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Characteristics of metakaolin-based geopolymer mortars activated by a novel alternative activator

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Abstract: The relatively high cost of geopolymers remains a key limitation to their broader implementation. To minimize the dependence on expensive commercial activators such as sodium or potassium silicate, the use of alternative and sustainable activators derived from industrial by-products has become increasingly important. In this context, partially dealuminated kaolin (DK), a residue from the aluminum sulfate industry, was utilized as a source of amorphous silica to partially replace cementitious materials. In the current research, DK was employed to synthesize sodium silicate solution as a substitute for the commercial product in the formulation of metakaolin-based geopolymer mortars. Several preliminary trials were conducted to identify the optimal proportion of DK to sodium hydroxide solution (12M). A detailed experimental program was then designed to evaluate the influence of different parameters on both the fresh and hardened properties of GDMK-M. Four mixes were prepared and cured at ambient temperature to examine the impact of three variables: the DK-to-NaOH ratio, the activator-to-MK ratio, and the water-to-solid ratio. The experimental findings were further validated using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). The outcomes demonstrated that the DK-NaOH combination can be considered a promising and sustainable alkaline activator capable of enhancing GDMK-M through the geopolymerization process.

Keywords: Alkali-activated materials; Geopolymers; Partially Dealuminated kaolin; Alkaline activator; Metakaolin.

1. INTRODUCTION

Reducing emissions is expected to remain among the most critical global challenges in the coming years. However, the world's energy problem is even larger due to the link between construction industries and greenhouse gas emissions, the lack of energy resources, and the continuous rise in energy prices.

The Portland cement production process releases large amounts of CO_2 into the atmosphere, contributing approximately 5-8% of the total global annual CO_2 emissions [1]. Due to current environmental requirements aimed to reduce the CO_2 impact, mainly from cement production. Beyond the issue of CO_2 emissions, Portland cement is unlikely to fully satisfy the demands of future construction. Moreover, the production of Portland cement consumes approximately 3 billion tons of raw materials annually and demands a substantial energy input of around 4700 MJ per ton [2]. Therefore, the need for alternative binders has been revealed.

Alkali-activated binders, particularly geopolymers, are increasingly viewed as sustainable alternatives to Portland cement. Due to their advantages in energy efficiency and environmental sustainability, they offer a 43–59% reduction in energy consumption compared to traditional concrete [3]. In addition, geopolymers demonstrate excellent mechanical performance and long-term durability.

Geopolymerization involves a chemical reaction between various alumino-silicate materials with silicate activator under highly alkaline conditions, yielding polymeric [Si-O-Al-O] bonds, indicating that any amorphous Si-Al materials could become sources of geopolymerization. Alkali-activated binders are reported to produce up to 80% lower CO_2 emissions than Portland cement [4]. However, their comparatively higher production cost remains a significant limitation in relation to Portland cement [5], [6].

Typical aluminosilicate precursors to produce geopolymers include fly ash, blast furnace slag, incinerator bottom ash, ladle slag, metallurgical slag, ceramic waste, high magnesium nickel slag, mine tailings, construction and demolition wastes, kaolin, and metakaolin. Local availability primarily influences the selection of precursor materials [7]. Today, fly ash and blast furnace slag are the major materials used in geopolymer synthesis. Nevertheless, in Egypt, such materials are not yet available in commercially viable quantities.

Metakaolin-based geopolymers are synthesized by combining metakaolin powder with specific ratios of concentrated alkaline hydroxide and sodium silicate solution [8]. The resulting properties of the geopolymer depend on the alkalinity levels, which significantly influence the final performance characteristics [9], [10]. Metakaolin, the aluminosilicate source material, is produced by heating of kaolin $[Al_2Si_2(OH)_4]$, one of the naturally abundant clay minerals in the earth’s crust, to temperatures of (600–850 ºC) for periods ranging from 1 to 12 h [11]. The heating causes the bonded hydroxyl ions of kaolin to be removed, resulting in a disordered structure. Previous studies have shown that metakaolin-based geopolymers exhibit excellent mechanical performance and possess favorable long-term durability characteristics [12].

