



Research Article

Enhancing geopolymer mortars: The impact of Algerian metakaolin substitution on mechanical performance and sustainability

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ARTICLE INFO

Article history

Received: 04 November 2024

Revised: 13 February 2025

Accepted: 01 May 2025

Keywords:

Binder Characterization;
Geopolymeric Mortar;
Mechanical Strength; Metakaolin
Substitution; Natural Pozzolan;
Rheological Behavior

ABSTRACT

Geopolymer binder has been received as sustainable substitute for the Portland cement that ensures lower carbon footprint and higher durability properties. This study focuses on the influence of metakaolin (MK) replacement in Benisaf natural pozzolan (BNP)-based mortars on the rheological and mechanical properties of these latter. The objective is to optimize the metakaolin content for improved mechanical performance while keeping suitable workability. Sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) were used as activating solutions to synthesize geopolymer mortars, where MK replaced BNP with five different ratios of 10%, 20%, 30%, 40% and 50%. X-ray diffraction (XRD), X-ray fluorescence (XRF), Scanning electron microscopy-energy dispersive x-ray (SEM-EDX) and particle size analysis were used for characterizing the raw materials. The tests focused on flowability in fresh state, and compressive strength (CS) at 14 and 28 days. The outcomes show that increasing MK quantity results in a reduction in workability, since its particle size is finer and its reactivity is higher, consequently the viscosity is higher and the fluidity is lower. As a result, compressive strength was increased considerably, with a maximum value of 24 MPa at 40% metakaolin substitution (150% higher than control geopolymer mortar). This improvement is due to increased availability of aluminosilicate, which allows a denser gel formation as well as a more favorable polycondensation. Over 40% of MK content resulted in incomplete dissolution and decreased strength. A quantitative evaluation of MK-BNP geopolymer interactions is presented, highlighting that 40% MK replacement can provide an optimal workability- strength equilibrium. This work aids the formulation of sustainable geopolymer mortars suitable for construction. Complementary investigations should emphasize practicability through large-scale applications, and durability and thermal stability as well.

Cite this article as: Hebbache K, Douadi A, Hamdi OM, Boutlikht M, Messai A, Khitas NEH, Belebchouche C. Enhancing geopolymer mortars: The impact of Algerian metakaolin substitution on mechanical performance and sustainability. Sigma J Eng Nat Sci 2026;44(4):2937–2951.

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This paper was recommended for publication in revised form by
Editor-in-Chief Ahmet Selim Dalkilic



INTRODUCTION

Concrete is one of the most used materials in construction, with a global consumption of 5 billion tons per year (almost 1 ton/person) [1]. This classifies concrete as the second most used resource following water [2]. The rising need for concrete in civil engineering is directly related to the increasing demand for cement, itself a vital binder in concrete formulation [3, 4]. Ordinary Portland Cement (OPC), that is used in much construction work, alone consumes about 7.36×10^6 kJ/ton cement produced, which is considered a very high rate [5, 6]. Moreover, the cement production releases large amounts of greenhouse gases such as carbon dioxide (CO_2) [7, 8], which are related to fuel combustion and decarbonation of limestone during clinkering process [9, 10]. Literature shows that cement manufacturing is responsible for around 8% of global CO_2 emissions [11], generating about one ton of CO_2 per one ton of cement [12]. These noticeable data underline the important environmental damages related to OPC, showing that it is crucial to find new kinds of sustainable alternatives with less associated carbon footprint emissions [13, 14].

Geopolymer binders have been explored as an alternative to OPC due to the prospect of reducing CO_2 emissions by up to 80% which makes them attractive for applications [15]. Geopolymers are inorganic polymers developed by the chemical process of aluminosilicate precursors with an alkaline solution (e.g., sodium or potassium hydroxide, with or without sodium/potassium silicate) [16]. When polymerization occurs, three-dimensional network of Si–O–Al bonds is formed and the built materials exhibits outstanding chemical resistance, mechanical properties and thermal stability [17]. In addition, geopolymers achieve strength from aluminosilicate gels instead of calcium silicate hydrates like OPC do, qualifying them to be a more attractive low-carbon substitute.

A geopolymer mortar, that is the result of chemical reactions between fly ash and alkaline activators, has been proposed as an environmentally-friendly alternative for traditional cement mortars [18, 19]. Geopolymer mortars proved to possess better strength properties, showing a compressive strength as high as 65% when compared to both OPC and PPC mortars, in addition to having bond strength approximately 50% higher [18]. It has been previously reported that the strength of geopolymer mortars is affected by some important variables such as curing temperature, alkaline activator concentration, and ratio of activator to fly ash [20]. Under ambient temperature curing conditions, optimal compressive strength has reached 31.39 MPa [19]. Including sand induces a slight increase in compressive strength, and the density of geopolymer mortar is between 2.0 and 2.23 g/cm³ [19]. These characteristics contribute to the potential suitability of geopolymer mortars as effective alternatives for traditional repair materials [18, 19].

Research on geopolymer binder has focused mainly on base materials, including fly ash [21–23] and metakaolin

[24–26], and synthesis parameters have been optimized over the recent decades. Three main pathways for research can be distinguished. The first focuses on the applicability of different pure or blended base materials for the production of geopolymers and investigates the intrinsic characteristics including types and ratio of components, and external agents such as curing conditions, affecting the chemical and microstructural characteristics [27]. The second pathway collects information regarding the compositions and microstructures of the base materials effect on the kinetics of reaction and polymerization process, and especially on the formation of mechanical properties like strength [28]. The last research focus investigates engineering characteristics and long-term behaviour of geopolymers, through their assessment in form of paste, mortar or concrete under diverse chemical exposures [29].

Although fly ash has attracted attention for use in geopolymer binder, the limited availability of fly ash due to the shrinking amount of coal plants is problematic and disposing of fly ash also raises environmental issues. As a result, there is significant interest in investigating alternative resources rich in aluminosilicate that are readily available and environmentally friendly. Natural pozzolan is a volcanic material with high content of silicon and aluminum that can be considered as a potential raw material for synthesizing geopolymer binders. Due to the easy extraction and low cost, it serves as a potential substitute, especially in areas where such pozzolans exist [30]. Natural pozzolan has a lower reactivity than the other materials such as metakaolin mainly because of its semi-crystalline structure and lower CaO and reactive Al_2O_3 contents. That is why the pozzolanic reaction rate is usually slower and its based geopolymers has a lower mechanical strength [31]. In order to overcome those limitations and improve also the performance of pozzolan-based geopolymers, a mixture design was explored, in which natural pozzolan is partially replaced with metakaolin. Metakaolin is an aluminosilicate material produced through calcination of kaolinite clay and characterized by its high reactive behavior especially because of the amorphous form and high alumina content [32]. This paper aims to study mechanical and rheological properties of geopolymer mortars incorporating different proportions (10%, 20%, 30%, 40% and) of metakaolin to replace natural pozzolan. In addition, the effects of important synthesis parameters like initial alkaline hydroxide solution concentration and $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio on the setting times and compressive strength of geopolymer mortars are assessed. Characterization of the raw materials is performed by techniques, such as X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX), and particle size distribution analysis, which are destined to reveal the chemical and mineralogical composition of the material [28].

