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

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**PAPER INFO**

**ABSTRACT**

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 (0.2K2O.8Na2O. Al2O3. 3.65SiO2.3H2O) 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.

**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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Please cite this article as: M. A. Ahmed Al-dujaili, I. A. Disher Al-hydarly, Z. Zayer Hassan, Optimizing the Properties of Metakaolin-based (Na, K)-Geopolymer Using Taguchi Design Method, International Journal of Engineering (IJE), IJE TRANSACTIONS A: Basics Vol. 33, No. 4, (April 2020) 631-638
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 construction 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 SiO2 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. Vaarsveld 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. MATERIAL 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 (Dwaikhlu, Western Iraqi Desert). The kaolin was calcined at 750 °C for three hours in air atmosphere using heating rate of 5°C/ min. Equation (1) was used to describe the composition of the prepared Geopolymer.

\[ 0.2K_2O:0.8Na_2O: Al_2O_3: nSiO_2: xH_2O \]

where (n) is the number of moles of SiO2 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 have 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 a 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 1). The physical, mechanical, and thermal properties were measured as an average of two-time measurements.

![Figure 1. Geopolymer cement paste specimen](Image)

| N  | H₂O (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              |

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 (σc = P/A). The physical, mechanical, and thermal properties were measured using Archimedes method.

![Figure 2. Flow chart that summarized the experimental work performed in the current study](Image)
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 at 750°C 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.

| Table 2. Wet chemical analysis of kaolin |
|----------------------------------------|
| SiO$_2$ (%) | Fe$_2$O$_3$ (%) | Al$_2$O$_3$ (%) | TiO$_2$ (%) | CaO (%) | MgO (%) | SO$_3$ (%) | P$_2$O$_5$ (%) | K$_2$O (%) | Na$_2$O (%) | 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 3. kaolin XRD form

Figure 4. Metakaolin (MK 750) XRD form

Figure 5. kaolin powder particle size

Figure 6. Metakaolin powder particle size
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 (0.2K₂O, 0.8Na₂O, Al₂O₃, nSiO₂). 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 is 15minutes.

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.

![Figure 8. The major impacts plot for the compressive strength at 7 days of batch GP2](Image)

| Level | SiO₂ | H₂O (ml)/10.73 MB-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           |
TABLE 4. Taguchi Analysis: Table of response for the 28-days compressive strength for GP2

| Level | SiO₂  | H₂O (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           |

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

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

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/cm³ and 1.25–1.72 g/cm³ 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.

| 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 decreases by increasing silica content, mixing time, (SS+PS)/(SH+PH) ratio and increase by increasing 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 GP1 geopolymer.

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خمیر زئوپلیمر یک ساختار متجانسی با بالاترین مقاومت فشاری و بالاترین سازگاری با محیط برای سیمان پرتلند معمولی است که به طور گسترده در دسترس جهانی است. در این مطالعه، تأثیر فعال کننده روی خصوصیات ژئوپلیمر تأثیرگذار است و تأثیر این پارامترها در زمان تنظیم، تراکم، تخلخل، مقاومت فشاری در خمیر زئوپلیمر یک ماده ساختاری نوآورانه است که با عمل شیمیایی مولکولی ایجاد می‌شود. این ماده که به طور گسترده در دسترس جهانی است، می‌تواند برای ساختن ژئوپلیمر به‌طور مثالی به عنوان بخشی از سیالیت‌های بسیاری مورد بررسی قرار گرفت. هدف از این مطالعه، بحث مکانیسم‌های متریال‌های زئوپلیمر با بالاترین مقاومت مقابل شارای دیگر- کمترین تخلخل و ناپایداری-کمترین زمان تکمیل لحیه و بالاتری است. این مطالعه نشان می‌دهد که برای ایجاد خمیر زئوپلیمر مبتنی بر بتاکائولین با بالاترین مقاومت، می‌توان از مولکول‌های موجود در محیط انتخاب کرد. این مطالعه نشان می‌دهد که می‌توان با استفاده از سیالیت‌های بالاترین مقاومت، خمیر زئوپلیمر شرایط طراحی کنید که می‌تواند به‌طور فیزیکی و شیمیایی مدت‌کاری را افزایش دهد.

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