In most cases, alkaline activators are considered the primary contributors to the environmental impact of alkali-activated binders, in addition to being their costliest constituent. Sodium silicate, for example, has a high energy and environmental cost (=0.30 kg CO_2/kg) when produced traditionally [13], [14]. Consequently, there is a growing need to explore and develop alkali-activated materials utilizing low-cost activators, ideally derived from waste or residual resources, and processed through environmentally benign methods.

Partially dealuminated kaolin (DK) is produced as a by-product during the process of extracting aluminum from calcined kaolin through sulfuric acid treatment. In Egypt, the daily production is about 250 tons of wet material, and a large amount has been stored at the factory or dumped in landfills [15]. Numerous studies have investigated the potential for recycling dealuminated kaolin (DK) across various applications. In this context, Mostafa et al. [16] dealuminated kaolin (DK) shows a high level of pozzolanic activity. Hamed et al.[17] reported that DK can be used as a valuable backfill component within the intricate process of radioactive waste disposal. It was reported that DK can be blended with cement kiln dust to produce binder material [18]. Moselhy [18] indicated that partially replacing cement in concrete specimens with DK up to 15% has benefits in enhancing compressive strength and reducing greenhouse gas emissions. Abdelalim et al.[19] found a reduction in the setting time and flowability of mortar mixtures, whilst the compressive and tensile strengths were improved when cement was partially replaced with DK up to 20%. The optimal ratio was 10%. Contrarily, the findings of Amin et al. [20] indicated that the addition of 10–30% DK negatively affected the compressive strength of cement pastes. In geopolymer systems, El-Naggar et al. [15] found a lower density of insulating geopolymer bricks with DK included up to 20%. Salam et al. [21] utilized dealuminated kaolin (DK) as a precursor for the production of geopolymer bricks, activated using varying concentrations of sodium hydroxide (NaOH). Their findings indicated that increasing the NaOH concentration led to enhanced compressive strength and reduced water absorption.

This study aims to evaluate the properties of geopolymer mortar produced by alkali-activating metakaolin (MK) using an alkaline suspension composed of dealuminated kaolin (DK), sodium hydroxide, and water, proposed as a substitute for conventional sodium silicate. To assess the physical performance of the hardened geopolymer mortars, mechanical strength and water absorption tests were conducted. Moreover, microstructural features were examined using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) with energy dispersive X-ray spectroscopy (EDX).

2. MATERIALS AND EXPERIMENTAL PROGRAM

2.1 Materials

Partially Dealuminated Kaolin (DK) was obtained from the Egyptian aluminum sulfate factory in Egypt. It is produced as a waste by-product during the extraction of aluminum from calcined kaolin using sulfuric acid. Metakaolin (MK) was procured from the local market. The chemical composition of both DK and MK are illustrated in Table 1. Fig.1. XRD patterns of (a) MK and (b) DK. MK is mainly composed of quartz with minor anatase (A), together with a considerable amorphous background in the range of 15–30° 2θ. DK shows similar phases with an amorphous background, but with sharper quartz peaks reflecting structural changes due to dealumination. The amorphous portion of silicate easily reacts with NaOH forming the alkaline silicate activator and control silica: alumina ratio in the geopolymerization process.

TABLE 1. Chemical composition of MK and DK.