Geopolymers mortars have been previously investigated especially the ones based on natural pozzolan and

metakaolin, but the published studies emphasize either mechanical strength or durability aspects without considering a thorough parametric investigation. In this regard, the current study intends to bridge part of these gaps by providing a systematic investigation on the influence of metakaolin substitution together with some important experiments' parameters on both fresh and hardened states characteristics. In addition, the use of advanced techniques for material characterization can complement investigations into microstructural evolutions that occur during geopolymerization. The present research combines these three elements, confirming recent studies in this field while providing new insights into the development of geopolymer formulations comprising pozzolans and metakaolin that finally maximize both performance and sustainability.

This study aims to expand the existing literature involving sustainable construction materials by providing an evaluation of pozzolan-metakaolin-based geopolymer mortars that are capable of significantly reducing the carbon emissions produced by cementitious materials without compromising structural performance.

MATERIAL AND METHODS

Characterization of Precursor Materials

The natural pozzolan used in this study, denoted as Bouhmedi Natural Pozzolan (BNP), was sourced from

volcanic tuff deposits at Bouhmedi in Beni-Saf, located in the northwestern region of Algeria (Ain Temouchent), and provided by the company SPMC Spa. Kaolin, on the other hand, was supplied by the Soalka company and is obtained from the Milia deposit in Jijel, located in northeastern Algeria as shown in Figure 1.

The active precursors used in geopolymerization preparation were derived from kaolin and pozzolan. Kaolin was calcined for 7 hours under 800 °C temperature to produce metakaolin (MK), and pozzolan was dried at 105 °C for 24 h prior to grinding using a ball mill. Then, BNP and MK were ground again aiming to achieve powders with better fineness. These powders were both sieved to a maximum particle size of 100 µm. The composition of the precursor materials is decisive in terms of their reactivity in an alkaline medium for the successful formation of geopolymer. The chemical properties of BNP and MK were obtained using x-ray fluorescence (XRF) spectroscopy through a Malvern Panalytical Epsilon 1 spectrometer and the results are summarized in Table 1. These materials both presents high contents of SiO₂ and Al₂O₃, key constituents for the geopolymerization process. The SiO₂/Al₂O₃ molar ratios of BNP (4.4) and MK (2.8) suggest that they are proper to synthesize geopolymer.

It is crucial to emphasize that the mineralogical variability of precursor materials significantly affects the physico-chemical and mechanical characteristics of the synthesized

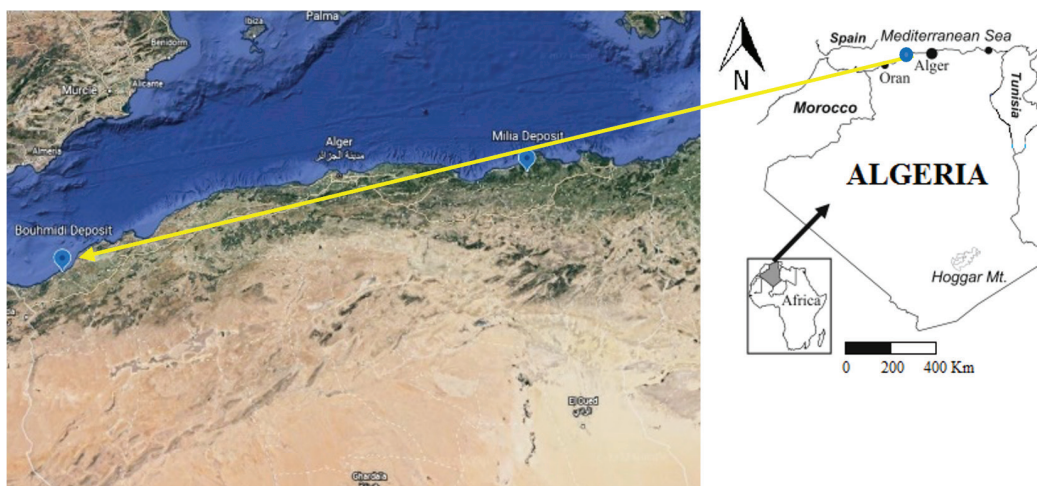


Figure 1. Location of Bouhmedi and Milia deposit.

Table 1. Oxide composition of BNP, Kaolin and MK as obtained by XRF

Composition (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	L.O.I
BNP	48.1	18.6	10.3	10.8	3.7	0.2	0.8	0.6	4.3
Kaolin	49.3	33.5	2.6	0.1	0.4	0	0.1	2.8	11.2
MK	54.4	31.1	7.4	0.1	0.4	0.3	0.2	4.2	1.9

geopolymer [31]. These precursors reactivity depends on their phase composition, crystallinity and the availability of reactive aluminosilicate species. Hence, X-ray diffraction (XRD) analysis was undertaken for the identification of the crystalline phases in raw materials and to evaluate their influence upon the geopolymerization process.

To this end, analysis was carried out with a Siemens D5000 diffractometer in the Brentano Bragg configuration and at an operational voltage of 45 kV using Cu K α 1 radiation source ($\lambda=1.54$ Å). The diffraction patterns were recorded and monitored in a continuous scan mode, with a 0.017° of step size covering a 2θ range of 3° to 75° , and measured for about 0.33 seconds per step to provide high-resolution characterization of the crystalline mineralogical phases existing inside natural pozzolan (BNP), metakaolin (MK) and kaolin as shown in Figure 2. The results suggested that BNP is constituted of multiple crystalline phases, dominated by several phases such as Anorthite (a calcium aluminosilicate), Alancime (a zeolitic phase), Diopside (a pyroxene mineral), and Hematite. Anorthite contributes to the pozzolanic activity of BNP, and Alancime can enhance alkali reactivity because of its content in structural water. Diopside shows limited reactivity in presence of alkaline conditions while Hematite affect the color and thermal stability of the ultimate geopolymer matrix.

By contrast, MK had a more heterogeneous character with the peaks from Quartz and Zeolite as the main crystalline phases. Quartz is a very stable silicate with low reactivity in geopolymerization process, which is only improved if further ground to a fine granulometry, while Zeolites have a high cation-exchange capacity and a microporous crystal structure that can improve alkali activation and can additionally define the mechanical performance and durability of the geopolymer.

Besides chemical composition, the physical properties of the precursor materials play an important role in optimizing geopolymer performance. Table 2 shows the specific surface area and density of BNP and MK. Measurements were made using a Blaine permeabilimeter (Controlab L0049) for specific surface area determination according

Table 2. The main physical properties of MK and BNP

Physical properties	MK	BNP
Absolute density (g/cm ³)	2.59	2.76
The Blaine's specific area (m ² /kg)	320	650
Particle size characteristics	D10 (μ m)	14.25
	D50 (μ m)	36.87
	D90 (μ m)	74.32

