

# OPTIMIZATION OF COMPRESSIVE STRENGTH AND BULK DENSITY OF MK-BASED GEOPOLYMER

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## ABSTRACT

This work aimed to synthesis metakaolin based geopolymer cement with different alkaline activator. The effect of activator type as well as W/MK ratio, Si/Al ratio and mixing time on compressive strength and bulk density were studied in this work. ANOVA test was employed to gained the main effect of processing parameters. The results showed that GP activated with sodium have a high bulk density than GP activated with sodium and potassium in which the maximum bulk density obtained were 1.56 g/cm<sup>3</sup> and 1.48 g/cm<sup>3</sup> for Na and K, Na based GP respectively. The optimum compressive strength achieved at 3, 14, and, 28 days were 65, 92, and 86 Mpa respectively. Short mixing time of 1 min was sufficient to obtain a GP with high density for Na and K, Na based GP. According to ANOVA test, the W/MK ratio has the major effect on the bulk density and strength of GP, also increasing the Si/Al ratio will lead to increase the GP density.

**Keywords:** Geopolymer, ANOVA, compressive strength, bulk density.

## 1. INTRODUCTION

Geopolymers are inorganic polymers, typically made at temperatures below 100°C. Mineral molecular chains or networks are joined by covalent bonds in these materials. The basic components are mostly minerals with geological origins, thus the term "geopolymer." [1]. Geopolymerization is a multi-step process that results in a geopolymer. The dissolution of Si as well as Al atoms from materials is the first stage in geopolymerization, which is followed by the formation of monomers through orientation of precursor's ions then monomers polycondensation into 3-D network polymeric structures are the three basic stages involved in geopolymerization in general. However, it should be noted that geopolymerization is as an exothermic reaction [2]. Geopolymers are made from alumina-silicate materials, which are high in silicon (Si) and aluminium (Al). Indeed, a large number of industrial by-products and minerals, including: kaolinitic clays, flyashes, silica fume, blast furnace slag, volcanic scoria, metahalloysite, granite, a mixtures of slag and fly ashes, mixtures of metakaolin and fly ashes, and mixtures of metakaolin and slag, have been reported as an aluminosilicate source for geopolymer synthesis [3]. Manufacturing of GP with predictable performance is difficult, owing to the fact that the composition, type, and raw materials reactivity all influence the chemistry of the final geopolymers, and therefore their properties. Meanwhile, Despite the widespread use of slag and fly ash FA being two of the most important constituents in the commercial geopolymer products, metakaolin has appeared as the most promising aluminosilicate source for GP synthesis. The main reason behind this, is that MK has a more consistent chemical compositions in compared to slag and FA, as a result, more reliable products which are appropriate for repairable building materials are produced. Due to their widespread use for production of blending cements as well as concrete, slag and FA are not abundant in many countries, as a result, it appears that using MK as a raw material is more promising and feasible [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]. It is well known that GP synthesis via the traditional path, includes two main constituents which include the solid part (aluminosilicate) beside the liquid one (alkaline solution). It is worth noting that a numerous factors are influence on the properties of GP like: the nature of starting materials that involve mineralogical and chemical composition, alkaline solution composition, particle size, mixing procedure, curing conditions, and liquid-to-solid ratio, Si/Al ratio, hydroxide concentration. Therefore, the properties of GP can be improved by adjusting these parameters [3]. A very alkaline media, according to Van Jaarsveld et al. [6], is needed for geopolymerization to take place. This is essential for silica and alumina to be dissolved. Alkaline solutions, either alone or in combination form, can be used to activate such an environment. Hydroxides (K<sup>+</sup>, Na<sup>+</sup>, and Ca<sup>2+</sup>) and silicates (K<sup>+</sup> and Na<sup>+</sup>) are the most often utilized alkaline compound. In most cases, a combination of hydroxides (solid dissolved in water) as well as silicates (solid dissolved in water or liquid) is employed. The resulting solution's silicate serves as an extra source of SiO<sub>2</sub>, on the other hand, the hydroxide ensures strong alkalinity. The alkaline solution plays a crucial role in the development of mechanical strength during geopolymerization [7]. The aim of current study is to investigate the effect of the type of alkaline solution on compressive strength and bulk density of GP. A mixture of sodium and/or potassium

based activation solution were developed for this purpose. However, the influence of Si/Al ratio, W/MK ratio, as well as mixing time with different levels were also studied to get the optimum condition that yield the maximum strength and density of GP.

