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Investigating the Effect of Different Parameters on Physical Properties of Metakaolin-Based Geopolymers

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HIGHLIGHTS

- The presence of K+ ions negatively influences GP's physical properties.
- W/MK ratio is the most important factor that affects GP's physical properties.
- Increasing the Si/Al ratio will lead to an increase in the density of GP.
- Short mixing time is necessary to achieve better GP properties.

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ABSTRACT

Recently, geopolymers have received widespread attention due to their ability to completely replace ordinary cement with better efficiency, lower cost, and less damage to the climatic environment. This paper aimed to prepare MK-based geopolymer cement at ambient temperature with different alkaline activators and processing parameters. XRD, PSA, DTA-TGA, SEM, and other techniques have characterized the prepared samples. ANOVA test was employed to identify the main effect of the processing parameters. Results showed that the incorporation of potassium ions has a negative effect on the physical properties of GP, in which the presence of such ions tends to decrease the density of GP. Furthermore, the apparent porosity and water absorption were increased. For Na and K, Na-activated GP, it was also concluded that the density of GP increases by increasing Si/Al ratios. Despite the Na-based GP processes having a larger density in compared to the K, Na-based ones. The results also suggested a strong effect of the W\MK ratio on physical properties, in which decreasing this ratio is necessary to achieve GP with better properties. The findings also revealed that a one-minute mixing period was sufficient for producing a homogeneous and dense GP paste.

1. Introduction

Concrete is only second to water in terms of global use. The demand for Portland cement grows in tandem with the demand for concrete as a building material. Climate change is mainly because of global warming, in addition to the environmental protection occupy considerable concerns. Global warming is caused by human activities that emit greenhouse gases, like (CO_2) into the atmosphere. CO_2 contributes to around 65% of global warming among greenhouse gases [1]. The cement industry is considered accountable for some of the CO_2 emissions. This is attributed to the fact that the manufacture of 1 ton of Portland cement release about 1 ton of CO_2 into the atmosphere. Hence, the design and production of alternative low-carbon binders are therefore recognized as one choice to reduce the emissions of CO2. In this respect, geopolymers show great promise for usage as an alternative binder to Portland cement in the concrete industry, based on the availability of raw material [2,3]. In 1978, Davidovits invented the term 'geopolymer.' A geopolymer is an inorganic polymeric material made by reacting aluminosilicate sources with a high alkaline silicate solution which is accompanied by curing at room temperature or slightly higher temperatures [4]. In recent years, Geopolymer has become a very common research subject. The key explanation for its success is that using geopolymers brings with it the ability to fully substitute Portland cement as the cement base [5]. Water affects the formation, structure, and properties of geopolymers. It is an integral part of geopolymer. Water provides a medium for dissolution, transport of dissolved ions, hydrolysis of oligomers, and polycondensation of oligomers [6]. According to Rahier et al. [7], the high and low water content decrease the reaction rate. This is believed to be due to the decreased OH^- concentration at high water content. In the other way, at low water content, even though the OH^- concentration increases, the reactive species (monodeprotonated monomer $H_3SiO_4^-$) for the reaction between silicate oligomers decreases and lowers the rate of geo-polymerization reaction. A high concentration of soluble silicate is required to develop an alumino-silicate gel that provides good interparticle bonding and

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good physical properties to geopolymers. Due to the high amount of alkali alumino-silicate gel formed due to the high reactive silica content, the resultant material acquired a high mechanical strength [8]. Duxson et al. [9] studied the effect of using different Si/Al ratios and alkali types on the mechanical characteristics of geopolymer cement cured at 40^{O}C for 20 hrs. The results found that the mixed-alkali specimens with a high Si/Al ratio exhibited significant strength increases compared to pure alkali specimens. The Si/Al ratio of 1.90 consistently exhibits the highest compressive strength of GPs specimens. The nature of the alkali metal cation in alkaline solution, which is usually a mixture of M_2SiO_3 and MOH with M = Na or K, plays a crucial role in the structure and thus the properties of geopolymer [10]. Jaarsveld et al. [11] revealed that the cations of alkali metal had a substantial impact on the dissolution of raw material and the nucleation process that results in a three-dimensional structure. The crystal morphology is affected by the size of the cations. In the same conditions, K^+ forms a geopolymer with more condensation than Na^+. In contrast, the silica and alumina oligomers dissolute greater in the presence of Na^+ cations compared to the presence of K^+ cations. Lizcano et al. [12] investigate MK-based geopolymer paste's mechanical and physical properties prepared from the alkali (KOH or NaOH) activation of SiO_2 and metakaolin mixture. They found that k-based geopolymer had a higher density than Na-based one. The lower density for Na-based GPs is that the activation solution based on sodium is more viscous than the activation solution based on potassium and polymerizes much faster, thus prohibiting good homogenization, long mixing, and degassing for Na-base activating solutions. The results also concluded that K^+ cations pair with larger silicate oligomers, while Na cations favor ion paring with smaller silicate monomers and dimers. This study aims to prepare geopolymer paste using mixtures of local Iraqi metakaolin and alkaline solution at room temperature and under different conditions to get the optimum physical properties. The performance of (Na) and (K, Na) based geopolymers are compared when exposed to different processing parameters, including W\MK ratio, Si\Al ratio, and mixing time. ANOVA test was employed in this work to identify the main effect of the processing parameters.

