International Journal of Engineering

Optimizing the Properties of Metakaolin-based (Na, K)-Geopolymer Using Taguchi Design Method

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P A P E R I N F O	A B S T R A C T
	Geopolymer paste is an innovative construction material which shall be produced by chemical action of inorganic molecules. It is a more environmentally friendly alternative to conventional Portland cement which is abundantly available worldwide. In this study, the influence of different alkaline activators (Na and K) on the mechanical and thermal behaviors of metakaolin-based geopolymer was investigated. The aims of this study is to find out the mixes and their process parameters, which are appropriate to produce Geopolymer paste with one of the highest compressive strength, highest - lowest porosity and highest-lowest initial and final setting time. Taguchi method is used in the design of the experiments for the metakaolin-based Geopolymer. Five factors were selected as process parameters that are more likely to affect the Geopolymer characteristics. These are the amount of Si, alkali type, alkali reagents ratio, mixing time, and water content. The effect of these parameters on the setting time, density, porosity, compressive strengths at 7 and 28 days. The results of study found that the Geopolymer paste with high compressive strength of (107.2MPa) can be obtained with the formula $(0.2K_2O.0.8Na_2O.\ Al_2O_3.\ 3.6SiO_2.xH_2O)$ using proper processing condition in which the alkali silicates to the alkali hydroxides molar ratio should be kept in the range of 2.26. The results revealed that the use of alkali solution of K-ions and Na-ions improves the compressive strength of the geopolymer remarkably as compared with the use of Na-ions solution along. In addition, it has been noticed that the setting time is reduced, for geopolymers with silica content of less than 3.8, when K-ions is used. Similarly, the bulk density of geopolymers is found to be reduced by adding K-ions.

doi: 10.5829/ije.2020.33.04a.14 |

1. INTRODUCTION

Cement is one of the essential building materials in the construction industry. Portland cement processing is energy-intensive and releases a significant volume of carbon dioxide. There are significant costs related to this energy consumption and environmental impact. Correspondingly, further study into cement products with reduced environmental consequences and increased economic advantages is needed [1]. This is because cement is one of the most common materials used to perform grouting either for soil or for the rehabilitation of concrete cracks [2].

Geopolymer cement is a ground-breaking material that can be used in the infrastructure of transport system,

manufacturing and offshore installations and a real alternative to traditional Portland cement. It depends on highly processed natural materials or industrial waste materials to substantially decrease its environmental footprint. Whilst, it is still being highly resistant to several durability problems that traditional concrete could cause [3]. Many efforts were applied toward developing high performance concrete for the building structures with enhanced performance and safety [4]. Geopolymers are the product of alkali activated aluminosilicate sources that excelled as an alternative to ordinary binders due to its sustainability, low cost and good mechanical properties [2].

Geopolymer is a relatively new material, which was first developed by Joseph Davidovits and patented in the

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1970s. They are inorganic polymeric materials with chemical compositions similar to zeolites. But their structure is either amorphous or semi-crystalline. They are produced from a mixture of several aluminosilicate materials (with high contents of Si and Al) along with an alkaline-activating solution (mostly Na+ and K+ silicates and hydroxides) [5].

Theoretically, any material containing aluminum and silicon can be a solid source of aluminosilicate for geopolymerization. However, the most–often-used materials are blast furnace slags and ash from the burning of coal and calcined clay. The quarry dust is a good replacement to weak soil or a good additive to help improve a problematic soil [5]. The most commonly used clay is kaolin which upon thermal activation changes into metakaolin. Ground improvement is a process that aims to enhance the engineering properties of the soils and generate an improved constriction material by increasing soil strength, durability, stiffness, and decreasing permeability and compressibility of sandy soils. Additive materials are one of the most important methods to improve the engineering properties of soil that are used to improve the engineering performance [6]. When, metakaolin is used as the aluminosilicate source, the resulting Geopolymer is purer and can be more easily characterized compared to the Geopolymer produced from agro industrial wastes. The Geopolymer produced with metakaolin are considered a ''model-system'' without the complexities introduced by the use of fly ash, slags, and other alternative raw materials, which contain several hard-to-characterize amorphous phases. Fly ash, for example, is not a well-defined material, but it comprises several crystalline and vitreous phases [7].

