

Article

Durability of Non-Heat-Cured Geopolymer Mortars Containing Metakaolin and Ground Granulated Blast Furnace Slag

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Abstract: This study presents the durability, strength and microstructure of non-heat-cured geopolymer mortars (GMs) containing metakaolin (MK), ground granulated blast furnace slag (GGBFS), potassium hydroxide (KOH), sodium metasilicate (Na_2SiO_3), CEN sand and network water. Optimum MK, GGBFS and activator solution ratios were investigated, and the compressive strength of non-heat-cured 28-day GMs reached 55 MPa. Analysis of GMs using scanning electron microscopy (SEM), energy-dispersive X-ray spectrophotometry (EDX) and X-ray powder diffraction (XRD) revealed aluminosilicate formation, potassium from KOH solution and calcium from GGBFS. It showed that the grains containing high silica in the form of quartz crystals were found in the gel formation. The strength and durability of MK- and GGBFS-based GMs exposed to freeze–thawing, a high temperature, wear loss, magnesium sulfate (MgSO_4), sodium sulfate (Na_2SO_4) and HCl solutions were found to be sufficient.

Keywords: geopolymer; metakaolin; ground granulated blast furnace slag; strength; durability



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1. Introduction

Due to the cement used in concrete production, high amounts of carbon dioxide (CO_2) are released into the atmosphere. A solution is sought by substituting waste pozzolanic materials such as fly ash (FA) and ground granulated blast furnace slag (GGBFS) instead of cement [1]. The use of geopolymer mortars (GMs) instead of cement-based materials may reduce CO_2 emissions [2]. Geopolymers can be produced without heat curing, which leads to lower energy consumption and CO_2 emissions. Furthermore, geopolymers cured at ambient temperatures do not cause uniform shrinkage, as seen in Portland cement [3].

Materials produced from the activation of pozzolanic materials using sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium metasilicate (Na_2SiO_3) solutions are called geopolymers [1–4]. Geopolymer formation is generally obtained by changing the chemical structures of minerals, and strength depends on various factors such as the pozzolanic components, curing time and temperature. The following are some of the superior features of geopolymers that can be listed: durability, fast strength and low thermal conductivity.

Geopolymers produced from calcined materials provide better strength than those produced from non-calcined materials. They are also advantageous in terms of durability compared to cement-based materials when interacting with harmful chemicals such as sulfates. Chemical deteriorations in geopolymers are generally determined as shrinkage and expansion. In general, the geopolymer paste shrinks due to the continued dissolution of the precursor to form monomers or small oligomers. The expansions are detected during the formation of zeolite-containing aluminum products.

High-strength geopolymers are produced by homogeneous minerals such as metakaolin (MK). It is formed by sintering kaolin at different temperatures and is often used in GMs due

to its sufficient alumina and silica content. Since very high temperatures are not required for the production of MK, it is inevitable that the amount of CO_2 to be released may decrease [5]. NaOH and KOH as well as Na_2SiO_3 are generally used for the preparation of alkaline solutions in the production of geopolymers [6–8].

GGBFS is a by-product of raw iron production, which generally contains silica, calcium, magnesium and alumina [9]. During the production of one ton of iron, approximately three hundred kg of slag waste is generated [10]. It is used to provide environmental and economic benefits in the production of binder materials for different purposes. Another benefit is its capacity to reduce thermal shrinkage cracks formed during hydration [11]. The setting time, strength and high-temperature resistance of geopolymers produced using GGBFS have an importance in terms of their use in buildings [12]. The increase in the use of GGBFS shortens the setting time in geopolymers [13].

Since the environment in Portland cement is alkaline, it is generally known that ordinary concretes are not resistant to acid. As a result of the acid effect, the hydration products decompose, become melted or disintegrate. In Portland cement products, the weak components are generally $\text{Ca}(\text{OH})_2$ and C-S-H gels. In general, geopolymer concretes are less affected by acid attacks since they contain low $\text{Ca}(\text{OH})_2$ as well as C-S-H gels and polymerization reaction products with a strong alumina-silicate structure [14]. For this reason, geopolymer concretes are more durable under acid than traditional Portland cement products [14,15]. High sodium sulfate (Na_2SO_4) and magnesium sulfate (MgSO_4) content can be found in some groundwaters. Sulphates, which come with the water seeping into the hardened concrete, cause the formation of chemical reactions that lead to expansion and cracking [15,16]. Resistance to sulfate in building materials is generally provided by impermeability and a low C_3A ratio. The absence of C_3A in GMs is an important factor in sulphate resistance [14–16]. For this reason, the sulphate resistance of geopolymer may be higher when compared to Portland cement. The ability of MK to increase sulfate resistance is due to its effect in improving the pore structure and its pozzolanic feature. The fineness of the materials used in geopolymer mortars reduces the porosity in the mortar matrix and provides structural resistance to chemical effects.

Abrasion is a physical and mechanical condition that occurs slowly, usually with forces in the form of friction and impact. It is commonly seen on roads, slab concrete and water structures. In order for concrete or mortar surfaces to be resistant to abrasion, high-strength aggregates and binders must be used. To this end, the abrasion of GMs was investigated with a Bohme disc in this study.

The durability of non-heat-cured MK-based GMs has not been sufficiently studied in the literature. For this reason, in this study, non-heat-cured GMs were produced using MK, GGBFS and alkali solutions to evaluate their strength development and durability. Scanning electron microscopy (SEM), energy-dispersive X-ray spectrophotometry (EDX) and X-ray powder diffraction (XRD) analyses were performed to determine the mechanism and formation of gels that provide strength enhancements in GMs. In addition, conventional characterization techniques using scanning electron microscopy (SEM) and X-ray powder diffraction (XRD) were applied to reveal the detailed phase distribution in the GMs. Test results can help develop a building material with sufficient durability.