2. Experimental Part

2.1 Synthesis of Geopolymer

The aluminosilicate source used in the current study was metakaolin powder. MK was obtained after calcinating kaolin clay for 3 hours at a temperature of 750 ^{O}C and with a 10^{O}C/min heating rate. Two types of activation solutions were used, which were sodium and potassium based, as shown in Table 1. First, the activation solution was prepared by mixing potassium hydroxide and/or sodium hydroxide solution with certain molarities for 5 minutes. After that, the sodium silicate powder with the required amount was added to the mixture of hydroxides with a constant ratio of Na_2SiO_3/NaOH of 1.5 and mixed for 10 minutes. The major source of silica used was nano silica gel powder which was added carefully to the mixture under continuous stirring. After adding all silica gel, the solution was heated to a temperature of 80C for 30 minutes. After ensuring the dissolution of silica, the solution was placed under the mechanical mixer, and MK was added carefully to the solution. At this time, geopolymer paste was formed, poured into a plastic mold with dimensions (diameter of Ø = 2 mm and length L = 4mm), and placed in a humid environment for 28 days before testing.

Table 1: The major batches used in the current work

Batches NO.	K_2O/Al_2O_3 (mole)	Na_2O/Al_2O_3 (mole)	Si/Al Atomic ratio
Batch.1	-	1	1.6-1.85
Batch.2	0.6	0.4	

MK-based GP cement was synthesized with different MK/ratio, Si/Al ratio, and Mixing times with equal levels of each of them to investigate these parameters' effect on GP's physical properties. Table 2. show the values of these parameters and their level. However, the major experiment employed in the current work is explained in Table 3.

Table 2: The parameters and their levels of values

NO.	Parameters	Level 1	Level 2	Level 3
1	W\MK ratio	0.46	0.74	1
2	Si\Al ratio	1.6	1.72	1.85
3	Mixing Times (min)	1	3.5	6

Table 3: The experiments that were employed in the current study

Experimental No.	W\MK ratio	Si\Al ratio	Mixing Time
1	1	1.6	6
2	1	1.72	3.5
3	1	1.85	1
4	0.74	1.6	3.5
5	0.74	1.72	6
6	0.74	1.85	3.5
7	0.46	1.6	1
8	0.46	1.85	1
9	0.74	1.72	3.5
10	0.46	1.72	3.5

2.2 Physical Properties Measurement

The following are the steps for calculating apparent porosity, water adsorption, and bulk density using the Archimedes method (ASTM C373-88) [13]:

1) The test specimens were dried for 24 hours at 110°C in a drying oven, then their dry mass (D) was measured after they were cooled to room temperature.
2) The specimens were submerged in distilled water, boiled for five hours, and then allowed to soak for an additional 24 hours to determine their suspended weight (S).
3) To record the saturated mass (M), the extra water must be carefully removed from the sample's surface using a moistened cotton towel.
4) The apparent porosity, bulk density, and water adsorption were calculated by employing the equations described below:

$$P = (M-D)/(M-S) * 100 \tag{1}$$

P: Apparent porosity
M: Saturated weight
D: Dry weight
S: Suspended weight
Bulk density can be expressed by the following equation:

$$B = D/(V) \tag{2}$$

B: Bulk density
V: Exterior volume

$$V = M-S \tag{3}$$

Water adsorption was calculated using the following equation:

$$WA\,(\%) = (M-D/D) * 100 \tag{4}$$

2.3 The Applied Techniques

2.3.1 Particle size analysis (PSA)

By employing a laser particle size analysis, (Bettersize 2000 instrument Ltd., China), the particle size distribution of kaolin and metakaolin powders was calculated. This test was performed at Babylon University/ Ceramics Engineering Department.

