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Impact of Sulfate in the Sand on the Compressive Strength of Metakaolin-Based Geopolymer Mortar

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ABSTRACT

The advancement of cement alternatives in the construction materials industry is fundamental to sustainable development. Geopolymer is the optimal substitute for ordinary Portland cement, which produces 80% less CO_2 emissions than ordinary Portland cement. Metakaolin was used as one of the raw materials in the geopolymerization process. This research examines the influence of three different percentages of sulfate (0.00038, 1.532, and 16.24) % in sand per molarity of NaOH on the compressive strength of metakaolin-based geopolymer mortar (MK-GPM). Samples were prepared with two different molarities (8M and 12M) and cured at room temperature. The best compressive strength value (56.98MPa) was recorded with 12M with lower sulfate content (0.00038%) at 28 days. Also, an inverse relationship is recorded between the increasing sulfate percentages in the sand and the compressive strength values of (MK-GPM). A higher reduction in the compressive strength results at 28 days (60.88% per 8M/NaOH) and (62.23% per 12M/NaOH) was associated with a higher percentage of SO_3 in the sand (16.24%).

Keywords: Geo-polymer, Sulfate, Molarity, Alkaline liquid, Compressive strength.

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تأثير نسبة الكبريتات في الرمال على مقاومة الانضغاط لمونة الجيوبوليمر المصنعة من الميتاكاؤولين

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الخلاصة

أحد الجوانب المهمة للتنمية المستدامة هو النهوض ببدائل الأسمنت في صناعة مواد البناء. يعد الجيوبوليمر أفضل مادة بديلة للإسمنت البورتلاندي العادي ، والذي ينتج عنه انبعاثات ثاني أكسيد الكربون أقل بنسبة 80٪ من الأسمنت البورتلاندي العادي. تم استخدام الميتاكوؤلين كأحد المواد الخام في عملية البلمرة الجيولوجية. يتناول هذا البحث تأثير ثلاث نسب مختلفة من الكبريتات (0.00038،1.532،16.24) ٪ في الرمل لكل مولارية من هيدروكسيد الصوديوم على مقاومة الانضغاط لمونة الجيوبوليمر المصنعة من الميتاكوؤلين (كمادة رابطة) .تم تحضير العينات بتركيزين مولاريين مختلفتين (8 م ، 12 م) وتمت معالجتها في درجة حرارة الغرفة. تم تسجيل أفضل قيمة لمقاومة الانضغاط (56.98 ميجا باسكال) بتركيز مولاري (12م) مع محتوى كبريتات (0.00038٪) في 28 يوم. وايضا تم تسجيل علاقة عكسية بين زيادة نسب الكبريتات في الرمل وقيم مقاومة الانضغاط للعينات حيث كان الانخفاض الأعلى في قيم مقاومة الانضغاط عند 28 يومًا (60.88٪ لكل 8 م / هيدروكسيد الصوديوم) و (62.23٪ لكل 12 م / هيدروكسيد الصوديوم) مرافقا لأعلى نسبة من ثاني أوكسيد الكبريت في الرمل (16.24٪).

الكلمات المفتاحية: الجيوبليمر ، الكبريتات ، المولارية , المحلول القلوي ، قوة الضغط.

1. INTRODUCTION

It is essential to develop a new binding material to replace conventional cement in the building industry that minimizes the emissions of CO_2, other harmful gases, and waste dust to combat environmental pollution. **(Muhsin and Fawzi, 2021; Chen, et al., 2021)**. Some researchers have developed novel materials to wholly or partially replace traditional cement, with geopolymeric material being the most promising binder material. **(Wang, et al., 2020; Marvila, et al., 2021)**. The geopolymer is a new three-dimensional reticular material mostly made of raw ingredients and alkaline liquids. Alumino-silicate-based materials with high silicon (Si) and aluminum (Al) content, such as MK, GGBFS, and other materials, were activated using an alkaline solution as a binder. **(Amouri and Fawzi., 2022; Kanagaraj et al., 2023)**