The activator requires an alkaline compound in the aqueous form. Hence, the traditionally used compounds are hydroxides (Na+ and K+), silicates (Na+ and K+), and silica gel. Normally, a mix between hydroxides (solid dissolved in water) and silicates (solid dissolved in water) and silica gel (solid dissolved in water) is used . The silicate of the produced solution is an additional SiO_2 source, while the hydroxide assures high alkalinity in the solution [8]. During geopolymerization, the alkaline solution plays an important role and affects the development of mechanical strength. Its selection depends mostly on its reactivity and the cost of the materials employed [9]. The reaction mechanism of Geopolymer, geopolymerization, is exothermic in nature due to polycondensation. Palomo et al. [9] have reported that alkali-activation, sometimes called geopolymerization. It is a chemical process that allows the transformation of vitreous structures (partially or totally amorphous and/or metastable) into very compact and luting composites. Van Jaarsveld et al. [10] have suggested that the geopolymerization to occur, a strongly alkaline medium is required, this is necessary for it to dissolve silica and alumina. As well as, to hydrolyze the surfaces of the particles of raw materials, such an

environment can be achieved using alkaline solutions in a simple or combined form for activation.

The properties of geopolymers depend on functional, crystallographic and microstructural feature which affect many significant properties, such as the setting time, density, porosity, mechanical strength, thermal properties, and chemical stability. These features are affected by many factors including the alkali type, alkali reactants ratio, solid/liquid ratio, molar ratio of Si/Al, mixing parameters, curing conditions, and water content [11]. Studying the effect of these parameters on the properties of geopolymer by the full factorial design needs to carry out many experiments. It develops to be complex and difficult when the number of factors increased. To overcome this issue, methods of design of experiment, such as Taguchi method, may be used in the design of the experiment for the metakaolin-based Geopolymer. Five factors were selected as process parameters that are more likely to affect the Geopolymer characteristics.

This method proves its efficiency in many fields, including biotechnology, environmental engineering, software testing, service system, education, and producing planning. Taguchi method is a methodical approach for expansion of various factors with considering performance, cost and quality [12]. Genetic algorithm (GA) method has been applied in order to obtain the optimum processing parameters [13, 14]. The current study aims to find out the mixes, and their processing parameters, which are suitable to produce Geopolymer paste with one of the following features: Highest compressive strength, highest/lowest porosity, highest/ lowest initial and final setting time.

2. MATERAL AND METHOD

2. 1. The Starting Materials   Kaolin, sodium silicate, sodium hydroxide, and silica gel have been used as starting materials to synthesis Geopolymer cement. Metakaolin which has been used in this study was obtained by the calcination of kaolin clay supplied from the local area (Dwaikhla, Western Iraqi Desert). The kaolin was calcined at 750 ˚C for three hours in air atmosphere using heating rate of 5ºC/ min. Equation (1) was used to describe the composition of the prepared Geopolymer.

$$0.2K_2O.0.8Na_2O. Al_2O_3. n SiO_2. xH_2 \qquad (1)$$

where (n) is the number of moles of SiO_2 in the formula of the Geopolymer.

For each mix formula, the effect of (n) and (x) have been studied in addition to the mixing time and the alkali reactants ratio using Taguchi method to design the experiments. Taguchi method suggested 25 experiments for Geopolymer mix (GP1). These four parameters have been selected based on the previous studies, including the

parameters which strongly affect the manufacturing of the Geopolymer. The upper and lower limits of each parameter has been selected based on the results of previous studies, as well as many primary experiments. The criteria used to select the values of these limits include: (1) The paste of the Geopolymer should be easily mixable. (2) The setting time should not be neither too short nor too long. 3) The resulting Geopolymer body should be free of macro-cracks. The magnitudes of the parameters are demonstrated in Table 1.

The alkaline liquid utilized in this investigation is mixture of hydroxide salts, involving potassium hydroxide (PH) and sodium hydroxide (SH), and silicate salts including sodium silicate (SS) and potassium silicate (PS). At first, water is placed in the beaker and then the hydroxide salts have been weighed and added to reach the desired molarity(M). The hydroxide salts pellet release heat as they dissolve in water. The hydroxide salts addition is completing the silicate salts added to the solution. At this time, a magnetic rod is placed in the solution, the solution is placed on a stirrer and stirred at 80˚C and 600 rpm.