2.3.2 X-ray diffraction (XRD)

The phases present in materials were specified by employing X-ray diffraction. An x-ray diffractometer was used for this test (XRD 6000, Shimadzo, Japan) performed at Babylon University/ Ceramics Engineering Department.

2.3.3 Differential thermal analysis / thermogravimetric analysis (DTA/TGA)

This analysis was performed to evaluate the thermal transformation, and the weight loss of Kaolin during heating, this test (SDT Q600 V20.9 Build 20, Universal V4.5A TA Instruments) was performed in Tehran, republic of Iran, at a temperature of 950 °C.

2.3.4 The scanning electron microscope (SEM)

The microstructure and morphology of kaolin and metakaolin powder have been observed using Thermo Scientific Axia ChemiSEM SEM.

3. Results and Discussion

3.1 Results of XRD

X-ray diffraction results for kaolin and metakaolin powders were demonstrated in Figures (1.a and b), respectively. The results of the XRD analysis for Kaolin in Figure (1.a) show that the material is rich in kaolinite, and small quantities of quartz were also characterized. However, the major peaks related to kaolinite and quartz were labeled with the letters K and Q. It was observed that after calcination Figure (1.b), the disappearance of the peaks related to kaolinite suggests a complete transformation of kaolinite to metakaolin. This is explained by the dehydroxylation of the water molecules that exist in the kaolinite structure by heat treatment. The major peak present in the metakaolin was at $2\Theta = 26.63^{\circ}$, which was attributed to residual quartz in the material.

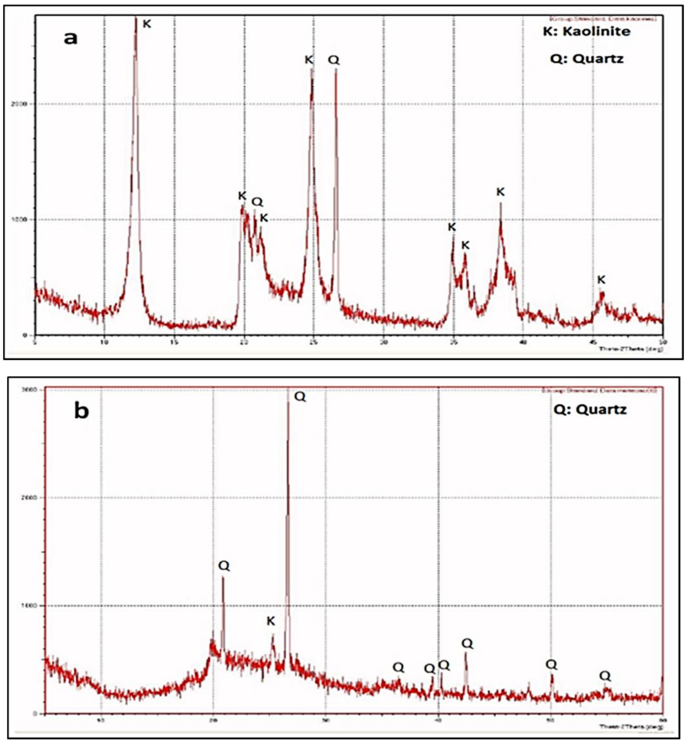


Figure 1: XRD analysis results for a) Kaolin and b) Metakaolin

The XRD pattern results of Batches 1&2 are illustrated in Figures (2.a and b), respectively.XRD patterns show that the metakaolin-based geopolymers resulted in two types of diffraction patterns. The first pattern corresponded to the crystalline materials contained in metakaolin (quartz) and small quantities of montmorillonite, while the second one was related to aluminosilicate amorphous materials. However, the peak related to quartz were decreased in intensity, indicating that the parent material was not totally dissolved into the inorganic polymeric material. According to the X-ray data, when the metakaolin was activated with an alkaline solution, there was no clear evidence of new crystalline phases. Instead, the same crystalline phases were identified in metakaolin and geopolymer. There is, however, a broad “amorphous hump” from a center at (2θ = 23°) to (2θ = to 35°). It has been noted that this hump is indicative of a geo-polymeric reaction [14].

