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Impact of Sulfate in the Sand on the Absorption and Density of Metakaolin-Based Geopolymer Mortar

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ABSTRACT
The advancement of cement alternatives in the construction materials field is fundamental to sustainable development. Geopolymer is the optimal substitute for ordinary portland cement, which produces 80% less CO_2 emissions. Metakaolin was used as one of the raw materials in the geopolymerization process. This research examined the influence of three different percentages of sulfate (0.00038, 1.532, and 16.24)% in sand per molarity of NaOH on the absorption and density of metakaolin-based geopolymer mortar (MK-GPM). Samples were prepared with two different molarities (8M and 12M) and cured at room temperature. The best results obtained for geopolymer mortar in the absorption and density test were (3.89%) and (2280 kg/m^3), respectively, recorded with 12M with the lowest sulfate content (0.00038%) at 90 days. Moreover, it has been observed that the absorption percentage increased along with sulfate content in the sand, and an inverse relationship was recorded between the increasing sulfate percentages in the sand and density values of (MK-GPM).

Keywords: geo-polymer, sulfate, molarity, alkaline liquid, absorption.

INTRODUCTION

Ordinary portland cement (OPC) is the main binding material utilized in concrete production. The environmental hazards associated with OPC production are well-known. In addition, the amount of energy required to produce steel and aluminum is similar to that required to manufacture OPC [1, 2]. Variations in the raw material availability have a significant impact on cement production [3, 4]. To reduce the cement manufacturing and therefore to decrease the CO_2 release in atmosphere and the construction cost, environmentally friendly materials are being created and incorporated into civil engineering projects [3, 4]. For manufacturing mortar and concrete, fractional replacement of cement by Pozzolanic (siliceous and aluminous) materials such as Metakaolin, Granulated Blast Furnace Slag, Fly Ash, Silica Fume and Rice Husk Ash has been attempted to reduce waste [5, 6].

The word "geopolymer" was coined in 1978 to refer to a category of mineral binders with a similar chemical composition to zeolites. On the other hand, geopolymers utilize polycondensation of silica and alumina predecessors, as opposed to conventional portland / pozzolanic cement to create the matrix. The primary components of geopolymers are raw materials and alkaline liquids. Alumino-silicate-based raw materials that are rich in silicon (Si) and aluminum (Al) are utilized. Geopolymerization yields a greater solids concentration than alumino-silicate gels or zeolite synthesis [7].

Because it is challenging to find well-graded sand with an acceptable sulfate concentration that may be utilized in mortar or concrete, sulfate-contaminated sand is a local issue in Iraq [8]. Concrete may be subjected to internal deterioration when it contains high sulfate content of concrete constituents with different types of cement.

Many studies are carried out on the durability properties of conventional concrete, but few studies are carried out on geopolymer concrete. Regarding the durability factors, geopolymer concrete indicates superior performance to traditional cement due to its higher early strength and lower permeability that grant it higher stability under aggressive environments [9]. Thus, this research studied the effect of different percentages of sulfates in sand and NaOH molarity on the absorption and density of metakaolin-based geopolymer mortar because the researches in this field are limited.

MATERIAL CHARACTERIZATION

Metakaolin (MK)

Kaolin has been acquired in western Iraq, (Dewekhla region, Al-Anbar Governorate). An air blast pulverized the kaolin, and the resulting particles were sieved to pass a 60-mm size. The kaolin powder was then burned for two hours at 750 degrees Celsius in a furnace. The metakaolin powder was finally cooled for 24 hours at room temperature. The chemical and physical analyses of metakaolin comply with ASTM C618 [10], as indicated in Tables 1 and 2.

Sodium hydroxide

The purity of commercially available NaOH flakes is 98%. NaOH is used in the production of geopolymer mortar solutions. To produce NaOH, caustic soda flakes are melted in water. Depending on the ratio of soda flakes to water, various molar concentrations can be achieved.