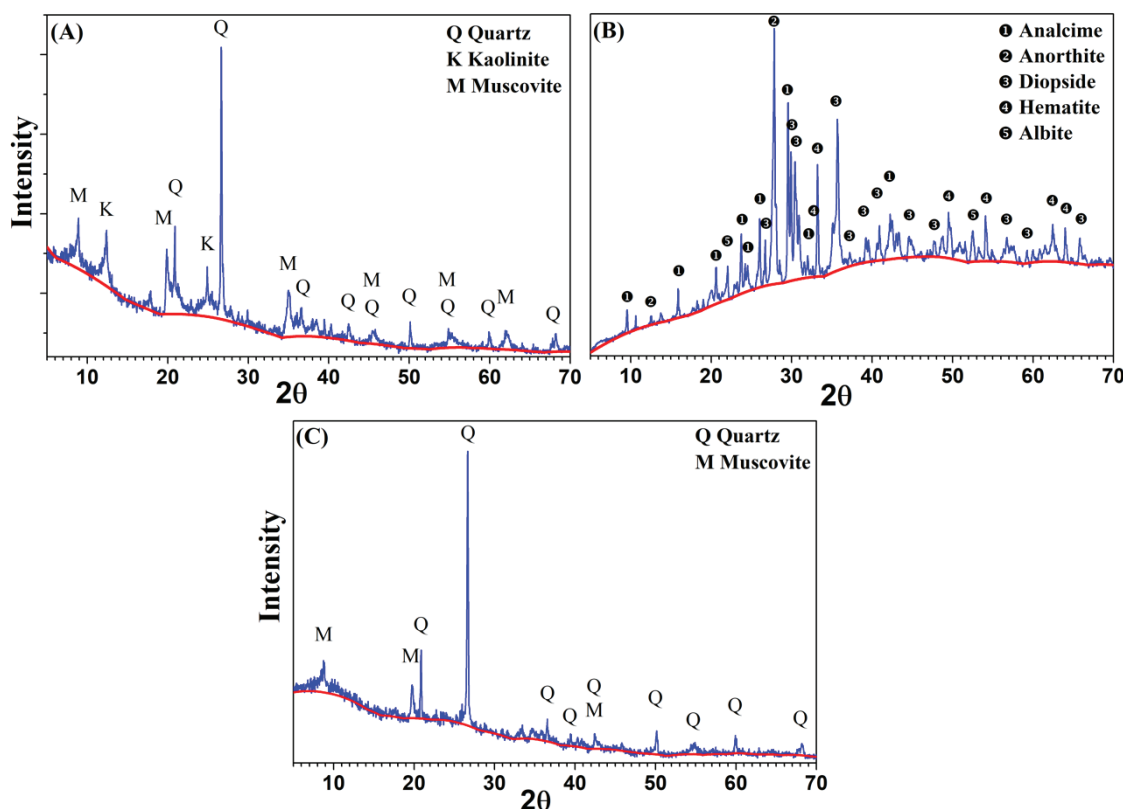


Figure 2. X-ray diffraction patterns of the Kaolin, MK and BNP samples.

to the European standard EN-196-6, whereas the density was measured by means of a pycnometer using Archimedes method. The laser scattering analyzer was used to analyze particle size distribution of BNP and MK, and both cumulative and partial distribution curves of particle sizes of each material are presented in Figure 3. The SEM (Scanning electron microscopy) revealed the morphological detail of the precursor materials that are determinant in their reactivity and packing density or arrangement within the geopolymeric matrix (Fig. 4).

Micrographs show that MK particles have a unique multilevel, plate-like morphology, which is consistent with thermally activated kaolinite. The noticed morphology gives it a high specific surface area and increases the dissolution kinetics during alkaline activation, which leads to an easy geopolymeric gel formation. On the other hand, BNP particles show an irregular, angular shape and a wide particle size distribution (1 to 25 μm) that reveal the

heterogeneity of this material, which in turn will affect the packing density and inter-particle porosity as well as the material reactivity. Angular particles presence indicates less natural weathering or mechanical grinding and possibly increased mechanical interlocking in the geopolymer matrix. Differences in the morphology of MK and BNP directly affect the flowability, workability, and microstructural evolution of the final geopolymer.

Alkaline Activators

In this study, the alkaline activators used to activate Bouhmedi Natural Pozzolan (BNP) and metakaolin (MK), are a mixture of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) solutions at different ratios. Commercial sodium silicate is sold as solid, powder or liquid in all forms with $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio of 1.6 to 3.85 [32, 33]. The sodium silicate used here was locally supplied by AFRICAVER EPE SPA (Jijel), as a viscous solution with silica modulus equal to 3.35. Sodium hydroxide (NaOH) which is an imported product supplied by the company of JSC KAUSTIK VOLGOGRAD, from NIKOCHEM group, was presented in white spherical pearls form. Those pearls were dispersed in distilled water with ongoing stirring to yield solutions of varying molar concentrations according to experimental requirements.

Preparation of Samples

Precursor materials (BNP and MK) were mixed with the alkaline activating solution using a mechanical mixer (Controlab L0031. 2) with a 5-liter capacity. The mixing design was based on an alkali/precursor mass ratio of 0.45 and a molar ratio $\text{SiO}_2/\text{Al}_2\text{O}_3$ on the order of 5. The activation liquids were mixed of sodium silicate and sodium hydroxide solutions, prepared 24 h prior to use, keeping a $\text{Na}_2\text{SiO}_3/\text{NaOH}$ mass ratio equal to 2.5, with the aid of a magnetic stirrer. To assess the effect of MK substitution levels on initial and final setting times, six mixes (0%, 10%,

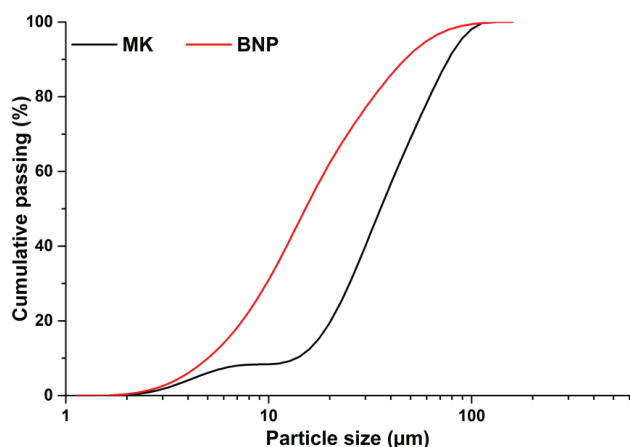


Figure 3. Particle size distribution from BNP powder and MK.

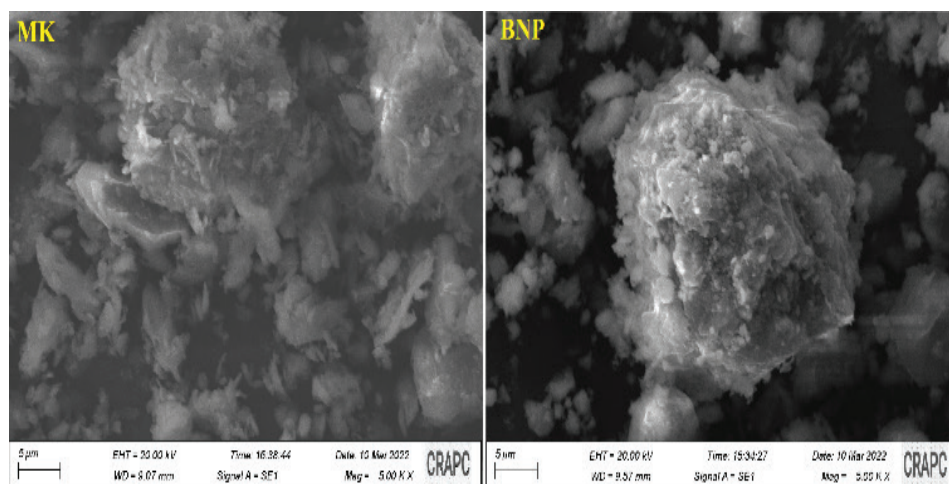


Figure 4. SEM images of MK (left) and BNP (right).