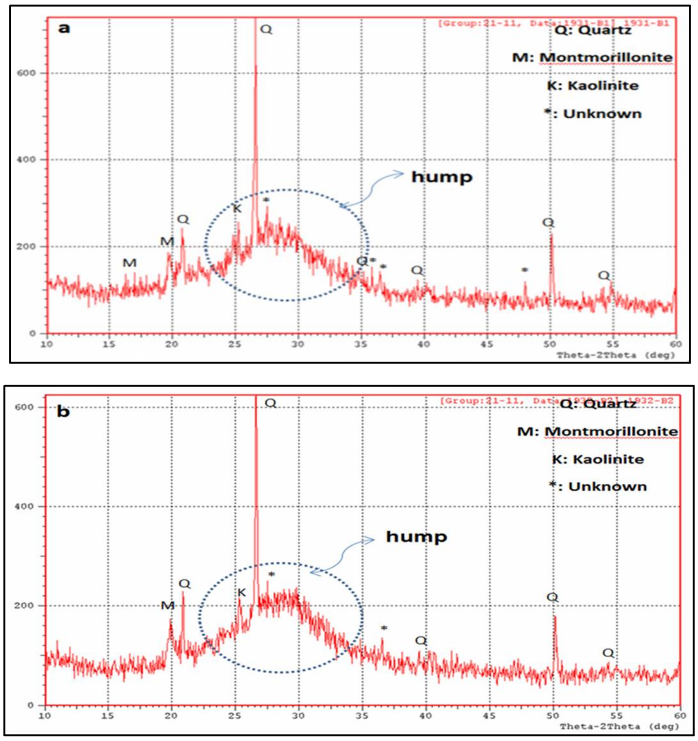


Figure 2: XRD analysis results for a). Batch 1 and b) Batch 2

3.2 Result of DTA/TGA Analysis

As shown in Figure 3, the thermal behavior of Kaolin was obtained via drawing DTA/TGA curves at a temperature range of (25 – 950 °C). A modest and hardly noticeable mass loss in the TGA pattern is connected with the loss of adsorbed water in the temperature range of 50–100 °C. However, in the DTA curve, the temperature range of 450–600°C was shown to have the most mass loss, represented by an endothermic peak. The TGA curve revealed a total mass loss of 21.78 %. The temperature for maximum mass loss was 516.76°C. According to DTA curves, Dehydroxylation of kaolinite to metakaolinite is responsible for the mass loss in this region [15]. It needs to point out that the mass loss that happened through the metakaolinization process was caused due to formation of disordered structures induced by the release of hydroxyl groups connected with layers of octahedral sheets [16,17]. The following reaction represents the thermal reaction process**:**

$$Al_2Si_2O_5\,(OH)_4 \xrightarrow{450^{o}C\,-\,700^{o}C} Al_2O_3.{}_2SiO_2 + {}_2H_2O(g) \qquad (5)[18].$$