Table 1. Metakaolin' chemical analysis

Oxide	Content, percent %	ASTM C618 requirements
SiO_2	45.59	Sum of value 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	
L.O.I	1.018	Max. 10%

Table 2. Metakaolin' physical properties

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

Sodium silicate

The ratio of Na_2O to SiO_2 and H_2O determines the concentration of Na_2SiO_3. The employed Na_2SiO_3 was manufactured in the United Arab Emirates.

Water

To prepare the NaOH solution, distilled water was used to melt caustic soda flakes and was in the geopolymer mix design to enhance its workability.

Fine aggregate

Two normal sands from the 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 Standards' limits. I.Q.S. (No.45/1984) [11] within the zone (2).

High-range water reducing admixture

To improve the workability of the geopolymer mortar, a high-range water lowering (superplasticizer) derived from adjusted sulfonated naphthalene formaldehyde condensed was employed. It conformed to ASTM C494 [12].

GEOPOLYMER MANUFACTURING

Alkaline solution preparation for geopolymer

Creating NaOH solutions at various concentrations – high volumes of sodium hydroxide flakes in distilled water were dissolved to produce different quantities with a purity of 98 per cent. The NaOH concentration usually varies from (5–16) in molarity [13]. The mass of solid sodium hydroxide in a solution changes depending on the concentration of the solution. For example, a 12 M NaOH solution has 12 x 40 = 480 g sodium hydroxide solids per liter, where 40 signifies the molecular weight of NaOH; O = 16, Na = 23 and H = 1.

Alkaline liquid preparation – after the NaOH solution has been prepared, it is combined with the sodium silicate solution. This mixture was then stirred for two to three minutes, considered an alkaline liquid. It was suggested that the alkaline liquid be produced by combining both solutions at least 24 hours before use [14].

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 that was produced, more water and a superplasticizer were added. The final mix was mixed for 4 to 5 minutes to achieve homogeneity, as shown in Table 3.

RESULTS AND DISCUSSION

Water absorption test

This test has been done based on the (ASTM: C-642 -13) [15]. The samples were weighed after removing each sample from water ($W1$); afterward, they were dried at 100 °C - 110 °C oven temperature for 24h and weighed ($W2$). The difference between the weights is the absorbed water's weight ($W3$). The percentage of water absorption was calculated using the following equation:

$$\begin{gathered}\text{Water Absorption \%} = (W3 / W2) \times 100 \\ W3 = W1 - W2\end{gathered} \tag{1}$$

where: $W1$ denotes the weight of the sample before the drying;
$W2$ denotes the dry sample's weight.

Increasing concentration of NaOH from (8M to 12M) led to decreasing the water absorption of geo-polymer as shown in the Figures (1), (2) and (3) due to increasing NaOH concentration leading to reduction of voids per cent and improvement of the density of samples because of increasing geo-polymerization rate, also increase molarity results in the reduction of the water content and improved micro-structure of the geo-polymer [16].

The mixes, including G3, G4, G5 and G6, show an increase in absorption values compared to the reference mixes (G1 and G2), as shown in Figures (4) and (5), because of the increase in the total effective (SO3%) content in these mixes at all ages of the test. The higher content of sulfates in mortar mixes makes the pores in specimens larger, which creates a poor transition zone

Table 3. Mix proportions for geopolymer mortar

Mix	$\%SO_3$ in sand	Total $\%SO_3$ of mix	Effective $\%SO_3$ of mix	M.K kg/m^3	NaOH kg/m^3	Na_2SiO_3 kg/m^3	Fine aggregate kg/m^3	Water kg/m^3	Sp by Weight	NaOH molarity	Na_2SiO_3/ NaOH
G1	0.00038	0.51	0.51	520	205	315	962	100	8	12	1.5
G2	0.00038	0.51	0.51	520	205	315	962	100	8	8	1.5
G3	1.532	3.343	1.82	520	205	315	962	100	8	12	1.5
G4	1.532	3.343	1.82	520	205	315	962	100	8	8	1.5
G5	16.24	30.55	14.45	520	205	315	962	100	8	12	1.5
G6	16.24	30.55	14.45	520	205	315	962	100	8	8	1.5

