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Physical Characteristics and Compressive Strength of Na-Geopolymer Paste Designed by a Taguchi Method

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Abstract. Geopolymer paste is a revolutionary building material that the chemical activity of inorganic molecules will create. It is an alternative to traditional Portland cement and is more Eco-friendly. This analysis aimed to classify the mixtures and their process parameters suitable for the development of Geo-polymer paste with one of the ultimate compressive powers, the highest-lowest porosity, and the lowest-lowest final and initial setting time. In the experimental design of the Geopolymer-based-metakaolin, a Taguchi methodology has been utilized. Five variables parameters were chosen that are mostly to influence the properties of the geopolymer. These are the quantity of Si, alkali, the proportion of alkali reagents, duration of blending, and water amount. These variables' influence has been calculated at 7 and 28 days on compressive strengths, porosity, density, and setting time. The analysis indicates that the strong compressive strength (115MPa) of Geopolymer paste could be achieved with the formula $(1\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 3.8\text{SiO}_2 \cdot x\text{H}_2\text{O})$ utilizing suitable processing conditions under which the molar ratio of alkali silicate to alkali hydroxide must be held within the range of 3.25-3.02.

Keywords: Geopolymer paste, Metakaolin-based Geopolymer, Taguchi method, Effect Alkali activator

1-INTRODUCTION

Geopolymers are relatively recent products that were first produced and patented in the 70s by Joseph Davidovits. These are inorganic polymeric products with a zeolite-like chemical structure, but their composition is either semi-crystalline or amorphous. They are formulated along with an alkaline-activating solvent (mainly Na⁺ hydroxides and silicates) from a combination of many aluminosilicate materials (with high Si and Al content).

Because of polycondensation, the geopolymer reaction process, geopolymerization, is exothermic. Alkali-activation, also called geopolymerization, was stated by Palomo et al. [1]. It is a chemical method that enables vitreous materials (partially or entirely amorphous and/or metastable) to be transformed into very compact composites that lute. Van Jaarsveld et al. [2] proposed that a highly alkaline medium is needed for geopolymerization to happen. This is essential for the breakdown of silica and alumina and the hydrolyzation of the raw material particles' surfaces. Such an atmosphere can be accomplished by utilizing alkaline solutions for activation in a clear or combination form.



To achieve the optimal processing parameters[3,4], a genetic algorithm (GA) technique was implemented. The Taguchi technique is one of the statistical techniques utilized to optimize the process parameters and enhance the efficiency of the components generated. This since achieves the procedure's desired outcomes; the Taguchi approach requires determining the right control factors. To perform a series of tests, Orthogonal Arrays (OA) have been utilized. The effects of these tests are utilized to evaluate the data and predict the generated components' consistency. [5].

The study of the effect these parameters on Geopolymers' properties by full factorial design requires carry out many experiments. It becomes complex and difficult when the factor number increases. To overcome this issue, experimental design methods, such as the Taguchi method, may be used. This method proves its efficiency in many fields, including biotechnology, environmental engineering, software testing, service system, education, and production planning. The Taguchi technique is a methodical technique for extending different variables concerning efficiency, cost, and quality. [6].

A measure of robustness utilized in Taguchi designs to define control variables that minimize uncertainty in a product or method by mitigating the impact of uncontrollable variables (noise factors). (noise factors). Throughout manufacturing or the procedure used, noise factors may not be managed but could be controlled throughout experimentation. A temperature could range to a low and high value between 40 °C and 80°C . The number of tests to be done rises by raising the number of levels to adjust a parameter.

The signal-to-noise (S/N) proportion is computed for every factor level combination. The equation for the smaller-is-better S/N proportion utilizing base 10 logs is:(5)

$$S/N = -10*\log(\Sigma(Y^2)/n) \quad (1)$$

Whereas: Y = responses for the given factor level combination and n = number of responses in the factor level combination.

However, in the nominal is best (I), the signal-to-noise (S/N) proportion is computed for each factor level combination. The equation for the nominal-is-best (I) S/N proportion utilizing base 10 log is:

$$S/N = -10*\log(s^2). \quad (2)$$

Whereas s = standard deviation of the responses for all noise factors for the given factor level combination.

The nominal is the best (II) of the signal-to-noise (S/N) proportion is computed for every factor level combination. The equation for the nominal-is-best (II) S/N proportion utilizing base 10 log is:

$$\frac{S}{N} = 10* \log \left(\frac{\bar{Y}^2}{S^2} \right) \quad (3)$$

Also, it could be a choice to utilize the adjusted equation for the nominal-is-best S/N proportion. The equation is:

$$\frac{S}{N} = 10* \log \left(\frac{\bar{Y}^2 - \frac{S^2}{n}}{S^2} \right) \quad (4)$$

Whereas: \bar{Y} = mean of responses for the given factor level combination. While, S = standard deviation of the given factor level combination responses, n = responses' number in the factor level combination.

Theoretically, a solid aluminosilicate source of geopolymerization could be any substance comprising aluminum and silicon. Nevertheless, blast furnace slags and ash from the burning of fossil fuels and calcinated clay are the most widely utilized components. The most widely utilized clay is kaolin that converts into metakaolin upon thermal activation. The resulting geopolymer is purer when metakaolin is utilized as the aluminosilicate source, which could have more easily defined characteristics than the Geopolymers derived from agro-industrial waste. Without the complications generated by utilizing fly ash, slags, and other alternate raw materials that include many hard-to-characterize amorphous phases, the geopolymers generated with metakaolin are deemed a " model method." For instance, fly ash is not a well-defined substance, but it involves many crystalline and vitreous stages. [7].