Table 3. Proportions of mixtures for the preparation of mortars

Mix	SiO ₂ /Al ₂ O ₃ molar ratio	SiO ₂ /Na ₂ O molar ratio	MK content
M-1	5.2	1.90	0
M-2	5.5	1.71	0
M-3	5.8	1.56	0
M-4	6.1	1.43	0
M-5	6.4	1.34	0
M-6	5.9	1.34	10
M-7	5.7	1.34	20
M-8	5.4	1.34	30
M-9	5.1	1.34	40
M-10	4.6	1.34	50

20%, 30%, 40% and 50%) were prepared per mix design. The water/BNP ratio was maintained at 0.4 to provide the proper paste consistency. To evaluate the influence of MK content and the molar ratio of SiO₂/Al₂O₃ on compressive strength, ten mortar mixes were prepared at five levels per parameter under standardized conditions as provided in Table 3. The fixed mass ratio of sand to precursor used was 3, according to European Standard EN 196-1.

The sample preparation consisted of three sequential operations: mixing, casting and curing. First, the sand and precursor powders were dry-mixed for 1 min in a mechanical mixer to reach homogeneity. Then, the activating solution was poured into the dry mixture dropwise and mixed at a low speed for 90 seconds. Subsequently, the mixer was paused so that the mixture clinging to the sides and bottom of the bowl could be scraped down before mixing again on high speed for another 90 seconds.

At the end of the mixing procedure, the mortar was cast into metal molds of 40×40×160 mm³ dimensions, in two layers, each compacted by 60 shocks by an automatic vibrating table (1 shock per second rate). The surface of each layer was leveled using a spatula before sealing the molds. The molds were stored in hermetically sealed plastic bags and placed in a climatic chamber set at 25°C and 95% relative humidity (RH) for controlled curing. With this approach, the geopolymerization process occurs under optimal conditions, so it allows to make a much more accurate assessment of the setting times and compressive strength of the different geopolymer mortars.

Workability

The geopolymer mortars workability was evaluated with the mini-cone slump test in accordance to EN 1015-3 as an indirect way to evaluate flowability [34]. This test was carried in an apparatus made by a truncated cone with base diameter (D1) of 150 mm, upper diameter (D2) equal to 50 mm and height (h) equal to 200 mm. The geopolymer mortar mixture was placed within the cone in two subsequent layers. The resulting homogeneous layers were compacted by

applying 30 shocks (using a mechanical vibrating table) to each layer, in order to expel entrapped air. The filling of the cone followed by its removal was used to measure the diameter of spread mortar in two orthogonal directions. The final spreading flow was determined by averaging the recorded values. To check the reproducibility and fidelity in the physical performance of workability, this test was performed two times per batch for each mortar. It should be noted that the rheological properties of the mortar can be gleaned from the flowability scores which also serve to indicate what fluidity level will work in casting such material. This becomes even more important for geopolymer-based systems, as small changes in precursor composition and alkaline activator concentration can drastically affect fresh state behavior.

Initial and Final Setting Time

The first and final geopolymer paste setting times were evaluated following European Standard EN 196-3 [35], as presented in Figure 5. The information conveyed through



Figure 5. Vicat device while measurement of initial and final setting time.

setting times make each of the materials to evolve from liquid to solid at the speed that polymerization takes place. The free penetration of the Vicat needle under its own weight at a distance of 6 ± 3 mm from the bottom of the paste was defined as an initial setting time. This is the point at which the geopolymer paste has sufficient strength that it no longer deforms, and thus begins to hardening.

Each test was performed in triplicate for the same mixture to avoid inaccuracies and mean values were reported. The initial and final setting times were also critically evaluated by varying the percentage of metakaolin and molar $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio (the most significant factors controlling the geopolymerization kinetics). Curing times provide important information regarding workability limit and potential field application of mixes, especially for large scale industrial use where time is an immediate concern.

Compressive Strength

The compressive strength of geopolymer mortars was measured to evaluate the effect of metakaolin content and $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio on some mechanical properties of the synthesized material. The values were measured in accordance with the methods outlined in the European Standard EN 196-1 [36] for determining the compressive strength of cementitious material (Fig. 6). As detailed in Section 2.3, prismatic mortar specimens measuring $40 \times 40 \times 160$ mm³ were prepared. All specimens, after curing were firstly tested in a 3-point bending to generate fracture, thus dividing each prism in equal halves ($40 \times 40 \times 80$ mm³). The two halves obtained after the fracture were then used for a compressive strength test.

Compressive strength (CS) testing was conducted at two significant curing periods, 14 days and 28 days, which correspond to the early and more mature stages of strength development. The halves were aligned in the compression testing machine so that a uniform application of load was

applied over the surface area. Specimen disruption occurred at a constant loading rate, and the peak force at failure was noted. In each mix, the CS was taken as the average of four halves of individual specimens in order to reduce the data variation and increase statistical significance. Such a method led to the full examination of how MK substitution and $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio affected the mechanical properties of geopolymer. The numbers of compressive strength reflect the ability of this material to resist compressive stress, while also giving a signal on the best MK content and amounts of activator needed to enhance all structural performance.

RESULTS AND DISCUSSION

Workability

Figure 7 shows the workability of alkali-activated natural pozzolan (BNP) mortars as a function of the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio, outlining its important role in the rheological behavior of the fresh mixtures. The flow has been measured from 160 mm to 251 mm, lining up with the limits generally accepted for the manufacture and use of geopolymer-based material. Interestingly, the flowability using the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio trend was somewhat different, an increase from 5.2 to 5.8 lead to a significant improvement of the flowability from 160 mm to 251 mm. The main reason for this improvement is the addition of water content with a higher dosage of sodium silicate, and thereby increasing the dispersion of solid particles and reducing internal friction (fluidity).

Moreover, when $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio exceeded to 5.8, workability started to reduce gradually and flow values decreased from 251 mm to 213 mm. This reduction is attributed to the availability of reactive SiO_2 species, which promotes geopolymerization and increases gel network building rates [37]. The formation of geopolymeric



Figure 6. Geopolymer mortar specimens during curing to evaluate their compressive strength.

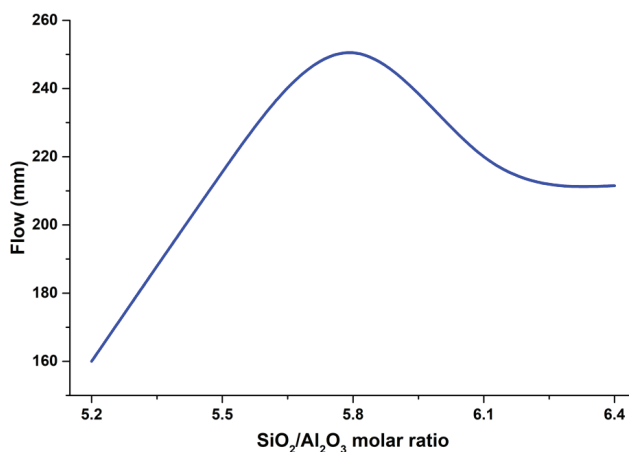


Figure 7. Effect of $\text{SiO}_2/\text{Al}_2\text{O}_3$ on the spread flow values of the prepared mortars.

gel occurs rapidly and entraps solid particles, which leads to increased viscosity and lower workability. These results highlight the fact that there exists an optimum range of $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios, beyond which excess silicate promotes initial setting and reduces fluidity due to excessive viscosity build-up of mortar [38]. Hence, the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio play a significant role for balancing workability and geopolymer reactivity of alkali-activated systems [39].