Compound	SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	TiO_2	Na_2O	K_2O	SO_3	P_2O_5	LOI	Total
MK	57.3	0.51	33.5	1.50	0.19	3.20	0.16	0.07	0.39	0.11	1.05	99.08
DK	82.0	0.16	6.70	0.53	0.08	3.50	0.02	0.04	0.95	0.02	4.70	99.13

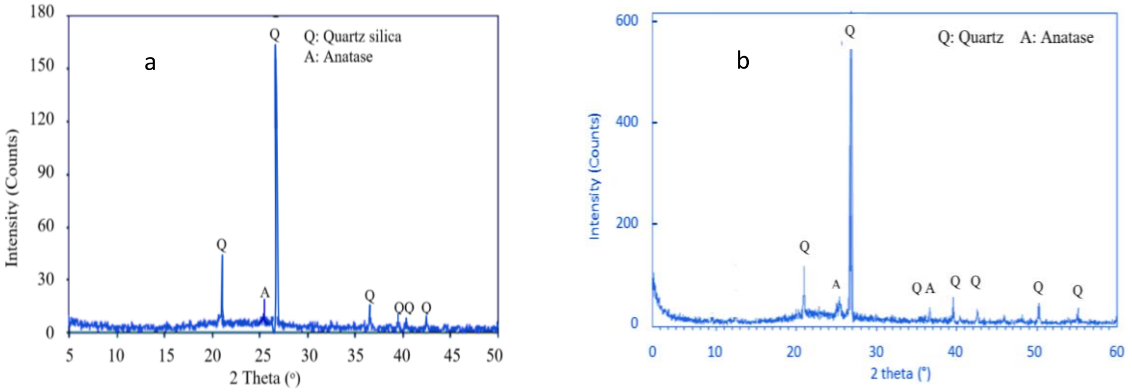


FIG 1. XRD analysis of (a) MK and (b) DK.

The fine aggregate used was clean siliceous river sand with a fineness modulus of 1.74, 1.5% water absorption, 2.5 specific gravity and was sieved through 4.75mm sieve. Finally, fine aggregate passed from sieve 2.36mm was used.

Sodium hydroxide pellets with a purity level of 98% was used, NaOH solution of a molarity 12 M was prepared by dissolving NaOH pellets in distilled water.

2.2 Preparing alkaline activator

Several trials were conducted until an adequate ratio of DK, water, and NaOH was obtained, defined by acceptable workability, proper setting behavior, and sufficient compressive strength. Seven different samples in two groups were used for the preparation of alkaline activator suspension. DK was used as it is, weighed, dissolved in water, and then NaOH solution (12M) was added to the mixture. The reaction was exothermic, so the suspension was cooled down to room temperature and left for 24 h after dissolution.

2.3 Mixture proportions

The experimental program was designed to conduct parametric studies on various mix design parameters for geopolymer mortars, including DK to sodium hydroxide ratio, alkaline activator to MK ratio, and water/solid ratio. A fixed sand/MK ratio of 3.0 was employed throughout the study. The experimental matrix in this study was established through a series of trials conducted to optimize a mix capable of delivering both high compressive strength and satisfactory workability. The mix proportions of the various mixes are presented in Table 2. In this study, the water/solid ratio was defined as the total mass of added water together with the water content of the NaOH solution, relative to the combined mass of MK, DK, and the solid portion of NaOH, while sand was excluded from the calculation.

TABLE 2. Mixture proportions.

Mix ID	**DK/NaOH (Activator)**	**Activator/MK**	**Water/solid**
M1	1.00	0.80	0.33
M2	0.89	0.85	0.34
M3	0.80	0.90	0.35
M4	1.14	0.75	0.32

2.4 Specimen preparation and curing method

Metakaolin and the alkaline suspension were initially mixed in the specified proportions for 10 minutes, followed by the addition of sand and continued mixing for an additional 5 minutes. The mixing duration was selected to ensure the preparation of a homogeneous and uniform mixture. The wet mixture was employed to measure the fresh properties of geopolymer mortar. The fresh mortars were immediately transferred to the molds, which were vibrated with a shaker for 1 min to eliminate the entrapped air. After vibrating, the molds were immediately transferred into the curing room (25 ± 2°C, 60 ± 5% RH), and the samples were covered with PE to prevent loss of moisture. After 24 hours, the specimens were removed from the molds and subsequently cured at ambient temperature until testing.

2.5 Testing of samples

2.5.1 Fresh properties

Fresh mortar flowability was determined through the Mortar Flow Table test, which was conducted according to the ASTM C1437 [22].