In the aqueous form, an alkaline compound is required by the activator. Thus, sodium hydroxide, sodium silicate, and silica gel are traditionally used substances. A combination of silicate (solid dissolved in water)

and hydroxide (solid dissolved in water) with silica gel (solid dissolved in water) is usually utilized. SiO₂ waste is the silicate of the solution formed, while the hydroxide ensures high alkalinity in the solution[8]. The alkaline solution has a significant function throughout geopolymerization and impacts the production of mechanical strength. Part of its selection relies on its reactivity and the expense of the products used. Geopolymers' mechanical performance is strongly impacted by the Si/Al proportion, hydroxide amount [9], geopolymerization reaction efficacy [10], water amount [11], and curing situation [12]. Depending on the resources used or based on their molar proportion, geopolymers may exhibit various thermal, chemical, and mechanical behaviors. The current research work aims to find the mixes, and their processing parameters, which are suitable to produce a Geopolymer paste with one of the following features: Highest compressive strength, highest/ lowest porosity, highest / lowest initial and final setting time.

2- Materials and methods

2.1 The Starting Materials

Metakaolin, sodium silicate, sodium hydroxide, and silica gel have been utilized as initial materials to synthesize Geopolymer cement. Metakaolin, which was 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 an air atmosphere using a heating rate of 5°C/ min. Equation (5) was used to describe the composition of the prepared geopolymer.



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) was studied, and the mixing time and the alkali reactants ratio using a Taguchi method to design the experiments. The Taguchi method suggested 25 experiments for the Geopolymer mix (GP1).

Four parameters were selected based on the previous studies, including the parameters that strongly affect the geopolymer's manufacture. The upper and lower limits of each parameter were selected depending on the finding of the prior studies and many primary experiments[13]. The criteria used to select these limits' values include: (1) The paste of the geopolymer should be easily mixable. (2) The setting time should be neither too short nor too long. 3) The resulting Geopolymer body should be free of macro-cracks. The parameters magnitudes are demonstrated in Table (1).

Table 1. The affected parameters the manufacture of the geopolymer

N	H ₂ O (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

2.2. Preparation of Geopolymer paste

The alkaline liquid utilized in this investigation was a mixture of sodium hydroxide (SH), and sodium silicate (SS). At first, the water is placed in the beaker, and then the sodium hydroxide is weighed and added to the water to reach the desired molarity(M). The sodium hydroxide pellet release heat as they dissolved in the water. When the sodium hydroxide addition is complete, the sodium silicate is added to the solution. At this time, they placed the solution on stirrers, a magnetic rod in the solution, and stirred at 80°C and 600 rpm. After all, the sodium silicate dissolved. Silica gel was added to the solution. Approximately one hour

later, all the silica dissolved completely. After that, the desired water volume was added to compensate for 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 (3550rpm) desired to mix time. Molds made of plastic with diameter = 2.1 cm and height = 4.2 cm have been utilized for molding the pastes of geopolymers. The specimens should be kept in a specific condition at a lab with temperature ± 2 (23 °C) for one day and demolded. Then, these samples have been cured under the specific condition for 7 or 28 days.

2.3. Characterizations

The chemical composition of the kaolin was found using a wet chemical method. The kaolin wet chemical analysis test is currently being used to analyze the content levels of heavy metals in kaolin. This analysis was performed at the Iraqi geological and mining survey. The starting materials' main oxides were declared in terms of their weight percentages (Table 2). The crystalline phases of the starting materials have been recognized by the X-ray diffraction (XRD) technique (Shimadzu 6000 diffractometer, Japan). $\text{CuK}\alpha$ X-ray as source radiation was used with 30 mA and 40 kV at a $5^\circ/\text{min}$ scanning speed. Nickel was used as a filter. Particle size analysis tests have been made to identify the distribution of kaolin and metakaolin powder's particle size by using a laser particle size analyzer. The synthesized samples were investigated with the Microvickers Hardness device model (TH-717) with a load of 9.8N and a holding time of 15 seconds. The diagonals distance was measured with the device of Vickers hardness.

The SEM was utilized to examine the features of the Geopolymer paste's microstructure; these features include the pores shapes, micro-cracks, and the presence of crystal. Geopolymer paste setting times are calculated utilizing the Vicat needle in compliance with ASTM C191. The penetration was recorded for each 15min period 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 is described as the time when the needle did not sink into the paste. Differential thermal analysis (DTA) was utilized to observe transition phases, crystallizations, and decomposition temperatures. According to the Archimedes method, the density, porosity, and water absorption of the synthesized samples were measured. Simultaneously, the compressive strength has been determined utilizing the formula ($\sigma_c = P/A$). The physical, mechanical, and thermal properties were measured as an average of two-time measurements.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis of Kaolin

Figure 1 demonstrates the kaolin powder XRD form. The pattern confirms the crystalline structure of the powder utilized. 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 temperature leads to the formation of metakaolin; this was confirmed for the kaolin heat treated a 750°C for 3h (MK-750) as demonstrated in Figure 2. The pattern asserts the amorphous structure of metakaolin powder. XRD analysis of metakaolin demonstrates a sharp peak that refers to free quartz in the kaolin powder.