This is shown in Figure 8, where partial substitution of BNP with MK reduces the workability of the mortars. The results show that flow values systematically change from 220 mm to 101 mm when the proportion of MK in the mixture increases. Such significant loss in workability is primarily originating from the unique morphological and physicochemical properties of MK, which as seen in Figure 4, possesses a microstructure that is plate-like with high specific surface area.

The layered structure of MK leads to an increased water retention capacity, as the fine particles tend to adsorb free water from the mixture, thereby reducing the effective water available for lubrication. In addition, the angular and rigid shapes of MK particles may allow for higher effective interparticle friction, which could contribute in decreasing flowability. The results in accordance with Hassani and Kazemian [40] findings that showed that the increase in red mud (RM) content in geopolymer mortar strongly reduced the flowability. At 50% RM substitution the spread diameter decreased by 57% (from 22 cm to 9.5 cm), which may be attributed to high surface area and water absorbent capacity of RM.

The results indicate that MK incorporation improves the mechanical and durability properties of geopolymeric mortars because of its high aluminosilicate reactivity. However, as far as excessive losses in workability are concerned, the dosage has to be properly optimized. Achieving a balance between improving mechanical performance and maintaining rheological stability remains as a significant

challenge in the design of geopolymers, which highlights the importance of designing tailored mix designs to achieve satisfactory processability and long-term performance.

Setting Time

The setting time of geopolymer mixtures is highly affected by a number of factors, primarily the nature of reactive products generated from precursor materials. These properties include free calcium oxide (CaO) content, specific surface area, crystallinity degree and chemical composition and concentration of the activating solution [33, 41]. Free CaO promotes the development of reactive amorphous structures enhancing geopolymerization, while a high degree of crystallinity caused by the low solubility in a crystal structure leads to decreased reactivity.

Setting times (Initial and final) of the alkali-activated pastes employed in the present study are shown in Figure 9. The results showed that the setting times of BNP alkali-activated mixtures were substantially greater than their ordinary Portland cement (OPC) counterpart. Such a delay can be rationalized by the kinetics of the reactions during geopolymerization, which differs from that of conventional Portland cement hydration. Moreover, the final setting times were addressed as they extended over several days to depict a slow polymerization and gradual generation of inside rigidity. In contrast, the initial and final setting times of mixtures with partially replaced BNP with MK were reduced to 129 minutes and 291 minutes, respectively. This decrease is owed to the higher amount of Al_2O_3 reactive species from metakaolin, which encourages faster aluminosilicate dissolution. As a result, this increase in the chemical reactivity of the particles favour formation and condensation of the geopolymer gel, which reduces the setting time and increases initial cohesion of material.

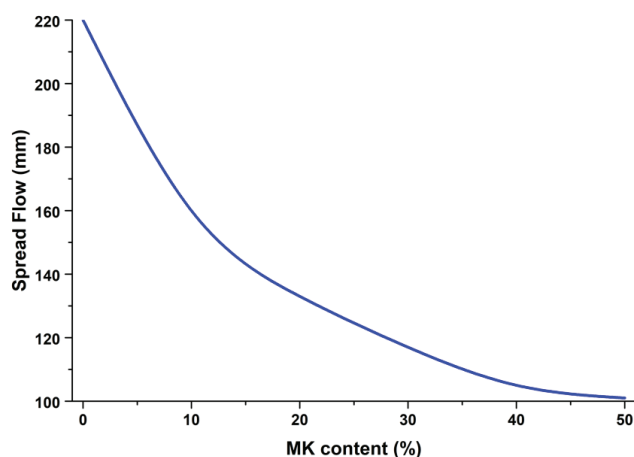


Figure 8. Effect of MK content on the spread flow values of the prepared mortars.

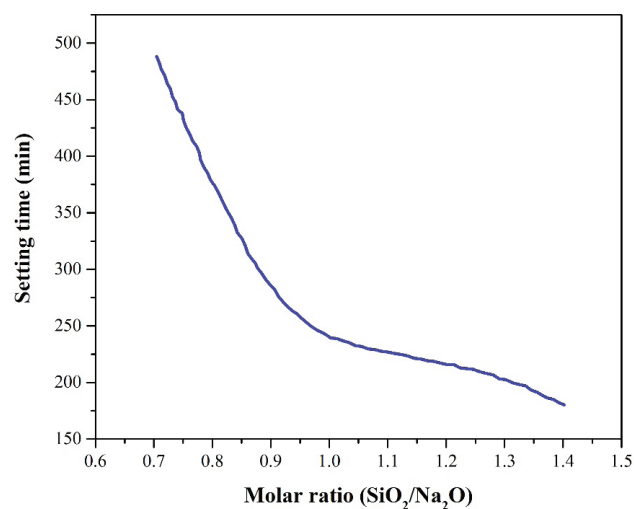


Figure 9. Effect of MK content on the initial and final setting time.

The observations aspects of determining geopolymerization kinetics, as well as the much-debated role of precursor characteristics and formulation parameters are encapsulated in these observations, which can both guide mixture optimization for workability as function of desired long-term performance.

According to Hamdi Oualid's doctoral thesis [42], two volcanic ashes from Djoungo, namely ZD and ZG (Galim), with CaO contents of 3.23% and 5.11%, respectively, were investigated to assess the effects of fineness and the $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio on geopolymerization. Geopolymer samples prepared from ZD volcanic ash could only be manipulated after 14 days of curing time likely as a consequence to its low free CaO content and lower fineness ($2.3 \text{ m}^2/\text{g}$) in comparison with ZG ($15.7 \text{ m}^2/\text{g}$) [43]. The kinetics of the geopolymerization reaction is largely influenced by particle fineness since a higher specific surface area allows for quicker dissolution of aluminosilicate species and more homogenous reactivity.

Furthermore, raising the $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio from 0.7 to 1.4 decreased the setting time of ZG-based geopolymer paste from 490 min to 180 min, as shows Figure 10. This is consistent with the results documented by Lalitha et al. [18], who reported that a lower $\text{SiO}_2/\text{Na}_2\text{O}$ ratio leads to prolonged setting time of blast furnace slag-based geopolymers. A higher $\text{SiO}_2/\text{Na}_2\text{O}$ ratio leads to the generation of more reactive oligomeric structures and keeps intermediate products that obstruct polymerization [8]. In addition, Yankwa Djobo et al. [44] stated that mechanically activating volcanic ash decreases its degree crystallinity, dramatically speeds up the geopolymerization reaction and reduces by over 95% the setting time, after grinding for only 60 minutes. This acceleration is due to an increasing number of reactive sites, and therefore better interaction with

the alkaline solution, thus accelerating ammonification of amorphous geopolymers.

The compressive strengths of geopolymer mortars at 14 and 28 days in terms of the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio are shown in Figure 11. A strong direct correlation between the compressive strength of the geopolymer mortar and the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio is observed. This behavior is related to the geopolymerization process, in which higher SiO_2 content leads to a more extensive three-dimensional aluminosilicate network being formed. The maximum compressive strength of 9.93 MPa was recorded for the highest $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of 6.4, suggesting that beyond a certain limit, there is more silicate species available for optimal network polymerization. This improvement in mechanical strength was related to the improved availability of reactive SiO_2 provided by sodium silicate, which contributed dissolution of aluminosilicate precursors as well as incoherent oligomerization of silicate chains. This leads to a more compact and integer gel, able to give development at the mechanical execution. Therefore, the condensation process in geopolymerization is more favorable, resulting in a well-developed aluminosilicate framework with enhanced structural integrity.