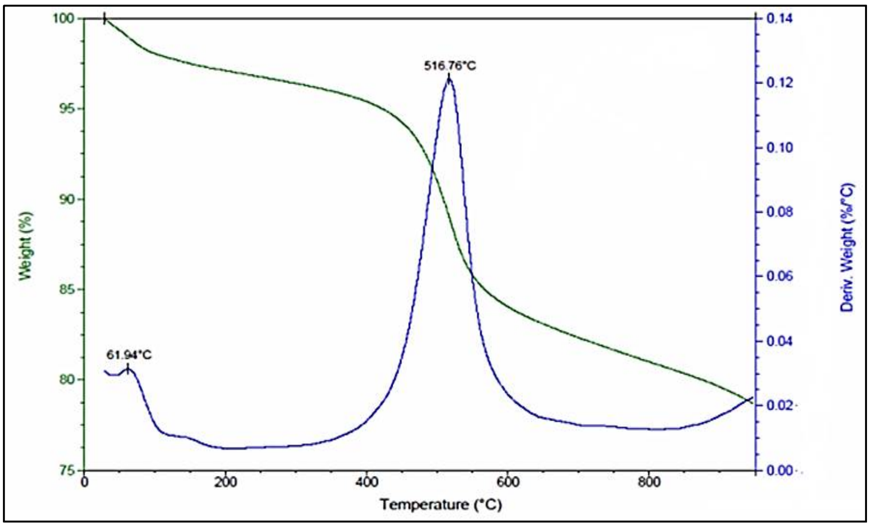


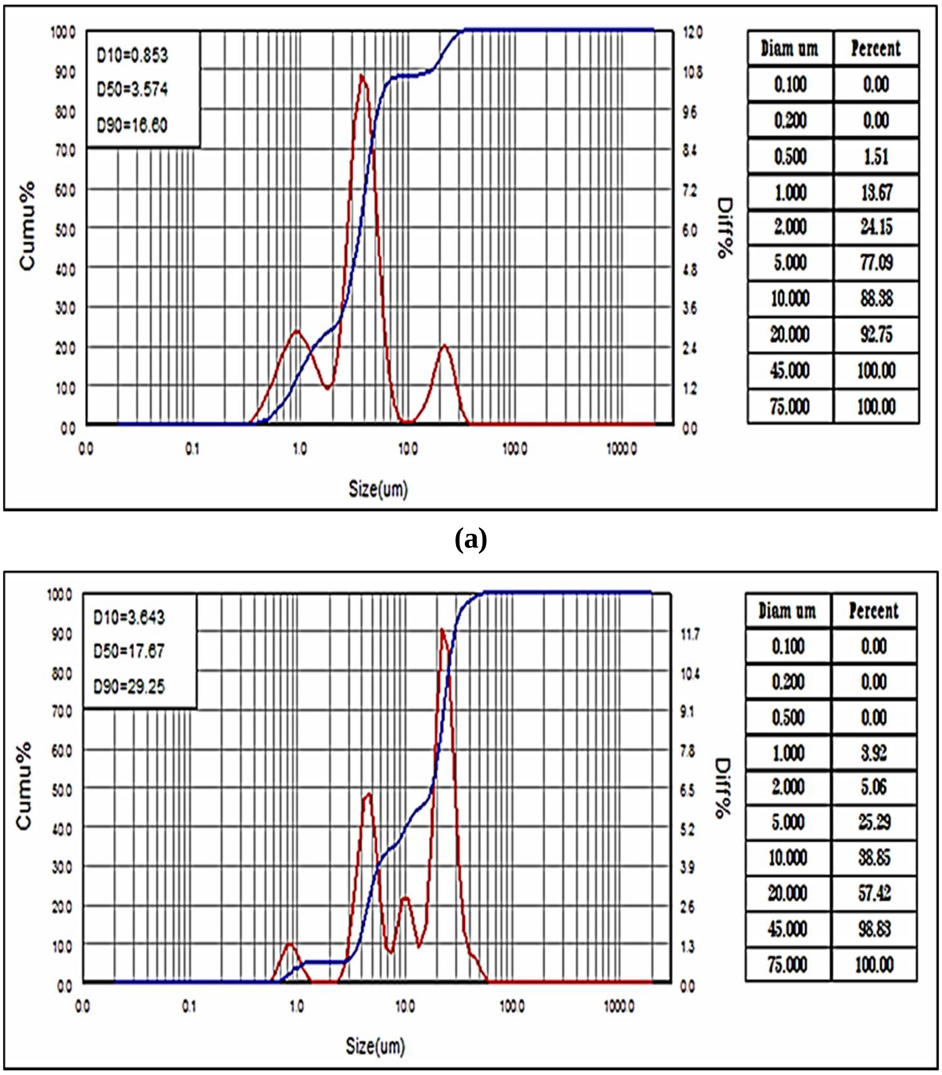
Figure 3: DTA/TGA plot for Kaolin

3.3 Particle Size Analysis Results

The laser particle size analyzer (Bettersize2000) was employed to determine the particle size of the samples. The Particle size significantly influences the physical and mechanical properties of geopolymer. Figure.4 (a and b) display the result of the analysis of the particle size distribution for the Kaolin and the metakaolin powders, respectively. Figure (4.a) indicates that Kaolin's particle size distribution is multimodel and is over the range (0.271µm-37.56µm). It can also be observed from Figure (4.b) that the particle size of the MK is distributed across the range (0.561µm-56.13µm) with a D50 of 17.67µm. Obviously, the particle size distribution for the MK was multimodal, where the first mode which focused at 0.8 µm, is regarding the smaller particle. While the mode related to the larger particles is centered at 24 µm, the percentage of the smaller particles in the first mode is much less than that of the other mode for larger particle size. In general, the particle size distribution in powder is significantly affected by the existence of agglomerations. Meanwhile, the agglomeration content increases the percentage of larger particle sizes.

3.4 SEM Results

Figure 5 (a,b) and (c,d) illustrate the SEM images for kaolin and metakaolin powders, respectively. It is clear from (Figures 5a and b) that kaolin powder is composed of particles of hexagonal shape, as described in the literature [19]. The figure also observed the formation of what is called a booklet structure. However, the formation of such structures results from the staking of kaolinite particles. On the other hand, the secondary van der Waals forces are responsible for such agglomeration formation. The hexagonal geometry of kaolinite particles is preserved following calcination, as evidenced by micrographs of metakaolin Figure 5 (c and d). On the other hand, the metakaolin particles do not form the booklet structure [20,21].