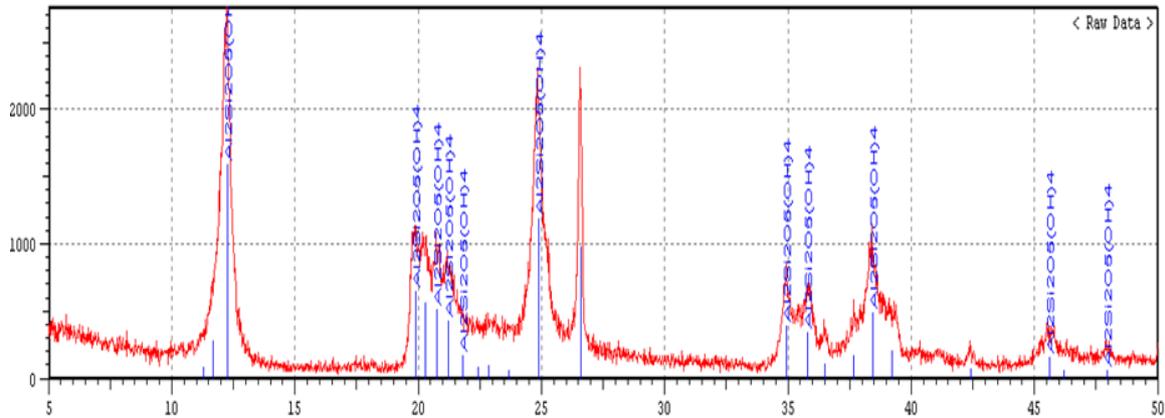


Figure 1. Kaolin XRD form

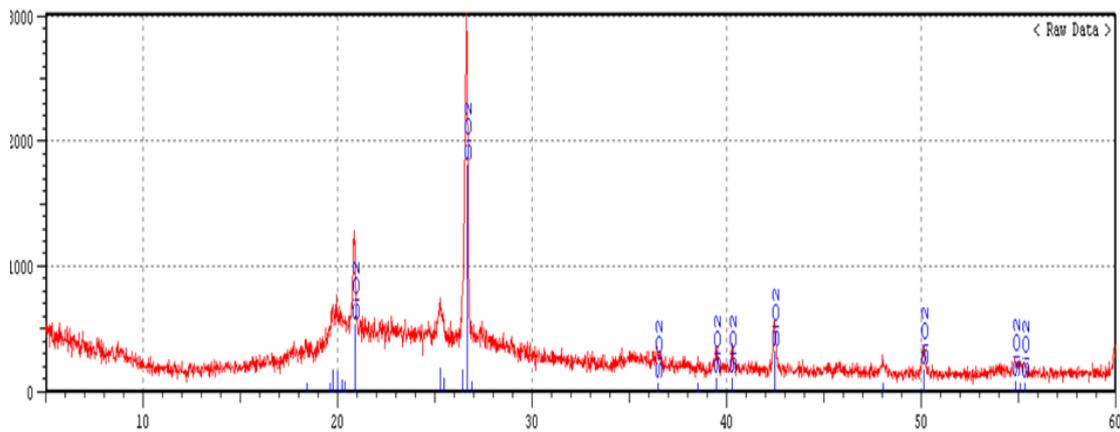


Figure 2. Metakaolin (MK 750) XRD form

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 SiO₂ amount in the clay is higher than the stoichiometric amount in the kaolinite, which is (48.77%). Furthermore, that confirms the XRD result regarding the presence of free quartz and helps calculate the amount of free quartz. Approximately, that should be excluded when the geopolymer's composition is calculated because of quartz's inertness, which makes quartz an inert component during the synthesis of the geopolymer.

Table 2. Demonstrates the kaolin wet chemical analysis results

SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	CaO	MgO	SO ₃	P ₂ O	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

3.3 Analysis of Kaolin Particle Size

Figure 3 demonstrates the distribution of kaolin powder particle size. The results demonstrated that the kaolin is composed of micro-sized particles mainly below 20 μm . The particle size distribution is multimodal with a D_{50} of 3.8 μm . The distribution of MK-750 particle size is demonstrated in Fig (4); it is well known that metakaolin has fine particle size as a compound with kaolin as it is produced due to the breaking of the kaolinite structure. However, the agglomeration and aggregation lead to large secondary particles, as confirmed in Figure 4.

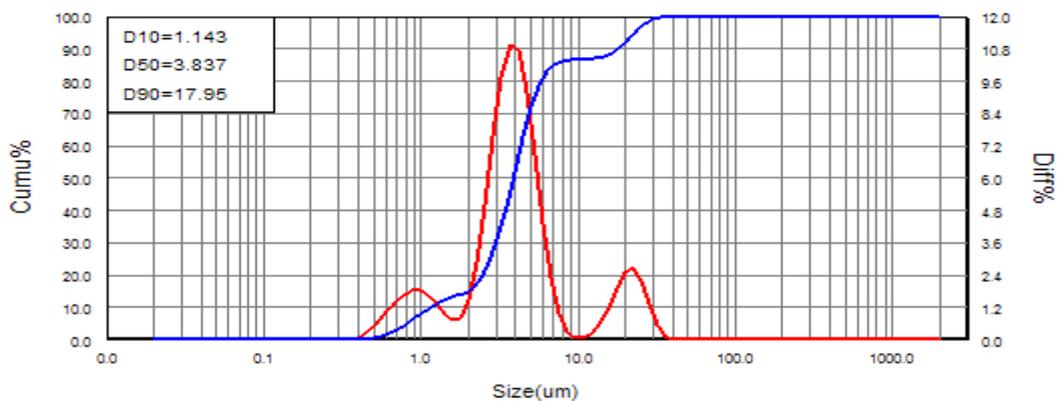


Figure 3. The analysis of kaolin powder particle size

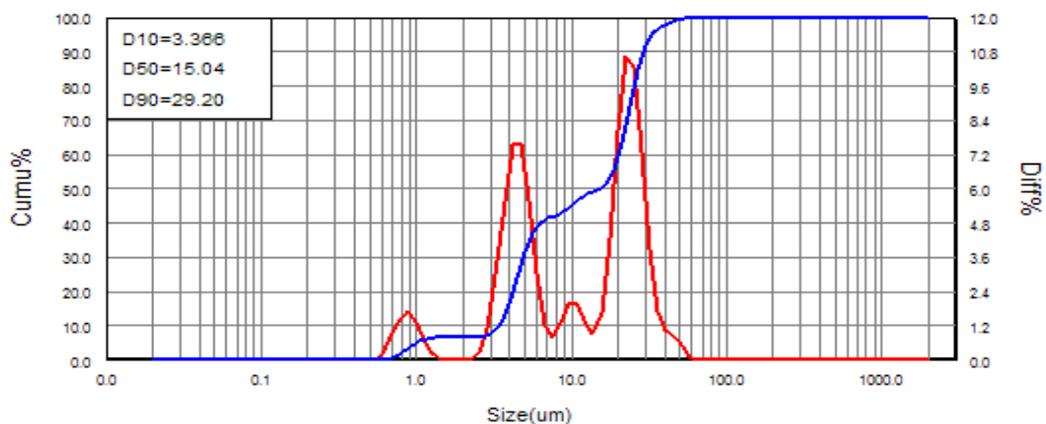


Figure 4. The analysis of Metakaolin powder particle size.

