength was obtained for the sample. Moreover, SEM images of the bare and boric acid-added gypsum plaster revealed that as boric acid addition was increased, particle size increased as well (Fig. 5). Having these bigger particles led to an increase in the porosity of gypsum plaster and decreased the mechanical strength of gypsum particles. It has been evaluated that the main reasons for the change in mechanical strength are the increase in pore sizes and the formation of particles of larger sizes. Similarly, Zhou et al. reported that the bending strength of concrete decreased with the increase in average pore diameter and total porosity [42]. On the contrary, in our previous study, reduced graphene oxide increased the skeletal density and decreased the porosity of the gypsum. Therefore, it changed the microstructural properties of gypsum and improved the mechanical strength of the gypsum plaster. 0.1 wt% B-rGO addition decreased the bending strength while it increased the compressive strength of the gypsum plaster [10]. Similarly, Davraz observed that B_2O_3 addition increased the compressive strengths and decreased the flexural strength of cementitious composite samples [12].

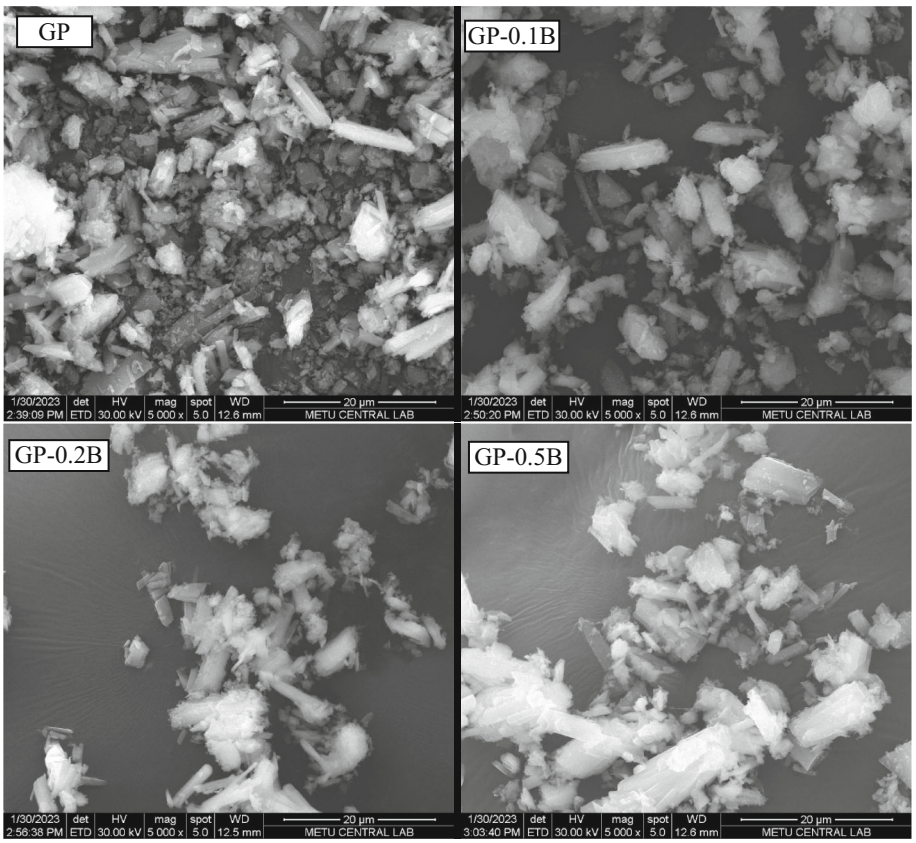


Fig. 5 SEM images of bare and boric acid-added gypsum plaster

3.3 Fire Resistance Test Results of Gypsum Materials

To measure the fire resistance of bare gypsum and boric acid-added gypsum plaster, the plaster was exposed to fire from two faces of the plaster plate. Two different procedure was used to add boric acid into the structure of the gypsum to observe the effect of the boric acid-adding procedure on the fire resistance of gypsum. In the first method, boric acid (0.1% by wt) was dissolved in water and mixed with the dry powder gypsum. The material was labeled as GP-0.1B as previously described. In the second procedure, boric acid (0.1% by wt) was dissolved in the required amount of water and the dissolved boric acid was sprayed on the two surfaces of the gypsum plaster plate (3300 × 75 × 10) at the end of the drying step of the 7th day. The new GP-0.1B/S material was dried at 40 °C for 3 days. The fire resistance time of GP and boric acid-added gypsum plasters (GP-0.1B and GP-0.1B/S) are given in Fig. 6. The fire resistance time in the bare gypsum plaster sample was measured as 40 s. At that time, the gypsum plaster plate cracked and broke. Boric acid addition increased the fire resistance of the gypsum plaster. Especially boric acid sprayed gypsum plaster GP-0.1B/S resisted the fire more than the GP-0.1B sample. Rojo et al. investigated the effect of standard fire on one face of gypsum plaster on the porosity distribution along the thickness of a large board sample. They concluded that the