(a)

Diam um	Percent
0.100	0.00
0.200	0.00
0.500	1.51
1.000	13.67
2.000	24.15
5.000	77.09
10.000	88.38
20.000	92.75
45.000	100.00
75.000	100.00

(b)

Diam um	Percent
0.100	0.00
0.200	0.00
0.500	0.00
1.000	3.92
2.000	5.06
5.000	25.29
10.000	38.85
20.000	57.42
45.000	98.83
75.000	100.00

Figure 4: Particle size analysis for a) kaolin and b) metakaolin

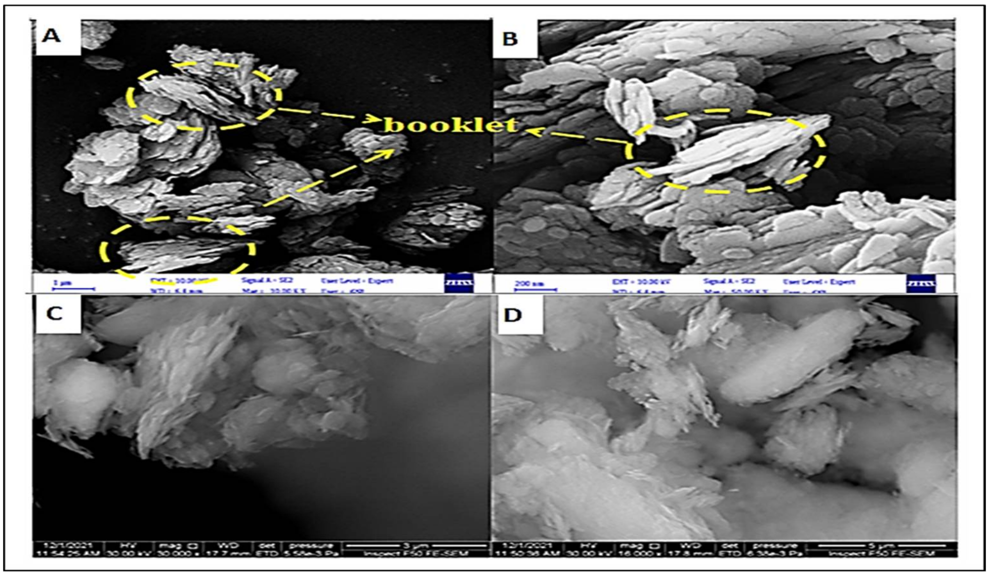


Figure 5: SEM image for (a) and (b) Kaolin; (c) and (d) Metakaolin

3.5 Physical Properties Results

3.5.1 Bulk density

The main effect plots of processing parameters on the bulk density of MK-based GP paste are illustrated in Figures 6 (a and b). It could be clearly seen that the bulk density generally decreased with increasing the water content because increasing the content of water was believed to provide the formation of high porosity in the GP since the excess water leaves pores after evaporation, and this would contribute to the poor compaction of the GP paste and thus lower density [22]. It was previously reported that the increase in Si/Al ratio usually leads to an improvement in the rate of geo-polymerization reaction. However, it can be seen from the Figure that by increasing the Si/Al ratio, the bulk density increase, which is attributed to the fact that increasing this ratio leads to the formation of a compact and dense geopolymer body with fewer pores. The impact of the Si/Al content on the bulk density of Na-geopolymer is higher than that of potassium-containing geopolymer. It has also been found that the bulk density of Na-based geopolymer is higher than that of K-containing geopolymer. This contradicts the previous study's findings by Lizcano et al. [12]. In any chemical synthesis

process, proper mixing of initial raw materials is critical. One of the most important parameters of mixing procedures is mixing time, which can be influenced by the mix proportion and the amount of water used [23]. Meanwhile, the optimum mixing time achieved to obtain a high bulk density for Batch.1&2 was 1 min. However, time > 1 min leads to a significant decrease in GP density.

