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ORIGINAL ARTICLE

Studying the metakaolin content, fiber type, and high-temperature effects on the physico-mechanical properties of fly ash-based geopolymer composites

Barış Bayrak[1] · Haluk Görkem Alcan[1] · Özge Çiğdem Özelmacı Durmaz[2] · Süleyman İpek[3,6] · Gökhan Kaplan[4] · Erhan Güneyisi[5] · Abdulkadir Cüneyt Aydın[4]

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Abstract

The study investigated the physicasl characteristics and mechanical performance of fly ash-based geopolymer composites when exposed to high temperatures. Geopolymer composites were produced using fly ash as an aluminosilicate-rich raw material and a combination of sodium silicate and sodium hydroxide as an alkaline activator. In this context, the study also examined the impact of partially replacing metakaolin (7.5% and 15% by weight). Furthermore, the study aims to examine the impact of adding fiber (basalt and carbon types) on the physical, mechanical, and high-temperature properties of geopolymer composites. The physical properties investigated were unit weight, apparent porosity, water absorption, and capillary water absorption, while the strength performances investigated were flexural and compressive strengths. To monitor the effect of high temperatures on the strength characteristics of the geopolymer composites, the mixtures were exposed to temperatures of 200 °C, 400 °C, and 600 °C. Besides, SEM images were provided to illustrate the degree of geopolimerization. The results indicated that metakaolin replacement yielded mixtures having higher unit weight, but lower apparent porosity and water absorption. The results indicated that metakaolin replacement yielded mixtures having a higher unit weight, reaching an increase of about 5%, but lower apparent porosity and water absorption, with decreases reaching 18.3% and 20%, respectively. The metakaolin-blended geopolymer composites resulted in better strength performance and resistance to high temperatures. Raising the metakaolin replacement level from 0 to 15% led to an increase of 17.3% in flexural strength. The compressive strength of the composites subjected to a temperature of 200 °C exhibited an increase of over 10%. Notably, this rate of increment was observed to be nearly 20% higher in nonfibrous composites. Fiber addition decreased the compressive strength up to about 21%, while increasing the flexural strength up to 65%. Strength performance improved at 200 °C, but decreased at higher temperatures up to 600 °C. The geopolymer composites experienced significant mass loss when exposed to high temperatures.

Keywords Basalt fiber · Carbon fiber · Geopolymer mortar · SEM analysis · Strength performance

✉ Süleyman İpek
suleyman.ipek@yasar.edu.tr

Barış Bayrak
bbayrak@kafkas.edu.tr

Haluk Görkem Alcan
hgorkemalcan@kafkas.edu.tr

Özge Çiğdem Özelmacı Durmaz
ocigdem.ozelmaci@hku.edu.tr

Gökhan Kaplan
gkaplan@atauni.edu.tr

Erhan Güneyisi
eguneyisi@harran.edu.tr

Abdulkadir Cüneyt Aydın
acaydin@atauni.edu.tr

[1] Faculty of Engineering and Architecture, Civil Engineering Department, Kafkas University, 36100 Kars, Türkiye

[2] Engineering Faculty, Civil Engineering Department, Hasan Kalyoncu University, 27010 Gaziantep, Türkiye

[3] Engineering Faculty, Civil Engineering Department, Yaşar University, 35100, İzmir, Türkiye

[4] Engineering Faculty, Civil Engineering Department, Atatürk University, 25030 Erzurum, Türkiye

[5] Engineering Faculty, Civil Engineering Department, Harran University, 63290 Şanlıurfa, Türkiye

[6] Faculty of Engineering and Architecture, Civil Engineering Department, Bingöl University, 12000 Bingöl, Türkiye

1 Introduction

In terms of environmental impact, approximately 40% of global resource consumption originates from the construction industry [1, 2]. Thanks to its superior mechanical and durability properties, as well as its low cost and ability to be produced with regional raw materials, concrete has an indispensable position in the construction industry [3]. However, conventional concrete's binder, cement, is a significant contributor to the aforementioned usage and gas emissions. Each ton of Portland cement produced emits almost 1 ton of CO_2 [4]. Over 4 billion metric tons of Portland cement are produced each year [5]. As a result, cement manufacturing contributes to more than 7–8% of all human-caused global carbon emissions [6, 7]. Considering the environmental challenges and high CO_2 emissions, it is significant to develop more environmentally friendly alternatives. Studies on this issue have been and are being carried out, but the problem still continues. Therefore, reducing cement consumption and limiting the carbon footprint is crucial.

When more environmentally friendly alternatives are considered in terms of CO_2 emissions and energy consumption, the cementitious material called geopolymer has become one of the most preferred options. Geopolymer is important in terms of the greenhouse gas effect, as it causes six times less CO_2 emissions during manufacturing than traditional concrete [8]. In addition to less environmental impact, geopolymer offers high compressive strength and flexural strength, minimal shrinkage, quick setting, and high heat resistance [9, 10]. Geopolymers are produced using a source of aluminosilicate material and alkali activator [11, 12]. Aluminosilicate materials have rich silicon (Si) and aluminum (Al) content, and some natural minerals and by-products can be used as source materials. Potassium and sodium are generally used in geopolymer production as alkali activators [13, 14].

The present work herein mainly aims to produce geolpymer, as an alternative to concrete in terms of its mechanical properties and environmental advantages. For this purpose, by-product materials fly ash (FA) and metakaolin (MK) were used in the production of geopolymer composites [15]. Fly ash, which is a by-product of the combustion of pulverized coal, is generated approximately 780 million tons per year, making it one of the largest solid wastes in the world. In contrast, the usage rate is around 17–20% [16]. MK is obtained by the calcination of kaolinite clay through a heat treatment process and a huge amount of MK is also produced in the world per year [17, 18]. The use of by-product materials is a positive solution for reducing gas emissions and natural resource use from an environmental perspective [19, 20]. However, the mechanical properties of geopolymers are still being tested and evaluated in terms of their applicability. Görhan and Kürklü [21] conducted a study by changing the MK ratio in FA-based geopolymer mortar samples and observed that as the MK ratio increased, porosity decreased and thus strength values increased. Gomez Casero et al. [22] conducted a study in which they evaluated the use of FA and MK in geopolymer production by partially substituting MK with FA in the range of 25–100%. Accordingly, they determined the optimal substitution rate of MK with FA and its effect on the compressive strength and thermal conductivity of geopolymer composites. As a result of the study, it was observed that slightly lower thermal conductivity and compressive strength were obtained in FA–MK blended geopolymers compared to geopolymers prepared by only FA or only MK. Nuaklong et al. [23] conducted a study to investigate the effect of MK substitution on FA-based geopolymer composites, containing 100% recycled coarse aggregate obtained from crushed laboratory specimens. As a result of the study, it was observed that increasing the use of MK leads to obtaining better strength, porosity, water absorption, and acid resistance.

Geopolymer composites exhibit brittle behavior under loads similar to traditional concrete. Studies have been conducted to enhance the deformation properties and toughness of geopolymer composites by incorporating fibers [24, 25]. Abbass and Singh conducted a study and used basalt fiber (BF) of 1%, 5%, 10%, 15%, 20%, and 25% by weight and obtained the higher impact strength in the composites using 10% BF by weight. In addition, increased compressive strength of geopolymer concrete was observed to be 88% at 7 days, 89% at 28 days, and 92% at 90 days compared to the control mixture. In another study, Şahin et al. [26] found that adding BF to geopolymer composite samples had a positive effect on all properties, particularly in the range of 0.8–1.2%. Researchers also reported increases of up to 25% in compressive strength and 50% in flexural strength. Ghanem and Bowling [27] conducted a study to investigate the effect of chopped carbon fiber (CF) on the workability properties and strength characteristics such as compressive, flexural, and splitting strengths. CF was used with ratios of 0.5%, 1.0%, 1.5%, and 2.0%. As a result of the study, a linear increase was obtained in splitting and flexural strength with effectiveness between 18.37–132.6% and 3.26–13.82%, respectively. The compressive strength effectiveness was found to be 13.65% with 1% CF content. It was observed that adding more CF did not improve the compressive strength. Furthermore, the addition of CF resulted in a decrease in the workability of concrete.

2 Research significance

As awareness of environmental pollution and the rapid consumption of natural resources increases, humans have come to realize that nature is not an infinite resource; therefore, solutions need to be developed in this context. Evaluating environmental performance in all types of production processes has become an important issue to prevent environmental pollution. The construction sector plays a significant role in greenhouse gas emissions and the consumption of natural resources. As concrete is extensively used in construction worldwide, it is crucial to develop alternative materials to reduce these effects. This study aims to develop a material that can serve as an alternative to concrete. This material should possess similar mechanical properties while also being environmentally friendly. The physical characteristics, mechanical performance, and resistance to the elevated temperature of geopolymer composites, produced using fully industrial by-products, were experimentally investigated in this regard. The primary aluminosilicate material used to produce geopolymer composites is FA. The alkaline activator medium consists of a combination of sodium silicate and sodium hydroxide. In this context, the study also examined the impact of partially replacing metakaolin (7.5% and 15% by weight), which is expected to improve the performance of geopolymer composites, to improve the physical properties, mechanical performance, and resistance to high temperature. A further key objective of this study is to examine the influence of fiber incorporation into geopolymer composites on their high-temperature durability. To this end, two prominent fiber types, namely basalt and carbon fibers, which are widely utilized in the construction industry, were selected for reinforcement of geopolymer composites. In addition to observing the impact of the fiber addition on the high-temperature resistance, the performance of both fiber types was also compared on the basis of their strength and high-temperature resistance performances.

