

Influence of Metakaolin Replacement on the Early Compressive Strength of Heat-Cured Fly Ash–Based Geopolymer Mortar

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Abstract

Geopolymer materials have gained significant attention as sustainable alternatives to traditional concrete, as they reduce carbon dioxide emissions associated with the cement production industry. This environmentally friendly material consists of industry waste materials that have been chemically activated by an activation solution. This study attempts to evaluate the effects of replacing metakaolin (MK) in fly ash (FA)-based geopolymer mortars with sand at 10%, 20%, and 30% with an emphasis on early strength within the first few hours. Na₂SiO₃ (2:1) and 12 M NaOH were combined to create an alkaline activator. After being formed into 50 x 50 x 50 mm cubes, the samples were first heat-cured at 90°C for initial durations. At two, five, and twenty-four hours, the bulk density and compressive strength were measured. The results showed that density gradually decreased over time, and that replacing some of the sand with MK significantly increased both early and later compressive strengths while initially reducing density. The increased active aluminosilicate content and quicker N-A-S-H gel formation are responsible for this improvement. MK20 showed the best workability, microcompactness, and reactivity. On the other hand, because of its higher viscosity and potential for microporosity, MK30 was marginally weaker than MK20 but showed greater strength in comparison to the reference. The results support the use of medium replacement proportions of MK in applications that require both structural stability and quick strength growth

Keywords: Geopolymer; Fly ash; Metakaolin; Heat curing; Early compressive strength.

Article history: Received: 21 Jan. 2026, Accepted: 20 Feb. 2026, Published: 20 March 2026

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1. Introduction

Due to growing environmental concerns about the production of ordinary Portland cement (OPC), the construction materials industry has evolved in recent decades toward more sustainable, lower-carbon cementing chemicals [1]. Geopolymers are one of the most popular alternative building materials. They produce a three-dimensional network of Si–O–Al–O connections using alkaline-based polymerization techniques using materials rich in silica and alumina [2]. These materials are being investigated for a new type of ecologically friendly mortar that exhibits remarkable mechanical strength and resistance to chemical and thermal stresses, all while significantly reducing carbon dioxide emissions [3]. Metakaolin (MK) is one of the most complex and reactive aluminosilicates that can be used for geopolymerization. It

is produced by calcining kaolin at temperatures ranging from 650 to 800°C, which disrupts the crystalline structure and turns it into an extremely reactive amorphous state [4].

The Synthesis of the N-A-S-H gel, which is required for the robustness and longevity of geopolymer systems, depends on alumina and silica, two important metakaolin constituents [5]. For geopolymer mortars to perform better, particularly in terms of early compressive strength, the proportion of metakaolin in the binder is crucial [6]. A higher concentration of metakaolin promotes the alkali reaction and the creation of geopolymer gel, which gives the material a noticeable early strength that may be observed during the first few hours of curing [7, 8]. This feature is crucial for applications that need to increase strength quickly, including structural repairs or the rapid industrial production of precast parts [9,10]. On the other

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hand, excessive metakaolin addition may lead to increased viscosity and decreased workability, which may be detrimental to final strength and structural homogeneity [11]. In order to provide better mechanical performance in conjunction with a low-porosity microstructure, it is therefore essential to strike the perfect balance between metakaolin content, workability, and hardening rate [12].

Many attempts were performed using metakaolin, fly ash, and different curing methods to obtain early high compressive strength; A. Nadarajah et al. designed a fly ash-GGBS-based geopolymer mortar using various blended mixtures. The results presented that high-volume content (60–80%) yielded an encouraging early-age compressive strength at room temperature. Metakaolin provided a further strength enhancement in geopolymer mortar, while high FA content is suitable for a repair mortar [13]. Another study achieved by Al-Dahash and Humad, which taking in to account the two-time period first one was at 7 days while another was at 28 days to investigate the compressive strength variation with curing time of geopolymer mortars composed of fly ash and metakaolin. Their results determine that the early-age compressive strength is enriched by considering heat-curing technique when it compared to curing at room temperature. The study concludes that in the case of including 40% of the fly ash with metakaolin, the matrix structure was enhanced with a reduction in fishers and tiny cracks [14].