2.5.2 Hardened properties

Compressive strength was measured according to ASTM C109/C109M [23] by means of a 2000 KN capacity testing machine. The test was conducted on three 50 mm cubes at the ages of 3, 7, and 28 days, and the averages were reported herein. Splitting tensile strength of the SCCs was determined on Ø100 × 200 mm cylinder specimens at 28 days as per ASTM C496/C496M [24]. The splitting tensile strength was determined by averaging the results obtained from three cylindrical specimens.

2.5.3 Mass transport properties

Sorptivity is considered a reliable indicator for evaluating the durability of geopolymer concrete, as the penetration of chemicals is strongly affected by water movement through capillary suction. The sorptivity rate was calculated according to ASTM C1585–13 [25].

2.5.4 Dry shrinkage properties

Prismatic specimens with dimensions of 25 × 25 × 285 mm were prepared for the drying shrinkage test in accordance with ASTM C1148 [26]. The samples were demolded after 1 day of curing in a standard curing room, and the initial length of specimens was recorded. Afterward, the specimens were placed in a controlled environment with a relative humidity of 60 ± 5% and a temperature of 20 ± 2°C. At specific curing intervals, their lengths were recorded, and the drying shrinkage was determined.

2.5.5 Microstructure analysis

The microstructure of the geopolymer matrix was analyzed after 28 days of curing at ambient temperature to investigate its phase composition, chemical structure, and morphological features. Scanning electron microscopy (SEM) analysis was carried out using a TESCAN VEGA3 operated at 20 kV with a probe current of 100 pA. The scanning electron microscope was equipped with an energy-dispersive X-ray spectroscopy (EDX) detector, enabling detailed elemental analysis of the geopolymer samples. To improve surface conductivity and imaging quality, the samples were coated with a thin layer of gold prior to SEM examination.

3. TEST RESULTS AND DISCUSSIONS

3.1 Workability

All fresh mortar mixes exhibited a stiff and cohesive consistency, attributed to their low liquid-to-solid ratio. As illustrated in Fig.2, the initial flow values exhibit a general increasing trend with the rise in the Activator/MK ratio. Mix M4 with a low Activator/MK ratio of 0.75, which had the lowest initial flowability of 80%. However, increasing the Activator/MK ratio up to 0.9 for M3 produced more initial flowability of 110%.

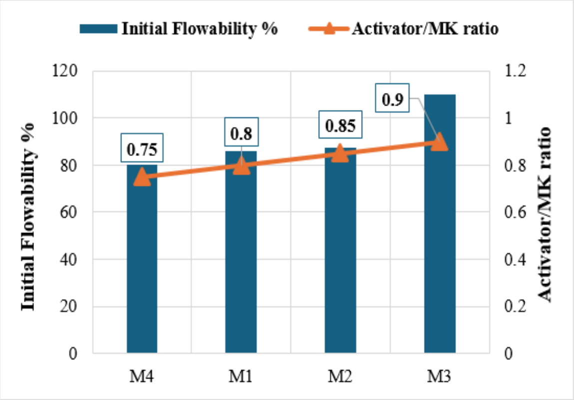


FIG 2. Initial Flowability % versus Activator/MK ratio.

3.2 Compressive strength

The compressive strength values of GDMK-M at 3, 7, and 28 days are illustrated in Fig.3. For each geopolymer mortar, substantial development of the compressive strength can be achieved within the first 3 days. A gradual increase in compressive strength was observed as the curing period progressed from 3 to 28 days. The compressive strength values are in the range of 23.41–36.09 MPa at 3 days. The highest early-age strength was observed in mix M2, while mix M4 exhibited the lowest value. At 3 days, mortar M2 achieved a compressive strength of 36.09 MPa, equivalent to approximately 77.8% of its 28-day strength. At 28 days, mix M2 exhibited the highest compressive strength, measuring 46.41 MPa, and mix M3 had the lowest (39.01 MPa). Also, as illustrated in Fig. 3, increasing the A/B ratio from 0.75 to 0.85 led to a noticeable improvement in compressive strength at all curing ages. The highest strength was observed at an Activator/MK ratio of 0.85 (M2), indicating an optimal balance between activator content and geopolymer formation. However, further increasing the ratio to 0.90 (M3) caused a slight decrease in strength, likely due to excess liquid leading to higher porosity. These results confirm that the Activator/MK ratio significantly affects the geopolymerization process and mechanical performance.