3 Experimental program

3.1 Materials, mix design, and manufacturing process

Here, in the manufacturing of geopolymer composite mixtures, FA (classified as class F according to ASTM C618 [28]) and MK, of which the physical properties and chemical compositions are presented in Table 1, were used as aluminosilicate-rich materials. Figure 1 shows the particle size distributions of both powder materials, as detected by a laser particle analyzer (Malvern Mastersizer 2000). The average particle sizes of FA and MK were 19.1 µm and 3.4 µm, respectively. In addition, the mineralogical properties of FA and MK determined by XRD are demonstrated in Fig. 2. The specific surface area of FA was determined by the Blaine method and measured as 335 m^2/kg, while that of MK was determined by the BET method and measured as 16,200 m^2/kg.

To enhance the sustainability of geopolymer composite mixtures, waste marble powder (WMP) with a specific gravity of 2.75 was utilized as a filler. The sieve analysis of WMP indicated that all WMP grains passed through 150 µm. The skeleton of the geopolymer mixtures is made of quartz sand with particle sizes changing between 1 and 3 mm, a specific

Table 1 The physical properties and chemical compositions of FA and MK

Oxide (% by mass)	FA	MK
CaO	2.9	0.6
SiO_2	56.7	56.6
Al_2O_3	27.6	36.5
Fe_2O_3	4.3	3.9
MgO	2.7	0.2
SO_3	1.9	0.3
Na_2O	0.4	0.2
K_2O	0.7	0.6
LOI (900 °C)	2.4	0.5
Specific gravity	2.28	2.54
Specific surface area (m^2/kg)	335*	16,200**

*Blaine method

**BET method

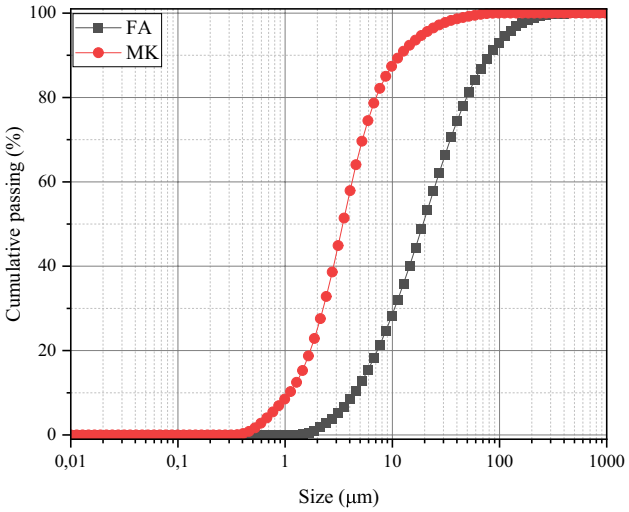


Fig. 1 The particle size distribution of the FA and MK

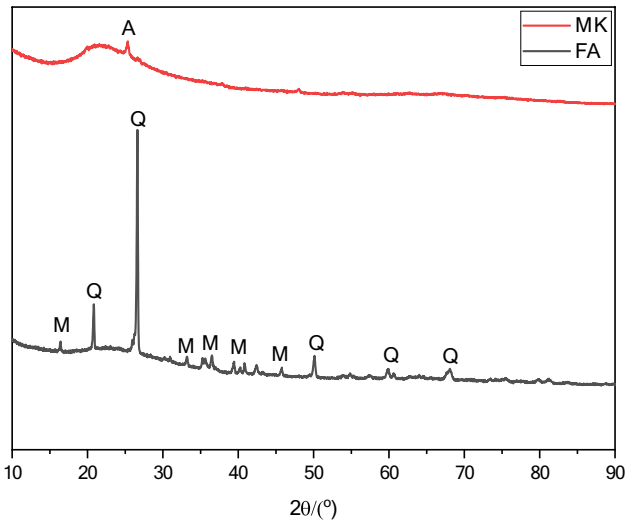


Fig. 2 XRD pattern of the FA and MK (*Q*: quartz, *M*: mullite, *A*: anatase)

gravity of 2.82, and water absorption of 1.3%. Microfibers CF and BF, with engineering properties presented in Table 2, were used to enhance the flexural performance and high-temperature resistance of geopolymer composites. The alkali activator was made from 99% pure sodium hydroxide (NaOH) and 30% solid-content sodium silicate ($SiO_2/Na_2O = 3$). NaOH was prepared as 12 M approximately 24 h before casting. Photographic views of ingredients used in the manufacture of geopolymer composites are given in Fig. 3. Geopolymer composites were produced without the use of a superplasticizer or excess water.

In the production of geopolymer composite mixtures, the total amount of aluminosilicate-rich materials (FA + MK) was designated as 800 kg/m^3. MK was replaced with FA at the levels of 7.5% and 15% by weight. In all mixtures, 525 kg/m^3 alkali solution ($Na_2SiO_3 + NaOH$) was used. The alkaline solution content constituted 65.6% of the aluminosilicate-rich materials. The content of the alkali solution was determined based on the workability of the mixtures. The ratio of Na_2SiO_3 to NaOH in the alkaline solution is 2.5. Additionally, CF and BF were added to the mixture at a fraction of 2% of the amount of aluminosilicate-rich materials, and in addition to the individual utilization, the fibers were also used as a hybrid with BF and CF at a fraction of 1%. The aggregate mixture is composed of 750 kg/m^3 WMP and 250 kg/m^3 quartz sand. A higher amount of WMP was used to reduce the carbon emission and porosity of the mixture. In total, 12 different geopolymer composite mixtures were designed and produced at different MK replacement levels and fiber type and weight fractions. The quantities of constituents used in the production of geopolymer mixtures are given in Table 3. The mixture code in this table denotes the replacement level of metakaolin with the abbreviation of MK and weight fractions of basalt and carbon fibers with BF and CF, respectively. For example, the MK15BF1CF1-coded mixture is a mixture manufactured with 15% MK, 1% BF, and 1% CF.

The production process of geopolymer composites starts with mixing aluminosilicate-rich materials (FA + MK) at low speed for 2 min. Then, an alkaline solution (mixture of $Na_2SiO_3 + NaOH$) was added to the dry mixture and mixed at low speed for 1 min and high speed for 1 min. Finally, fine aggregates (mixture of WMP + quartz) were added to the geopolymer paste and mixed at low speed for 1 min and high speed for 1 min. At the end of 6 min, no vibration was applied as the geopolymer mixtures obtained had a self-compactability

Table 2 Engineering properties of the basalt and carbon fibers

Fiber	Length (mm)	Diameter (μm)	Density (g/cm^3)	Elongation (%)	Modulus of elasticity (GPa)	Fracture strength (MPa)
BF	12	10	2.70	2.7	100	3000
CF	10	7	1.71	2.1	220	3530

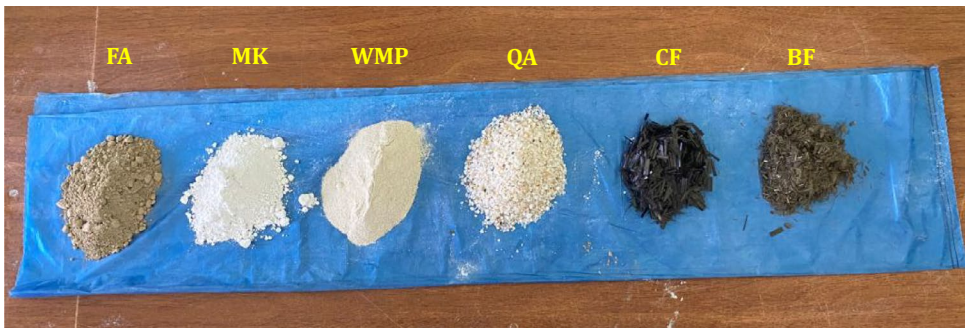


Fig. 3 Photographic view of ingredients used in the manufacture of geopolymer composites (*FA*: fly ash, *MK*: metakaolin, *WMP*: waste marble powder, *QA*: quartz aggregate, *CF*: carbon fiber, *BF*: basalt fiber)

Table 3 Mixture codes and quantities of geopolymer constituents (kg/m^3)

Mixture code	FA	MK	WMP	QA	NaOH	Na_2SiO_3	BF	CF
MK0BF0CF0	800	0	750	250	150	375	–	–
MK7.5BF0CF0	740	60						
MK15BF0CF0	680	120						
MK0BF2CF0	800	0	750	250	150	375	16	–
MK7.5BF2CF0	740	60						
MK15BF2CF0	680	120						
MK0BF0CF2	800	0	750	250	150	375	–	16
MK7.5BF0CF2	740	60						
MK15BF0CF2	680	120						
MK0BF1CF1	800	0	750	250	150	375	8	8
MK7.5BF1CF1	740	60						
MK15BF1CF1	680	120						

characteristic. The flow diameter of the mixtures varies between 250 ± 20 mm. The geopolymer composite mixtures poured into the molds were immediately placed in the oven and the thermal curing regime at 80 °C for 20 h was applied to the specimens. At the end of 20 h, the oven was turned off, but the samples were allowed to cool in the oven to prevent thermal shock in the samples. 50-mm cubic and 40- by 160-mm prismatic specimens were taken from each geopolymer composite mixture to carry out the designated experimental tests.