3.4 DTA Analysis of Kaolin

Figure 5 demonstrates the differential thermal analysis (DTA) for kaolin. The endothermic peak at 530 $^{\circ}\text{C}$ was recorded for the kaolin calcination. To form metakaolin, this event is finished at 575 $^{\circ}\text{C}$, and no other thermal event could be observed, indicating that 750 $^{\circ}\text{C}$ is a suitable choice for the formation of metakaolin.

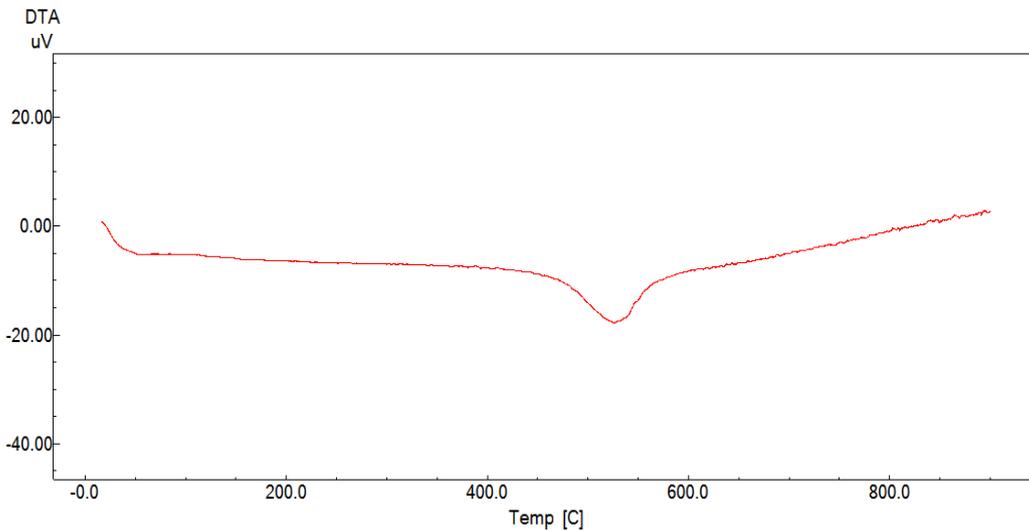


Figure 5. Different thermal analysis (DTA) for kaolin

3.5 Geopolymer paste Result

3.5.1 Compressive Strength

3.5.1.1 Compression Strength at 7 Days

Table (3) illustrates the Taguchi analysis's findings for the compression strength after seven days since the casing date and gives the rank for the process parameters utilized in the current study. The results indicate that the most influential factors on the compressive strength are silica content mixing time water content SS/SH ratio. It can be observed that the compression strength after seven days since the casing date for all the tested sample is higher than 30MPa and reaches up to (61MPa). These values are too high as compared with that reported in pieces of literature as demonstrated in Table (4)

Table 3. Taguchi Analysis of response for 7-days compressive strength for GP1

Level	SiO ₂	H ₂ O/10.73 MK-750	SS/SH	Mixing time
1	33.42	42.60	32.86	41.82
2	39.22	32.60	35.00	35.62
3	45.46	33.22	42.64	35.68
4	31.60	39.70	36.80	30.00
5	32.82	34.40	35.22	39.40
Delta	13.86	10.00	9.78	11.82
Rank	1	3	4	2

These results indicate the advantage of the current study over the previous studies.

Table 4. Values of compressive strength at 7-days compared with other studies

Compressive strength at 7-days (MPa)	Reference
61	Current study
8.61	[14]
31	[15]
28.53	[16]
18.94	[16]
32	[17]
21.33	[18]

Figure 6 demonstrates the major impact plot for the compression strength at 7 days of Geopolymer paste of batch GP1 which satisfies the formula ($\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot n\text{SiO}_2$). In general, the strength increases by increasing the silica content up to 3.6, where the best strength can be obtained. The silica content controls the Si/Al proportion that impacts hydrolysis, dissolution, and Geopolymer condensation reaction. Depending on prior studies, many academics stated that the optimal proportions of $\text{SiO}_2/\text{Al}_2\text{O}_3$ were between 3 and 3.8 that was somewhat less compared to the ranges that Davidovits stated them [19]. Moreover, De Silva et al. proved that [20] the Geopolymer system condensation response with little proportion of Si/Al tends to occur between types of silicate and aluminate, producing primarily structures of poly(sialate). In the system of geopolymer that has a great Si/Al proportion, Geopolymer structures of poly(sialate-disiloxo) and poly(sialate-siloxo) are made due to condensation response of predominant between silicate types, making silicates (oligomeric), which reacted with $\text{Al}(\text{OH}_4)^+$. Greater numbers of siloxo units have been contributing to improving strength.

Rahier, et al. [21] report that low and high amounts of water in producing Geopolymers will negatively affect the rate of reaction. This was supposed as a result of the reduced concentration of OH at a high amount of water. In other words, at low amounts of water, despite increasing OH concentration, the reactive types (H_3SiO_4^- monomer monodeprotonated) for reacting between oligomers' silicate reductions and reducing the reaction rate of geopolymerization.

For the concentration of SS/SH, the strength also generally increases by increasing the SS/SH ratio due to the acceleration of polymerization; the highest strength was obtained at the ratio of 3.02. Mixing time is one of the variables that affect the Geopolymer strength where it works to homogenize the alkali solution with the powder; the result demonstrates the best compressive strength at a time of 5 min.