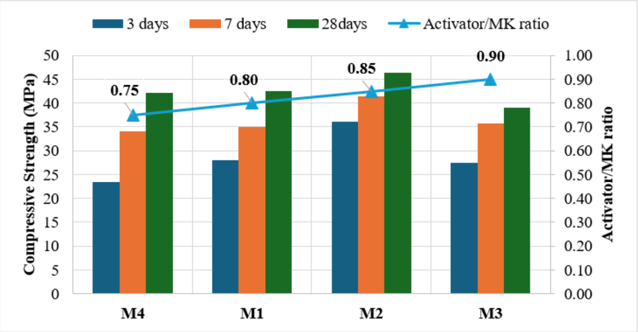


FIG 3. Compressive strength development and effect of Activator / Binder ratio on compressive strength for GDMK-M.

3.3 Splitting strength

Fig.4 illustrates the relationship between compressive strength and splitting tensile strength of GDMK-M mixtures after 28 days of curing. As shown, Mix M2 achieved the highest values for both compressive (46.41 MPa) and splitting tensile strength (4.1 MPa), indicating a denser and more cohesive geopolymeric matrix. Mixes M1 and M4 presented similar compressive strengths (around 42–43 MPa) but with slightly lower splitting tensile strengths of 3.2 MPa and 3 MPa, respectively. The lowest mechanical performance was observed for Mix M3, which attained compressive and tensile strengths of 39.01 MPa and 3.5 MPa, respectively. These results confirm that the splitting tensile strength trend aligns with the compressive strength, reflecting the influence of mix design on the overall structural integrity and crack resistance of the geopolymer mortar.

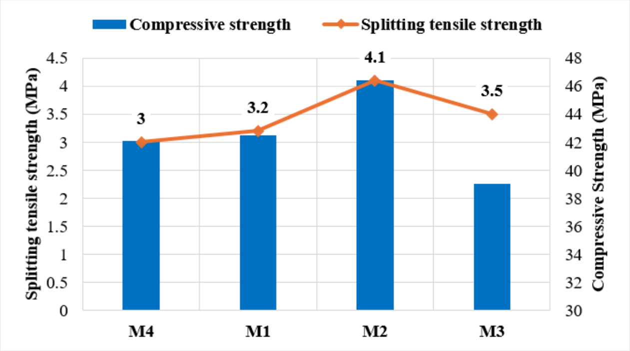


FIG 4. Splitting tensile strength versus compressive strength for GDMK-M at 28 days.

3.4 Mass transport properties

Fig.5 shows the relationship between sorptivity and compressive strength for GDMK-M mixes at 28 days. The results clearly indicate an inverse relationship: mixes with higher compressive strength tend to exhibit lower sorptivity values. Mix M2, which recorded the highest compressive strength (46.41 MPa), also showed the lowest sorptivity, indicating a denser microstructure with reduced capillary pores and improved resistance to water ingress. Conversely, Mix M3 had the lowest compressive strength (39.01 MPa) and the highest sorptivity, suggesting a more porous matrix and lower durability. Mixes M1 and M4 showed intermediate values for both properties, confirming that enhanced compressive strength is generally associated with better durability performance due to lower permeability. This results in agreeing with that obtained by Thokchom et al. [27].