3.2 Test methods

After the samples were cooled in the oven, their hardened density, apparent porosity, and water absorption were determined on the 50-mm cubic specimens following the ASTM C642 [29]. To ascertain the hardened density of geopolymer composite mixtures, the volume of a geopolymer composite sample (V) was initially determined, and then the oven-dry sample was weighed (D). The hardened density was subsequently determined by dividing the oven-dry weight by the volume. The apparent porosity of the geopolymer composite mixtures was determined by saturated weight (W) minus dry weight divided by volume. On the other hand, the water absorption capacity of the geopolymer composite mixtures was calculated by dividing the saturated weight minus oven-dry weight by the oven-dry weight. The following three expressions, respectively, used to determine the hardened density, apparent porosity, and water absorption capacity of the geopolymer composites:

$$Hardeneddensity\left(\frac{kg}{m^3}\right) = \frac{D}{W} \quad (1)$$

$$Apparentporosity(\%) = \frac{W-D}{V}x100 \quad (2)$$

$$Waterabsorptioncapacity(\%) = \frac{W-D}{D}x100 \quad (3)$$

ASTM C348 [30] (for flexural strength) and ASTM C349 [31] (for compressive strength) were followed to determine the mechanical properties of the geopolymer composites. Prismatic specimens with dimensions of 40 × 40x160 mm and cubic specimens with dimensions of 50 × 50x50 mm were used to carry out flexural strength and compressive strength tests, respectively. The mechanical properties were determined immediately after the specimens cooled once the oven-curing regime was completed. The capillary water absorption of geopolymer composites was measured on 50-mm cubic specimens in regard to EN 1015-18 [32]. The samples were exposed to capillary action for 24 h, and the amount of water absorbed per unit surface area was then determined. The geopolymer composite specimens were also exposed to high temperatures at 200 °C, 400 °C, and 600 °C. A laboratory-type muffle furnace was used for high-temperature resistance. The heating rate of the muffle furnace was set to 10 °C/min. The samples were kept for 2 h after reaching the target temperature. After the samples were cooled in the muffle furnace, they were tested for compressive strength and mass loss. 50-mm cubic specimens were used for the determination of high-temperature resistance.

3.3 Microstructure examination

To determine the microstructural properties of PAG mixtures, SEM analysis was performed. Microstructure analysis was carried out at Kastamonu University Central Research Laboratory Application and Research Center. SEM images of the specimens at different magnifications were determined by the FEI Quanta FEG 250 device.

4 Results and discussion

4.1 Physical properties

4.1.1 Unit weight and apparent porosity

The changes in the unit weight and apparent porosity of the geopolymer mixtures are presented in Fig. 4a, b, respectively. Replacing FA with MK significantly impacted the unit weight and apparent porosity values of the geopolymer composite mixtures, as demonstrated by the results. Increasing the MK replacement level resulted in higher unit weights and lower apparent porosity of the mixtures, as demonstrated in Figs. 4a, b, respectively. In the nonfibrous geopolymer composite mixtures, MK0BF0CF0, MK7.5BF0CF0, and MK15BF0CF0-coded mixtures have unit weights of 2112 kg/m^3, 2198 kg/m^3, and 2215 kg/m^3, respectively, while the apparent porosity values were measured as 8.55%, 7.62%, and 7.34%, respectively. The increase in unit weights can be attributed to the relatively higher specific gravity of MK. The decrease in apparent porosity can be explained by the filling effect contribution of the finer MK particles. Replacing the FA with MK at the level of 15% resulted in increases in unit weight with varying rates between 2 and 5% in all geopolymer composite mixture series. The highest increase with a rate of approximately 5% was obtained in the nonfibrous geopolymer composite series. Replacing FA with MK at a level of 15% resulted in an increase in the apparent porosity of the geopolymer composites, with rates ranging from approximately 9% to 18%. A comparable outcome was reached by Görhan and Kürklü [21]. The researchers observed that elevating the MK substitution ratio led to a reduction in the apparent porosity ratio. Moreover, the findings of the Nuaklong et al. [23] study also demonstrated a decrease in porosity associated with augmented MK content. The most effective contributions due to the MK replacement to the apparent porosity were seen in the nonfibrous and hybrid–fibrous geopolymer composite series with rates of 14.2% and 18.3%, respectively. Figure 4a, b, respectively, indicates the impact of adding 2% BF, 2% CF, and 1% BF-1% CF on the unit weight and apparent porosity of the geopolymer composite mixtures. It is evident that using CF and BF, either separately or as a hybrid, does not result in significant differences, and the overall trend is a reduction in unit weight in all cases. Fiber addition did not have a systematic or stable influence on the unit weight and apparent porosity of geopolymer composite mixtures. However, the general trend was to reduce the unit weight and increase the apparent porosity.

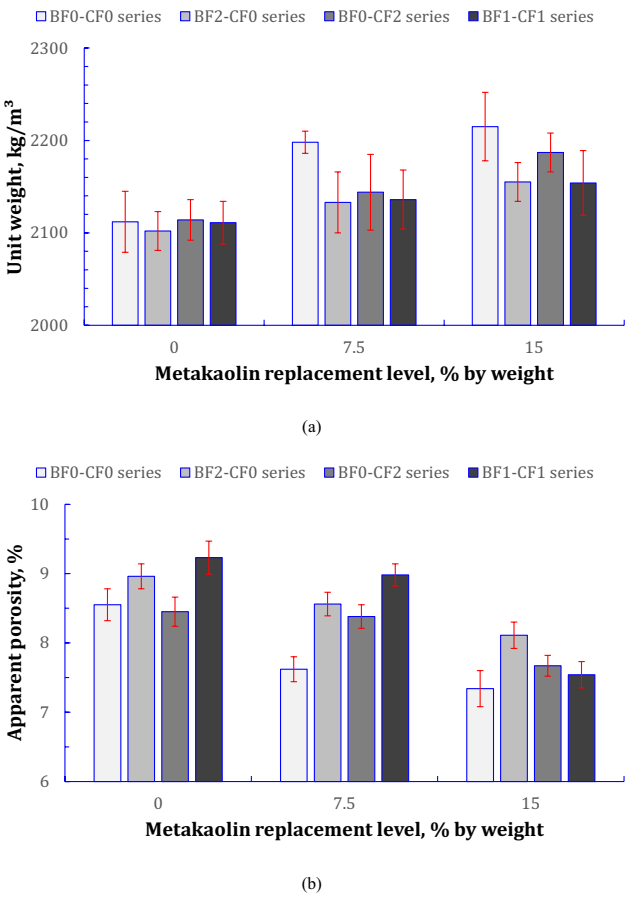


Fig. 4 **a** Unit weight and **b** the apparent porosity of the geopolymer composites

4.1.2 Water absorption and capillary water absorption

The effects of the substitution of MK with FA and adding fiber on the water absorption and capillary water absorption of geopolymer composite mixtures are, respectively, presented in Fig. 5a, b. The results indicate that incorporating MK and increasing the replacement level generally lead to a slight decrease in water absorption. The average water absorptions of geopolymer composite mixtures manufactured with only FA were in the range of almost 5.5% and 6.0%, while those manufactured with MK at replacement levels of 7.5% and 15% have water absorption values changing between 4.8% and 5.7% and between 4.7% and 5.1%, respectively. The water absorption values and Fig. 5a demonstrate a consistent trend in water absorption with the incorporation of MK, as observed in the apparent porosity. Similarly, Görhan and Kürklü [21] reached a comparable conclusion regarding the influence of MK incorporation on the water absorption performance of such composites. The findings of this study indicate that an increase in MK content is associated with a reduction in water absorption rate. As observed by Nuaklong et al. [23], the trends in water absorption are analogous to those of porosity, exhibiting a decline in water absorption