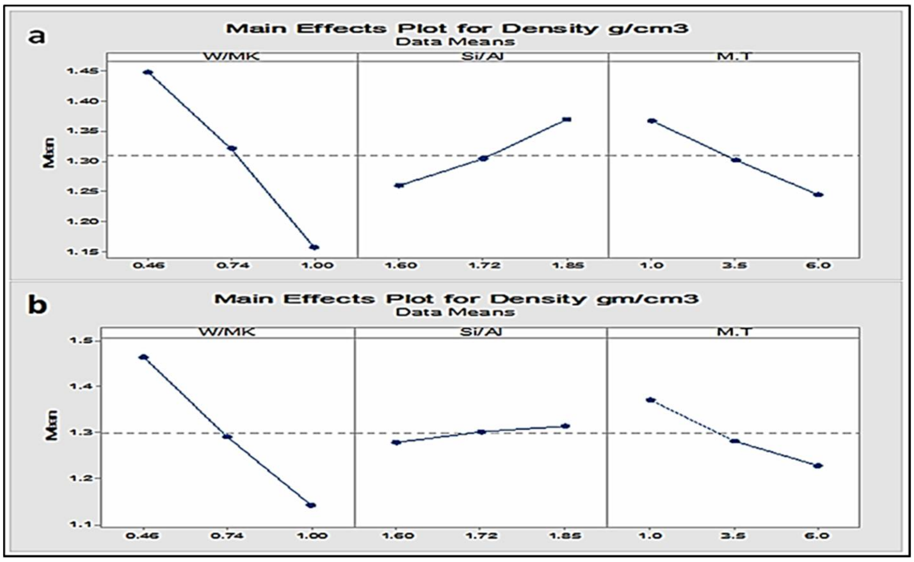


Figure 6: The main effect of parameters on the density of a) Batch 1 and b) Batch 2

3.5.2 Apparent porosity

Figures 7(a and b) clarify the main effect plot of inputs parameters on the apparent porosity of GP cement. It is clear from the figures that the porosity increased with increasing the amount of water. However, there are two possible causes for pores in solid samples: The primary cause is that the air voids are introduced during mixing. Under the ambient circumstances, this is inevitable (such types of pores can be avoided using vacuum mixing). On the other hand, the second probable reason is that during sampling, the unreacted particles of metakaolin in the structure may drop off from the shell, causing the development of pores in the GP structure. Whether pores are created as a result of mixing or the detachment of unreacted particles, they are all weak areas of the structure that negatively affect mechanical properties [24]. However, water evaporation during the curing period may also contribute to the formation of pores. It can also be noted that the Si/Al ratio of ≥ 1.72 was adequate to achieve low water absorption. In the case of mixing time, we observed that prolonged mixing for up to 6 min would increase the porosity of GP. However, these results correspond with that reported by Chindaprasirt et al. [23] who stated that the increasing mixing time will lead to increased air voids and large capillary pores forming. Thus, the strength of the geopolymer paste drop.

3.5.3 Water absorption

The key element that will affect the durability of geopolymer cement and concrete is water movement through the structure. Due to this issue, a water absorption test is performed to determine how much water is absorbed by the concrete [25]. A noticeable increase in water absorption was observed by increasing the W/MK ratio, as shown in Figure8 (a and b). This is because, as the amount of water increases, the pore size and pore volume of geopolymer cement increase, giving good water absorption but decreasing their mechanical strength [26]. Meanwhile, low water absorption is a good indicator of less open porosity. Porosity can lead to sample crack and low compressive strength [27]. If the pores are disconnected and have less pore size, the percentage of water absorption is respectively low [28]. The main effect of the Si/Al ratio on water absorption is similar to its effect on apparent porosity. Increasing this ratio up to 1.72 will reduce the water absorption due to compact and dense GP body with fewer pores. However, mixing for up to 6 min leads to increased GP cement's water absorption for the two batches. This suggested that short-term mixing may be employed to make homogeneous, degassed GP pastes.