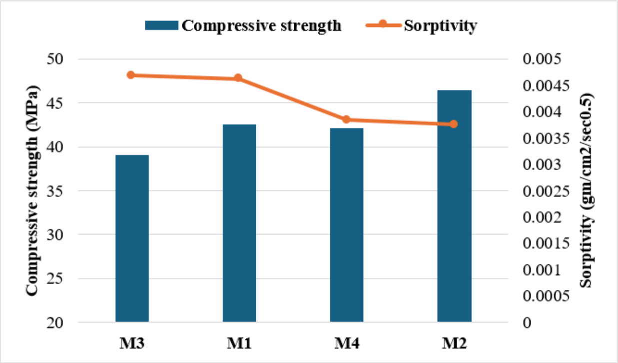


FIG 5. Sorptivity versus compressive strength for GDMK-M at 28 days.

3.5 Dry shrinkage

As shown in Fig.6, the development of dry shrinkage for the GDMK-M mixes increases significantly at early ages and then grows more gradually over time. For example, the 14-day dry shrinkage of mortar M3 reaches 53.37 micro strain, accounting for about 61.2% of its 90-day value, indicating that increasing the alkaline activator content accelerates early shrinkage. In contrast, mortar M2 records only 3.625 micro strain at 14 days (around 17.26% of its 90-day shrinkage), reflecting slower moisture loss and better dimensional stability. Among all mixes, M2 exhibits the lowest overall shrinkage, highlighting its superior resistance to drying compared to the other blends. Moreover, it is noteworthy that the drying shrinkage of these geopolymer mortars is much lower than that typically reported for conventional Portland cement mortars [28], [29]. Overall, these results confirm that mix composition significantly influences shrinkage behavior and demonstrate that M2 is the most suitable for applications requiring enhanced dimensional stability.

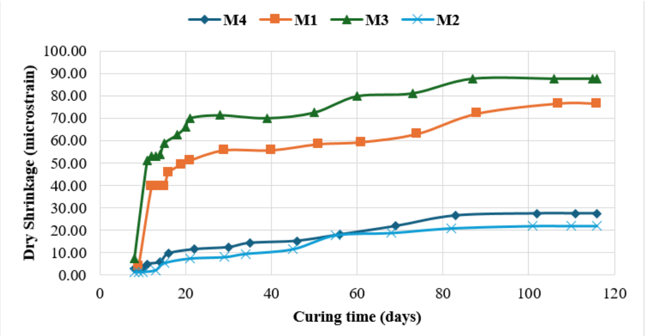
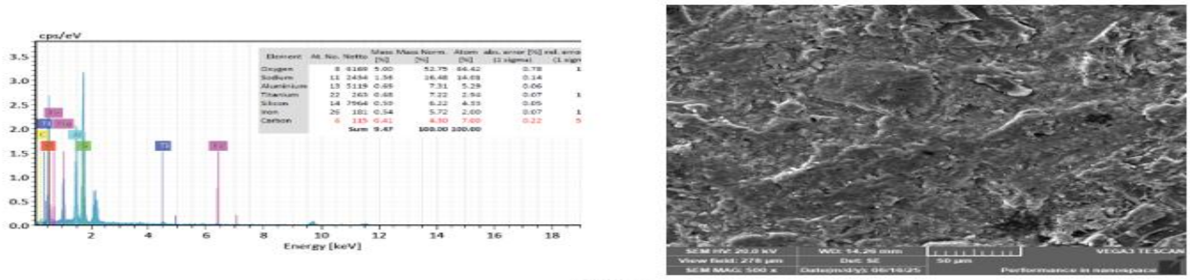