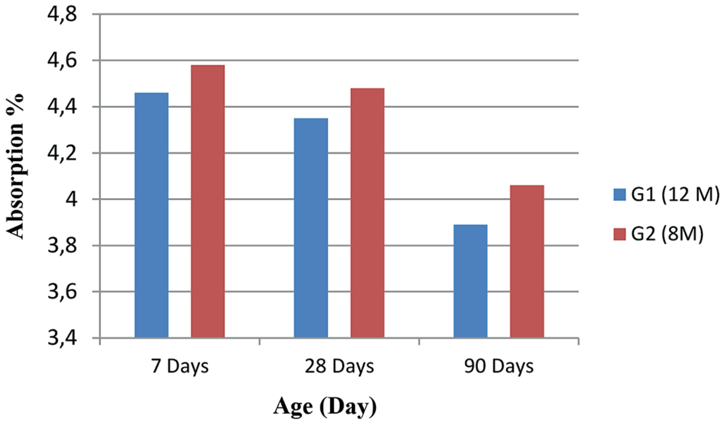

Figure 1. The effect of NaOH molarity on the absorption of G1 and G2 at different ages

between the geo-polymer paste and fine aggregate and absorbs high quantities of water [17].

In addition, Figures (4) and (5) reveal an important fact: the water absorption of all geopolymer mortars decreased with time; after 90 days, it was lower than after 28 days, and after seven days, it was lower than after 28 days. This long-term decline in water absorption is attributable to an increase in pozzolanic reaction and a greater degree of geopolymerization.

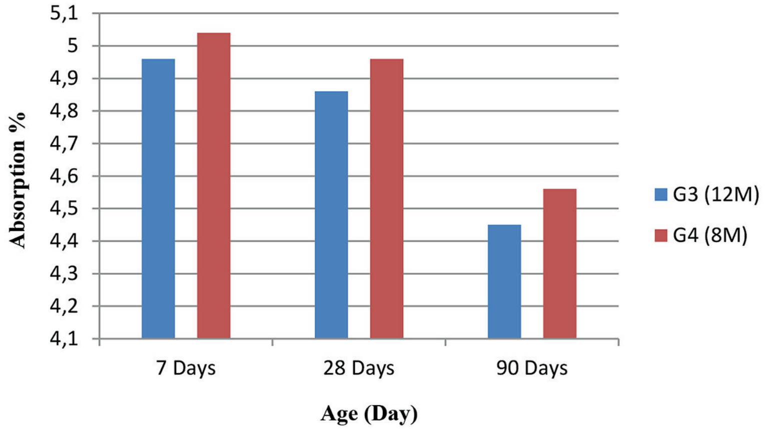

Figure 2. The effect of NaOH molarity on the absorption of G3 and G4 at different ages

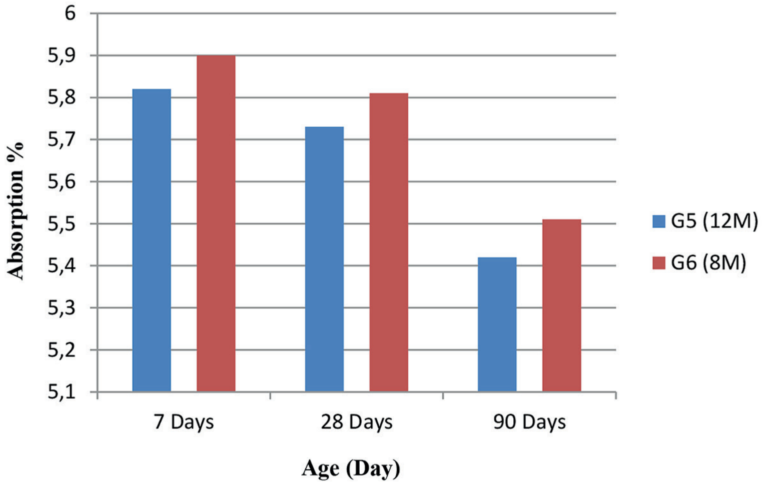

Figure 3. The effect of NaOH molarity on the absorption of G5 and G6 at different ages