On the other hand, specimens with mixture M-1 did not develop any significant compressive strength after 28 days of curing and they were brittle (Fig. 11). This lack of mechanical performance indicates the importance of silicate species during geopolymerization. This is mainly due to the presence of sodium hydroxide in an alkaline solution. Because NaOH is effective in dissolving the amorphous aluminosilicates in Bouhmidi pozzolan (BSP) but does not provide any additional silicate species, polycondensation reactions during hydration are incomplete. Consequently, a continuous aluminosilicate gel cannot be formed in which case a coherent geopolymeric matrix cannot develop. The inability of silicate to facilitate condensation of the dissolved species leads to precipitation of poorly polymerized

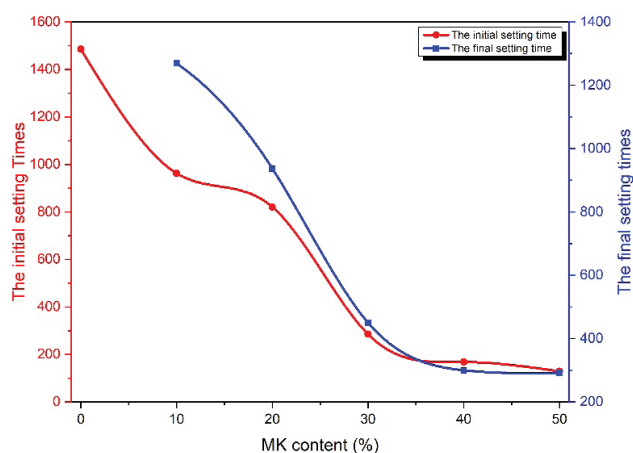


Figure 10. Effect of $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio on the setting time [Adapted from Hamdi's doctoral thesis [42]. The thesis is openly accessible through the USTHB institutional repository: <https://dspace.usthb.dz/handle/123456789/10212>].

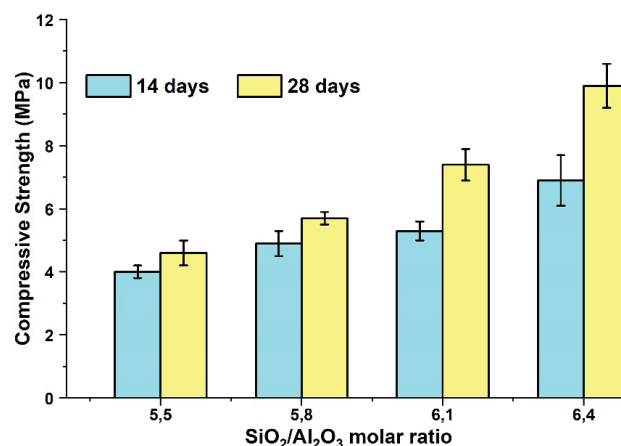


Figure 11. Effect of $\text{SiO}_2/\text{Al}_2\text{O}_3$ on the compressive strength of geopolymer mortars.

products that lack structural cohesion. This effectively prevents the last stage of the geopolymerization reaction taking place, resulting in mechanically unstable samples.

Figure 12 emphasizes the impact of metakaolin (MK) mass percentage on the compressive strength (CS) of the geopolymer mortars. In particular, this aluminate would massively change the geopolymerization kinetics and microstructural development of the hardened matrix when MK was used as a secondary aluminosilicate source. The incorporation of MK to replace BNP up to 40% by mass improved the compressive strength values of the binary systems with strengths approaching 24 MPa. Such enhancement is associated with the more rapid dissolution of MK when compared to BSP, which provides a larger amount of Al^{3+} and Si^{4+} species contributing to faster geopolymeric gel formation phases. On the contrary, the mortars incorporating MK in excess of 40% exhibited lower compressive strength. The decrease in mechanical performance relates to the high alumina content which disturbs the appropriate $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio required for obtaining a structured gel. Excess Al^{3+} ions trigger a marked viscosity decrease in the fresh paste, which can hinder the homogeneous dispersion of the reactants and reduce the extent of polycondensation. These findings are concurrent with the observation reported in Hamdi Oualid's doctoral thesis [42], who proposed that excess alumina (originated from MK) rapidly decreases the initial viscosity of the alkaline suspension because of its high reactivity. By lowering the viscosity, premature setting may occur which in itself could decrease the interaction time between aluminosilicate precursors and alkaline activators before setting occurs, having a final impact on mechanical strength development. This prompt response would likely not give the suspension enough time to sufficiently disperse the BNP particles, which would in turn detract from the mechanical strength of the geopolymeric pastes [45]. Additionally, Yankwa Djobo et al. [44] observed that, in

the case of dissolution, MK particles react faster compared to their natural pozzolan equivalents. This difference in reactivity will then impact the microstructural development of the final geopolymer matrix as any excess MK present in the activator can lead to gel network heterogeneities and therefore unreacted residual particles. As a result, the degree of geopolymerization becomes more and more governed by the quantity of MK in the system [44].

Application and Broader Impact of the Findings on Geopolymer Mortars

This study describes the macro-mechanical behaviour of geopolymer mortars as a function of $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio and metakaolin (MK) replacement. These results have significant implications for sustainable construction materials, specifically related to Portland cement carbon footprints. The progressive increases of $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio are generally associated with higher compressive strength, thus underlining how silicate species should be taken into account for controlling the reaction degree. All the above data can be integrated to elaborate green substitutes to cement-based mortars based on high-performance polymeric binders. By manipulating the molar ratio and activator composition, engineers and materials scientists can create geopolymer blends with improved mechanical properties or durability.

Additionally, the study also highlights binary systems based on metakaolin potential. It was found that in mixes involving more than 40% MK, that partially substitutes Bouhmedi pozzolan (BSP), a higher compressive strength was reached (up to 24 MPa). This finding is particularly valuable for utilizing industrial by-products and natural pozzolans in geopolymer formulations, providing a feasible path for resource-efficient material design. However, further reductions in strength beyond this substitution limit suggest that the control of mix proportions is essential to avoid very high levels of alumina inhibiting polycondensation. In an application view these insights may be used in the precast elements manufacture as well as repair mortars and high-durability coatings for aggressive environments. In addition, a basic understanding of geopolymer chemistry could help optimize the various combinations based on maximum sustainable development with final mechanical performance in infrastructure.

Furthermore, these research findings are in accordance with global efforts to produce low carbon construction materials with reduced reliance on Portland cement [46]. Considering that the characteristic of Portland cement-based technologies actively limits circular economy, one potential solution consists of geopolymer mortars as they are versatile and can also be produced with industrial by-products. Future work can be oriented to study their robustness under real environmental conditions to validate their potential field scalability.

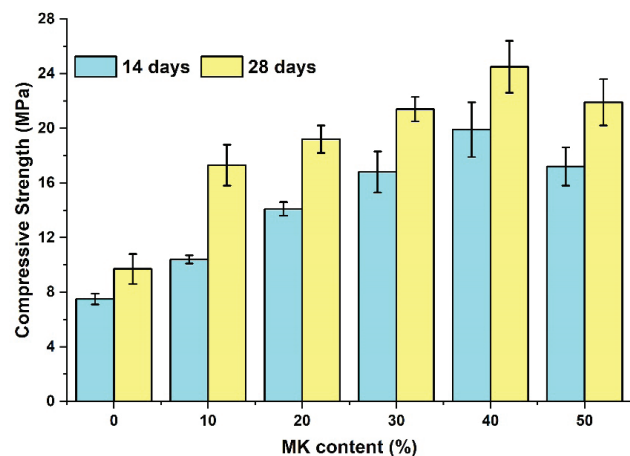


Figure 12. Effect of MK content on the compressive strength of geopolymer mortars.