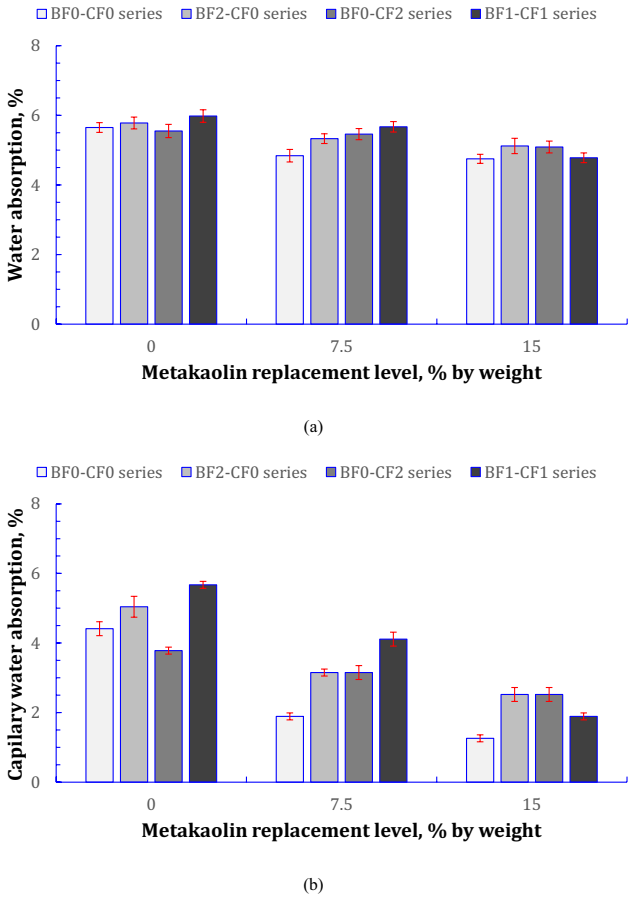


Fig. 5 **a** Water absorption and **b** capillary water absorption values of the geopolymer composites

with an increase in MK. On the other hand, it can be clearly stated that incorporating MK and increasing its replacement level effectively resulted in a decrease in capillary water absorption. The capillary water absorption of geopolymer composites manufactured with only FA was more than 3.8%, while those manufactured with MK at a replacement level of 15% had capillary water absorption values of less than almost 2.5%. The reduction in water absorption and capillary water absorption is due to the formation of a less porous structure in the geopolymer paste and a thinner interfacial transition zone between the aggregate and geopolymer paste resulting from the incorporation of MK. However, the addition of fiber caused a slight increase in water absorption and a more significant increase in capillary water absorption. The results also showed that the addition of fiber to the MK-blended geopolymer composites resulted in greater increases in water absorption and capillary water absorption values than the addition of fiber to the plain geopolymer composites.

4.1.3 The relationship between physical properties

Bilateral correlations between the physical properties of geopolymer mixtures were examined and whether there was a relationship between these properties was investigated within the scope of this study. The relationships between apparent porosity-unit weight, unit weight-water absorption, apparent porosity-water absorption, and capillary water absorption-water absorption are represented in Fig. 6a–d, respectively. Upon examining the relationship between unit weight-apparent porosity and unit weight-water absorption of the geopolymer composite mixtures, a moderate linear correlation was observed with an R-squared value of 0.755. As can be seen in Fig. 6a, b, there is an inverse relationship between apparent porosity and unit weight and unit weight and water absorption. In other words, as the apparent porosity and water absorption of the geopolymer composites increases, the unit weight decreases. On the other hand, there was a strong linear correlation with R-squared values of roughly 0.93 and 0.96 between the apparent porosity–water absorption and capillary water absorption–water absorption, respectively. As can be seen in Fig. 6c, d, there is a direct relationship between apparent porosity and water absorption and capillary water absorption and water absorption. All these findings indicate that a systematic experimental program was carried out during the study.

4.2 Strength performance

Determining the mechanical performance of such composites is a crucial aspect of experimental research. This is because many properties of both cement-based and geopolymer-based composites are directly related to their mechanical properties. In this study, compressive and flexural strengths of FA/MK-blended nonfibrous and fibrous geopolymer composites were determined and the results are shown in Fig. 7a, b, respectively. The plain geopolymer composite mixture achieved an average compressive strength of around 34.3 MPa. Incorporating MK led to even higher compressive strength. This situation may be formed due to two possible effects of MK utilization: one is due to the formation of a less porous structure in the geopolymer paste and the second is the formation of a thinner interfacial transition zone between the aggregate and geopolymer paste resulting from the incorporation of MK. However, the fibrous geopolymer composite mixtures exhibited lower compressive strength performance compared to their nonfibrous counterparts. The beneficial impact of MK was also evident in the fibrous geopolymer composites. Replacing FA with MK at a 15% level led to significant improvements in the BF, CF, and hybrid fiber-reinforced geopolymer composites: approximately 27%, 15%, and 32%, respectively. However, the compressive strength results indicated that

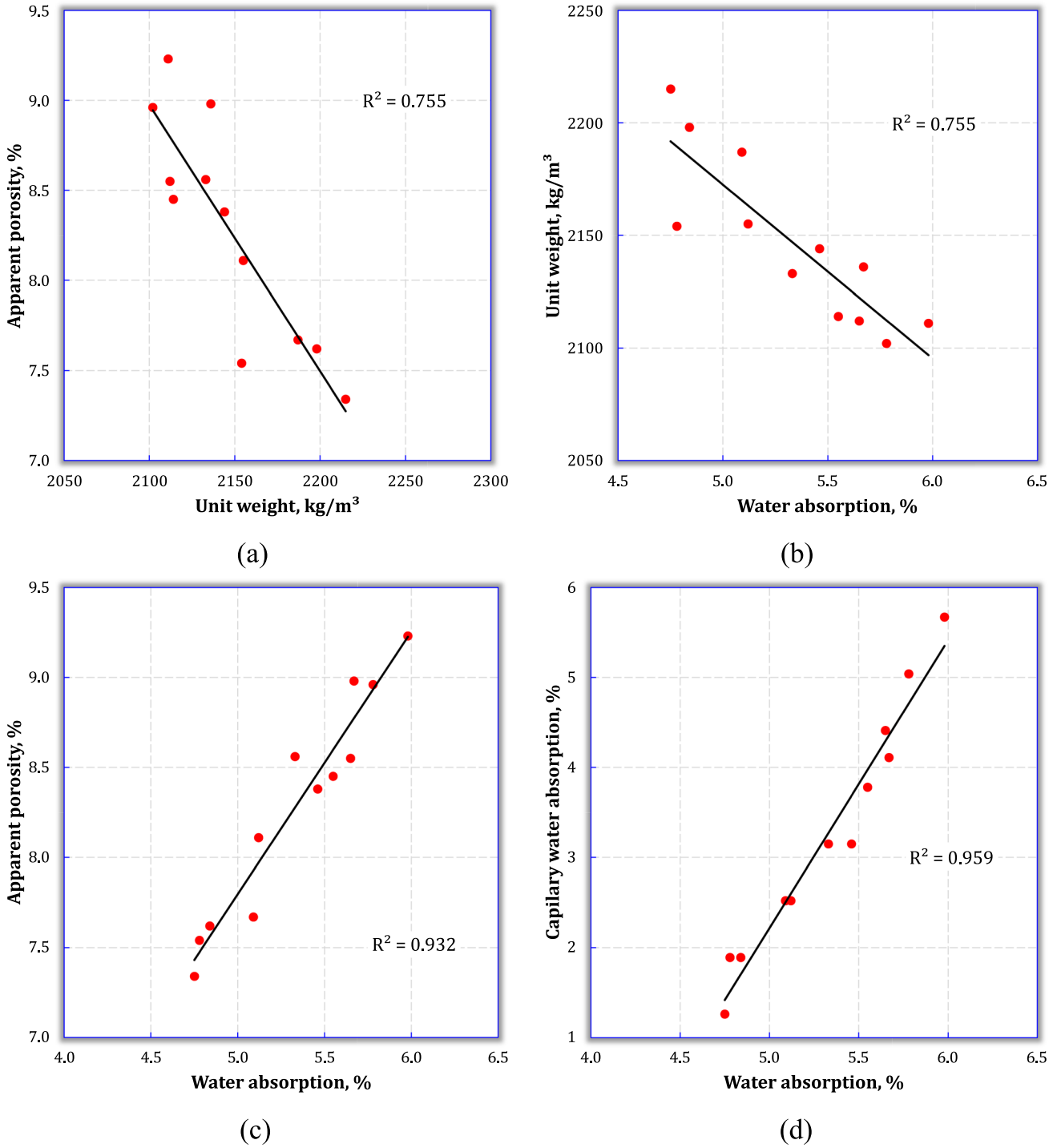


Fig. 6 The relationship between **a** apparent porosity and unit weight, **b** unit weight and water absorption, **c** apparent porosity and water absorption, and **d** capillary water absorption and water absorption

adding microfiber into the geopolymer composites has a tendency to decrease the compressive strength performance. Fiber particles may lump together and cause agglomeration, which could be a main factor in this situation. The agglomeration of fiber particles may also cause the formation of microporous structures in the geopolymer paste. The average highest compressive strength of 43.0 MPa was achieved in the MK15BF0CF0-coded geopolymer composite mixture, while the average lowest compressive strength of 30.3 MPa was obtained in the MK0BF1CF1-coded mixture.