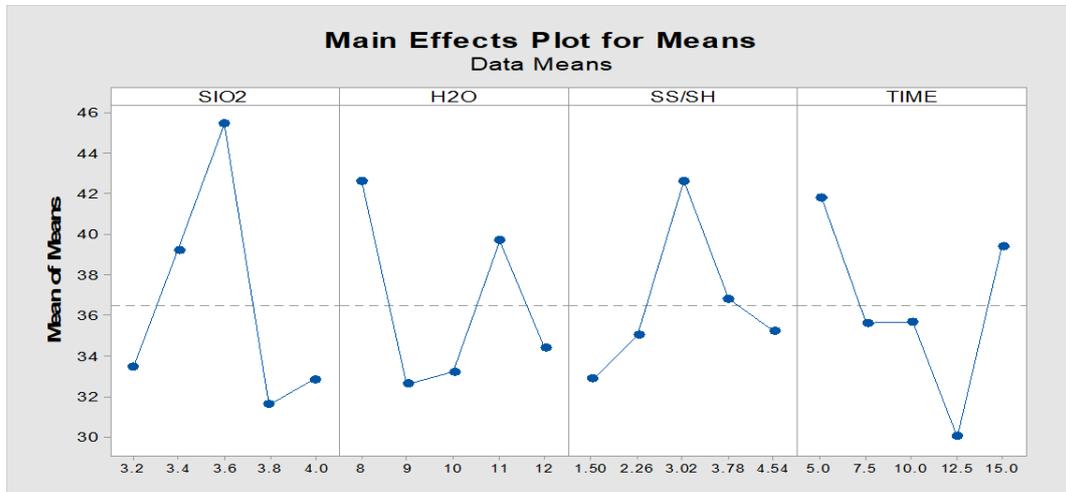


Figure 6. The major impacts plot for the compressive strength at 7 days of batch GP1

3.5.1.2 Compression Strength after 28 Days

The Taguchi analysis outcomes for the 28-days compressive strength are shown in table (5) for batch GP1. It could be observed that the silica amount was still the most effective factor in the strength of the sample under compression. However, compared with the 7-days strength under the same batch compression, the water content gets the second-order among the other effective factors; this might be connected to the amount of water on the porosity of the resulting Geopolymer body. On the other hand, the mixing time and SS/SH proportion's influence is limited to 10-16MPa only.

As shown in Figure 7, the 28-days compression strength increases when the silica content increases up to 3.8. Similarly, it increases with increasing the water content up to 11ml; this is maybe due to facilitating the mixing process as reported by Ghosh, et al. [22]. The achieved 28-day compressive strength was as high as (114MPa), which is a very high value compared with the results reported by many researchers, as demonstrated in Table (6).

Table 5. Taguchi Analysis of response for 28-days compressive strength for GP1

Level	SiO ₂	H ₂ O/10.73 MK-750	SS/SH	Mixing time
1	48.56	58.56	64.00	76.62
2	71.62	66.84	69.32	63.42
3	68.70	65.00	73.14	60.44
4	77.60	78.42	62.70	71.18
5	75.28	72.94	72.60	70.10
Delta	29.04	19.86	10.44	16.18
Rank	1	2	4	3

Table 6. Values of compressive strength at 28-days compared with other studies

Compressive strength at 28-days (MPa)	Reference
114	Current study
48.1	[23]
79	[24]
33-42	[25]
32	[26]
50	[27]
62	[15]
48.45	[18]
60-85	[13]

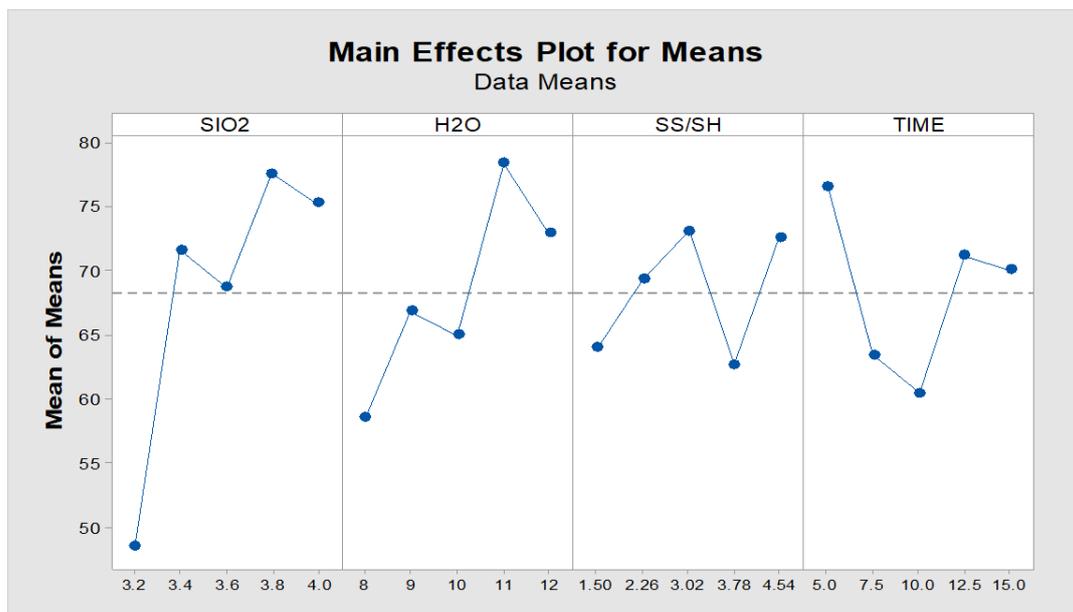


Figure 7. The major impacts plot for the compressive strength at 28 days of batch GP1

3.5.2 Setting Time

Figure 8 demonstrates the major impact plot for the setting time of GP1. In batch GP1, silica content is the most influential factor in the initial and final setting time. When geopolymer contains sodium ions only, as in GP1batch, the setting time decreases by increasing the silica content up to 3.8. This indicates that the polymerization process rate, in the presence of Na ions only, increases when the silica content is increased.