Statistical Analysis

A thorough statistical analysis using JMP 14 Pro was performed to model and predict the fresh (flow, setting time) and hardened (compressive strength) properties of the mortars (Fig. 13). The study was based on the methodology of central composite design, by varying the Si/Al molar ratio from 1.34 to 1.9 and the Si/Na molar ratio from 5.4 to 6.2. Regression models were used to examine parameter affects and optimize mix formulations for optimal performance.

The fitting results of the obtained models, developed to predict the studied responses using second-degree polynomial equations, indicate that the coefficient of

determination (R^2) exceeds 0.85, as given in Table 4. This proves that the mathematical models provide a reliable prediction of the investigated responses. Moreover, as a confirmation of visual representation derived from fit plot, all root mean square error (RMSE) values are very low which indicates minimum mismatch between the predicted and experimental values. This confirms the reliability and validity of the models for predicting properties of mortars and in capturing both their effects.

Furthermore, the analysis of variance (ANOVA) for each of the modeled responses is presented in Table 5. For degrees of freedom $df1=P-1=5$ and $df2=n-P=3$, the

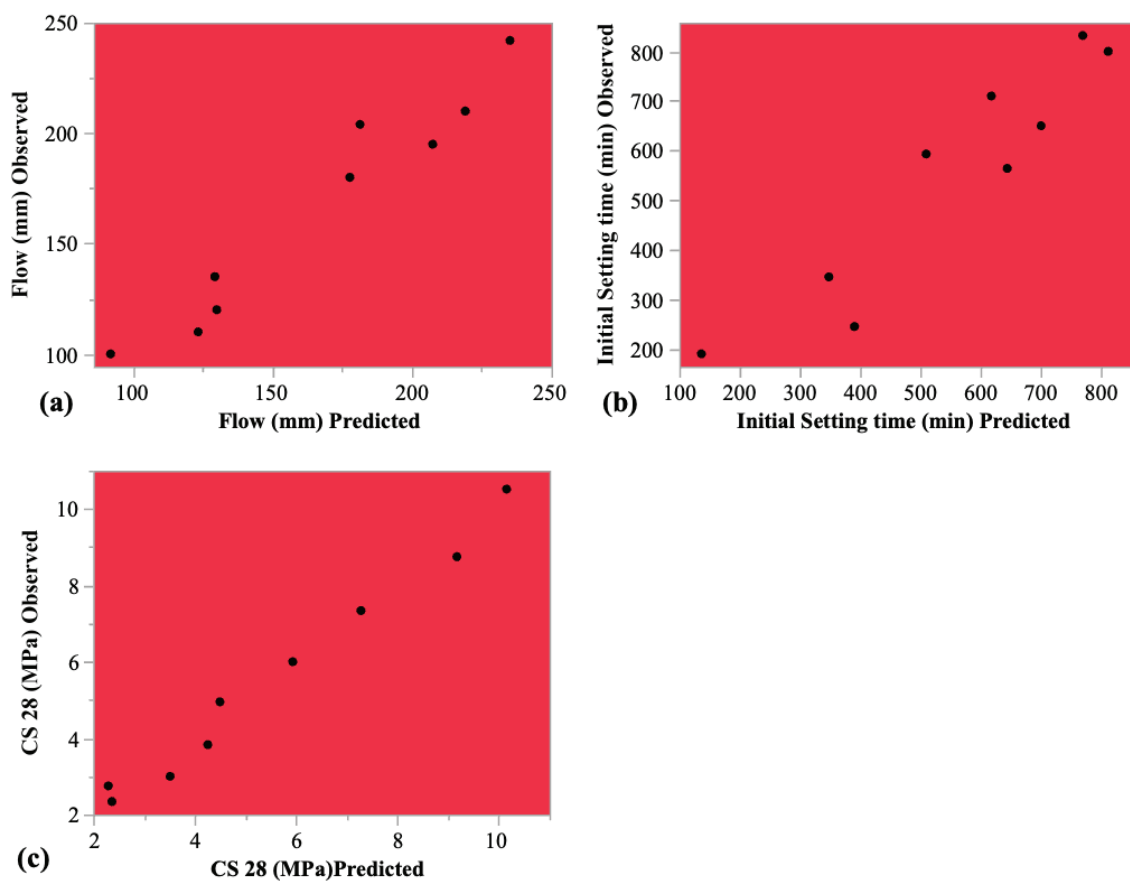


Figure 13. Measured versus predicted; (a) Flow, (b) time of initial setting (c) compressive strength at 14 and 28 days.

Table 4. Summary of the statistical analysis

Designation	Flow	Initial setting time	CS at 28 days
R^2	0.94	0.881697	0.982166
Adjusted R^2	0.85	0.684526	0.952441
Root-Mean-Square Error (RMSE)	19.79	132.361	0.62775
Mean response	166.22	547.6667	5.497778
Observations (or weighted sums)	9	9	9

Table 5. ANOVA analysis for the studied responses

	Source	df	Sum of squares	Mean square	F ratio
Flow	Model	5	391711.67	78342.3	4.4717
	Error	3	52558.33	17519.4	Prob. > F
	Uncorrected Total	8	444270.00		0.1238
Initial setting Time	Model	5	65.105744	13.0211	33.0427
	Error	3	1.182211	0.3941	Prob. > F
	Uncorrected Total	8	66.287956		0.0080*
CS at 28 days	Model	5	65.105744	13.0211	33.0427
	Error	3	1.182211	0.3941	Prob. > F
	Uncorrected Total	8	66.287956		0.0080*

p-values obtained are 0.1238 for flow, 0.0080 for initial setting time, and compressive strength. The Fisher F-values calculated for flow time and initial setting time, as well as compressive strength, are 132.52 and 7.88, respectively. In this case, df corresponds for the degrees of freedom, P is the number of coefficients in the model and n is the number of experiments performed. These p-values suggest the significance of models. A p-value under the threshold 0.05 points to a highly significant model as seen for the initial setting time and compressive strength ($p=0.0080$), whereas for flow; despite its low RMSE the p-value of this regression exceeds this limit indicating less statistical significance (0.1238). However, the Fisher F-values are higher than the critical F-values, confirming that the established models adequately describe the experimental data and can be used for response prediction.