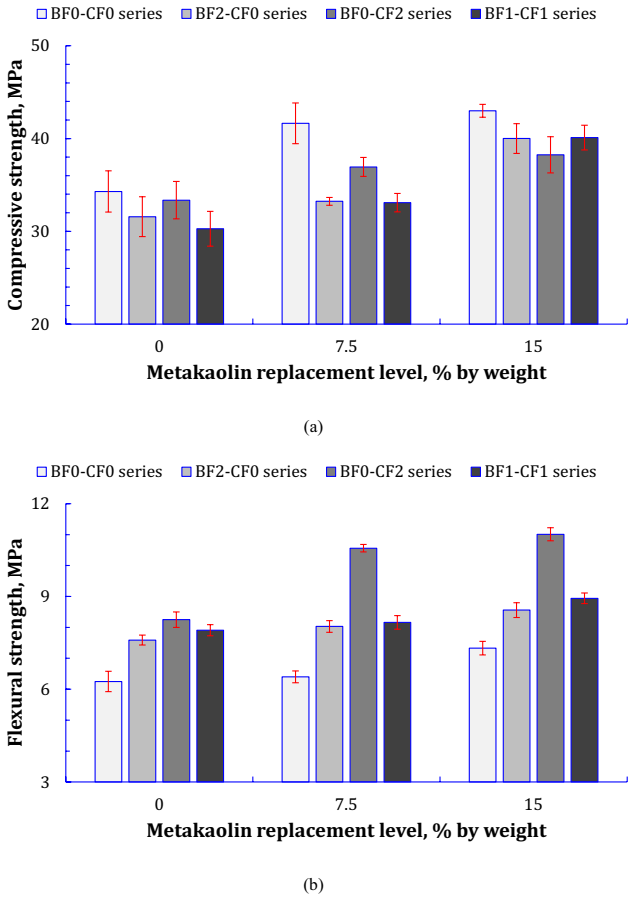


Fig. 7 **a** Compressive strength and **b** flexural strength of the geopolymer composites

The plain geopolymer composite (not containing MK and fiber) has an average flexural strength of about 6.25 MPa. Replacing FA with MK had a similar effect on the flexural strength of the geopolymer composites as it did on the compressive strength. Raising the MK replacement level from 0 to 15% led to a remarkable 17.3% boost in flexural strength. Geopolymer composites reinforced with CF achieved an impressive increment rate of over 30% by replacing FA with MK at a 15% level. The BF and hybrid fiber-reinforced geopolymer composites exhibited an increase in flexural strength of approximately 13% with the increase of MK replacement level from 0 to 15%. The flexural strength of the geopolymer composites significantly increases with the addition of a 2% weight fraction of fiber, as demonstrated in Fig. 7b. The highest flexural strength increment was achieved in the geopolymer mixture series reinforced with CF. The average highest flexural strength of 11.0 MPa was achieved in the MK15BF0CF2-coded geopolymer composite mixture, while the average lowest flexural strength of 6.25 MPa was obtained in the MK0BF0CF0-coded mixture. Although the addition of BF improved the flexural strength of the geopolymer composites, its performance in enhancing flexural strength falls behind that of CF. The fiber dimensions and mechanical performance may be the reasons behind this scenario. Table 2 shows that CF particles are shorter and have a smaller diameter than BF particles. Additionally, CF has a much higher fracture strength and lower density than BF. At the same weight fraction, there will be more CF particles than the BF particles. For this reason and due to its higher fracture strength of CF, the CF-reinforced geopolymer composites yielded higher flexural strength. However, generally, the increase in flexural strength can be attributed to the fact that the fibers can reduce crack expansion by resisting the propagation of cracks, thus improving the brittleness, crack resistance, and flexural strength of the geopolymer mixture. The flexural strength values obtained for the geopolymer composites produced in this study were approximately 15%–30% of their compressive strengths.

Jithendra et al. [33] reported a comparable impact of MK incorporation on the strength performance of geopolymer composites. They replaced ground granulated blast furnace slag with MK at levels of 5%, 10%, 15%, and 20% and proposed the 15% replacement ratio as the optimal proportion when considering the mechanical performance of the composites. Görhan and Kürklü [21] reported a similar effect of MK incorporation on the strength performance of geopolymer composites and concluded that the incorporation of MK into geopolymer mixtures has been observed to enhance the strength performance of these mixtures, which can be attributed to a reduction in porosity. Similarly, the findings of the study conducted by Nuaklong et al. [23] unequivocally demonstrated that the incorporation of MK enhanced the flexural strength characteristics of geopolymer composites. A review of the literature revealed that the incorporation of fibers can effectively mitigate the formation and propagation of cracks in geopolymer composites, while enhancing the crack resistance, brittleness, toughness, and consequently the flexural strength of geopolymer composites [20, 34]. Mermerdaş et al. [20] observed a sustained enhancement in the flexural strength of fly ash-based geopolymer composites when the volume fraction of fibers (of steel, basalt, and glass types) was incrementally augmented from 0% to 1.2%.

4.3 High-temperature effect

The study evaluated the impact of exposure to high temperatures on both nonfibrous and fibrous geopolymer composites. The evaluation was based on the composites' compressive strength and mass loss. Figure 8a–d demonstrates the change in compressive strength of geopolymer composites exposed to temperatures of 200 °C, 400 °C, and 600 °C. The compressive strength of the composites increased as the temperature was raised to 200 °C when compared to those kept at ambient temperature. The compressive strength of the composites exposed to a temperature of 200 °C increased

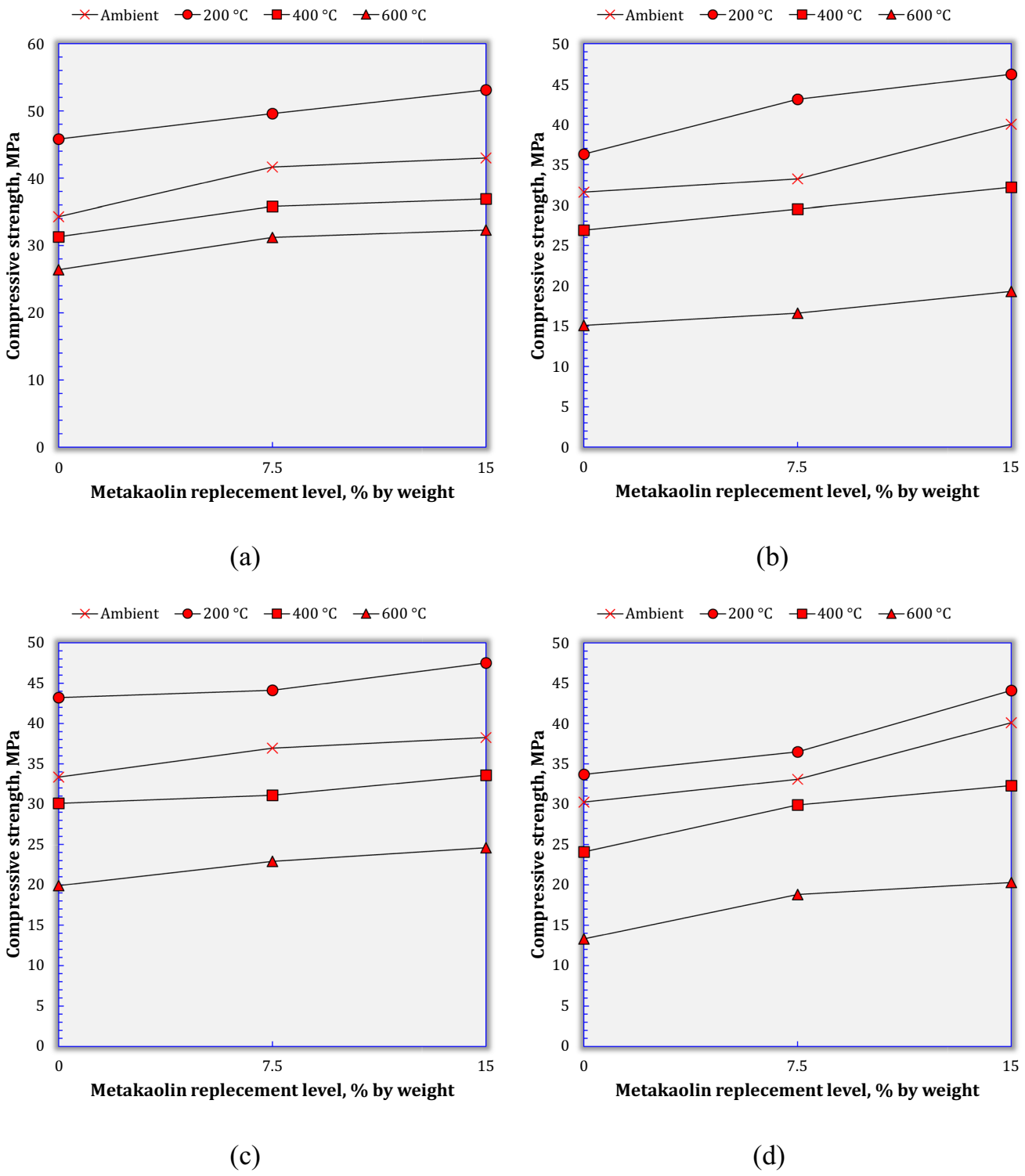


Fig. 8 Variation in compressive strength geopolymer composites exposed to high temperature: **a** nonfibrous series, **b** BF2-CF0 series, **c** BF0-CF2 series, and **d** BF1CF1 series