## 2. EXPERIMENTAL PART

The kaolin clay used in this study was supplied from Dwaikhla region/ Al-Anbar Governorate. The kaolin was heated at a temperature of 750°C for three hours and with heating rate of 10 °C/min so as to obtain metakaolin powder. The chemical composition of metakaolin oxides in wt.% was calculated using the X-ray fluorescence (XRF) instrument (Spectro Xepos), This test was conducted at Department of Geology \ College of Science\ University of Baghdad. It can be observed from the **Table.1** that the metakaolin is basically formed by SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>.

**Table.1 Show chemical analysis of MK**

As we mentioned earlier, that the process of preparation of geopolymer cement involve using two sources which are aluminosilicate powder and alkaline solutions, the aluminosilicate source was represented in this study by employed metakaoline powder while the alkaline solution include using a mixture of potassium, sodium hydroxide in addition to potassium, sodium silicate. The preparation of geopolymer cement was performed according to following steps: the first step is to prepare the activator solution by initially dissolving the proper amount of NaOH and / or KOH in a distilled water which were stirred

Chemical composition in wt.%	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	SO <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	Cl	Others
<b>MK</b>	54.53	1.58	37.81	1.86	0.53	0.20	0.17	0.05	0.51	0.10	0.01	0.22

magnetically at a speed of (800) rpm. The required amount of Na<sub>2</sub>SiO<sub>3</sub> was then added to the NaOH and KOH solution while it was continuously stirred at 800 rpm with a constant ratio equal to Na<sub>2</sub>SiO<sub>3</sub> / NaOH = 1.5 and left until clear solution obtained. Silica gel was then added to the solution with a certain amount, and after all silica dissolved completely, the solution is left for its temperature to decreased naturally to room temperature. The second step of the elaboration is the mixing of metakaolin with the activator solution (after cooled to room temperature), which was conducted using mechanical mixer with a constant speed of (2000 rpm) at ambient temperature in order to obtain a geopolymer paste with a good homogenization. The paste was then poured into plastic cylinder molds with a diameter of Ø = 2 mm and length L = 4mm. The samples were cured for 24 h at the room temperature of 25°C. After de-molding, the geopolymer samples were left for 3, 14, and 28 days at ambient temperature prior to mechanical and physical tests. Metakaolin based geopolymer cement was prepared with two basic batches. However, the two basic batches prepared are shown in the **Table.2** below:

**Table.2 The value of basic batches.**

Batches NO.	K <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub> (mole)	Na <sub>2</sub> O/ Al <sub>2</sub> O <sub>3</sub> (mole)	Si/Al Atomic ratio
G1	-	1	1.6-1.85
G2	0.6	0.4	

MK-based GP cement was prepared with different processing variables in order to studying the effects of these variables which involved water/metakaolin (W/MK ratio), Si/Al ratio and mixing times and with three levels for each them, as shown in the **Table.3**, on the outputs parameters that include the mechanical and physical properties of GP cement. **Table.4** show the experiment that were used throughout this study.

**Table.3 The inputs parameters with their levels**

NO.	Parameters	Units	Levels		
			1	2	3
1	<b>W\MK ratio</b>	Mass ratio	0.46	0.74	1
2	<b>Mixing Times</b>	Minute	1	3.5	6
3	<b>Si\Al ratio</b>	Atomic ratio	1.6	1.72	1.85

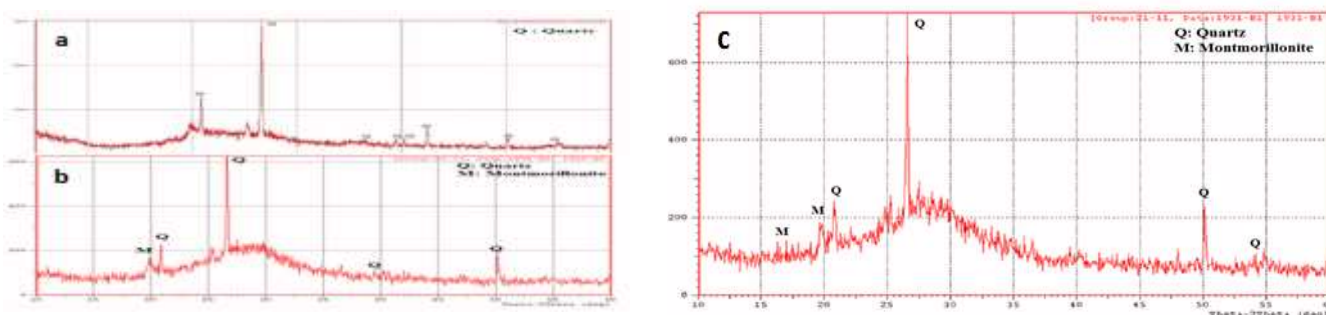
**Table.4 The experiment used throughout this study**