After all silicate salts are dissolved, silica gel was added to the solution. Approximately one hour later, all silica was dissolved completely. After that, a desired volume of water was added to compensate the water lost due to evaporation and the solution was cooled naturally to room temperature. After the alkaline solution is cooled to room temperature, the metakaolin (MK- 750) was added to the solution and mixed using a mechanical mixer at a fixed speed of (3550rpm) for the desired mixing time. Moulds made of plastic with diameter =2.1 cm and height =4.2 cm have been utilized for molding the pastes of Geopolymer. The specimens were kept in a specific condition at lab at a temperature of 23 °C± 2 for one day and then demolded. These samples have been cured at a specific condition for 7 or 28 days. Figure 1 shows the specimen of geopolymer cement paste after 24 hours past casting. Figure 2 illustrates the experimental work performed in the current study.
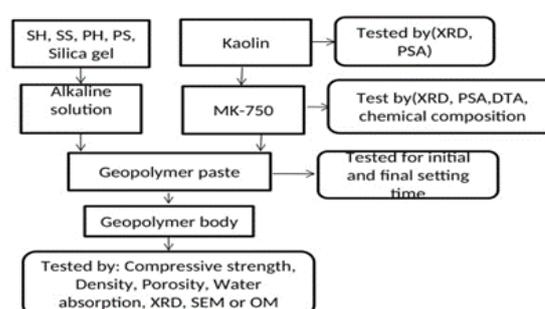
2. 3. Characterizations   The chemical composition of the kaolin was found using the wet chemical method. This analysis was performed at the Iraq geological and mining survey. The main oxides of the starting materials were declared in terms of their weight percentages (Table

Figure 1. Geopolymer cement paste specimen

Figure 2. Flow chart that summarized the experimental work performed in the current study

2). The crystalline phases of the starting materials were identified by the X-ray diffraction (XRD) method (Shimatzu 6000 diffractometer, Japan). Cukα X-ray as a source radiation was used with 40 kV and 30 mA at a scanning speed of 5°/min. Nickel was used as a filter. Particle size analysis test has been made to identify distribution of particle size of kaolin and metakaolin powder by using laser particle size analyzer. The synthesized samples were investigated with Microvickers Hardness device model (TH-717) with a load of 9.8N and holding time of 15 seconds. The diagonals distance was measured with the device of Vickers hardness.

SEM was used to examine the features of the microstructure of the Geopolymer paste, these features include the pore shapes, microcracks, and the presence of crystals. Geopolymer paste setting times are calculated utilizing the Vicat needle in compliance with ASTM C191. The penetration was recorded for each 15min intervals until a 5 cm or less penetration was achieved. Interpolation at a depth of 5 cm calculated the initial setting. The final setting time was also calculated, which described as the time when the needle did not clearly sink into the paste. Differential thermal analysis (DTA) was utilized to observe transitions phase, crystallizations, and temperatures of decomposition.

The density, porosity, and water absorption of the synthesized samples were measured using Archimedes method. While the compressive strength is calculated by using the formula ($\sigma c =P/A$). The physical, mechanical, and thermal properties were measured as an average of two-time measurements.

TABLE1. The effected parameters the manufacture of the Geopolymer

N	H_2O (ml)/ 10.73 MK-750	SS/SH (wt/wt)	Mixing Time (min)
3.2	8	1.5	5.0
3.4	9	2.26	7.5
3.6	10	3.02	10.0
3.8	11	3.78	12.5
4	12	4.54	15.0

3. RESULTS AND DISCUSSION

3. 1. XRD Analysis of Kaolin   Figure 3 demonstrates the kaolin powder XRD form. The pattern confirms the crystalline structure of the powder. The common characteristic peaks of kaolin were observed according to (ICCD=00-001-0527) and (ICCD=00-033-1161) for kaolinite and SiO_2 minerals, respectively. It is well-known that the heat treatment of kaolin at moderate temperatures leads to the formation of metakaolin. This was confirmed for the kaolin heat treated a 750ºC for 3h (MK-750) as demonstrated in Figure 4. The pattern asserts the amorphous structure of metakaolin powder. XRD analysis of metakaolin demonstrates there is sharp peak refers to quartz that refers to free quartz in the kaolin powder.