2. Materials and Methods

2.1. Materials

Mixtures were prepared using MK, GGBFS, KOH, Na_2SiO_3 and CEN sand, as shown in Figure 1. MgSO_4 and Na_2SO_4 were used in tests to determine the sulfate effect. The specific weights of MK and GGBFS were 2.6 g/cm^3 and 2.9 g/cm^3 , respectively, as shown in Table 1.



Figure 1. Materials: (a) standard sand; (b) KOH; (c) Na_2SiO_3 solution; (d) MK; (e) GGBFS.

Table 1. Chemical properties of MK and GGBFS.

Compound%	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	LOI
MK	50.00	45.00	0.50	1.00	0.50	0.40	0.51	0.24	1.10
GGBFS	40.55	12.83	1.10	0.75	35.58	5.87	0.68	0.78	0.80

The grain size distribution of the sand is shown in Table 2. Na_2SiO_3 and KOH were used in alkaline solutions at 0.65M and 8M, respectively. Molarity values were determined according to the compound ratios, such as CaO/SiO_2 , $\text{Al}_2\text{O}_3/\text{SiO}_2$, $\text{CaO}/\text{Al}_2\text{O}_3$, and the trial mixtures we produced. The chemical properties of Na_2SiO_3 and KOH are provided in Tables 3 and 4. The data sheets for the analysis in Tables 1–4 were obtained from the companies from which the materials were obtained. While MK, KOH and Na_2SiO_3 chemical properties were determined with the XRF analysis technique, the standard sand grain size distribution was determined with the sieve analysis method.

Table 2. Grain size of CEN sand.

Properties						
Sieve size (mm)	0.08	0.16	0.50	1.00	1.60	2.00
Remaining %	99	87	72	34	6	0
Limit	99 ± 1	87 ± 5	67 ± 5	33 ± 5	6 ± 5	0

Table 3. Chemical properties of Na_2SiO_3 solution.

Na_2O (%)	SiO_2 (%)	Density (20 °C, g/mL)	Fe (%)	Heavy Metal (%)
8.75	27.43	1.371	0.005	0.005

Table 4. Chemical properties of KOH.

KOH (%)	K_2CO_3	Cl (%)	SO_4 (%)	NaOH (%)	Fe (%)
92	0.2	0.0031	0.0017	1.0	0.0001

2.2. Methods

Standard sand, water, KOH and Na_2SiO_3 ratios were kept constant, and various ratios of MK and GGBFS were used, as seen in Table 5. Mixture ratios were determined by producing trial mixtures, examining literature data and based on compound ratio values in Table 5. In order for the alkaline solution to be homogeneous, KOH and Na_2SiO_3 were prepared by mixing them with tap water at 50 °C in a closed container to prevent evaporation. When determining the mixing water ratio, calculations were made by deducting the excess water in the Na_2SiO_3 solution from water mixing. For strength and abrasion tests, 108 specimens were produced in $40 \times 40 \times 160 \text{ mm}^3$ and $50 \times 50 \times 50 \text{ mm}^3$ dimensions

in accordance with TS EN 196-1 and ASTM C 267-01 standards [17,18]. Specimens were stored uncovered in laboratory conditions for 28 days, as shown in Figure 2. After curing, unit weight, compressive and flexural strength were obtained on the GMs. In addition to durability tests (such as sulfate and acid effects, freeze–thawing, high temperature and abrasion), SEM, EDX and XRD analyses were performed.

Table 5. Mixing (%weight) and compound ratios of GMs.

Mix	No	MK	GGBFS	Water	KOH	Na ₂ SiO ₃ Solution	Sand	CaO/SiO ₂	Al ₂ O ₃ /SiO ₂	CaO/Al ₂ O ₃
MK-GGBFS	1	9.1	13.7	9.6	4.5	1.5	61.5	0.48	0.55	0.88
	2	6.8	16.0	9.6	4.5	1.5	61.5	0.57	0.48	1.20
	3	4.5	18.2	9.6	4.5	1.5	61.5	0.67	0.40	1.65
	4	2.3	20.5	9.6	4.5	1.5	61.5	0.77	0.33	2.33



Figure 2. Twenty-eight-day non-heat-cured GMs.

SEM analyses were performed with a JEOL JSM 5600 brand SEM device (JEOL, Tokyo, Japan). In the EDX analysis, X-ray mapping and quantitative elemental analyses were performed in selected areas. A gold/palladium coating device operating under high vacuum was used to prepare samples for the analysis of insulating samples.

The crystal structures of solids were examined, and the phases in their content were determined with the XRD (X-ray diffraction) method. X-ray scattering techniques do not destroy the sample and do not change the properties of the examined sample. The coarse sample was ground with a tungsten carbide cobalt (WC-Co) ring grinder to below ~63 µm. The sample was then kept in a ~105 ± 5 °C oven for 4 h to dry. The samples were prepared according to the appropriate method and measured with a Rigaku brand Miniflex 600 model XRD device (Rigaku, Tokyo, Japan) and in the standard scanning range (2θ = 5° – 70°).

2.3. Durability of Non-Heat-Cured GMs

In durability tests, GMs were kept in 5% Na₂SO₄, 5% MgSO₄, 1.5 M and 2 M HCl solutions for 10 weeks. The solutions were renewed every 2 weeks during the experiment by adding water or chemicals, as seen in Figures 3 and 4. At the end of this period, the specimens removed from the solutions were left to dry in the laboratory for 24 h; then, flexural and compressive strength tests were performed according to TS EN 196-1 standard [17]. In this study, freeze–thaw and high-temperature resistance of specimens were investigated, as seen in Figures 5 and 6. Freeze–thaw tests were carried out in an automatic freeze–thaw cabinet at +20 °C and –20 °C at 6 h intervals in 60 cycles for 30 days. High-temperature tests were carried out in furnaces at 600 °C–800 °C. The temperature was increased by 10 °C per minute and held constant for 1 h when the target temperature was reached. The specimens were then removed from the oven and allowed to cool to 20 °C. Flexural and compressive tests were carried out after calculating unit weights.