Statkauskas et al. [15] studied the effect of curing temperatures on fly ash and metakaolin geopolymer mortars with bemban fiber. A hybrid geopolymer mortar was produced using a NaOH concentration of 8M. After casting, heat curing was conducted at 80°C and 100°C for 12 hours in the oven. The samples were then treated at room temperature. They concluded that the maximum compressive strength (29.9 MPa) was obtained at a temperature of 100°C [15]. A work conducted by Yilmaz et al. which aimed to evaluate the mechanical properties of low-calcium fly ash-based geopolymer mortars. The study considered various curing times at several temperatures. Two conditions were considered, condition one conducted at room temperature (20±3°C). However, the other condition included several air temperatures, which were conducted at 40°C, 60°C, and 80°C for 24 h, 48 h, and 72 h, respectively, after that, normal curing criteria were applied. It has been noticed that from the curing results, the compressive strength improved by taking into account the 72 h [16]. In the same field of study, a work conducted by Mohammed and Fawzi, who used 70% fly ash and 30% metakaolin to produce a geopolymer mortar. Moreover, the work includes 14 M NaOH, varied with sodium silicate in a 2.5:1 percentage. The curried temperature that was assumed in this work was 60°C in an oven for 24 hours for the formed samples. The results obtained from the study showed that there is an enhancement reached by 59% and 55% in compressive strength values. It is worth mentioning that curing times were after 7 and 28 days [17].

The novelty of this study lies in investigating the impact of metakaolin content on the mechanical

characteristics of geopolymer mortar during the early hours of curing in order to understand the relationship between the early reactivity of MK and the pace of strength development during the early stages of hardening.

Therefore, finding the ideal balance between metakaolin %, workability, and hardening rate is crucial for ensuring improved mechanical performance while retaining a low-porosity microstructure. This study aims to examine the effects of varying metakaolin fractions on the mechanical properties of geopolymer mortar in the initial hours of curing. The goal is to clarify the relationship between metakaolin's early responsiveness and the pace of strength development in the early stages of hardening.

Methodology

Used materials

To guarantee accurate results and stable qualities during preparation, carefully chosen raw ingredients were utilized. Fig. 1 illustrates the usage of fly ash (FA) as the main source of aluminosilicate in the geopolymer mortar. When activated with alkalinity, it contributes to the production of a (N–A–S–H) gel since it is a low-calcium type (F), which offers it chemical stability and mild reactivity. Sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) solutions were combined in a 2.3:1 ratio with a 12 M NaOH concentration to create the alkaline activator.

To provide the necessary alkaline activator, the NaOH solution was made 24 hours before to mixing in order to guarantee its chemical and thermal stability. It was then combined with the silicate solution. The fine aggregate used is air-dried natural sand and was used at a constant volume ratio of 70:30 with fly ash to achieve adequate cohesion and reduce porosity. To improve workability and reduce water demand in mixes with metakaolin content, a superplasticizer was used with a ratio of 1.5% by weight of fly ash. It was added during mixing to ensure homogeneous distribution of components and increase flowability without affecting early setting time.

Finally, metakaolin (MK) was used as the primary source of alumina and silica in some mixes. The metakaolin used has 40–45% aluminum oxide (Al₂O₃) and 60–55% silicon oxide (SiO₂), making it an effective material for accelerating geopolymer reactions and improving mechanical resistance in the early hours of curing.