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

### 3. RESULTS AND DISCUSSION

#### 3.1 XRD RESULTS

The XRD pattern results of metakaolin, G1, and G2 are illustrated in **Figure. (1 a, b and c)**. The major peak present in the metakaolin was at  $2\theta = 26.63^\circ$  which was attributed to residual quartz in the material. However, the XRD analysis results for G1 and G2 revealed that metakaolin-based geopolymers resulted in two types of diffraction patterns, which were corresponded to crystalline materials that were contained in metakaolin (quartz) in addition to a small quantity of montmorillonite and aluminosilicate amorphous materials, However, the peak related to quartz were decreased in intensity, indicating that the parent material were not totally dissolved into the inorganic polymeric material. According to the X-ray data, when the metakaolin was activated with alkaline solution, there is no clear evidence of new crystalline phases. The same crystalline phases were identified in metakaolin and geopolymer except montmorillonite. There is, however, a broad “amorphous hump” from a center at  $23^\circ 2\theta$  to  $35^\circ 2\theta$ . It has been noted that this hump is indicative of geopolymeric reaction[8].



**Figure:1 XRD pattern results of a). metakaolin, b). G1, and c). G2**

#### 3.2 FTIR RESULTS

The FTIR spectra of kaolin and metakaolin are illustrated in **Figure (2.a and b)**, In kaolin, a characteristic spectrum of kaolin bands at  $3695\text{ cm}^{-1}$  and  $3626\text{ cm}^{-1}$  are appeared which attributed to the vibration of external hydroxyl and inner hydroxyl, respectively[9]. Water in kaolinite give a broad band at  $3448\text{ cm}^{-1}$  corresponding to the  $\text{H}_2\text{O}$ -stretching vibrations[10].  $\text{H}_2\text{O}$  stretching was also found at  $1635\text{ cm}^{-1}$  [11]. Bands at  $1134\text{ cm}^{-1}$  and at  $979\text{ cm}^{-1}$  were assigned to Si-O bonds in the  $\text{SiO}_4$  molecules [11].The other bands at  $933\text{ cm}^{-1}$  and  $902\text{ cm}^{-1}$  represent the Al-OH bending vibrations [9,10]. The bands at  $794\text{ cm}^{-1}$ ,  $756\text{ cm}^{-1}$ , and  $694\text{ cm}^{-1}$  were attributed to Si-O symmetric stretching [11]. The bands at  $578\text{ cm}^{-1}$  and  $501\text{ cm}^{-1}$  was assigned to Si-O-Al where the Al is in octahedral coordination [12, 11]. Vibration at  $416\text{ cm}^{-1}$  can be related to the deformation mode of Si-O or Al-O bonds[13]. Some minor peaks were detected in the present study especially at  $2368\text{ cm}^{-1}$  and  $2337\text{ cm}^{-1}$  due to the absorption of the  $\text{CO}_2$ . After calcination, the bands at  $3695\text{ cm}^{-1}$  and  $3619\text{ cm}^{-1}$  that appear in the kaolin spectrum were not observed in the metakaolin spectrum as shown in **Figure 2.b**, due to loss of water from kaolin, since these bands are related to the presence of OH groups. According to Du and Yang [14], calcination leads to the break of the kaolin crystalline structure, which can be verified by means of the metakaolin spectrum, suggesting that the thermal treatment was adequate to convert kaolin to metakaolin.

The FTIR spectra of the synthesized geopolymers pastes are shown in **Figure.(2.c and d)** for G1 and G2 respectively. Significant broad bands are observed in the range of ( $3448\text{ cm}^{-1} - 3456\text{ cm}^{-1}$ ) and ( $1651\text{ cm}^{-1} - 1635\text{ cm}^{-1}$ ), which were

corresponding to adsorbed water molecules in the large voids of polymeric skeleton allied with the reaction products. The bands located between  $2368\text{ cm}^{-1}$  and  $2276\text{ cm}^{-1}$  are assigned to stretching vibrations of O–C–O bond indicating the existence of carbonate which is occurred due to atmospheric carbonation. The major band between  $1026\text{ cm}^{-1}$  and  $1018\text{ cm}^{-1}$  is attributed to asymmetric stretching vibration of Si–O–T bands, where T is tetrahedral silicon or aluminum. The band at approximately  $478\text{ cm}^{-1}$  is attributed to O–Si–O bending mode. Finally, vibration at  $432\text{ cm}^{-1}$  can be related to the deformation mode of Si–O or Al–O bonds [9].