by more than 10%. Especially in nonfibrous composites, the rate of increment was nearly 20% higher. However, applying temperatures above 200 °C to geopolymer composites caused a significant loss in compressive strength. At 400 °C exposure, the compressive strength fell below the compressive strength of the mixtures kept at the ambient

temperature. In the composites exposed to a temperature of 400 °C, the compressive strength fell below the compressive strength of the mixtures kept at the ambient temperature. A strength loss of approximately 9% was observed when the temperature of 400 °C was applied to the geopolymer composites. The strength loss in fibrous geopolymer composites reached 20%. Increasing the temperature further resulted in a cumulative loss of strength, as expected. Strength losses of around 45–50% were observed in fibrous geopolymer composites when exposed to a temperature of 600 °C, while it was about 25% in the nonfibrous geopolymer composites. The study found that the incorporation of MK did not have a significant impact on the high-temperature resistance of the geopolymer composite. However, the addition of fibers resulted in a significant loss of strength. In their investigation of the behaviors of 56-day geopolymer composites, Şahin et al. [26] observed no significant change in the compressive and flexural strength of the geopolymer composite mixtures at 200 °C. However, they did note a significant decrease in the strength performance of the geopolymer composites at high temperatures.

Figure 9a–d shows the mass loss values in the geopolymer composites resulting from exposure to elevated temperatures. The results indicate that when the composites were exposed to a temperature of 200 °C, they experienced mass losses changing between nearly 1.1% and 2.5%. The general trend was that the composites containing MK had lower mass loss rates, while those produced entirely with FA exhibited higher mass loss rates. Upon investigation of Fig. 9a–d, it is evident that fiber-reinforced geopolymer composites showed greater mass loss than nonfibrous composites when exposed to a temperature of 200 °C. The nonfibrous geopolymer composites experienced a mass loss rate of less than 1.6% in the case of 200 °C exposure, while the fibrous composites experienced a mass loss rate between 1.6% and 2.5%. On the other hand, a remarkable increase in mass loss was observed when the geopolymer composites were exposed to higher temperatures. The rates of mass loss ranged from 4.0% to 7.7% after exposure to 400 °C and from 4.5% to 9.3% after exposure to 600 °C. The positive impact of incorporating MK was observed during exposure to higher temperatures. Among all the geopolymer composite series, those with MK blended mixtures showed better resistance to high temperatures in terms of mass loss. However, the addition of fiber still resulted in lower resistance to high temperatures, regardless of the type of fiber. A comparable conclusion was reached by Şahin et al. [26]. Upon examination of this study, it becomes evident that an increase in temperature is accompanied by a discernible loss of mass. Here, Fig. 10 shows the relationship between mass loss and compressive strength values of geopolymer composites exposed to elevated temperatures. There was a strong inverse correlation with an R-squared value of almost 0.84 between the compressive strength and mass loss of composites.

4.4 SEM analysis

Microstructural investigation was carried out on four geopolymer composite mixes, coded MK0BF0CF0, MK0BF2CF0, MK15BF0CF2, and MK15BF1CF1, details of which are given in Table 3. The SEM images of each mixture are presented in Figs. 11, 12, 13, 14, respectively. Each figure includes photographs enlarged 500, 2500, and 10,000 times. Figure 11a–c shows the microstructure of a nonfibrous geopolymer composite made with only FA. As can be seen in Fig. 11a, there is a compact, homogenous, and dense geopolymer matrix containing some unreacted FA particles. Capillary cracks and microvoid formations were observed, as shown in Fig. 11b, c. Both quartz aggregate and marble powder were detected, as shown in Fig. 11a, b, respectively. In addition to these, Fig. 11b, c shows the inspection of ettringite formation. Figures 12 and 13 show that the fibrous geopolymer composites, coded MK0BF2CF0 and MK15BF0CF2, have a similar microstructure. However, these geopolymer composite mixtures were found to contain a higher amount of unreacted fly ash particles, see Figs. 12b and 13b. Furthermore, upon individual investigation of the fibers in Figs. 12c and 13c, it was observed that the CF particle has a smooth surface, whereas the BF particle has a rough surface. It was also noticed that the geopolymer matrix of fibrous mixtures contains more microvoids than the nonfibrous mixture. On the other hand, the hybrid fiber-reinforced geopolymer composite containing 15% MK has a geopolymer matrix containing a significant amount of ettringite formation and a lot of capillary cracks, see Fig. 13a–c. The matrix of this mixture contained a crack exceeding 6 µm. However, the number of unreacted FA particles was found to be significantly lower than in the other two fibrous geopolymer composite mixtures.

5 Conclusions

The study's results and aforementioned conclusions led to the following conclusions:

- Replacing FA with MK systematically increased the unit weights of geopolymer mixtures. The substitution of MK for FA at a level of 15% led to an increase in unit weight, with a rate of change varying between 2 and 5% across all geopolymer composite mixtures. The greatest increase, amounting to approximately 5%, was observed in the nonfibrous geopolymer composite series. Besides,

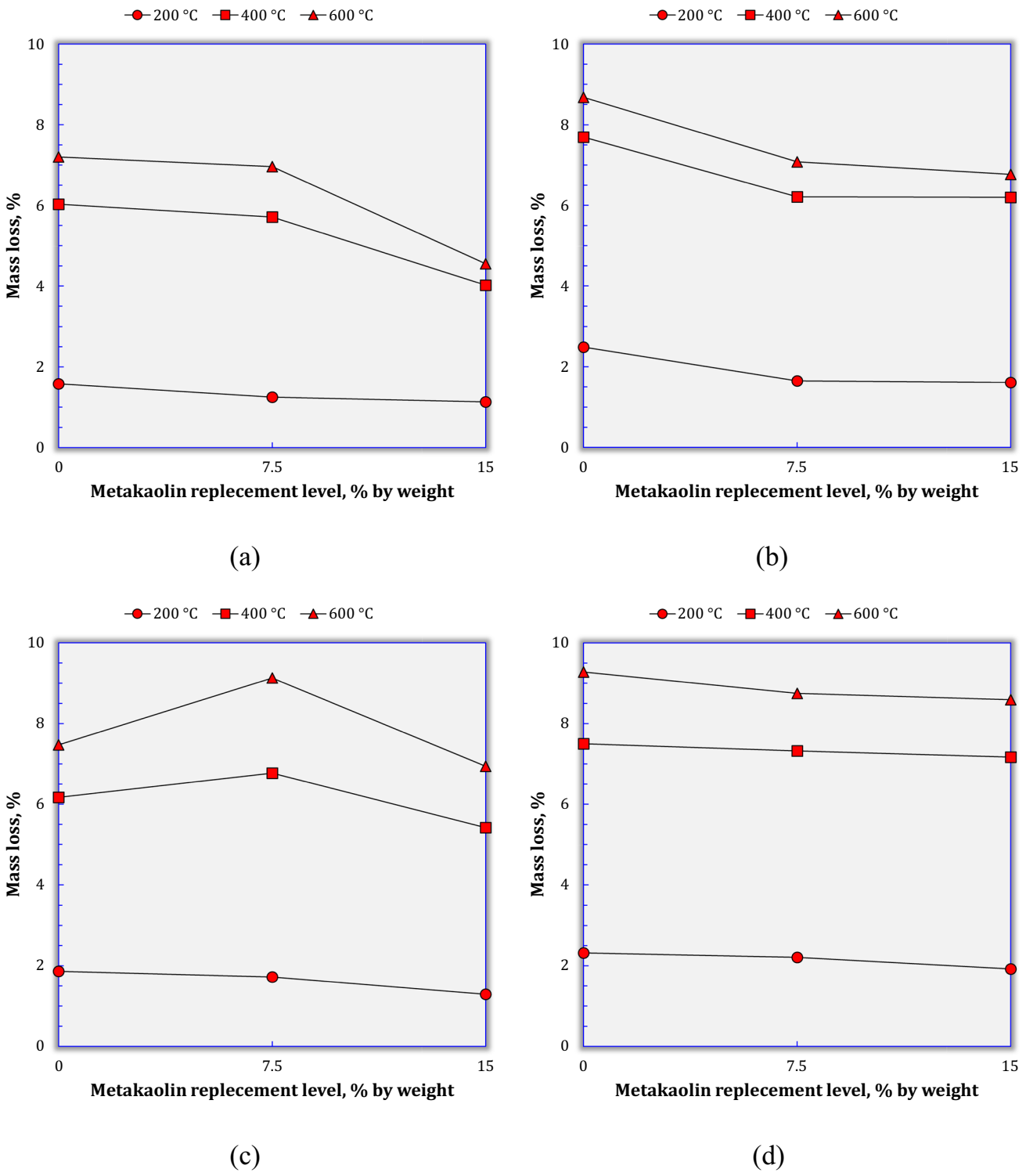


Fig. 9 Weight loss of geopolymer composites exposed to high temperature: **a** nonfibrous series, **b** BF2-CF0 series, **c** BF0-CF2 series, and **d** BF1CF1 series

it was seen that the addition of CF and BF separately or as a hybrid did not lead to very different results and reduced the unit weight in all cases.