3. 2. Results of The Chemical Analysis of Kaolin
Table 2 demonstrates the outcomes of the wet chemical analysis of the kaolin. It can be seen that the amount of SiO_2 in the clay is higher than that of the stoichiometric amount in the kaolinite (48.77%).This confirms the XRD result regarding the presence of free quartz and helps approximate calculation of the amount of free quartz, , that should be excluded when the composition of Geopolymer is calculated., This is because of the inertness of quartz which makes quartz an inert component during the synthesis of Geopolymer.

3. 3. Analysis of Kaolin Particle Size   Figure 5 demonstrates the distribution of kaolin powder particle size. The results demonstrated that the kaolin is composed of micro-sized particles mainly below 20µm. The particle size distribution is multimodal with D_{50} of 3.8µm. The distribution of MK-750 particle size is demonstrated in Figure 6. It is well known that metakaolin has fine particle size as compound with kaolin as it is produce due to the breaking of the kaolinite

structure. However, the agglomeration and aggregation lead to the formation of large secondary particles, as confirmed in Figure 6.

3. 4. DTA Analysis of Kaolin   Figure 7 demonstrates the differential thermal analysis (DTA) for kaolin. The endothermic peak at 530 °C was recorded for the kaolin calcination. To form metakaolin, this event is finished at 575°C and no other thermal event could be observed indicating that the 750°C is a suitable choice for the formation of metakaolin.
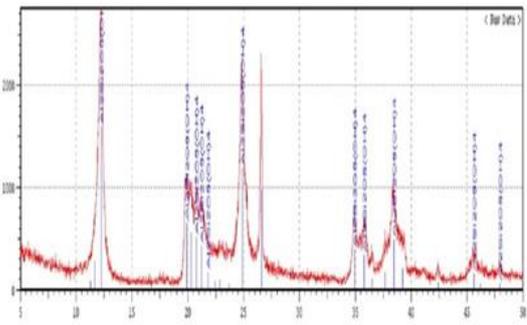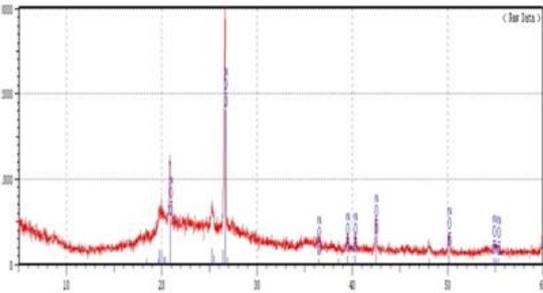

Figure 3. kaolin XRD form

Figure 4. Metakaolin (MK 750) XRD form

TABLE 2. Wet chemical analysis of kaolin

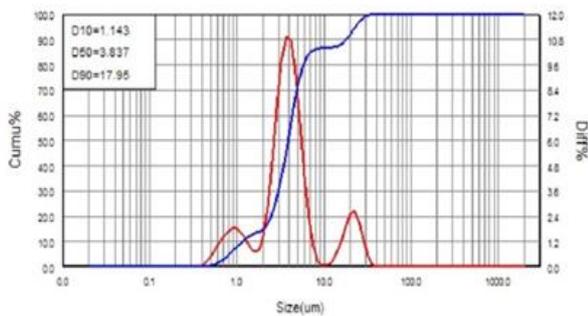| SiO_2 (%)	Fe_2O_3(%)	Al_2O_3(%)	TiO_2(%)	CaO (%)	MgO (%)	SO_3(%)	P_2O (%)	K_2O (%)	Na_2O (%)	Cl (%)	LOI (%)
48.77	1.76	34.27	1.47	0.43	0.08	0.11	0.02	0.43	0.17	0.03	12.46