FIG 6. Dry shrinkage for different GDMK-M

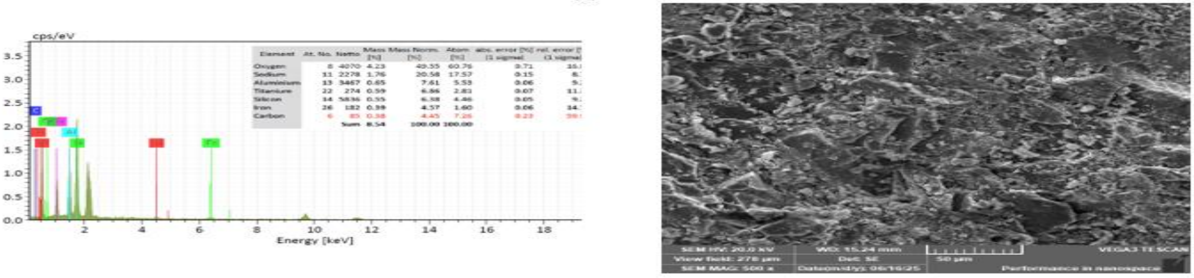
3.6 SEM with EDX

The SEM micrographs were used to investigate the microstructure of the GDMK-M mixes M2 and M3, and energy dispersive spectroscopy (EDX) was performed on the same images to analyze the chemical composition of their geopolymerization products. The EDX results for these mixes are presented in Fig.7, which also shows the corresponding SEM images at identical magnification. The analysis revealed oxygen (O), aluminum (Al), silicon (Si), carbon (C), and sodium (Na) as the major elements, with minor contributions from iron (Fe), titanium (Ti), and calcium (Ca), attributed to the raw material composition. The SEM images reveal that mix M2 exhibits a denser and more homogeneous microstructure with fewer pores and white spots; these spots likely represent Na_2CO_3 formed by the carbonation of NaOH, as confirmed by EDX. In contrast, mix M3 shows a more porous and heterogeneous matrix with a higher presence of these white spots, indicating less effective geopolymerization. This explains the superior mechanical properties and lower shrinkage of M2 compared to the weaker performance and higher shrinkage observed in M3.



Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error (1 sign
Oxygen	8	4169	5.00	52.75	66.42	0.78	1
Sodium	11	2434	1.36	16.48	14.03	0.14	
Aluminium	13	5119	0.65	7.31	5.29	0.06	
Titanium	22	263	0.68	7.22	2.94	0.07	1
Silicon	14	7964	0.59	6.22	4.33	0.05	
Iron	26	181	0.54	5.72	2.00	0.07	1
Carbon	6	115	0.41	4.30	7.00	0.22	5
		Sum	9.47	100.00	100.00		

(a) M2



Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [(1 sigma)
Oxygen	8	4070	4.23	49.55	60.76	0.71	16.
Sodium	11	2278	1.76	20.58	17.57	0.15	6.
Aluminium	13	3467	0.65	7.61	5.53	0.06	9.
Titanium	22	274	0.59	6.86	2.81	0.07	11.
Silicon	14	5836	0.55	6.38	4.46	0.05	9.
Iron	26	182	0.39	4.57	1.60	0.06	14.
Carbon	6	85	0.38	4.45	7.26	0.23	39.
		Sum	8.54	100.00	100.00		

(b) M3

FIG 7. SEM/EDX analysis results for (a) M2 and (b) M3

4. CONCLUSIONS

From the investigations carried out in the present study, it can be concluded that:

- Geopolymer products can be successfully obtained using a DK–NaOH mixture, which represents a promising and sustainable alternative alkaline activator.
- DK-NaOH gel can be used as an alternative to commercial sodium silicate in the geopolymerization process.
- The workability was found to be improved with the increase of activator to MK ratio, with the maximum initial flowability obtained at Activator/MK = 0.9.
- The compressive strength generally increases with an increase in activator-to-binder ratio up to 0.85.
- A sustainable compressive strength can be achieved within the first 3 days (up to 36 MPa), and a gradual increase occurs with the curing time up to 46.41 MPa at 28 days.
- Overall, the splitting tensile strengths reflected the same patterns observed in compressive strength for all mixes.
- An inverse relationship was observed between water sorptivity and compressive strength, where lower sorptivity corresponded to higher strength values.
- The development of drying shrinkage in GDMK-M follows a trend similar to that of strength development; specifically, drying shrinkage increases markedly at early ages and then tends to stabilize over time.
- SEM–EDX analyses confirmed the experimental findings, revealing that lower sorptivity corresponded to a denser microstructure, consistent with the enhanced compressive strength.

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