Figure 8. (A) initial setting time and (B) the final setting time of Geopolymer batch GP1

3.5.3 Bulk Density

Figure 9 show the Taguchi major impact plot for the bulk density of the geopolymer patches. The results indicate that the density is impacted mainly by the water content, which decides the geopolymer's porosity. The SS/SH ratio seems to have a minor impact on the bulk density of the prepared geopolymer. The impact of the silica amount on the bulk density of Na-geopolymer is high. It has been found that the bulk density of Na-based geopolymer is high. This result is in contrast with the previous study Lizcano, et al. [28], where it has been specified that the bulk density magnitude note down for metakaolin Geopolymers' Na-based are (1.25–1.72 g/cm³) and K-based are (1.39–1.82 g/cm³).

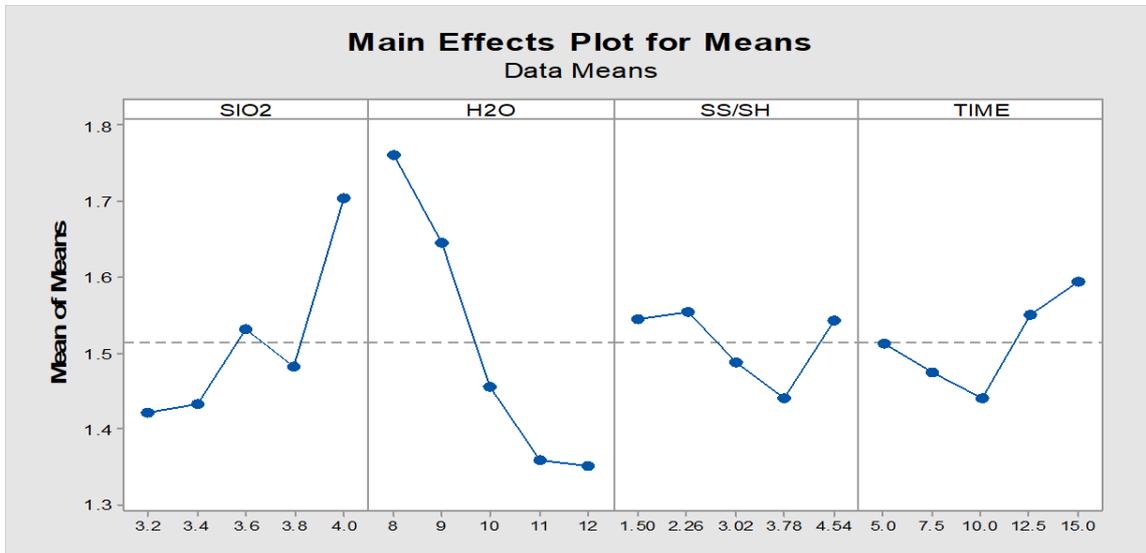


Figure 9. The major impacts plot for the bulk density of GP1

3.5.4 Porosity and Water Adsorption

Figures 10 and 11 demonstrate the major impact plots for the prepared geopolymer's water absorption and porosity, respectively. As expected, the water content was found to be the most effective factor on the porosity; hence, the water absorption caused the formation of pores due to:

- 1-The removal of the extra water, which is commonly added to enhance the workability of the paste.
- 2-The removal of the water which is produced during the condensation polymerization.

Tables (7) and (8) list the value of the process parameters, suggested by Taguchi analysis, to obtain geopolymer with the highest or lowest porosity, respectively, from each batch.

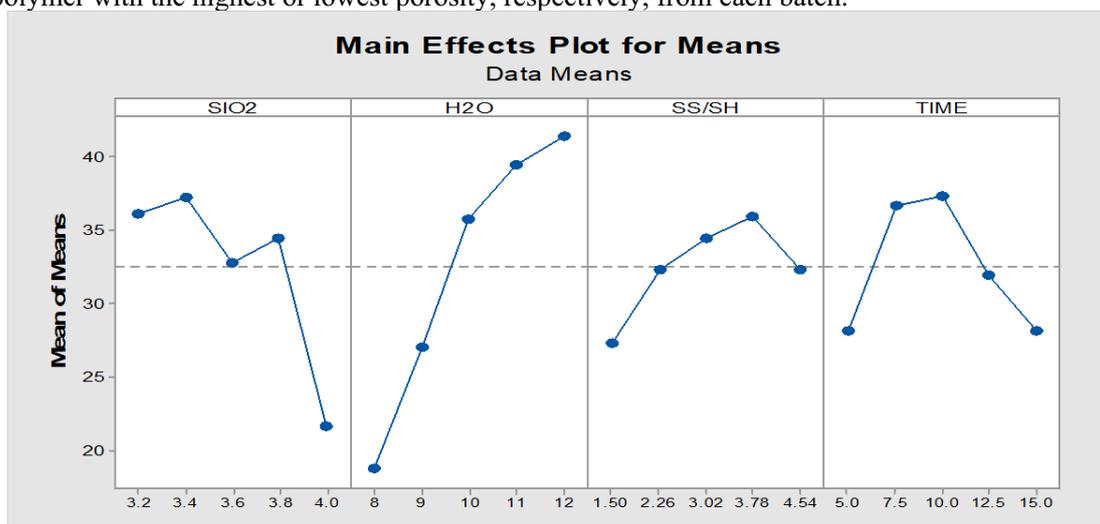


Figure 10. The major impact plot for the porosity of GP1 batch

Table 7. the process parameters required to prepare geopolymer with the highest porosity

Batch	SiO ₂	H ₂ O/10.73MK-750	SS/SH	Mixing time
GP1	3.4	12	3.78	10

Table 8. the process parameter required to prepare geopolymer with the lowest porosity.