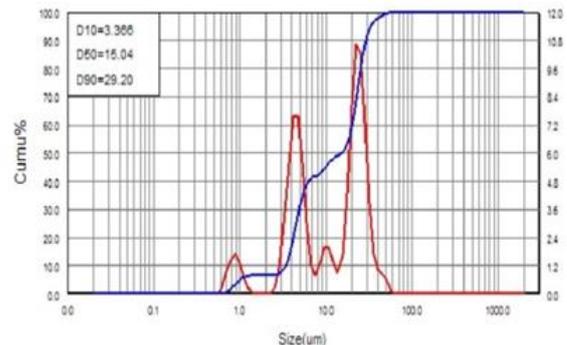**Figure 5**. kaolin powder particle size
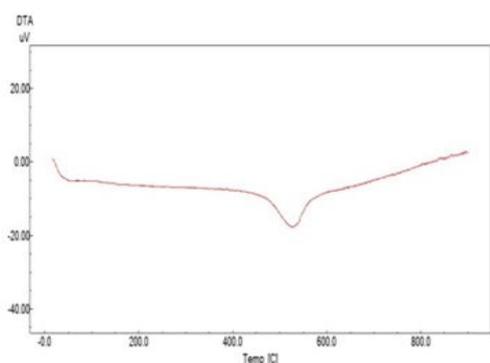

Figure 6. Metakaolin powder particle size

Figure 7. Different thermal analysis (DTA) for kaolin

3. 5. Geopolymer Paste Result
3. 5. 1. Compressive Strength
3. 5. 1. 1. Compression Strength at 7 Days   Table 3 demonstrates the results of Taguchi analysis for the 7-days compressive strength of the GP1-batch geopolymer ($0.2K_2O. 0.8Na_2O. Al_2O_3. nSiO_2$). The results indicate that the rank of the most influential factors on the compressive strength is silica content, mixing time, water content, (SS+PS)/(SH+PH) ratio. This indicates that the selected factors keep their order of importance while the potassium content is increased.

Figure 8 demonstrates the major impact plot for the compressive strength during 7 days of geopolymer paste of batch GP1. In general, the strength increases by increasing the silica content and decreases by increasing water, where the highest strength is obtained when the content of silica (3.4- 3.8) with (8ml-10ml) water. Concentration of (SS+PS)/(SH+PH), 2.26 and mixing time of 5minutes seems to be the best choice.

The gained strengths under compression at seven-days for GP1-batch extend to magnitudes as great as (93MPa). This indicates that the 7-days strength increases when the sodium ions are replaced partially by potassium ions. Smaller ions of Na+ lead to simplify coupling with anion of silicate to produce oligomers that have a small size. While the larger ions of K+ pairing with anion of silicate prepares oligomers that have larger

size. Consequently, K-based geopolymers displays 42 percent greater strength under compression compared with Na-based geopolymers [15]. In addition, larger ions of K+ help to improve the geopolymers setting [16].The outcome is in contrast with that reported by Liew Yun-Ming et al. who stated that the compressive strength decreases when different types of ions are utilized [12].

3. 5. 1. 2. Compression Strength after 28 Days
The results of 28-days compressive strength analyzed by Taguchi method for GP1 batch are given in Table 4. As can be seen, the mixing time and the (SS+PS)/(SH+PH) ratio have the major impact on the compressive strength.

This indicates that the dissolution, hydrolysis, and condensation processes, which are mainly affected by the silica content occurs at high rates but is influenced by other factors such as mixing time and (SS+PS)/(PH+SH) ratio. Thus, the process is mainly controlled by other factors i.e., mixing time and (SS+PS)/(SH+PH) ratio. Moderate mixing time improves the strength of the geopolymer as it works to homogenize the alkali solution with the powder. The best mixing time in batch GP1 at 28 days is15minutes.

Figure 9 demonstrates that the silica content should be kept in the range of (3.4-3.6) to obtain the highest compressive strength for GP1 batch. The highest experimentally achieved 28-days compressive strength was 107.2MPa, while the lowest was 59MPa. These values are too high as compared to most other results reported in literature [17-19]. The 28-day compressive strength was as high as 107MPa which is a very high value as compared with the results reported by many researchers as demonstrated in Table 5.