Fig. 1 Used fly ash

Mix proportions

Standard mixing ratios were adopted as shown in Table 1. Metakaolin (MK) was replaced by 10%, 20%, and 30% by weight of sand, while the alkaline activator ratio and plasticizer dosage were kept constant. A 12 M sodium hydroxide solution was prepared 24 hours in advance and mixed with sodium silicate at a 2:1 ratio to form the alkaline activator. The solid components (fly ash (FA) and sand, along with metakaolin MK0/MK10/MK20/MK30) were dry mixed for 2–3 minutes in a mechanical mixer. The alkaline activator was then gradually added, continuing to mix for 4–5 minutes until a homogeneous consistency was achieved. The superplasticizer was added as part of the liquid addition phase to adjust workability without affecting early setting time. The mortar was poured into 50×50×50 mm steel molds in two layers with light compaction to remove entrapped air and level the surface as shown in Fig. 2. The samples were then kept inside the molds and transferred directly to a curing oven at 90°C for 5 hours as shown in Fig. 3. Compressive strength and bulk density tests were performed at ages 2 h and 5 h directly after curing, while the remaining specimens were stored at 23 ± 2°C and tested at 24 h. The mass of each cube was documented before testing to verify mixing and curing consistency between different mixes.

Table 1: Mix proportion of geopolymer mortar

Mix	MK%	FA	Sand	MK	Na ₂ SiO ₃	NaOH
MK0	0	1217.3	521.7	0	308.6	134.2
MK10	10	1217.3	469.5	52.2	308.6	134.2
MK20	20	1217.3	417.4	104.3	308.6	134.2
MK30	30	1217.3	365.2	156.5	308.6	134.2



Fig. 2 Preparation of Sample



Fig. 3 Samples under heat curing at 90°C

Results

Bulk density

Fig. 4 and Table 2 show the bulk density results at 2, 5, and 24 hours, showing a gradual decrease in density over time under the early heat-curing regime. This behavior can be explained by two simultaneous mechanisms: the first is related to the loss of a portion of free water and the rise in the mortar temperature inside the oven to 90°C during the first hours, which leads to a decrease in the sample mass after heating is complete and a subsequent decrease in temperature; the second is related to the development of the microstructure and the Synthesis of an early N–A–S–H gel, where structural coalescence is accompanied by the removal of fine trapped air bubbles in the first minutes, and the remaining pores are then stabilized as the matrix hardens [18]. Therefore, the density at 5 hours appears lower than at 2 hours. The reference mix MK0 starts with the highest density at 2h (2.22 g/cm³) with a relatively limited decline until 24h (-4.95%).

As sand is replaced by metakaolin, the initial density gradually decreases with increasing MK, because MK is finer and has a lower bulk density than sand, and its high surface area increases viscosity and the potential for initial microporosity. The largest relative declines between 2h and 24h are seen in MK10 and MK20 (-11.52% and -12.68%), indicating an equilibrium that promotes subsequent moisture loss after initial hardening. In contrast, MK30 exhibits the lowest early relative decline (-2.19% between 2h and 5h) despite its lower absolute density, indicating faster hardening that limits subsequent moisture removal. Overall, the results demonstrate that replacing sand with MK reduces initial density and increases the system's sensitivity to early moisture loss, with the maximum relative effect at intermediate replacement ratios [19, 20].

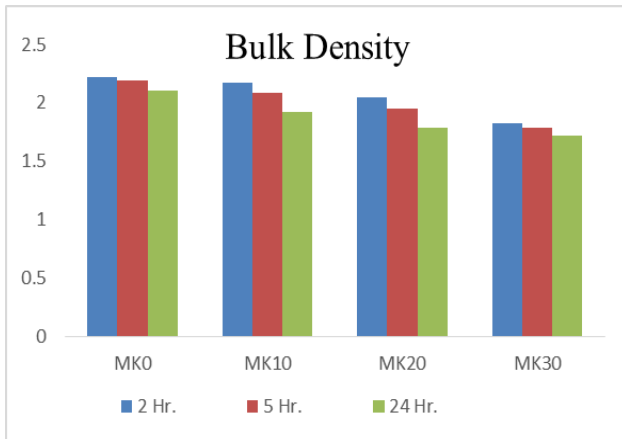


Fig. 4 Bulk density of the prepared mixes

Compressive strength

Fig. 5 and Table 3 show that compressive strength increases steadily from 2h to 5h and then 24h under heat curing at 90°C, with the metakaolin-replacement blends outperforming the reference. This improvement is attributed to the fact that replacing a portion of the inactive sand with finer, more reactive MK particles increases the active aluminosilicate content and provides additional nucleation sites to accelerate the Synthesis of the N-A-S-H gel [21]. The matrix quickly interlocks, and this