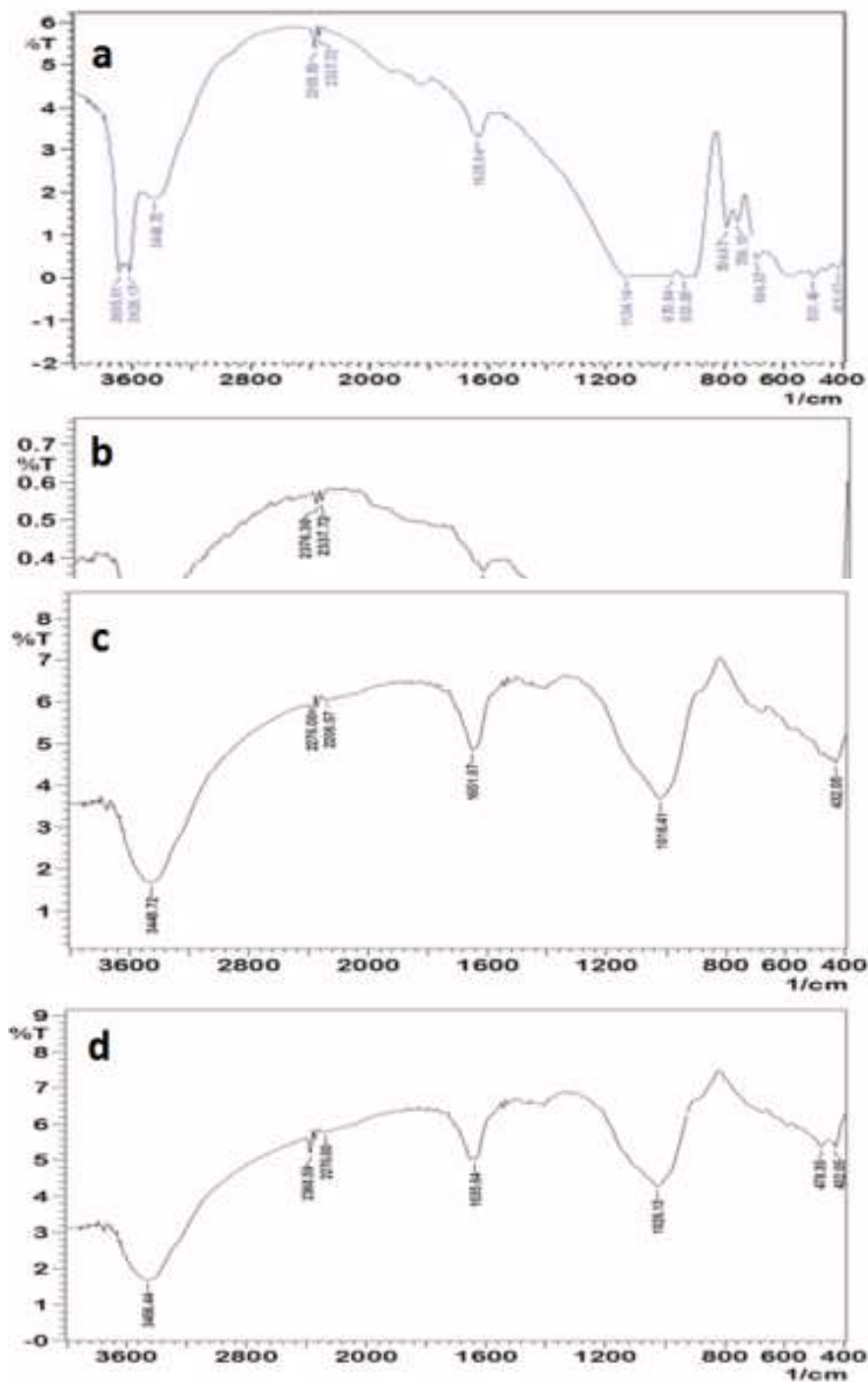


Figure.2 Show the FTIR spectra of a). Kaolin, b). Metakaolin, c). G1, and d). G2

### 3.3 COMPRESSIVE STRENGTH RESULTS

#### 3.3.1 Compressive strength at 3 days

The main effects plot of processing variables on the compressive strength of metakaolin based GP cement for G1 & 2 at 3 days are shown in **Figure.(3 a and b)**. The water provide a medium to facilitate the geopolymerization reaction. It can be observed that the water content have the dominant effect on the compressive strength of GP, decreasing the water content will provide high a molar concentration in alkaline solution [15]. However, based on several studies, increasing the molar concentration of solution has a significant effect in rising the Na- based GP strength especially at early age [16], and this clearly observed in **Figure 3.a**. The effect of water on GP strength for G2 is different from G1 through observing the **Figure 3.b**, it can be noticed that increasing the W/MK ratio until 0.74 has a major effect in increasing the strength for GP activated with potassium. However, excess water content (> 0.74) lead to remarkable drop in the strength due to increasing the pores of GP body [17]. It is