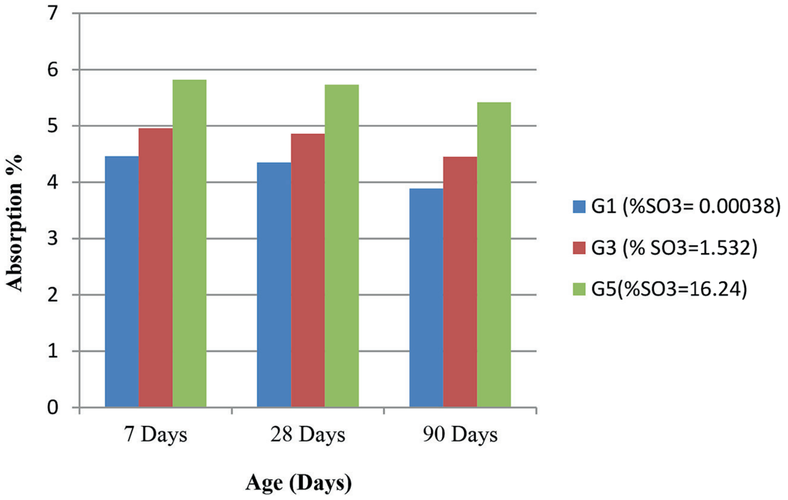

Figure 4. The effect of %SO_3 on the absorption of G1,G3 and G5

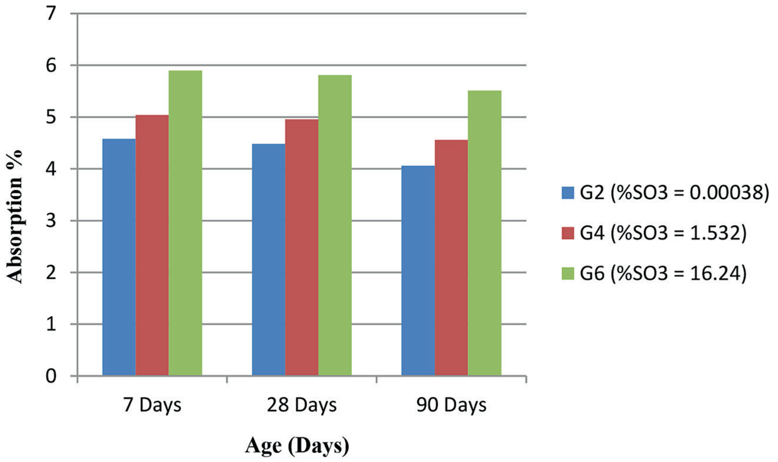

Figure 5. The effect of %SO_3 on the absorption of G2, G4 and G6

Dry density

Using the standard method (ASTM C 138-17) [18] of weighing samples and dividing their mass (in kilograms) by their volume, the density of the particulate particle composites was estimated in kg/m^3. The dimensions of the sample cube dimensions are 50×50×50 mm, as shown in Figure (6).

Figure 6. The samples from the dry density test

As shown in Figures (7), (8) and (9), the density value of geo-polymer mortar increased when the molarity increased from 8M to 12M because the density is a function of weight; therefore, an increase in molarity means an increase in the amount of solute in a fixed volume, and on top the density increases [19].

Figures (10) and (11) show the effect of sulfate in the sand on the density. The results show that the mixes exhibit a decrease in density with the increase in total effective (SO_3%) content in mortar mixes at all ages of the test. Generally, the results show a reduction in density relative to reference geopolymer mortars which contain (0.00038%) SO_3 content in sand.