Second-order polynomial equations were developed to predict the flow time, initial setting time, and compressive strength of the studied geopolymer mortars. These equations (1, 2, and 3) accurately describe the physical and mechanical properties of the mortars within the investigated range.

$$\begin{aligned}
 \text{Flow (mm)} = & 219.11 + 28.66 \times \left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) + 29 \\
 & \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) + \left(\left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times 10 \right) \\
 & + \left(\left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times \left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times -12.66 \right) + \left(\left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \right. \\
 & \left. \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times -66.66 \right) \quad (1)
 \end{aligned}$$

$$\begin{aligned}
 I.S.T \text{ (mm)} = & 509.66 + -189.5 \times \left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) + \\
 & -148.16 \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) + \left(\left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \right. \\
 & \times -92.5 \left. \right) + \left(\left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times \left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times 70.5 \right) \\
 & + \left(\left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times -13.50 \right) \quad (2)
 \end{aligned}$$

$$\begin{aligned}
 CS \text{ 28 (MPa)} = & 5.93 + 0.49 \times \left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) + 2.94 \\
 & \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) + \left(\left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times 0.455 \right) \\
 & + \left(\left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times \left(\frac{\text{Si}}{\text{Na}} - 1.62 \right) \times -1.93 \right) \\
 & + \left(\left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times \left(\frac{\text{Si}}{\text{Al}} - 5.80 \right) \times 1.27 \right) \quad (3)
 \end{aligned}$$

Figure 14 illustrates the evolution of flow spread, setting time and compressive strength of the studied geopolymers as a function of the Si/Na and Si/Al ratios. It is observed from the flow spread results that low Si/Al ratios combined with increasing Si/Na ratio leads to a slight increase in flow spread from 0.92 mm to 136.7 mm. This trend can be explained by a decrease in interparticle interactions and more effective dispersion of particles into the alkaline matrix, resulting in an increase in fluidity of the mixture. At higher Si/Al ratios, a more significant increase in flow spread (up to 241 mm) caused by the increasing Si/Na ratio was observed. This behaviour can be associated with structural changes of the geopolymer gel since, at high concentration of silicates, the formation of long-chain and less cross-linked aluminosilicate chains is enhanced. This

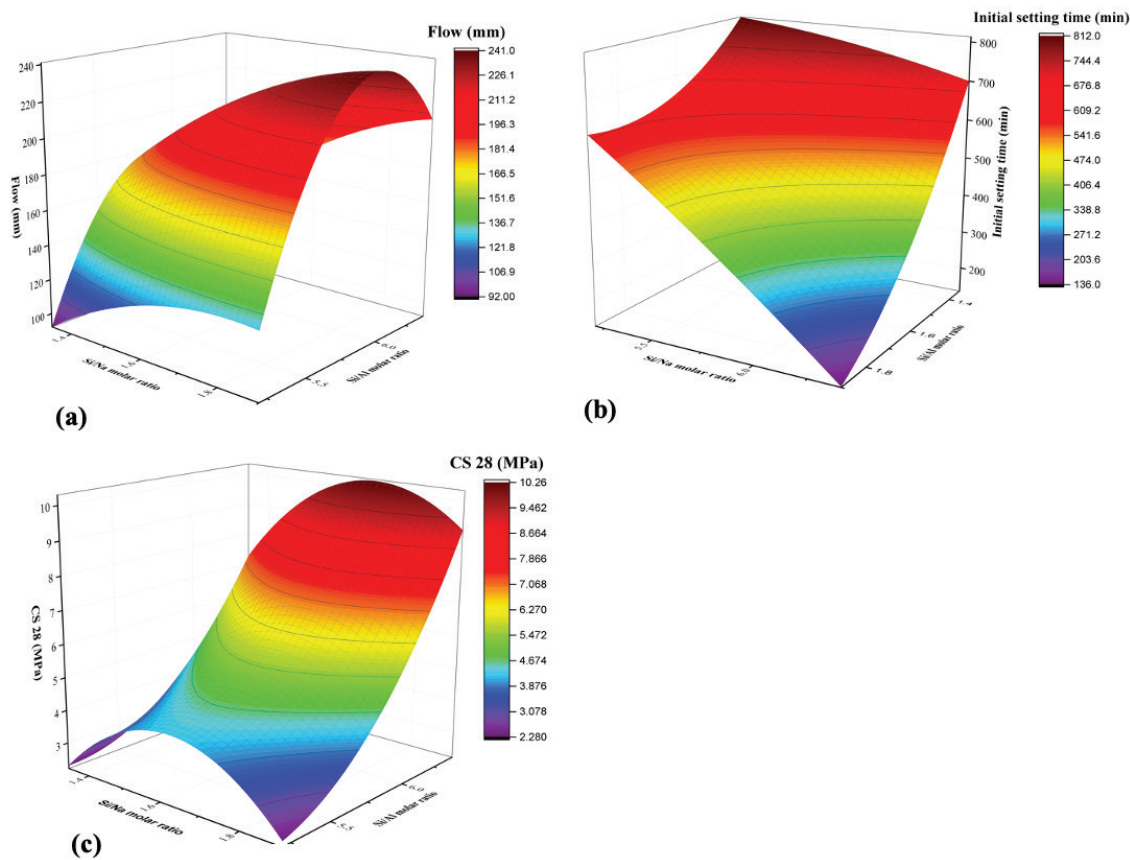


Figure 14. Surface response of studied mortars (a) flow; (b) Initial setting time and (c) Compressive strength at 28 days.

changes the structure of the paste and reduces its viscosity, which makes it more workable.

Conversely, the setting time results indicate that at low Si/Al ratios, an increase in the Si/Na ratio leads to a significant reduction in the initial setting time, decreasing from 812 minutes to 136 minutes. The increase in sodium ions facilitates the solubilization process of aluminosilicate precursors, leading to faster geopolymerization reactions and accelerated setting. But with the increase of Si/Al ratio, the setting retardation effect is less significant. This may be attributed to the increased availability of silicate species for geopolymeric integration and development of a more ordered and crosslinked network. The denser network reduces both the mobility of ions and the reaction rate, which counteracts with Na on setting time reduction. Figure 14. c indicates that the compressive strength of mortars increased from 2 MPa to 10 MPa as the Si/Al ratio increased. The higher Si/Al ratio facilitates the polymerization and densification of the geopolymeric matrix, which leads to a more cross-linked and denser aluminosilicate network that improves mechanical performance.

The Si/Na ratio initially increases compressive strength (CS), but then the values drop subsequently. This trend can be attributed to the twofold action of sodium ions in

the geopolymerization process. At medium Si/Na ratios sodium, even at low levels, functions as an activator producing the dissolution of the precursor and a more compact microstructure which increases strength. At a higher Si/Na ratio, an excess of sodium can lead to the formation of unreacted or weakly bonded silicate phases, increasing porosity and reducing the overall mechanical performance of the mortars.

CONCLUSION

This study provides important information on the performance of geopolymer mortars, highlighting the effect of $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio and the addition of metakaolin (MK) as a partial replacement for natural pozzolan (BNP). The following key observations were made:

1. The compressive strength (CS) of geopolymer mortars increases significantly with the increase in $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio, which reaches a maximum value of 9.93 MPa at a molar ratio greater than 6.4
2. Higher reactivity of MK is possibly responsible for 40% replacement, resulting in the CS values as high as 24 MPa.

3. The replacement more than 40%, the strength from MK was decreased, which demonstrates that its receding friability may not be complete and so it loses initial viscosity.
4. MK can play a crucial role in substituting Portland Cement in geopolymer formulations, which could promote sustainable construction.
5. The Central Composite Design (CCD) method statistical analysis explains a good association between experimental and predicted values. The RMSE of flowability, setting time and CS at 28 days were 0.88, 0.94, 0.98, respectively, which confirms the accuracy of predictive models as high-quality data with negligible error for a regression problem using machine learning for all the considered parameters in this research. In addition, p-values for flowability, setting time and CS were 0.010, 0.0008 and 0.0008, respectively, substantiate statistical significance of the models as well. The results corroborate the robustness of the validated models, as they were able to very precisely predict the three responses studied.

In the future aspect, proper designed and pattern of processing parameters needs to be optimized for fine performance in geo polymer mortars as construction materials.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE

Artificial intelligence was not used in the preparation of the article.

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