also observed that amount of Si/Al ratio play an important role in controlling the GP strength. Meanwhile, Si/Al ratio up to 1.72 for the two batches are necessary to achieve a high strength at early age. it can be also observed from figures that the mixing time has a great effect on the compressive strength where there is noticeable increase in the compressive strength with increase the mixing time up to 3.5 min.

However, water is indispensable during geopolymerisation, especially for the destruction of solid particles and the hydrolysis of dissolved  $Al^{3+}$  and  $Si^{4+}$  ions. Six possible reactions in the complicated system are assumed as Eqs. (1-6) shown, Water is the reactant Eqs.(1-3), But in Eqs. (4-6), the main reactions may change from hydrolysis (consuming water) to polycondensation (releasing water), water plays as a product in this period, if too much, will hinder the polycondensation kinetically [18].

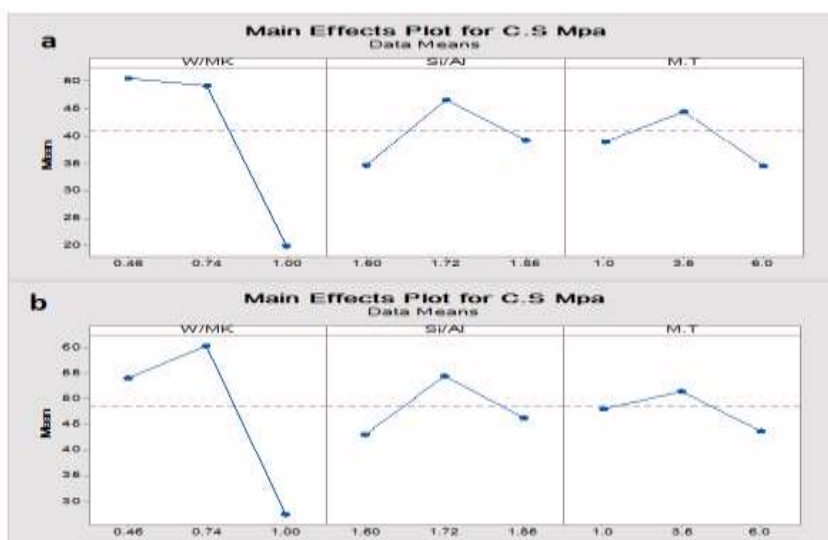
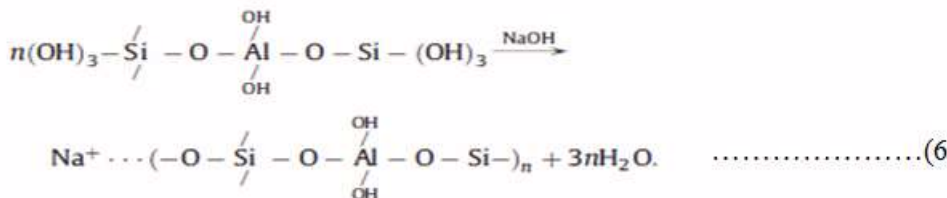
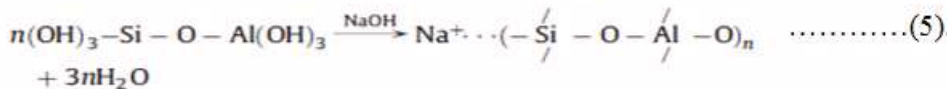
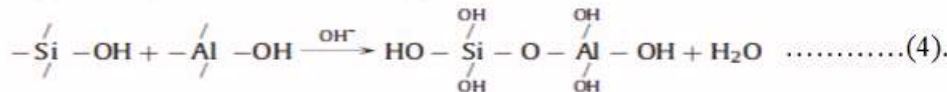
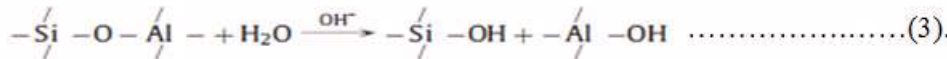
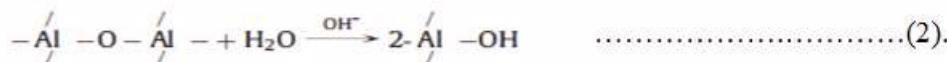
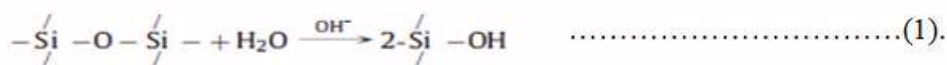