This may be construed as excessive (SO_3) ions diffusing in the geopolymer structure,

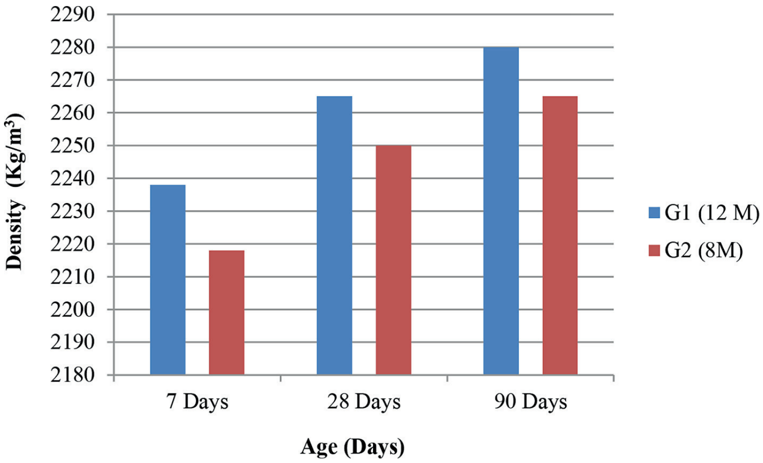

Figure 7. The effect of NaOH molarity on the density of G1and G2 at different ages

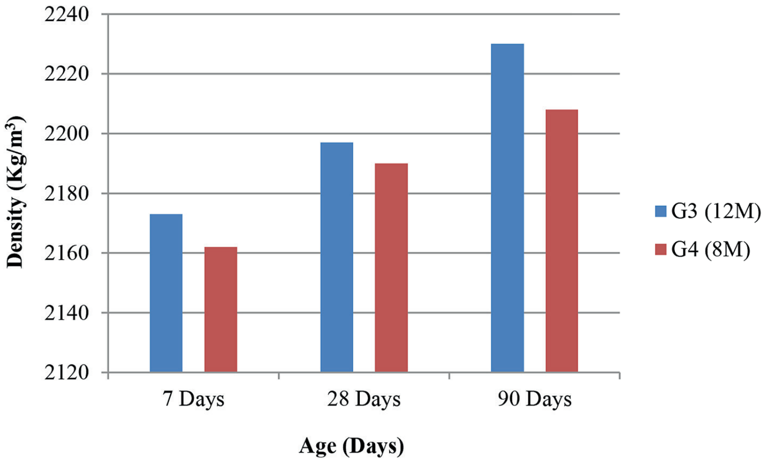

Figure 8. The effect of NaOH molarity on the density of and G4 at different ages

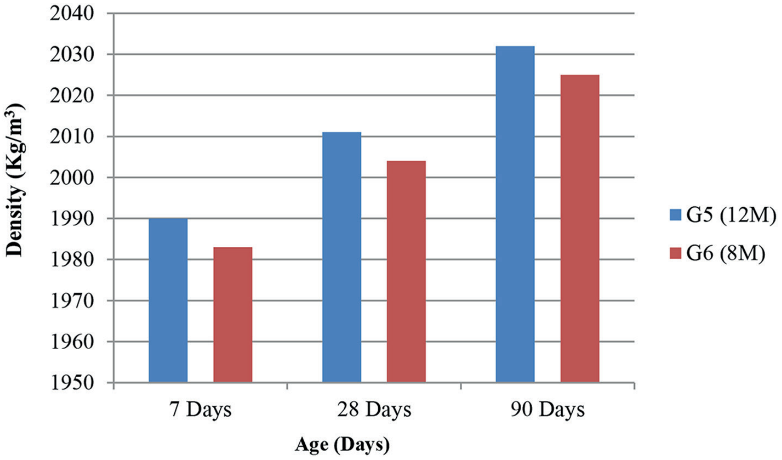

Figure 9. The effect of NaOH molarity on the density of G5 and G6 at different ages