Batch	SiO ₂	H ₂ O/10.73MK-750	SS/SH	Mixing time
GP1	4.0	8	1.5	15

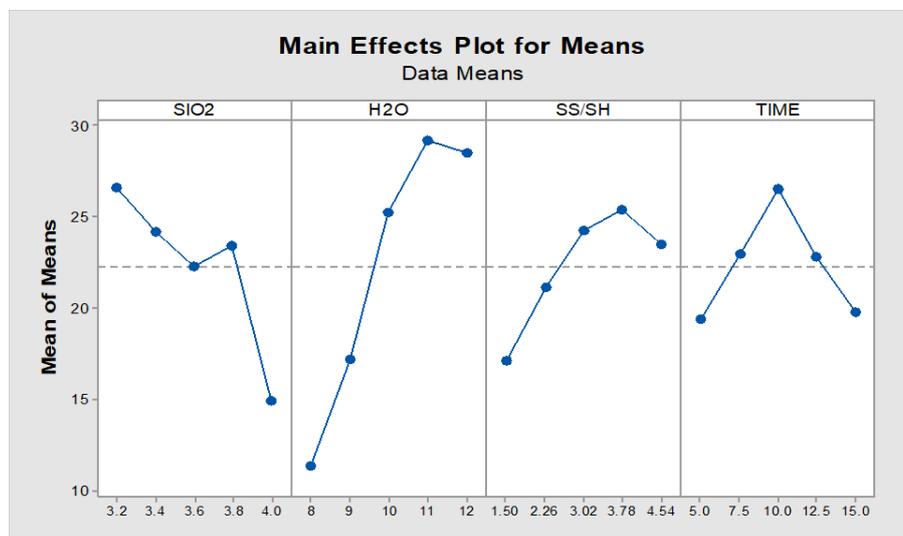


Figure 11. The major impact plot for the water absorption of GP1batch

3.6 Results of Optimum Mixes

3.6.1 Compressive Strength of Geopolymer paste

Table (9) illustrates the value of the processing variables required to obtain the optimal compressive strength according to Taguchi analysis.

Table 9. Value of the processing variables to obtain the optimal compressive strength according to Taguchi analysis

Batch	SiO ₂	H ₂ O/10.73MK-750	SS/SH	Mixing time
GP1	3.8	11	3.02	5

Table (10) demonstrates the compressive strength of Geopolymer cement at 7 days and 28 days. The result reveals that the suggested optimum mix achieved compressive strength higher than that obtained from the experimental works; this confirms that the Taguchi method can suggest the optimum conditions to get optimum results.

Table 10. Compressive strength of geopolymer paste prepared according to the optimum mix

Batch	Compressive strength at 7 days (MPa)	Compressive strength at 28 days (MPa)
GP1	96	115

3.6.2 Vickers Hardness of Geopolymer Paste

Table (11) shows the Vickers hardness of Geopolymer cement past, prepared according to the optimum mixes.

Table 11. Vickers Micro-hardness of Geopolymer cement

Batch	Vickers hardness (HV)
GP1	28.83

3.6.3 Physical properties of Geopolymer Paste

Table (12) demonstrates the result of the density, porosity, and water absorption of Geopolymer paste. The consequences show that batch GP1 has the greatest density magnitude and lowest porosity. This result explains the reason behind the hardness result as well as the compressive strength.

Table 12. Density, water absorption, and porosity of Geopolymer paste

Batch	Density (g/cm ³)	Water absorption %	Porosity %
GP1	1.4	2.9	4

3.6.4 Setting Time of Geopolymer Paste

Table (13) shows the geopolymer paste's final and initial setting time. The results indicate that the batch GP1 setting times within the range accepted for cement applications.

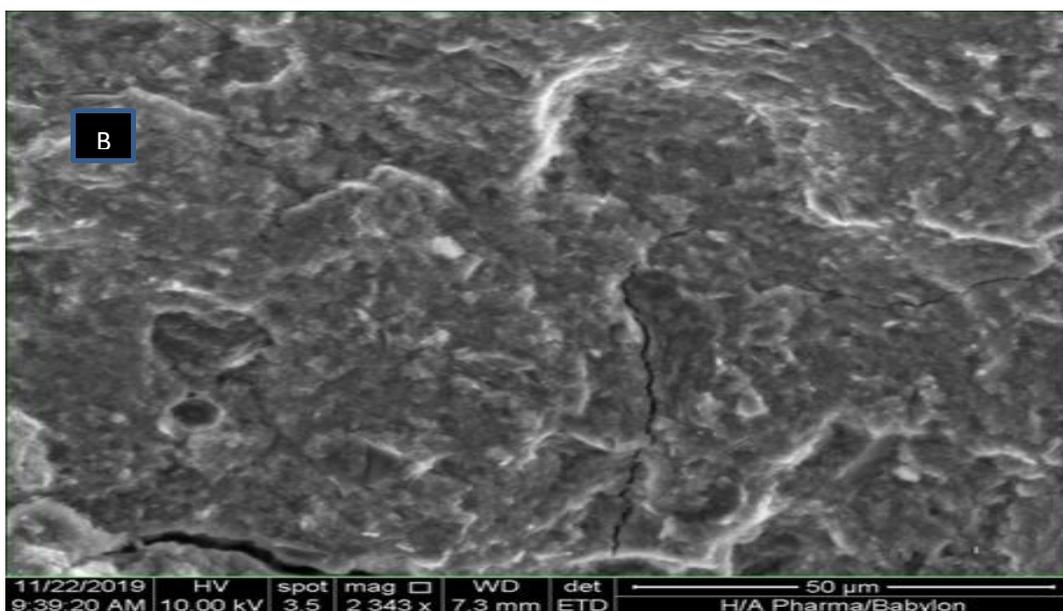
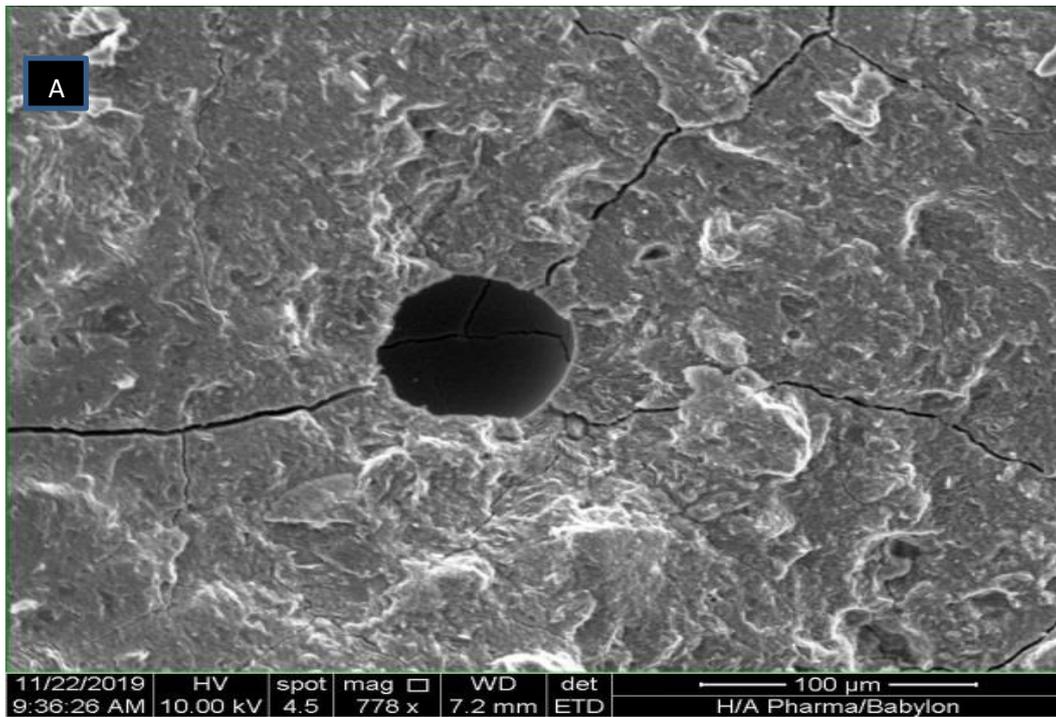
Table 13. The initial and final setting time of the Geopolymer paste