Figure.3 illustrate the main effect of variables on compressive strength at 3 days for a). G1 and b). G2



### 3.3.2 Compressive strength at 14 & 28 days

Figures (4a, and b) and Figures (5a, and b) demonstrated the main effect plot of each variable on the compression strength of GP at 14 and 28 days for G1 and G2 respectively. A poly(sialate) structure will arise at low Si concentrations (low Si/Al ratio) [19]. In the geopolymer system, the bond Si-O-Al is weaker than the bond Si-O-Si because of the high aluminate content. However, the Si-O-Si bond is stronger than the Si-O-Al and Al-O-Al bonds. It can be observed from Figure.4a that the strength of GP rises and subsequently falls when the Si/Al ratio is increased, with the maximum strength being obtained when the Si/Al ratio is 1.72. This is in line with prior study findings from other researchers. According to Steveson et al. [20], geopolymers with a Si/Al ratio of 1.50 to 1.95 have the best mechanical characteristics. A GP structure of poly(sialate-siloxo) or poly(sialate-disiloxo) will be created in a system with a high concentration of Si (Si/Al ratio is high) [19], contributing to the production of more compact, dense, and homogenous microstructures. To put it another way, a complex network will be developed, resulting in increased strength. The amount of Si-O-Si bonds and hence the compressive strength increase significantly when the silicate source concentration is greater and this clearly seen for G2 Figure.4.b. For G1 the optimum Si/Al ratio observed was 1.72 at 14 & 28 days, the same value were obtained for G2 at 28 days, beyond this value, a noticeable decrease in strength was observed due to fact that extra silicate species produces hindrance in the reaction between silicate and aluminate species. Ultimately the dissolution did not occur or occur in a reduced manner then, finally the material got less strength and due to this, a part of silica remained unreacted in the produced geopolymer gel [21]. In [22], author reported that a high quantity of silica produces hindrance in dissolution and once the dissolution slowed down ultimately the material got less strength. According to Duxson et.al. when the Si/Al ratio is high (Si/Al > 3), compressive strength decreased [23].

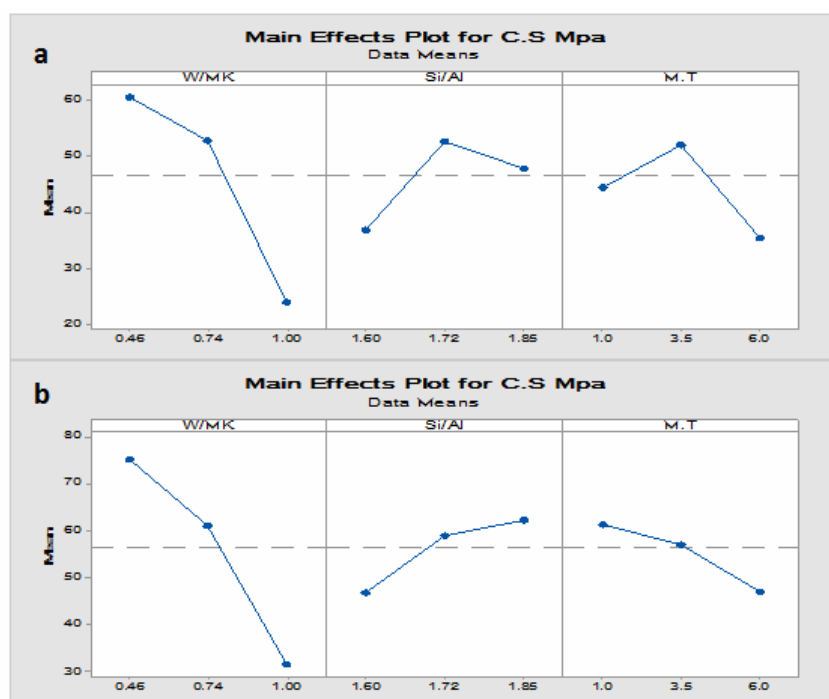


Figure.4 illustrate the main effect of variables on compressive strength at 14 days for a). G1 and b). G2