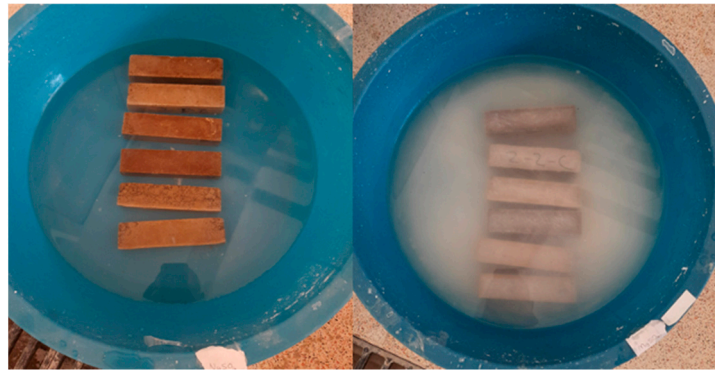


Figure 3. Non-heat-cured GMs held in 5% Na_2SO_4 and 5% MgSO_4 solutions.



Figure 4. Non-heat-cured GMs held in HCl solutions.



Figure 5. Freeze–thaw tests of non-heat-cured GMs.



Figure 6. High-temperature tests of non-heat-cured GMs.

The abrasion of GMs was investigated with the Bohme test according to the TS 2824 standard, as shown in Figure 7. Cubic specimens were subjected to 16 cycles of 22 turns, removing sanding dust and freshening them after each cycle. The total volume losses were determined by measuring the specimens with a caliper.



Figure 7. Abrasion test of non-heat-cured GMs.

3. Results and Discussion

Strength, durability and microstructure of GMs were investigated in this study. Initially, the strength of GMs was determined on control specimens. Then, strength and weight losses of GMs, which were exposed to sulfate and acid solutions for 10 weeks, were determined. Additionally, SEM, EDX and XRD analyses of GMs were performed. According to the results, sufficient strength of GMs revealed that GMs may be produced without being heat-cured.

3.1. Influence of Sulfate, Acid, Freeze–Thawing and High Temperature on the Weight Loss of GMs

Weight losses were determined by measuring the samples on a precision balance before and after exposure to sulfate, acid and freeze–thawing and by calculating the weight difference. The increase in the GGBFS ratio used in the mixtures caused an increase in the unit weight of the GMs, as seen in Figure 8. This is explained by the fact that the GGBFS unit weight is higher than the MK unit weight and as the dense structure formed as a result of better alkali activation of fine particles. Thus, the unit weight of the control specimens varied between 2.1 and 2.3 g/cm³ for 2MK-GGBFS and 4MK-GGBFS, respectively. When the specimens kept in sulfate solutions were compared with the control specimens, small decreases were observed in the unit weights depending on the mixing ratios, while higher decreases were observed in unit weights of the specimens kept in the acid solution. This indicates that GMs are not significantly affected by sulfate solutions. As seen in Table 6, a lower weight reduction was detected in specimens with high MK content. This showed that alkali activation and gel structures were better in mixtures containing high MK content. Weight loss rates increased as the amount of MK of GMs retained in Na₂SO₄ and MgSO₄ solutions decreased. No greater change was observed in the weight loss rates of the specimens kept in the acid solution. This is explained by the absence of C₃A in GMs.

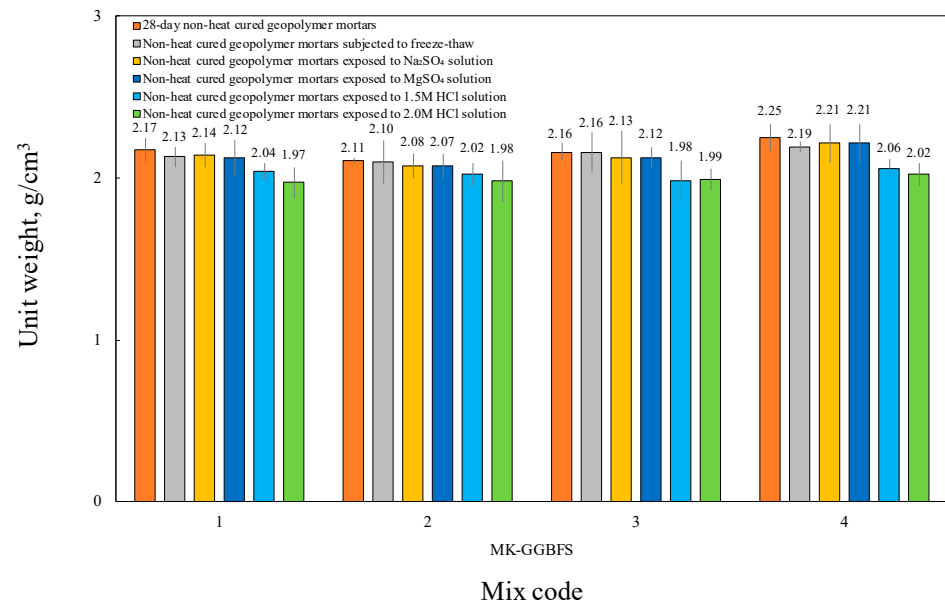


Figure 8. Unit weight of GMs exposed to sulfate, acid and freeze–thawing.

Table 6. Weight loss of GMs exposed to sulfate, acid and freeze–thawing.