At room temperature, the geo-polymer hardens and gains mechanical strength and durability. Geo-polymer qualities are determined mainly by the characteristics of the underlying ingredients (chemical composition, crystalline phase content, silicon and aluminum solubility, distribution of particle sizes, and existence of inert particles). Sodium silicate (Na_2SiO_3), NaOH, and potassium hydroxide are commonly employed in alkaline solutions for geo-polymer production **(Al-Jaberi et al., 2021).** Because it is challenging to find well-graded sand with an acceptable sulfate concentration that may be utilized in

mortar, sulfate-contaminated sand is a local issue in Iraq **(Fawzi et al., 2015).** Sulfate is known to offense away at concrete and mortar, causing them to expand, weaken, and decay. "Internal" and "External" are two forms of attacks in concrete or mortar. Sulfate found in mortar materials induces internal attack, while sulfate in the aquatic environment around the mortar causes the exterior attack **(Kheder et al., 2010; Atahan and Dikme, 2011)**.
Depending on calcium concentration, the degradation mechanism of geo-polymers in a sulfate environment is variable. Due to the similarities of hydration products, increased calcium alkali-activated compounds have equivalent degradation mechanisms to OPC, as explained by **(Shi, 2003; Alcamand et al., 2018)**. Limited alkali-activated systems allow ions to move between the sulfate solution and the network structure, which may be more resistant to sulfate assault due to reduced crystalline phase formation during an expansion **(Wang et al., 2020; Chen et al., 2021).** The development of internal attack, according to the authors **(Brunetaud et al., 2012; Campos et al., 2016)**, is caused by the presence of considerable amounts of oxygen and humidity, which cause the oxidation of mineral sulfide, resulting in sulfate ions and iron hydroxide in the atmosphere.
Geopolymer concrete performs better than conventional concrete due to its higher early strength and lower permeability, increasing stability under aggressive environments **(Karakoc et al., 2016; Amran et al., 2021).** So, studying the effect of internal sulfate attack on the geopolymers is a topic that needs more investigation because the research in this field is limited.
The current work investigates the effect of sulfate in the sand, NaOH molarity, Na_2SiO_3/NaOH, and solid/liquid ratio on geopolymer mortar's Compressive Strength.

2. EXPERIMENTAL WORK

2.1. Materials

2.1.1. Metakaolin

Kaolin clay was obtained in the Al-Anbar Governorate's Dewekhla district. The kaolin is ground and then calcined in a furnace at 700 $^oC \pm 20$ ^oC for 1 hour to make Metakaolin. The Metakaolin was then ground to 23 m^2/g. The chemical composition of Metakaolin that meets the Pozzolan ASTM C618-19 and physical composition are given in **Tables 1. and 2**.

Table 1. Metakaolin's Chemical Composition

Oxide	Content, %	ASTM C618 requirements
SiO_2	45.59	The sum of values more than 70%
Al_2O_3	35.16	
Fe_2O_3	1.97	
CaO	0.48	
MgO	0.42	
SO_3	0.41	Max. 4%
Na_2O	1.029	
K_2O	0.2658	
LOI.	1.018	Max. 10%

Table 2. Metakaolin's physical properties

Physical property	Value
Specific gravity	2.62
Physical form	Powder
Surface area m^2/kg	1730
Color	Off-white

2.1.2. Sodium Hydroxide NaOH

NaOH, with a purity of 98%, in flak form, is commercially-available. The solids should be dissolved in distilled water to make a solution with the needed concentration. According to the ratio of caustic soda flakes to water, various molar concentricity levels were possibly achieved. The concentration of sodium silicates is determined by the ratio of Na_2O to SiO_2 and water.

2.1.3. Sodium Silicate, Na_2SiO_3

The Na_2SiO_3 utilized in this study was produced in the United Arab Emirates. The characteristics of sodium silicate are listed in **Table 3.**

Table 3. Characteristics of sodium silicate

Description	Value
SiO_2 % by weight	32-33
Na_2O % by weight	13.1 – 13.7
The ratio of SiO_2/ Na_2O	2.4 ± 0.05
Density - 20° Baumé	51 ± 0.5
Appearance	Hazy
Viscosity (CPS) 20°C	600 – 1200
Specific Gravity	1.5340 – 1.5510

2.1.4. Water

For preparing the NaOH solution, distilled water was utilized to dissolve caustic soda flakes and used in the geopolymer mix design to enhance its workability.

2.1.5. Fine Aggregate

Two normal sands from Al-Ekhadir and Al-Obeidi regions were used with three percentages of sulfates (0.00038, 1.532, and 16.24) % in mortar mixtures of this work. The grading and physical characteristics of the two types were within Iraqi Standard's limits (**Iraqi Standard IQS No.45, 1984)** within the zone (2).