At 28 days, the effect of water for the two batches is quite different, the optimum W/MK ratio obtained that yield the highly compressive strength was 0.74 and 0.46 for G1 and G2 respectively. However, this value is different from that obtained at 3 & 14 days. Increasing the amount of water, in spite that it decreasing the alkali concentration, it will decrease the viscosity of solution, since this viscosity would prohibit to achieve a good mixing and this lead to achieve a low strength Figure.5a[24]. On the contrary of G2, it was observed that the strength greatly increase with decreasing the water content. Furthermore, the increase of alkaline concentration leads to a high dissolution of the silica and alumina in which more cations ( $Si^{4+}$  and  $Al^{3+}$ ) will be generated, which in turn contributes to the creation of new bonds during the gel-building phase Figure.5b[16]. However, when the activation concentration solution exceeds a certain limit, the polycondensation process was hindered and the compressive strength would be reduced. From the results of this study, the initial concentration of NaOH & KOH solution did not exceed the limit value, and the strength value monotonously increased with the increasing concentration[15]. The effect of mixing time for the two batches is quite different, for G1 the optimum mixing time achieved was 3.5 min at 14 and 28 days while for G2 was 1 and 3.5 min at 14 and 28 days respectively. However, mixing factor has a major effect in decreasing the particles sizes of metakaolin powder due to mechanical mixing and this lead to increase the surface area of MK particles, making them more active, which when reacted with the alkaline solution, more geopolymer would be produced [17].

### 3.4 Bulk density

Figures (6 a and b) illustrate the major influence of processing variables on the bulk density of GP paste, It was obvious that as the W/MK ratio was increased, the bulk density declined. This was thought to be due to the establishment of large porosity in the GP, because after evaporation, extra water leave pores, contributing to poor GP paste compaction and consequently decreased density [25].

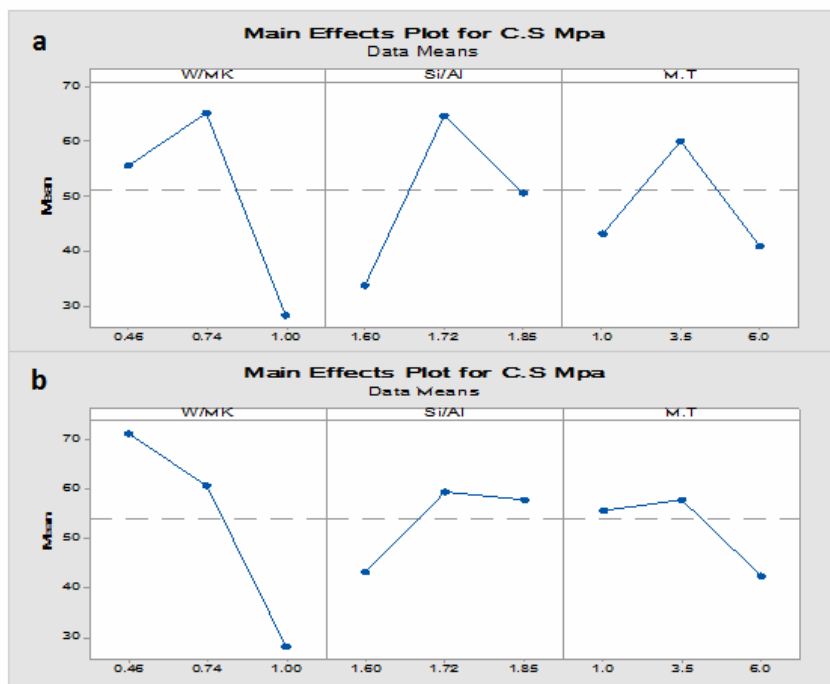


Figure.5 illustrate the main effect of variables on compressive strength at 28 days for a). G1 and b). G2

It has previously been observed that increasing the Si/Al ratio improves the rate of geopolymerization process. However, increasing the Si/Al ratio leads to the production of a compact and dense geopolymer structure with fewer pores [19], as seen in the Figures (6 a and b). The bulk density of Na-geopolymer is more affected by Si/Al concentration than that of potassium containing geopolymer. Meanwhile, Na-based geopolymer was found to have a higher bulk density than GP incorporated K<sup>+</sup>. However, This finding is in contradiction to Lizcano et al earlier 's research [24]. In terms of mixing time, 1 minute was the ideal mixing time for G1 and G2 to attain a high bulk density. However, when the period is more than 1 minute, the GP density drops significantly.