Mix Code	No	Weight Loss (%)				
		Na ₂ SO ₄	MgSO ₄	2 M HCl	1.5 M HCl	Freeze–Thawing
MK-GGBFS	1	1.3	2.2	9.2	5.9	1.8
	2	1.6	1.9	6.1	4.2	0.5
	3	1.6	1.7	7.9	8.3	0.0
	4	1.7	1.7	10.2	8.4	2.6

As shown in Figure 9, there was no significant change in the unit weights of the GMs after freeze–thawing. This is due to the sufficient strength of the GMs. Also, unit weights generally decreased under the influence of high temperature; however, at 800 °C, the losses were even greater as seen in Table 7. The reason for the weight decreases due to the high temperature is explained by the removal of water in the mortar structure and the deterioration of the gel structure.

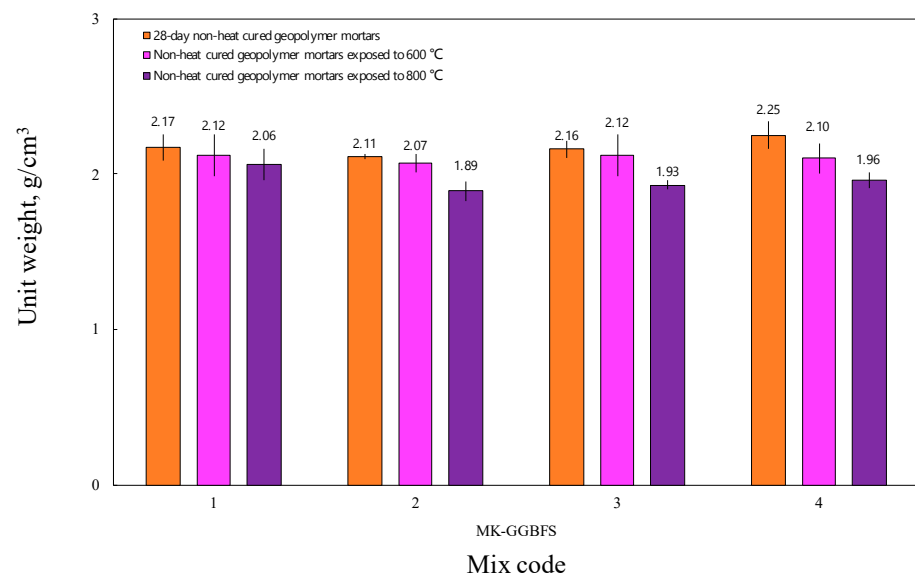


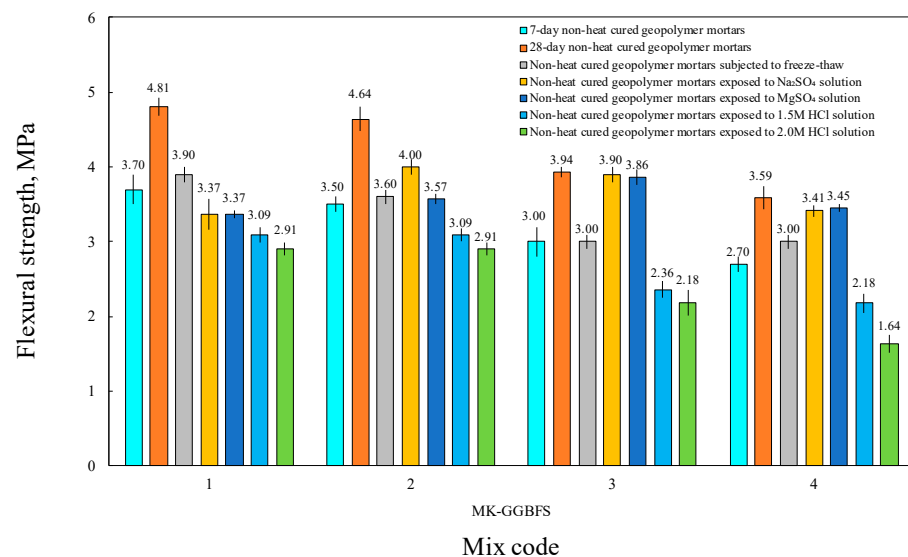
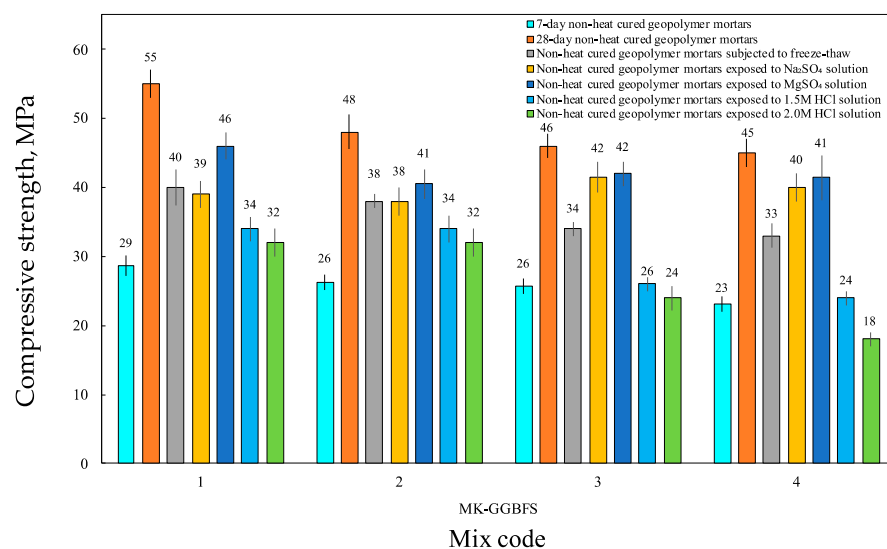
Figure 9. Unit weight of GMs exposed to high temperature.

Table 7. Weight loss of GMs exposed to high temperature.