- Increasing the MK replacement level decreased apparent porosity owing to the filling effect of the finer MK particles. The substitution of FA with MK at a level of

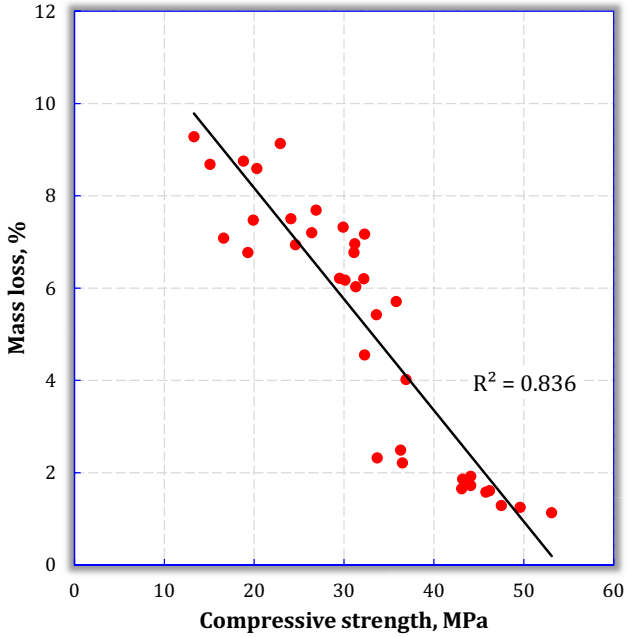


Fig. 10 The relationship between mass loss and compressive strength values after exposure to high temperature

15% led to an observable enhancement in the apparent porosity of the geopolymer composites, with observed increases ranging from approximately 9% to 18%. The most significant contributions to the apparent porosity resulting from the MK replacement were observed in the nonfibrous and hybrid–fibrous geopolymer composite series, with rates of 14.2% and 18.3%, respectively. However, the addition of fiber did not lead to very stable results on apparent porosity.

- Incorporating MK and increasing its replacement level generally caused a slight decrease in water absorption and a significant decrease in capillary water absorption. The average water absorption of geopolymer composite mixtures produced with FA was observed to be within the range of approximately 5.5%–6.0%. In contrast, the water absorption values of geopolymer composite mixtures manufactured with FA and MK at replacement levels of 7.5% and 15% were found to fluctuate between 4.8% and 5.7%, and between 4.7% and 5.1%, respectively. The capillary water absorption of geopolymer composites manufactured with FA was in excess of 3.8%, while those manufactured with FA and MK at a replacement level of 15% exhibited capillary water absorption values of less than 2.5%. On the other hand, fiber addition caused a slight increase in water absorption and an important increase in capillary water absorption, regardless of fiber type.
- MK incorporation and increasing its content led to a systematic increase in the compressive and flexural strengths

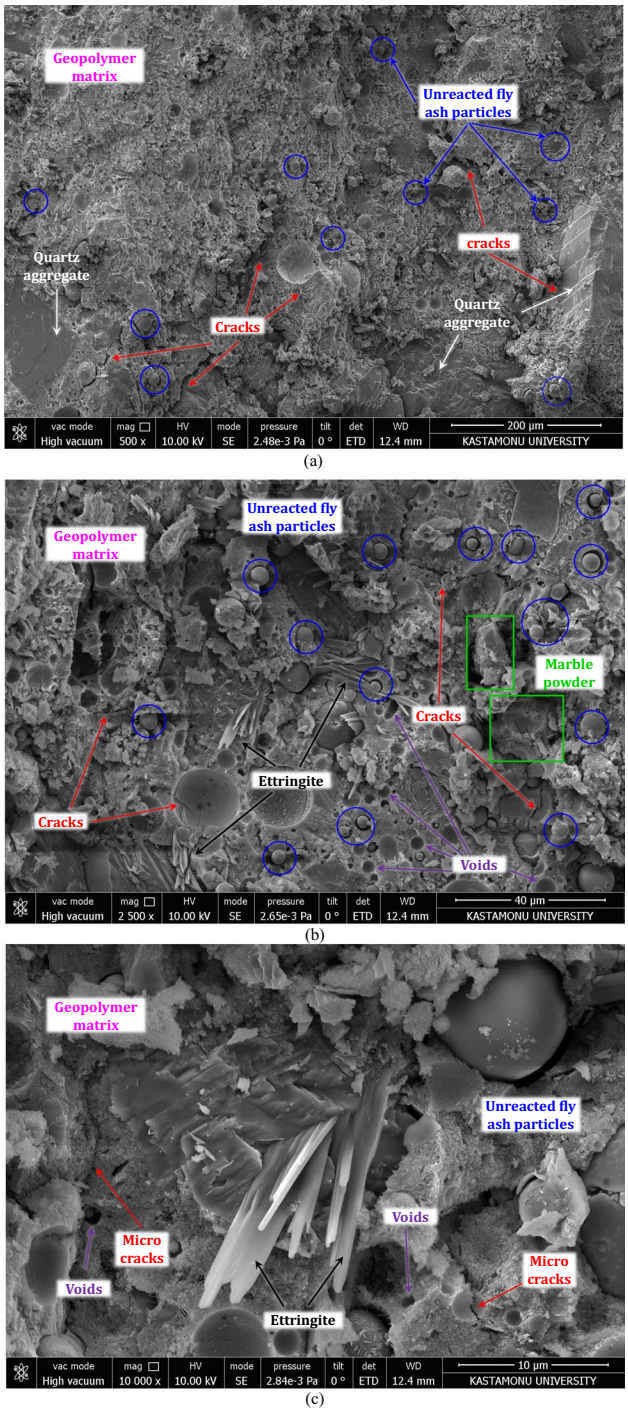


Fig. 11 Microstructure examination of MK0BF0CF0-coded mixture: **a** 500x, **b** 2500x, and 10,000×SEM images

in all mixtures. An increase in the MK replacement level from 0 to 15% resulted in a noteworthy 17.3% enhancement in flexural strength. Geopolymer composites reinforced with CF achieved an impressive increment rate of over 30% when FA was replaced with MK at a level of 15%. The BF and hybrid fiber-reinforced geopolymer

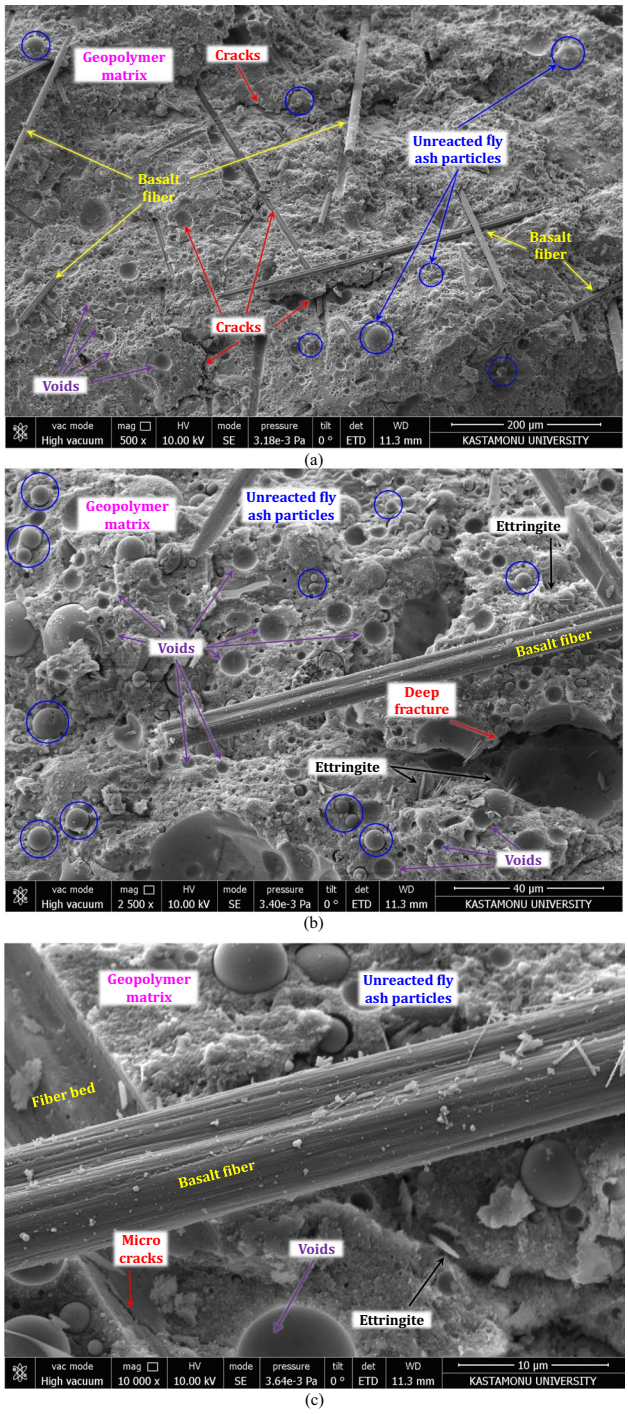


Fig. 12 Microstructure examination of MK0BF2CF0-coded mixture: **a** 500x, **b** 2500x, and 10,000 × SEM images