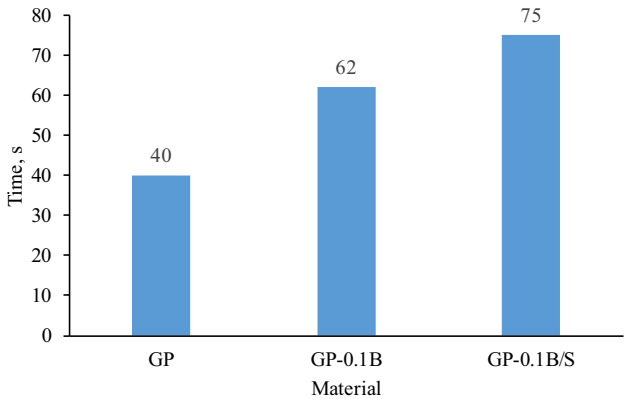


Fig. 6 Fire resistance time of GP and boric acid-added gypsum plasters (GP-0.1B and GP-0.1B/S)

cracks were observed due to the crystal morphology changing at very high temperatures [43]. Uslu et al. [44] tested the effect of adding $CaCO_3$, $C_3H_6N_6$ and H_3BO_3 to water-based indoor paints and concluded that the addition of these flame-retardant additives improved the flammability of the paint. In the study of Tasi et al. [45], the thermal shielding and the fire retardancy properties of the construction coating were enhanced by adding hexagonal boron nitride material. It was reported in the literature that boron compounds have high potential as flame retardants in polymer-based materials and

cellulose-based products due to their low toxicity, molecular diversity, and different pathways of flame-retardant action [46]. Borazan and Gokdai tested the flame-retardancy properties of polyester composite materials with different boron compounds. They observed that boron oxide enhanced the flame-retardant property of pine cone/polyester composite [20]. Rajpoot et al. reported that calcium borate addition to the cotton fabric increased the flame resistance efficacy of the fabric [21].

3.4 X-ray Radiation Attenuation Test Results of Gypsum Materials

To evaluate the shielding properties of bare gypsum and boric acid-added gypsum plaster, X-ray radiation attenuation tests were performed from one face of the plaster plate. As discussed in the Characterization Result section, boric acid addition increased the porosity of gypsum samples. Zayed et al. reported that the increase in porosity with the addition of boric acid into the serpentine concrete significantly decreased the attenuation properties against γ-rays [17]. To minimize the boric acid addition effect on structural changes in gypsum plaster, 0.1% by wt boric acid-added gypsum sample (GP-0.1B) was selected for the X-ray radiation attenuation tests. The test results showed that a small amount of boric acid addition (0.1% by wt.) slightly increased the X-ray radiation attenuation of bare gypsum plasterboard by 0.7%. The attenuation ratio value of gypsum materials was compared with a thickness of reference lead having the same attenuation ratio. The equivalence thickness was found as 83.5 ± 3.5 μm Pb for the case of the GP-0.1B sample. On the contrary, boric acid addition significantly decreased the γ-rays attenuation properties of cement, and limited boron proportions must be used because of physical change in gypsum plaster and the decrease in its mechanical strength [17].

4 Conclusions

In this study, boric acid was evaluated as a gypsum plaster additive. XRD patterns of bare and boric acid-added gypsum plasters showed that boric acid addition did not change the calcium sulfate dihydrate structure of gypsum plaster. ATR-FTIR supported XRD analysis results by revealing almost similar functional groups of the bare gypsum and boric acid-added gypsum. However, boric acid addition increased the pore volume and pore size of the gypsum, resulting in a decrease in the bending and compressive strength of the gypsum plaster. The small amount of boric acid did not change the mechanical strength of the gypsum plaster much; however, it almost doubled the fire resistance time of the gypsum plaster plate, especially using the sprayed method. Furthermore, boric acid addition slightly enhanced the X-ray radiation attenuation properties of bare gypsum plaster. The results indicated that boric acid can be used as a fire resistance and a radiation shield additive for gypsum plaster.

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Author Contributions EA was involved in the Formal analysis and Investigation, Writing-Original draft preparation, and Validation. HA was involved in the Conceptualization, Methodology, Formal analysis and Investigation, Writing-Reviewing and editing, Funding acquisition, Resources, and Supervision. DDEK was involved in the Conceptualization, Methodology, Formal analysis and Investigation, Writing-Reviewing and editing.

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Data and Code Availability Not applicable.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

Ethical Approval Not applicable.

Human and Animal Rights This article does not include any studies of human participants performed by the authors.

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