Mix	No	Weight Loss (%)	
		600 °C	800 °C
MK-GGBFS	1	2	5
	2	2	10
	3	2	11
	4	7	13

3.2. Influence of Sulfate, Acid and Freeze–Thawing on the Strength of GMs

As seen in Figures 10 and 11, the 28-day flexural strength of GMs is between 3.5 and 5.0 MPa, while the compressive strength is between 45 and 55 MPa. The lowest and highest strengths were seen in 4MK-GGBFS and 1MK-GGBFS, respectively. The decrease in the MK ratio and the increase in the GGBFS ratio caused a decrease in strength. This is explained by the effect of changing the mixing ratio of two different pozzolans to the chemical composition ratio required for alkali activation.

**Figure 10.** Flexural strength of GMs exposed to sulfate, acid and freeze–thawing.**Figure 11.** Compressive strength of GMs exposed to sulfate, acid and freeze–thawing.

Strength losses were determined in GMs under the influence of acid, sulfate and freeze–thawing. Decreases in compressive and flexural strengths were observed in sulfate, acid and freeze–thaw exposure depending on the mixture components. Lower strength losses were observed in GMs with high MK content. Thus, MK had a positive effect against acid and sulfate, forming strong bonds in the gel matrix. The stability of GMs in the acidic solution depended on the crystal phase formation in aluminosilicate. This resulted in the formation of more crystalline phases. The morphology of the GMs was also effective in the resistance of the binder to the acidic solution. The low calcium content of the GMs was one of the main factors that provided durability. The active calcium in GGBFS dissolved, causing the gel phase to transform into C-A-S-H gel. This resulted in an increase in strength, providing a better void filling capacity than N-A-S-H. The formation of the compact C-A-S-H gel phase was associated with the amount of active calcium [19–22]. As the amount of GGBFS increased, the unreacted phases increased, resulting in a slight decrease in strength. Thus, unreacted particles in GGBFS provided an appropriate level of filling effect while damaging the entire structure when used in high doses [23,24]. According to the freeze–thaw test results, the strength loss was higher in the GMs with high GGBFS content. The high MK content led to high geopolymerization and, as a result, a denser internal structure. Due to the highly active pozzolanic property of MK and the fine structure of GGBFS, GMs provided positive results against freeze–thaw exposure.

3.3. Influence of Abrasion on the GMs

As seen in Figure 12, wear losses in GMs varied between $3 \text{ cm}^3/50 \text{ cm}^2$ and $12 \text{ cm}^3/50 \text{ cm}^2$. As the amount of MK decreased, the wear loss increased. The lowest wear loss was observed in 1MK-GGBFS, and the highest wear loss was observed in 4MK-GGBFS. Abrasion resistance showed a direct relationship with the strength of GMs and increased as the strength increased.

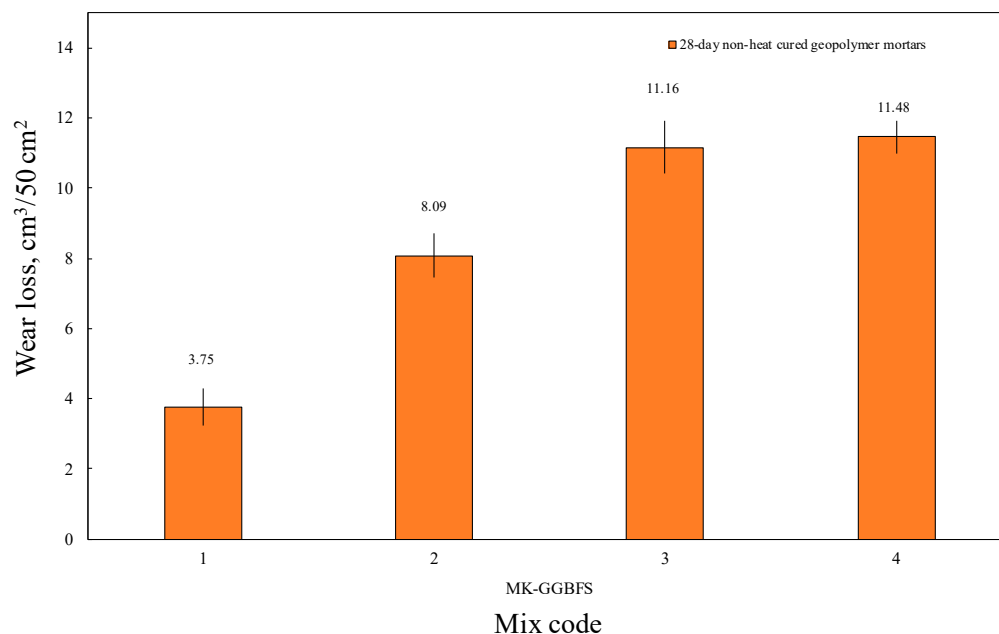


Figure 12. Wear loss of GMs exposed to abrasion.

3.4. Influence of High Temperature on the GMs

As seen in Figures 13 and 14, the compressive strength of GMs exposed to 600°C decreased by 27%, 25%, 5% and 60%, while their flexural strength decreased by 50%, 33%, 24% and 74%, respectively. The compressive strength of GMs exposed to 800°C decreased by 70%, 86%, 82% and 80%, while their flexural strength decreased by 74%, 82%, 76% and 86%. The low loss of strength is explained by the fact that non-heat-cured GMs complete

their curing with the effect of high temperature. Significant decreases in strength occurred at 600 °C and 800 °C. According to the previous studies, cracks generally formed due to the effect of a high temperature, creating large voids, with strength decreasing due to voids formed in the matrix [12,16]. A dehydration reaction occurred in GMs at a high temperature and humidity. This resulted in disruptions of the microstructure and an eventual loss of the GMs' mass.