Batch	Setting time (min)	
	Initial	Final
GP1	130	270

3.6.5 Microstructure of Geopolymer Paste

A few spherical voids and micro-cracks that can be traced to air bubbles throughout the geopolymerization process and micro-cracks attributable to fracture are seen in Figure 12 for batch GP1. It is stressed that the large structure observed tends to validate the amorphous properties of these geopolymers. These bubbles added to the overall porosity. In the tests, the unreacted metakaolin particles can not be detected; the unreacted raw metakaolin particles could serve as structural faults decreasing the Geopolymer paste's compressive power [29]. Slower and more uniform reaction kinetics may be identified with completely reacted matrices with homogenous distribution and a dense gel process. The slower method involved in

Geopolymer paste's strength production helps the precursor to melt continuously, resulting in high strength of a more homogeneous composition at later ages [30].



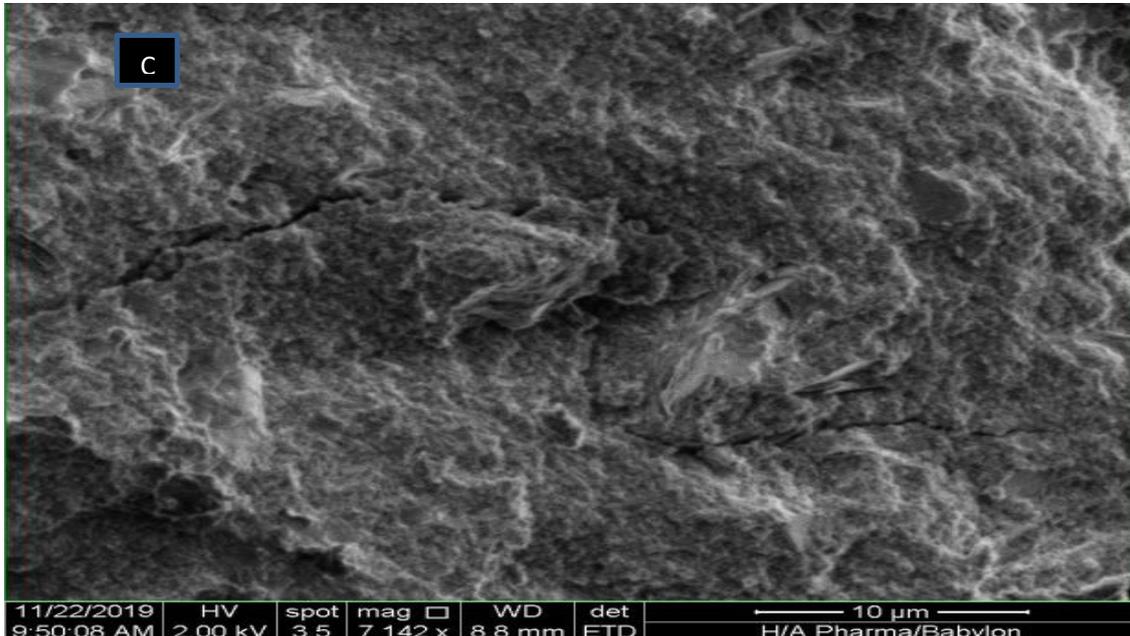


Figure 12. SEM images of geopolymer paste for batch GP1

4. CONCLUSIONS

From the findings of the current study, the following can be concluded:

1. The Taguchi method is an appropriate method to design the experiments of metakaolin-based Geopolymers.
2. Controlling the silica content, water content, SS/SH ratio, and the mixing time is required to produce a geopolymer with a compressive strength that exceeds 100MPa.
3. The porosity of geopolymer can be low as 3% or high as 40%, depending on the mix composition and processing conditions.
4. The alkali hydroxides to silicates molar proportion must be kept between 3.02-3.25 in order to obtain high compressive strength.

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