3. 5. 2. Setting Time   Figure 10 demonstrate the major impact plot for the setting time of GP1. In batch GP1, silica content is the most influential factor on the initial as well as final setting time. When the geopolymer contains both sodium and potassium ions, the setting time was found to increase when the silica content is increased up to 4. This result clearly demonstrates that the presence of potassium ions in the geopolymers, as the case of batch GP1, reduces the rate of ,polymerization of geopolymer.
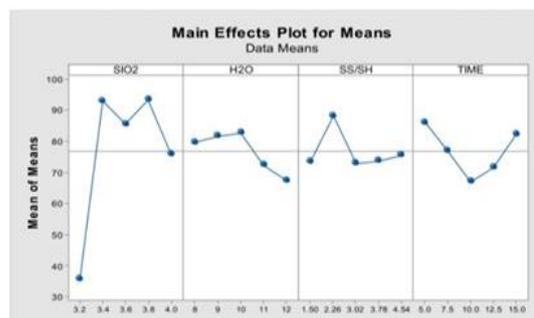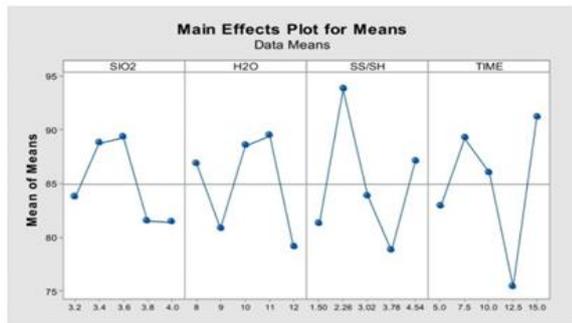
TABLE 3. Taguchi Analysis: response for the 7-days compressive strength for GP1

Level	SiO$_2$	H$_2$O (ml)/ 10.73 MK-750	(SS+SP)/ (SH+PH)	Mixing time
1	35.88	79.54	73.48	85.98
2	92.88	81.62	88.04	76.88
3	85.46	82.66	72.82	67.00
4	93.30	72.28	73.66	71.50
5	75.92	67.34	75.44	82.08
Delta	57.42	15.32	15.22	18.98
Rank	1	3	4	2

Figure 8. The major impacts plot for the compressive strength at 7 days of batch GP2

TABLE4. Taguchi Analysis: Table of response for the 28-days compressive strength for GP2

Level	SiO$_2$	H$_2$O (ml)/ 10.73 MK-750	(SS+SP)/ (SH+PH)	Mixing time
1	83.68	86.80	81.20	82.86
2	88.74	80.80	93.76	89.20
3	89.28	88.48	83.80	85.98
4	81.48	89.42	78.74	75.36
5	81.38	79.06	87.06	91.16
Delta	7.90	10.36	15.02	15.80
Rank	4	3	2	1

(a)

(b)

Figure 10. (a) Initial setting time and (b) Final setting time of geopolymer batch GP1
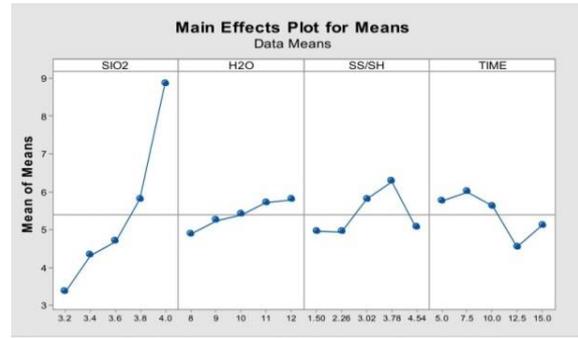

Figure 9. Major impacts plot for the compressive strength at 28 days of batch GP1

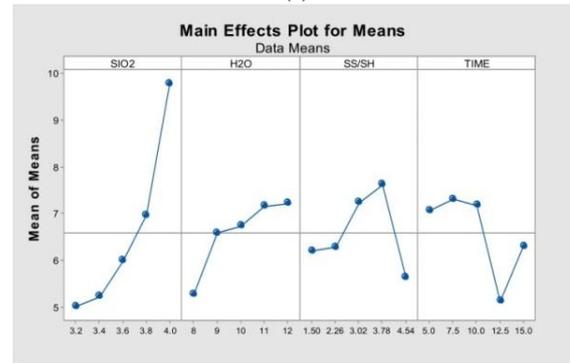**TABLE 5.** Compressive strength at 28-days compared with other studies

Compressive Strength at 28-days (MPa)	Reference
107	Current study
48.1	[20]
79	[19]
33-42	[21]
32	[22]
50	[23]
62	[24]
48.45	[25]
60-85	[26]

As expected, for batch GP1, the increase of the water content was found to increase both the initial and the final setting time. This was expected as the polymerization process of geopolymer is a water producer process; thus, increasing the water enhances the backward reaction according to Le Chateliers principle. It is important to note that the impact of the water content on the setting time is found to be lesser when the potassium content is increased. This may be related to the desired amount of water required to build the geopolymer structure, this amount seems to be higher in the case of potassium containing geopolymers.