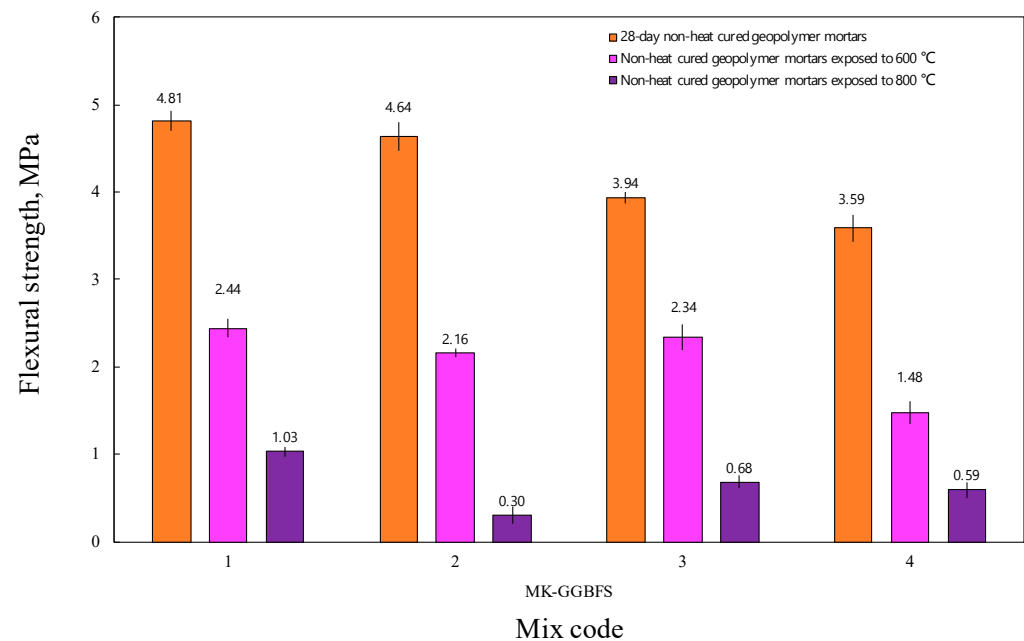


Figure 13. Flexural strength of GMs exposed to 600 °C and 800 °C.

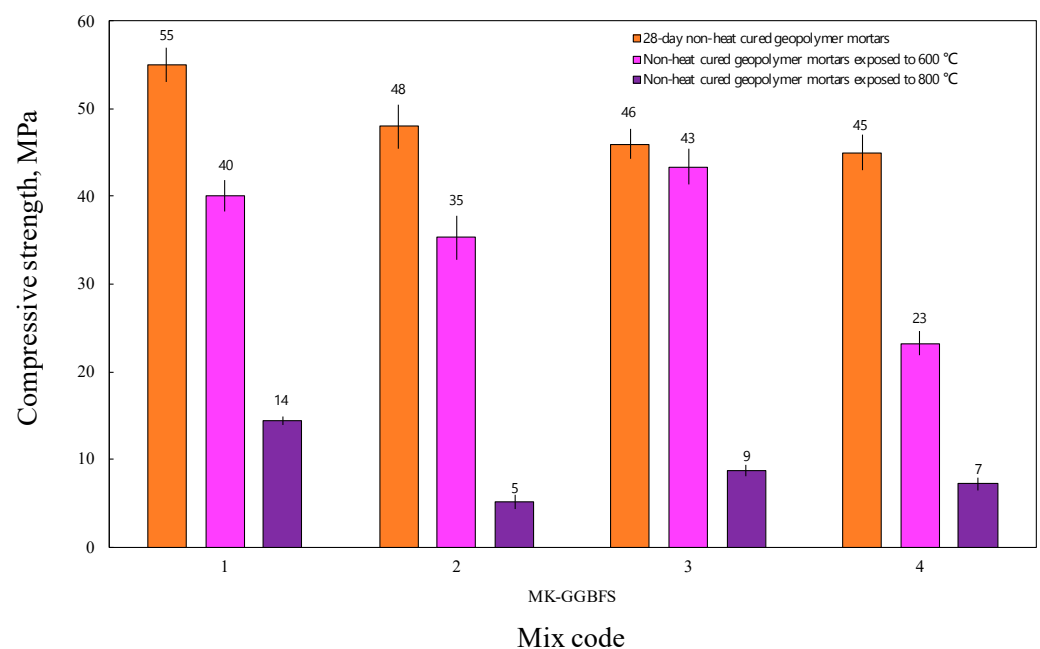


Figure 14. Compressive strength of GMs exposed to 600 °C and 800 °C.

3.5. SEM and XRD Analyses of GMs

In the study, SEM, EDX and XRD analyses were carried out on the samples with the highest and lowest GM strengths. It was observed that the homogeneous, dense and continuous matrices and grains, usually composed of aluminosilicate gel, came together in agglomeration. Thus, stratified grains were found together, and gaps were observed

between grains. Furthermore, binder liquid phases were determined in the intermediate regions. As seen in Figures 15 and 16, the internal structure of GMs was quite dense, and this showed that the strength test results were compatible with each other and that the aggregate–paste interface was sufficient. MK and GGBFS caused high geopolymerization and formed layered structures in different regions. It was observed that the different particle sizes of the materials entering the mixture affected geopolymerization. Thus, the high MK content in the mixture caused a denser internal structure.