### 3.5 Optimum results of compressive strength & bulk density

Table.5 Show the optimum compressive strength achieved for G1&2. at 3, 14, and 28 days. It can be clearly seen that the compressive strength at 3 & 14 d for G2 is larger than that for G1, in which its 28d strength was better than that for GP activated with potassium. However, the high early compressive strength of GPs achieved at ambient temperature, due to the faster formation of gels, considered an important property for MK based GP cement to be used in many application that required highly age strength. It is need to point out that this result (C.S at 28 days) is not corresponded with that reported by Lizcano et.al. [24] how stated that the compressive strength of GP was increased with incorporation of potassium ions, since because of the larger size of K<sup>+</sup> ions, it will pairing with anion of silicate prepares oligomers have a larger size. However, the outcome is in agreement with that reported by Liew Yun-Ming et al [26]. who stated that the compressive strength decreases when different types of ions are utilized.

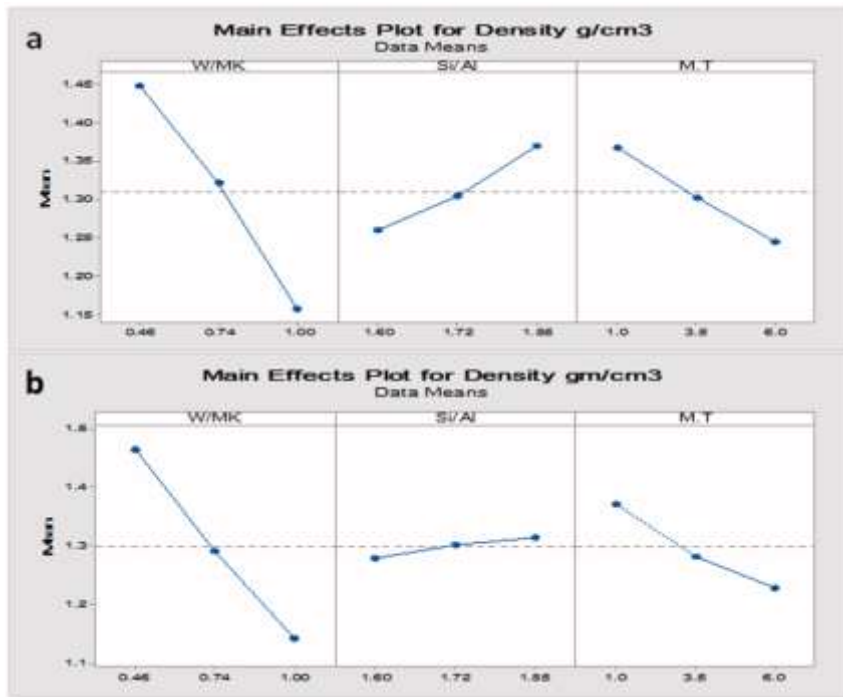


Figure.6 illustrate the main effect of variables on bulk density for a). G1 and b). G2

Table.5 The optimum results of C.S of GP

Batch. no	C.S at 3d	C.S at 14d	C.S at 28d	B.D g/cm <sup>3</sup>
G.1	58.22	72.43	86.18	1.56
G.2	65.32	92.82	83.35	1.48

In the case of bulk density, the optimum density achieved for GP activated with sodium was 1.56 g/cm<sup>3</sup>, while for GP activated sodium and potassium the density was 1.48 g/cm<sup>3</sup>. Which mean the incorporation of potassium ions lead to decrease the GP density. However, this behavior may attributed to the workability of GP paste, in which presence of K<sup>+</sup> ions lead to produce more workable paste in relation to Na<sup>+</sup> ions.

#### 4. CONCLUSION

In this paper, geopolymer cement was prepared by activation of metakaolin with two types of alkaline activator based on sodium and potassium. Based on the experimental results, the W/MK ratio seemed to have the maximum effect on the compressive strength and bulk density of GP. The maximum compressive strength achieved at 3, 14, and 28 days were 65.32, 92.82, and 86.18 Mpa. The bulk density of GP activated with Na was higher than that for K, Na activated one. Increasing the Si/Al ratio and decreasing the W/MK ratio with decrease the time of mixing was necessary to obtain high density.

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