2.1.6. High-Range Water Reducing Admixture

To improve the workability of the geopolymer mortar, a high-range water lowering (superplasticizer) derived from modified sulfonated naphthalene formaldehyde condensed was employed. It was confirmed to **(ASTM C494/C494M, 2017**)

2.2. Alkaline Solution Preparation for Geopolymer

2.2.1. Creating NaOH Solutions at Various Concentrations:

High volumes of sodium hydroxide flakes in distilled water were dissolved to create different quantities with a purity of 98%. Generally, NaOH concentration usually varies from (5 – 16) in molarity, as mentioned **(Hussein, 2021)**. In this study, NaOH concentration values were (8M and 12 M). The mass of solid sodium hydroxide in a solution changed depending on the solution's concentration. For example, a 12 M NaOH solution has 12 x 40 = 480 g sodium hydroxide solids per liter, where 40 represents the molecular weight of NaOH, (O = 16, Na =23, and H = 1).

2.2.2. Alkaline Liquid Preparation

After the preparation of the NaOH solution, it is added to the sodium silicate solution. Then, stir this mix for (2-3) min., which is considered an alkali liquid. It was recommended to produce the alkaline liquid by mixing the two solutions at least 24 h before utilization **(Lloyd and Rangan, 2010)**.

2.3. Procedure for Mixing

The raw material (MK) and sand were combined to the tune of two or three minutes. After combining the dry ingredients with the alkaline liquid produced, more water and a superplasticizer were added. The final mix is mixed for 4 to 5 minutes to achieve homogeneity.

2.4. Casting Process

Three layers of geo-polymer paste were poured into steel molds after completing the mixing procedure. Depending on the mold form, each layer was stirred and vibrated on a vibrating table for one to two minutes. The vibrating was utilized to release the trapped air.

2.5. Curing

After casting, geopolymer mortar samples are allowed to cure at room temperature, as shown in **Fig. 1.**

Figure 1. Curing of samples

2.6. Laboratory Examination

Compression strength testing was performed on all geopolymer mortar mixes according to **(ASTM C109/C109M, 2020).** Cube specimens with 50 x 50 x 50 mm dimensions were tested using the hydraulic compression machine. This test takes ages 7 and 28 as reference ages after curing.
Regular patterns in standard specifications fundamentally depend upon the compressive strength of the mortar or concrete in assessing other mechanical characteristics because it's a significant property in concrete or mortar mixtures **(Voigt et al., 2006)**.

2.7. Proportions of the Mix

The combination percentage used in the current study is shown in **Table 4**. This investigation used (520 kg/m^3) of the source material (MK) in the mixes, and the molarity value was (8M and 12M). The solid/liquid ratio was (1), the Na_2SiO_3/NaOH ratio was (1.5), and sand was used with SO_3% of three percentages (0.00038, 1.532, and 16.24) %.

Table 4. Mix proportions of all mixes

Mix	NaOH molarity	SO_3% in sand
G1	12	0.00038
G2	8	0.00038
G3	12	1.532
G4	8	1.532
G5	12	16.24
G6	8	16.24

3. RESULTS AND DISCUSSION

3.1. Compressive Strength

Table 5. clarifies the compressive strength results for all mixes. The highest compressive strength is obtained at 28 days in mix 1 with NaOH (12M) and sand containing %SO_3 (3.8E-4). It could be noted from **Figs. 2 to 4** that the geo-polymer mortar's compressive strength increases as the NaOH concentration increases, as explained by **(Azad et al., 2021; Prasad et al., 2022)**, where the geopolymerization process, which is responsible for the composition of the metakaolin, results in the stimulation of Silica and Alumina to generate the requirements of alumino-silicate gel for a stronger alkaline solution **(Chindaprasirt and Rattanasak., 2017)**. As a result, a higher concentration of NaOH increases the solution's ability to leach alumina and silica, developing sufficient binding among the solid particles in the resulting system structure and significantly impacting the mechanical properties of the hardened geo-polymer **(Khaled et al., 2023)**. Gel formation has two opposing effects: first, it depletes the ions, causing additional ions to leach from the MK particles. Second, solution thickening reduces ion mobility, especially near the material's surface, slowing ion leaching.