It can be seen that the setting time is reduced when the mixing time is increased. This is expected as the mixing process enhances the dissolution process, which is the first step in the polymerization process. It is important to note that mixing time, as well as the (SS+PS)/(SH+PH) ratio, seems to have a minor impact on the setting time as compared with silica content.

3. 5. 3. Bulk Density   Figure 11 demonstrates the Taguchi major impact plots for the bulk density of the geopolymer patches. The results indicate that the density is influenced mainly by the water content, which determines the porosity of geopolymer. The influence of the mixing time increases when the potassium content is increased. The (SS+PS)/(SH+PH) ratio seems to have a minor impact on the bulk density of the prepared geopolymer. The impact of the silica content on the bulk density of K-geopolymer is less. It has been found that the bulk density of K-based geopolymer is less. This result is in contrast with the previous study of Lizcano,et.al. [27], where it was stated that the bulk density values recorded for K-based are 1.39–1.82 g/cm^3 and 1.25–1.72 g/cm^3 for Na-based metakaolin geopolymers.

3. 5. 4. Porosity and Water Adsorption   Figures 12 and 13 demonstrate the major impacts plots for the porosity and water absorption of the prepared Geopolymer, respectively. As expected, the water content was found to be the most effective factor on the

porosity and, hence, the water absorption. This is because of the formation of pores due to:

1- Removal of the extra water, which is commonly added to enhance the workability of the paste.

2- Removal of the water which is produced during the condensation polymerization.

Tables 6 and 7 list the values of the process parameters, suggested by Taguchi analysis to obtain Geopolymer with highest or lowest porosity, respectively, from each batch**.**
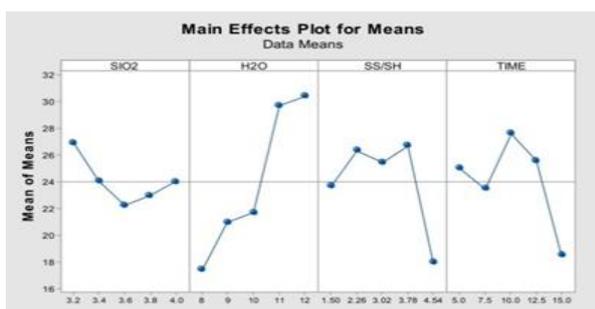

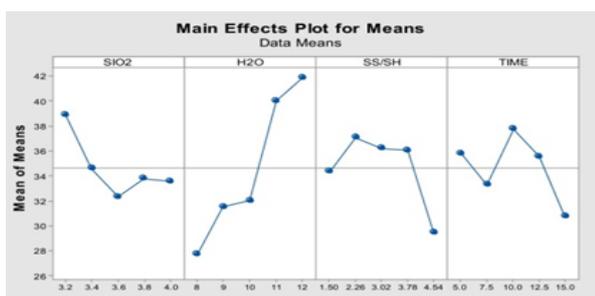**Figure 11.** The major impacts plot for the bulk density of GP1

Figure 12. The major impact plot for the porosity of GP1 batch
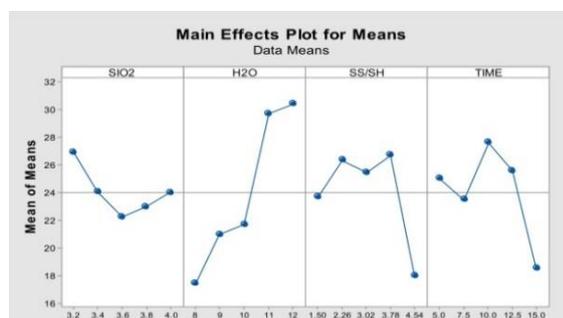

Figure 13. The major impact plot for the water absorption of GP1 batch

TABLE 6. The process parameters required to prepare Geopolymer with highest porosity