structural increase is reflected in the early strength despite a corresponding decrease in bulk density. The values demonstrate a peak performance at MK20, where a balance is achieved between the "filler effect" and improved microscopic compaction on the one hand, and maintaining good workability and compaction on the other, which reduces microporosity and enhances structural mobility [22]. At MK30, the strengths remain higher than the reference but are slightly lower than at MK20 due to the higher viscosity and the increased likelihood of microporosity or localized heterogeneity, which reduces the strength [23]. Overall, the results support that metakaolin replacement improves early and advanced compressive strengths via a chemical-structural mechanism consistent with the density results, with an optimum ratio close to 20% for applications requiring rapid strength development. As shown in Fig. 6, compression resulted in a diagonal shear with cracks extending to the edges and central crushing beneath/above the platens. This mode of failure is characteristic of compression in brittle mortar: platen friction causes a multiaxial stress state that supports shear bands oriented at approximately 45 degrees, as well as confined crushing at contacts and spalling at edges

Table 2: Bulk density results of the mixes

Mix	2 h (g/cm³)	5 h (g/cm³)	Δ% (5h vs 2h)	24 h (g/cm³)	Δ% (24h vs 2h)	Δ% (24h vs 5h)
MK0	2.22	2.19	-1.35%	2.11	-4.95%	-3.65%
MK10	2.17	2.09	-3.69%	1.92	-11.52%	-8.13%
MK20	2.05	1.95	-4.88%	1.79	-12.68%	-8.21%
MK30	1.83	1.79	-2.19%	1.72	-6.01%	-3.91%

Table 3: Compressive strength results of the mixes.

Mix	2h MPa	5h MPa	Δ% 5h vs 2h	24h MPa	Δ% 24h vs 2h	Δ% 24h vs 5h
MK0	10.2	13.4	31.37%	22.6	121.57%	68.66%
MK10	11.8	19	61.02%	26.03	120.59%	37.00%
MK20	13.26	21.6	62.90%	32	141.33%	48.15%
MK30	12.9	20.1	55.81%	29.13	125.81%	44.93%

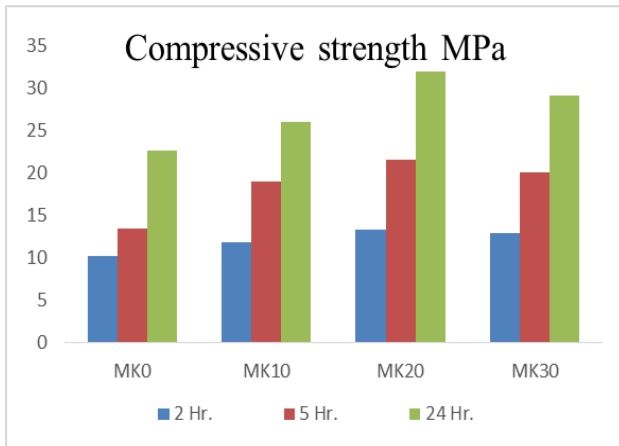


Fig. 5. Compressive strength of the prepared mixes



Fig. 6 Sample after compressive strength test

Conclusions

The conclusions of the present study are:

- 1- Replacing sand with metakaolin (10, 20 and 30) % increased compressive strength at 2h, 5h, and 24h compared with the reference mix.
- 2- The increase in strength is due to accelerated geopolymerization and denser early gel development (N–A–S–H). This is because MK particles are finer and more reactive with aluminosilicates.
- 3- The MK20 mix had the ideal general balance, achieving the highest early-age strength while still having a workable rheology and good compaction, thereby limiting fine entrapped porosity.
- 4- At MK30, strength continued above the reference but was relatively lower than at MK20. This was mainly because of higher yield stress/plastic viscosity and an increased probability of micro-voids.
- 5- In heat curing at 90°C, aluminosilicate dissolution and N–A–S–H gel condensation are accelerated, shortening setting time and markedly boosting early-age strength.

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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