Table 5. The compressive strength results (Na_2SiO_3/NaOH equal to 1.5, S/L equal to 1)

Set. No	Molarity	%SO_3 in sand	Total SO_3% of the mix	Effective SO_3% of the mix	Comp. St at 7 days	Comp. St at 28 days
G1	12	0.00038	0.51	0.51	44.98	56.98
G2	8	0.00038	0.51	0.51	42.56	54.88
G3	12	1.532	3.343	1.82	31.98	39.35
G4	8	1.532	3.343	1.82	29.80	36.52
G5	12	16.24	30.55	14.45	17.62	22.29
G6	8	16.24	30.55	14.45	16.21	20.73

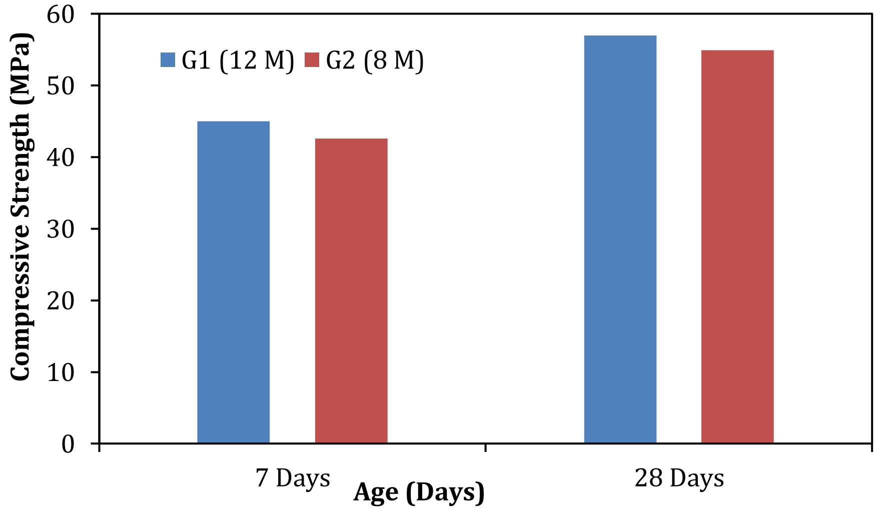

Figure 2. The influence of NaOH molarity on the compressive strength of G1and G2

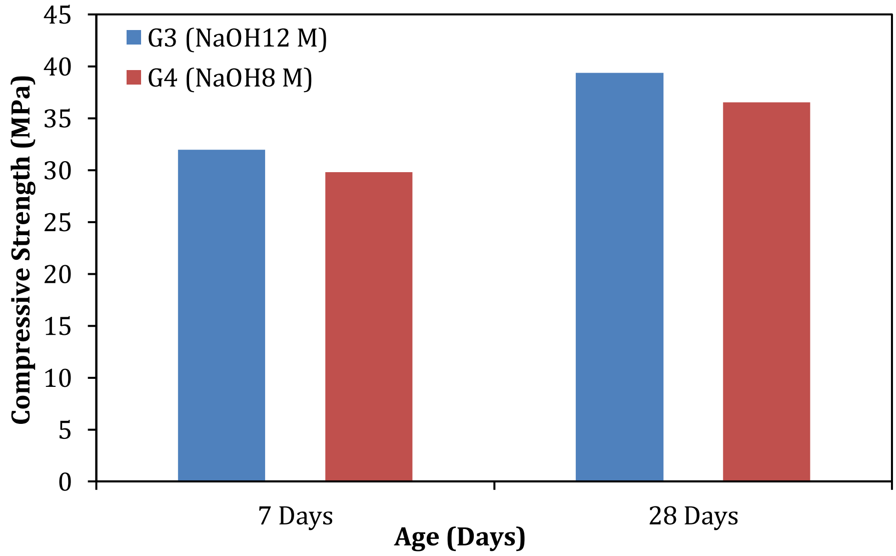

Figure 3. The influence of NaOH molarity on the compressive strength of G3and G4

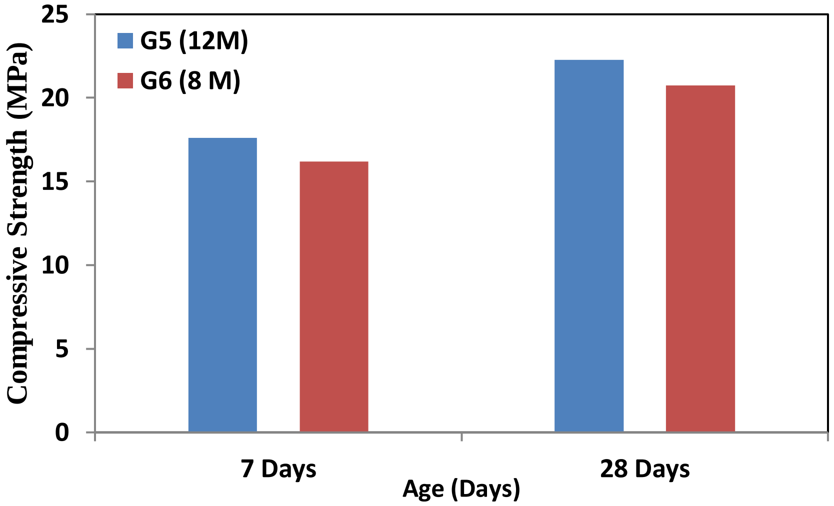

Figure 4. The influence of NaOH molarity on the compressive strength of G5 and G6

From the results in **Figs. 5 and 6.**, it can be observed that when the sulfate content in sand rises, the compressive strength decreases. Because the excessive SO_3 leads to the occurrence of an internal sulfate attack which is an exchange process in which cations (i.e., sodium) exchange with the components of N-A-S-H gel in metakaolin (low-calcium alkali-activated system), this chemical reaction reduces the adhesion forces of the N-A-S-H structure resulting in increased porosity and micro-crack development and therefore a reduction in compressive strength **(Alcamand et al., 2018)**.