Batch	SiO₂	H₂O/ 10.73 MK-750	SS/ SH	Mixing time
GP1	3.4	12	3.78	10

TABLE 7. Process parameters required to prepare Geopolymer with lowest porosity

Batch	SiO₂	H₂O/ 10.73 MK-750	SS/ SH	Mixing time
GP1	4	8	1.5	15

4. CONCLUSIONS

From the results of the current study, the following can be driven:

Taguchi method is an appropriate method to design the experiments of metakaolin-based Geopolymer. Controlling the silica content, water content, SS/SH ratio, and the mixing time is required to produce geopolymers with a compressive strength exceeding 100MPa. In general, the strength increases by increasing the silica content and decreases by increasing water.The highest strength is obtained when the silica content is 3.4-3.6 with 8-11ml water. As for the concentration of (SS+PS)/(SH+PH), 2.26 seems to be the best choice. This indicates that the strength increases when the sodium ions are replaced partially by potassium ions. The porosity of Geopolymer decrease by increases silica content, mixing time, (SS+PS)/(SH+PH) ratio and increase by increase water content. Generally, the presence of Na and K ions together enhances the mechanical properties of metakaolin- based geopolymers. The added ion of K-ion to the Na-alkali solution reduces the setting time when the silica content is less than 3.8 and vice versa. The bulk density of metakaolin-based geopolymer reduces when K-ion is added to the mix on the expense of Na-ion

5. FUTURE WORKS

Based on the results obtained in the current work, the following studies are recommended: 1) The effect of using higher potassium contact on the properties of Geopolymer. 2) Geopolymer concrete prepared by using GP1. 3) Mechanical properties of the concrete prepared using GP1geopolymer.

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Persian Abstract

چکیده

خمیر ژئوپلیمر یک ماده ساختاری نوآورانه است که با عمل شیمیایی مولکول‌های معدنی تولید می‌شود. این ماده یک جایگزین زیست‌سازگار با محیط برای سیمان پرتلند معمولی است که به طور گسترده در دسترس جهانی است. در این مطالعه، تأثیر فعال کننده‌های مختلف قلیایی (Na و K) بر رفتارهای مکانیکی و حرارتی ژئوپلیمر مبتنی بر متاکائولین مورد بررسی قرار گرفت. هدف از این مطالعه، یافتن مخلوط‌ها و پارامترهای فرآیند تولید آن‌ها برای دست‌یابی به خمیر ژئوپلیمر با بالاترین مقاومت فشاری، بالاترین– کمترین تخلخل و بالاترین–کمترین زمان تنظیم اولیه و نهایی است. از روش تاگوچی در طراحی آزمایشات برای ژئوپلیمر مبتنی بر متاکائولین استفاده می‌شود. پنج عامل به عنوان پارامترهای فرآیند انتخاب شدند که احتمالاً روی خصوصیات ژئوپلیمر تأثیر می‌گذارند. این‌ها مقدار Si، نوع یون قلیایی، نسبت معرف‌های قلیایی، زمان اختلاط و مقدار آب هستند. تأثیر این پارامترها در زمان تنظیم، تراکم، تخلخل، مقاومت فشاری در ۷ و ۲۸ روز بررسی شدند. نتایج مطالعه نشان می‌دهد که خمیر ژئوپلیمر با مقاومت فشاری بالا (۱۰۷٫۲MPa) را می‌توان با فرمول (0.2 $SiO_2.xH_2O$, $2/6K_2O.0.8Na_2O. Al_2O_3$) با استفاده از شرایط پردازش مناسب به دست آورد که در آن سیلیکات قلیایی می‌شود. نسبت مولی هیدروکسیدهای قلیایی باید در محدوده ۲٫۲۶ نگاه داشته شود. نتایج نشان داد که استفاده از محلول قلیایی یون‌های پتاسیم و سدیم باعث افزایش مقاومت فشاری ژئوپلیمر در مقایسه با استفاده از محلول یون‌های سدیم همراه می‌شود. همچنین، توجه شده است که زمان استفاده برای ژئوپلیمرهای دارای محتوای سیلیس کمتر از ۳٫۸، هنگام استفاده از یون‌های K کاهش می‌یابد. به طور مشابه، تراکم فله ژئوپلیمرها با افزودن یون‌های K کاهش می‌یابد.