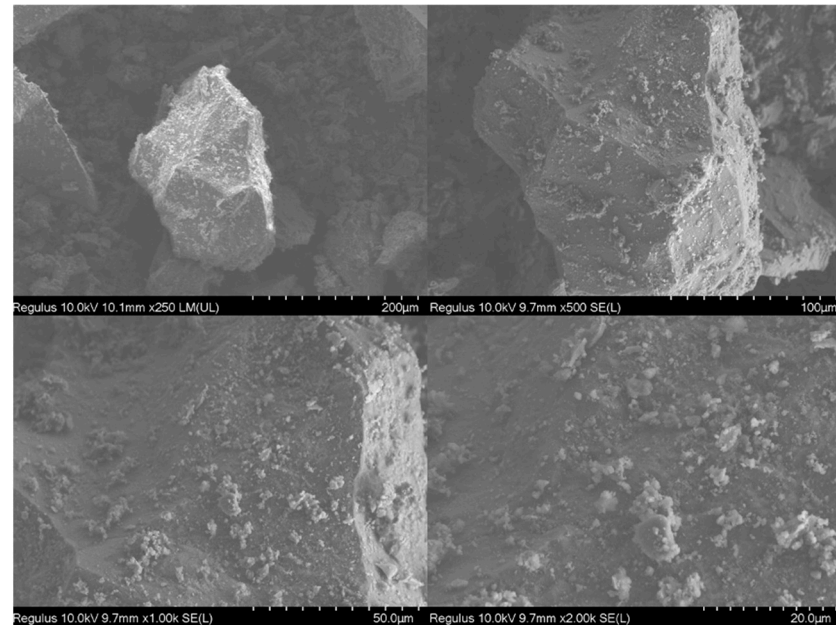


Figure 15. SEM analysis of GMs (1MK-GGBFS).

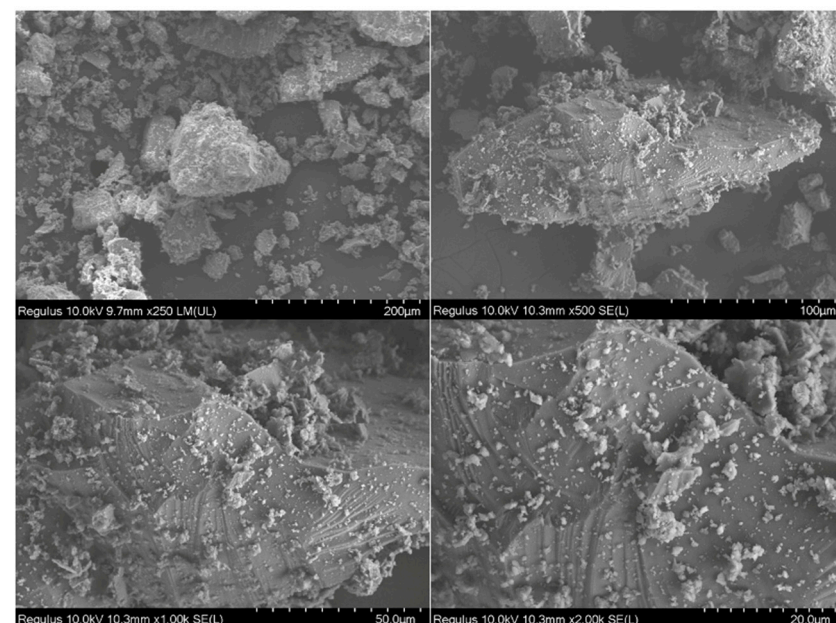


Figure 16. SEM analysis of GMs (4MK-GGBFS).

Since the highest strength was obtained in 1MK-GGBFS, the densities in the internal structure supported this situation. The void ratio in the internal structure was higher, and this situation had emerged as a decrease in strength. In general, the morphology of the samples was denser, and the porosity decreased with the increase in the MK ratio. It was

observed that KOH and Na_2SiO_3 with constant molarity contributed to the formation of the reticulated structure formed during geopolymerization. Furthermore, according to the formation of cracks, it was noted that 1MK-GGBFS was in a better condition than the other samples.

EDX analysis was carried out to obtain the elemental formation of GMs' gel structures, as shown in Figures 17 and 18. It was found that there was a predominantly aluminum, silica-based formation; the presence of potassium due to the KOH solution used; and calcium originating from the GGBFS. Silicon content was higher in all regions selected in the EDX analyses when the sample was examined as both an oxide and compound. The fact that the potassium oxide and aluminum oxide ratios were not very variable indicates that there was a reaction around the silicon particles. High potassium content was an indicator of geopolymerization, which mostly occurs on the surface.

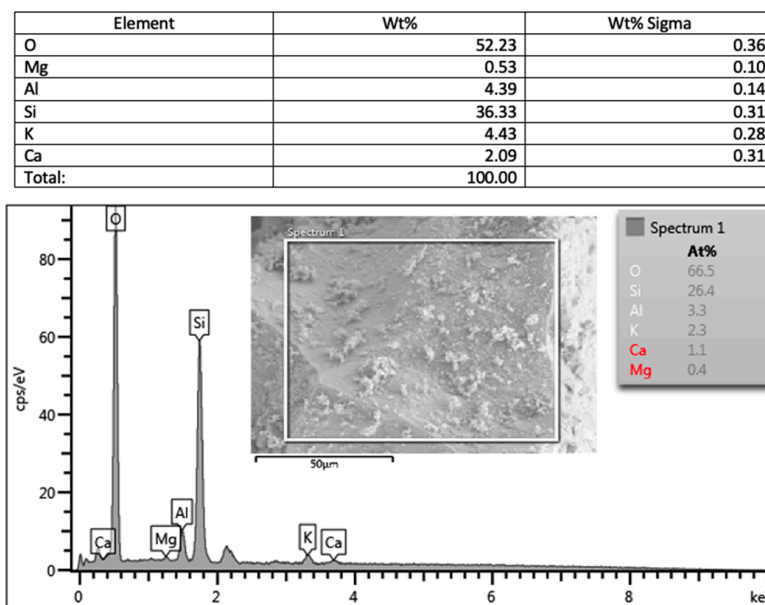


Figure 17. EDX analysis of GMs (1MK-GGBFS).