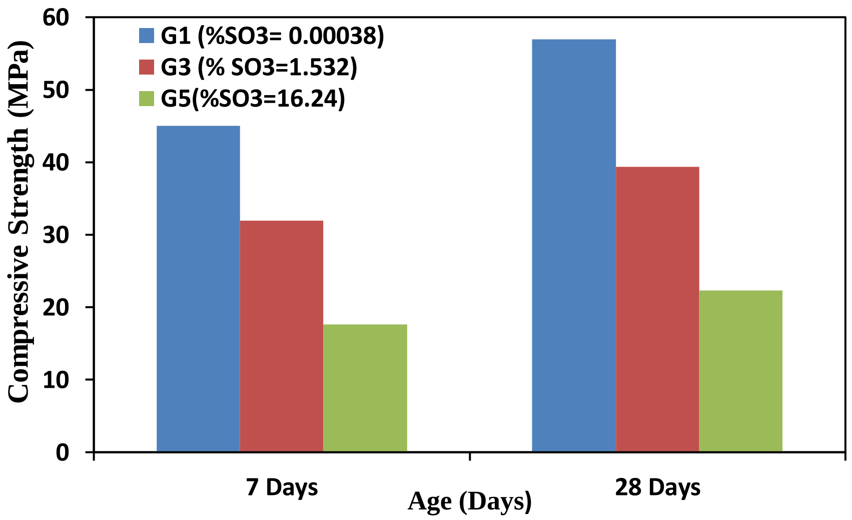

Figure 5. The influence of %SO_3 on the compressive strength of G1, G3 and G5

From **Table 6.** the loss in compressive strength of mix G3, which contained (1.532) % of SO_3 in the sand, was (28.9 and 30.94) at 7 and 28 days, respectively, compared to the reference mix. Likewise, for the rest of the other mixtures, as given in **Tables 6 and** 7, it is concluded that the reduction in compressive strength of concrete is affected by the increase in the total effective sulfate content more than the total sulfate within the mixture this is consistent with the findings of **(Mas et al., 2012; Fawzi et al., 2015)**. For example, when total sulfates in the mix (12) increase by (30.55%), the short and final ages of mortar strength are decreased by (61.91and 62.23) %, respectively, when compared to the reference mortar with a total effective sulfate of (0.51%).

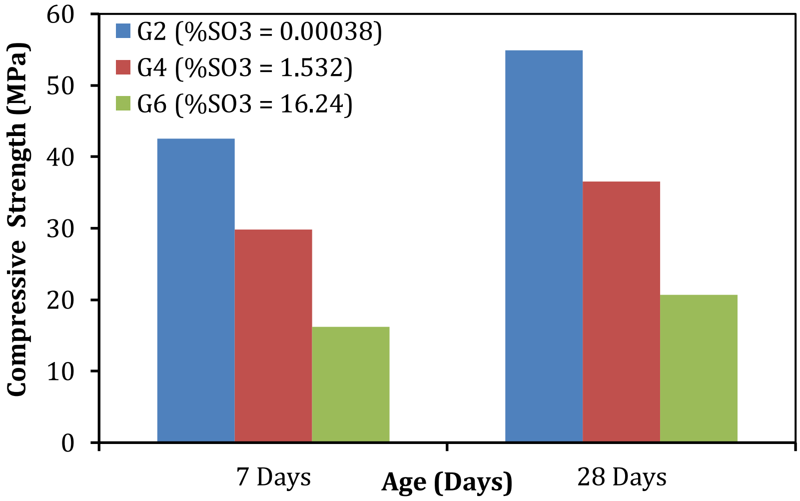

Figure 6. The effect of $\%SO_3$ on the compressive strength of G2, G4, and G6.