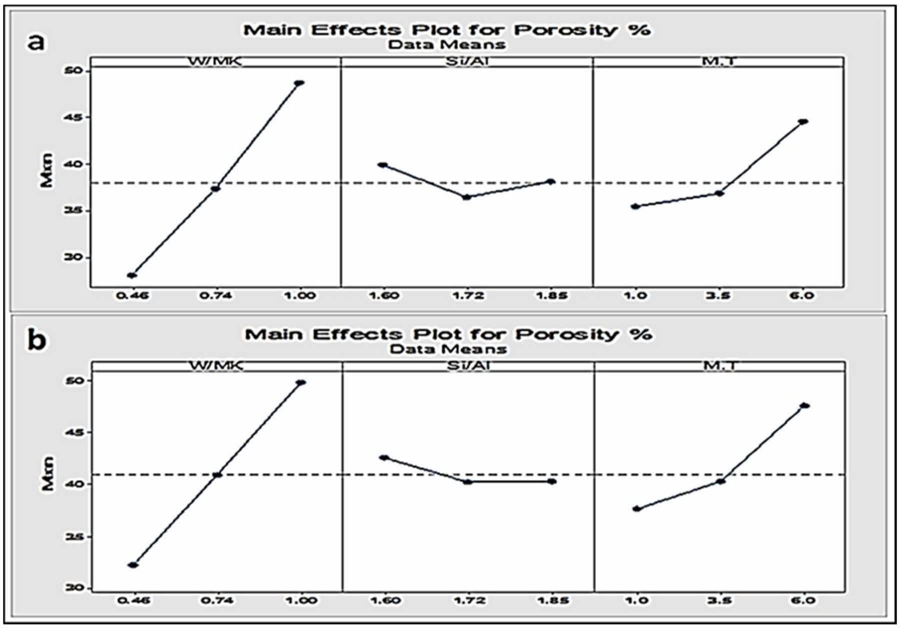


Figure 7: The main effect of parameters on porosity of a). Batch 1 and b) Batch 2

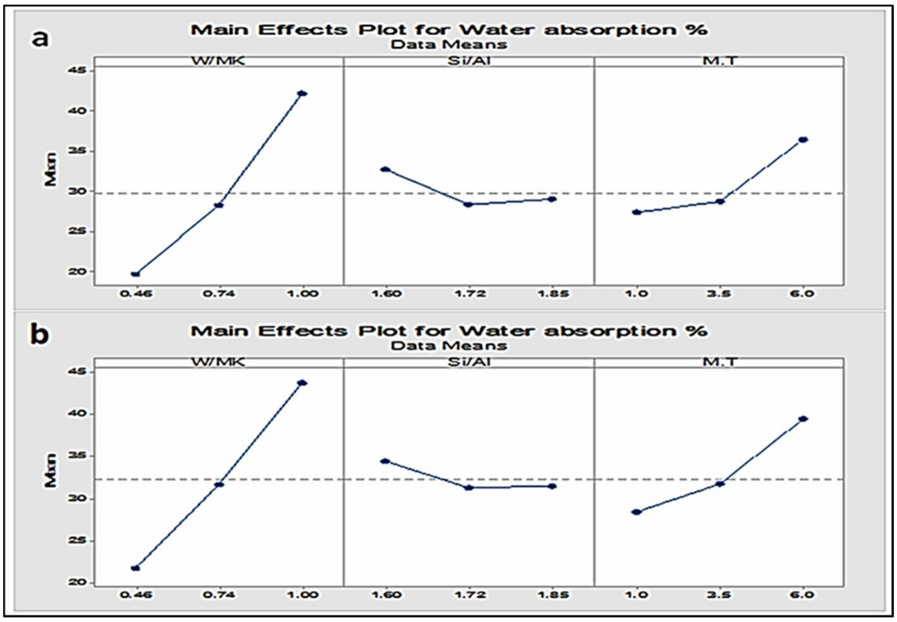


Figure 8: The main effect of parameters on water absorption of a) Batch 1 and b) Batch.2

3.5.4 Optimum results of physical properties

The optimum results achieved for bulk density, apparent porosity, and water absorption for each batch are summarized in Table 4. It can be concluded from the results shown in Table that the K-ions substitution of Na-ions will increase the porosity of the produced geopolymer despite the viscosity of Na-based GP being larger than that of K-based one. However, the inherent porosity arises from the presence of intrinsic interconnected meso/macro-pores in the geopolymer matrix, the unreacted metakaolin particles, or entrapped air [29]. In any case, when compared to sodium ions, potassium ions tend to produce more porous structures. This finding is associated with the influence of each ion on the workability of GP pastes, in which more workable pastes would be produced using potassium ions compared to the sodium ones [30].

Table 4: The optimum physical properties

Batch. no	Density gm/cm3	Porosity %	water absorption %
Batch.1	1.56	26.8	18.09
Batch.2	1.48	31.71	21.41

4. Conclusion

In this work, MK-based GP cement was prepared with different alkaline activators and with different processing parameters. In general, incorporating the K-ions will negatively influence GP properties. It was concluded that at higher Si/Al ratios, the density GP increases regardless of the type of alkaline activator. According to the ANOVA test, The W/MK ratio seemed to be the most important factor affecting GP properties. So, adjusting this ratio is necessary to achieve GP with adequate physical properties. The results also indicated that the short duration mixing had been enough for producing a dense GP paste with low pores.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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