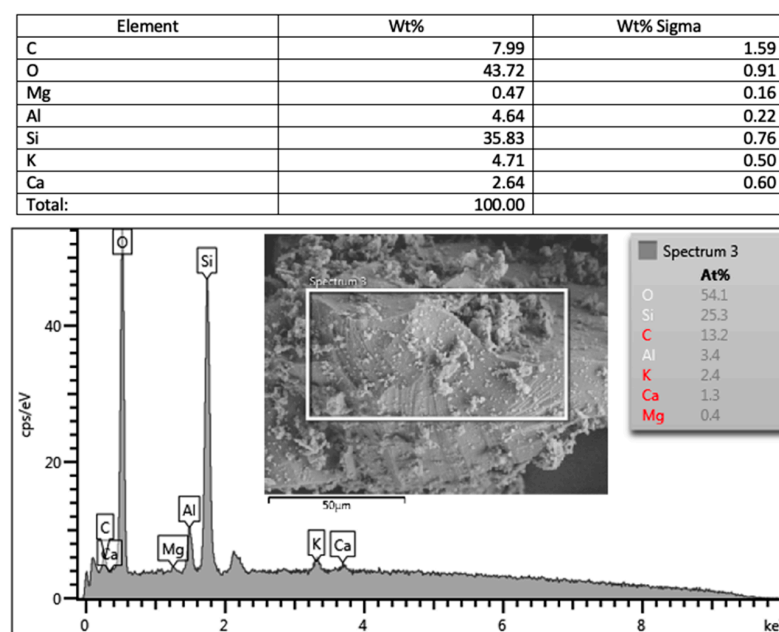


Figure 18. EDX analysis of GMs (4MK-GGBFS).

As can be seen in Figure 19, the phases in the solids were observed by examining the crystal structures with the XRD method. X-ray scattering techniques were used on samples. According to the XRD analysis, quartz, albite, leusite, lycetite, calcyan and aluminum silicate minerals were observed in the samples. The highest peaks were seen in quartz, silica and albite.

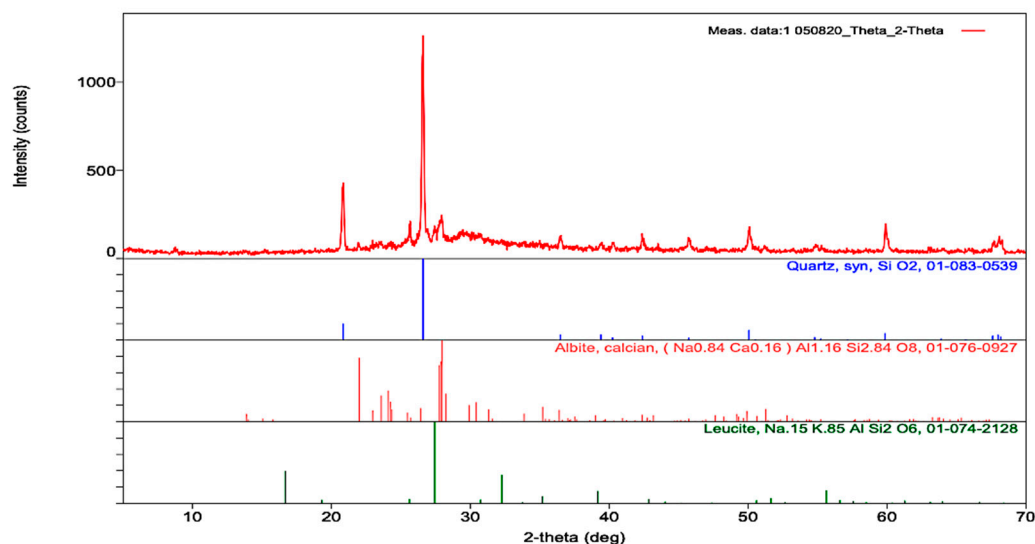


Figure 19. XRD analysis of GMs (1MK-GGBFS).

4. Conclusions

In this study, the use of GGBFS in alkali-activated MK materials was investigated. Strength, durability and characterization tests were conducted to obtain the effect of GGBFS and MK for geopolymerization. The results revealed that GMs gained a homogeneous and dense appearance due to the good adhesive properties of MK and GGBFS. Higher MK content accelerated the reaction rate, while adequate strengths were obtained in non-heat-cured GMs. The increase in GGBFS content led to a decrease in strength. This was explained by the insufficient ratio of the chemical composition required for activation.

No color change or crack formation was detected in GMs kept in the sulfate and acidic solutions. Considering the exposure times to sulfate and acidic solutions, strength and weight losses were acceptable. Strength loss was found to be lower in GMs with higher MK content. Furthermore, the resistance to freeze–thaw exposure and abrasion increased with the increase in MK content. However, the wear losses in all specimens remained below the values predicted by the standards.

The reason for the low strength losses of GMs kept at 600 °C was that the high temperature created an additional curing effect in non-heat-cured GMs. While the strength losses were determined to be higher in GMs at 800 °C, it was revealed that GMs with high MK content were more resistant to a high temperature.

EDX analysis showed that there was a predominant aluminum, silica and potassium presence in the samples due to the KOH solution used and due to calcium originating from the GGBFS. In the internal structure, it was seen that the particles with high silicon content were quartz and that they were found in the gel structure.

SEM analysis revealed a dense and continuous internal structure composed of aluminosilicate gel, with the grains adhering to each other in the form of agglomeration. The stratified grains were close together, and there were small gaps between the grains. Binder liquid phases were determined in the intermediate regions.

According to the XRD graphs, the presence of quartz, albite, leusite, lycetite, calcyan and aluminum silicate was observed in the GMs. The highest values were seen in quartz, and albite was found in all GMs.

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Conflicts of Interest: The authors declare no conflict of interest.

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