The above can be explained as owing to the action of sulfate. As the sulfate percent in sand increases, the effect of the effective $SO_3\%$ increases. Accordingly, the reduction in compressive strength increases also **(Liu et al., 2018; Chen et al., 2020)**

Table 6. Reduction in compressive strength of G1, G3, and G5 at various ages.

Set. No.	$SO_3\%$ in sand	Total $SO_3\%$ of mix	Effective $SO_3\%$ of mix	Comp. St at 7 days	Comp. St at 28 days	Reduction in Compressive strength (%)	
						7 days	28 days
G1	0.00038	0.51	0.51	44.98	56.98	-	-
G3	1.532	3.343	1.82	31.98	39.35	28.90	30.94
G5	16.24	30.55	14.45	17.62	22.29	60.82	60.88

Table 7. Reduction in compressive strength of G2, G4, and G6 at various ages.

Set. No.	$SO_3\%$ in sand	Total $SO_3\%$ of mix	Effective $SO_3\%$ of mix	Comp. St at 7 days	Comp. St at 28 days	Reduction in Compressive strength (%)	
						7 days	28 days
G2	0.00038	0.51	0.51	42.56	54.88	-	-
G4	1.532	3.343	1.82	29.8	36.52	30.29	32.55
G6	16.24	30.55	14.45	16.21	20.73	61.91	62.23

To calculate the total $SO_3\%$ content in mortar constituents, the following formula according to (**Iraqi Standard IQS No. 45, 1984)** was used:

$$Total\ SO_3 = \frac{binding\ material}{binding\ material} \times SO_3\%\ in\ binding\ material + \frac{fine\ aggregate}{binding\ material} \times SO_3\ in\ fine\ aggregate \quad (1)$$

For geopolymer, the formula will be:

$$Total\ SO_3 = \frac{binding\ material}{binding\ material} \times SO_3\%\ in\ binding\ material + \frac{fine\ aggregate}{binding\ material} \times SO_3\%\ in\ fine\ aggregate + \frac{NaOH}{binding\ material} \times SO_3\%\ in\ NaOH + \frac{Na_2SiO_3}{binding\ material} \times SO_3\%\ in\ Na_2SiO_3 \quad (2)$$

The present study used the total effective sulfate in concrete to calculate the optimum SO_3 content. The following is an experimental formula was used to calculate the total effective SO_3% content in mortar constituents **(Al-Sammari and Rouf., 1987; Al-Rawi et al., 2002)**:

$$SO_3(effective) = 0.9 - 0.25\sqrt{F.M} \quad (3)$$

F.M represents the Fineness Modulus

4. CONCLUSIONS

This work concerns using sand with three different percentages of SO_3 in geopolymer mortar production. This approach is one of the effective methods to reduce CO_2 emissions to the atmosphere by up to 80%. Based on the analysis and discussion of the obtained results in this study, the following conclusions can be drawn:

- Due to its better features and sustainable ingredients, metakaolin-based geopolymer mortar is preferred over conventional mortar.
- Raising the NaOH concentration from (8 to 12) M increases the compressive because of a decrease in total water and a greater Geopolymerization rate, making the geopolymer paste more viscous.
- The geopolymer mortars with the most significant percentages of sulfate concentration in the sand (16.24 %) had the greatest reductions in compressive strength (60.88and 62.23) at the time of (28) days because the higher percentages of SO_3 led to the occurrence of an internal sulfate attack which is a chemical reaction resulting in increased porosity and micro-crack development and therefore a reduction in compressive strength.
- An increase in the total effective sulfate content of the mortar decreases the compressive strength of the mortar, but the total effective sulfate content is more linearly related to this reduction than the total sulfate content; therefore, the total effective SO_3 is a useful indicator of the potential of using sand in the mortar.

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