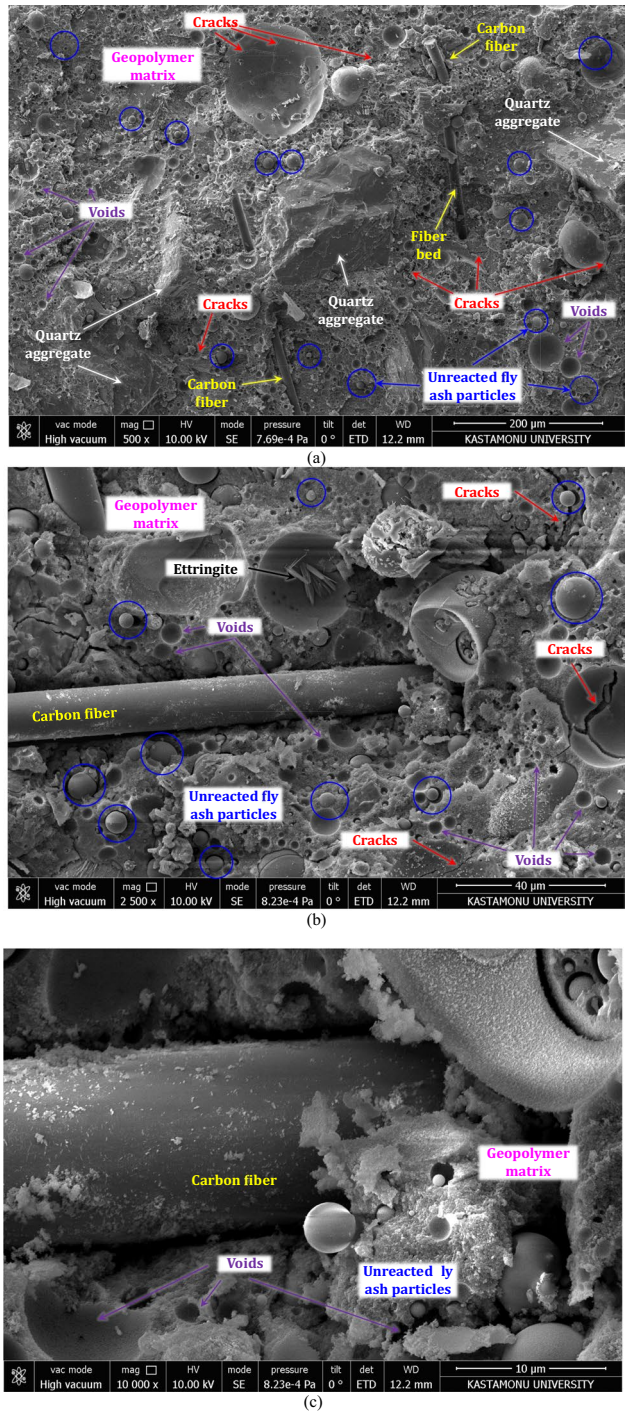


Fig. 13 Microstructure examination of MK15BF0CF2-coded mixture: **a** 500x, **b** 2500x, and 10,000 × SEM images

composites exhibited an increase in flexural strength of approximately 13% with the increase of MK replacement level from 0 to 15%. However, fiber addition reduced the compressive strength, regardless of MK content. On the contrary, fiber addition improved the flexural performance of the geopolymer composites. The highest flexural strength increment was achieved in the geopolymer mixture series reinforced with CF. The highest flexural strength of 11.0 MPa was achieved in the MK15BF0CF2-coded geopolymer composite mixture, while the lowest flexural strength of 6.25 MPa was obtained in the MK0BF0CF0-coded mixture.

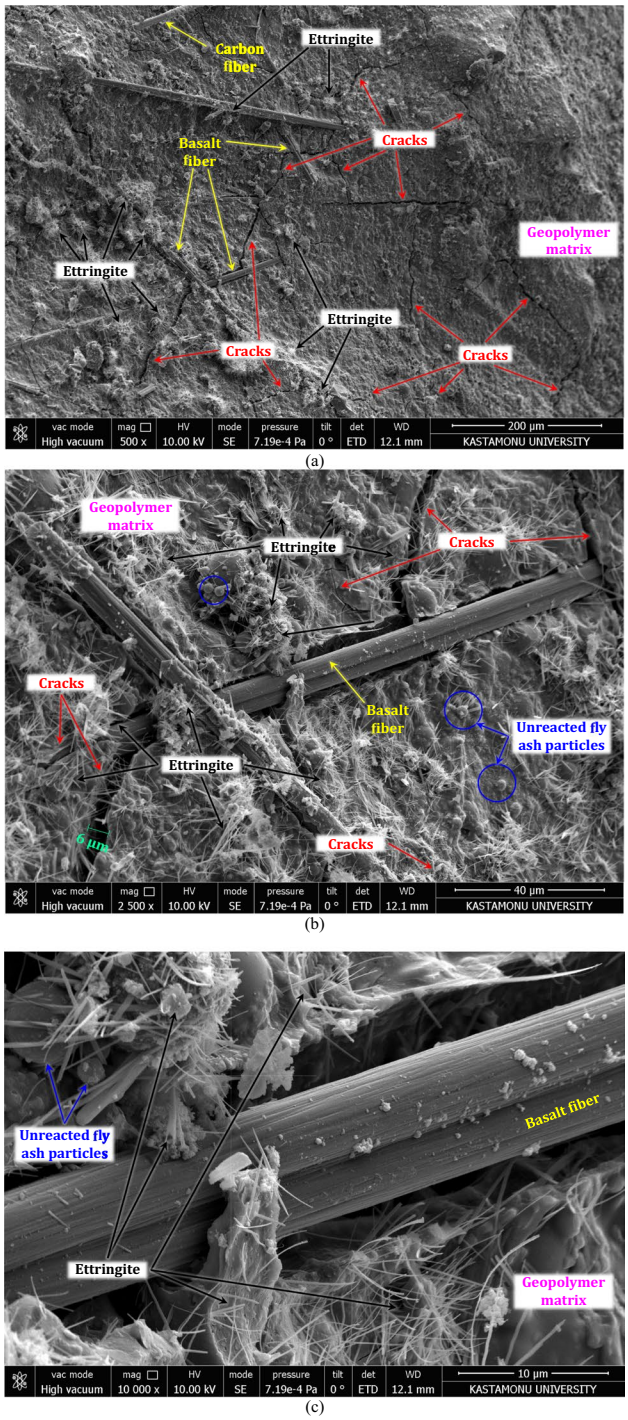


Fig. 14 Microstructure examination of MK15BF1CF1-coded mixture: **a** 500x, **b** 2500x, and 10,000×SEM images

- The application of high temperature to the geopolymer composites increased the compressive strength when the applied temperature was 200 °C. However, further increases in applied temperature resulted in significant decreases in compressive strengths. The compressive strength of the composites subjected to a temperature of 200 °C exhibited an increase of over 10%. In particular, the rate of increment was observed to be approximately 20% higher in nonfibrous composites. The application of a temperature of 400 °C resulted in a loss of strength in the geopolymer composites, with a reduction of approximately 9% observed. A 20% reduction in strength was observed in fibrous geopolymer composites. The investigation revealed that fibrous geopolymer composites exhibited a strength loss of approximately 45–50% when subjected to a temperature of 600 °C, whereas the loss in nonfibrous geopolymer composites was approximately 25%. No remarkable beneficial effect of MK incorporation and fiber addition was observed in the resistance of geopolymer composites to high temperatures in terms of compressive strength loss.
- The geopolymer composites experienced significant mass loss when exposed to high temperatures. The findings revealed that when the composites were subjected to a temperature of 200 °C, they exhibited mass losses that ranged from approximately 1.1% to 2.5%. The MK-blended geopolymer composites exhibited better resistance to high-temperature exposure in terms of mass loss, particularly those without fiber. However, the fiber addition resulted in lower resistance to high temperatures, regardless of the type of fiber.

5.1 Recommendation for future studies

The primary objective of this study was to conduct an empirical investigation into the characteristics of fly ash-based geopolymer composites. Additionally, the effects of metakaolin replacement and reinforcement by diverse fiber types on the physico-mechanical and microstructural properties of geopolymer composites and their high-temperature resistance were examined. For this reason, an environmental impact assessment was not the primary focus of the current study. However, as this is an important aspect of such studies, the authors suggest that researchers engaged with this topic consider the environmental impact of these composites.

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Data availability Some or all data presented in the current study can be provided by request.

Declarations

Conflicts of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in the paper.

Research involving human participants and/or animals The study does not involve human and/or animals.

Informed consent The study does not require an informed consent.

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