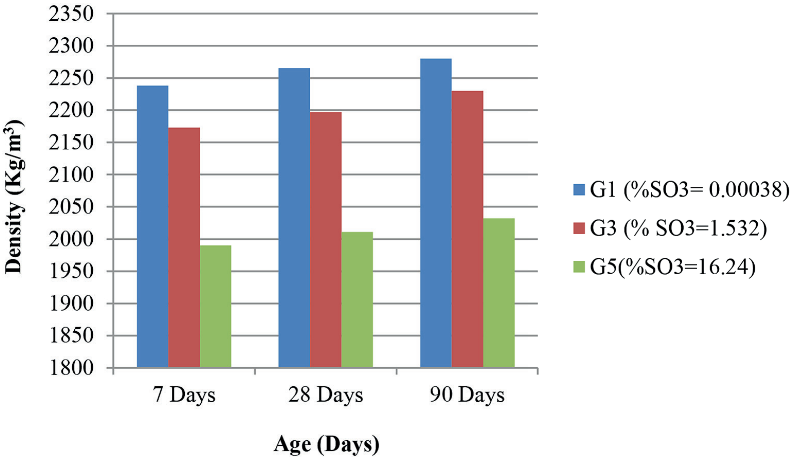

Figure 10. The effect of %SO$_3$ on the density of G1, G3 and G5

producing dissolution of siloxane linkages (–Si–O–Si– links), which reduced the silica to aluminium (Si/Al) atomic ratio in the samples and silica (Si) leaching in the geopolymer gel structure [20]. This caused GPM specimens to lose more density.

The increase in results of geopolymer mortar constructed with high density and pores filled with the binding material matrix increases the density with gepolymerisation products, which increases the strength of mortar with age.

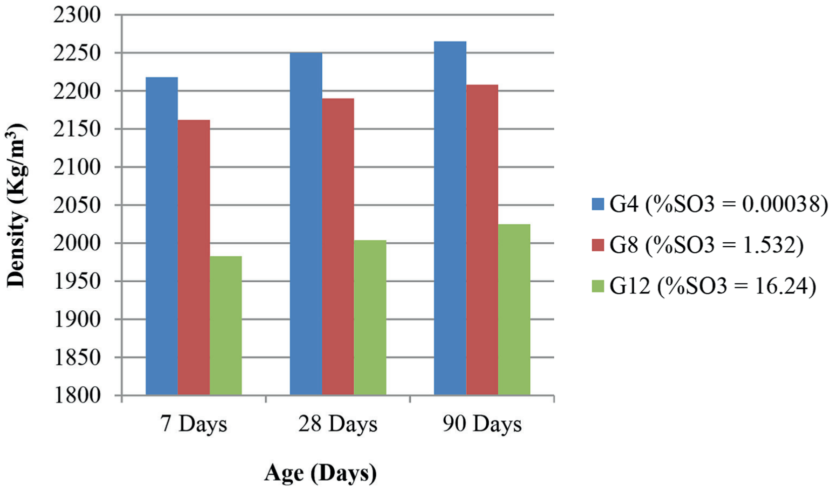

Figure 11. The effect of %SO_3 on the density of G2, G4 and G6

CONCLUSIONS

According to the findings of the current research on the MK-based geopolymer mortar, the following are the principal conclusions. Geopolymer is an environmentally preferable alternative to OPC for structural applications. MK-based geopolymer mortar is superior to ordinary cement mortar due to its eco-friendly components and enhanced properties. Water absorption is affected by NaOH concentration, where higher molarity (12M) leads to reduced water absorption due to fewer voids content. It can be noted that the higher absorption values coupled with increasing SO_3, were the greatest values in mixes with SO_3 was 16.24%, because higher sulfate content made the pores become larger and weakened the transition zone of the geopolymer mortar. The density value of geo-polymer mortar increased when the molarity increased from 8M to 12M because of the increase in the amount of solute in a fixed volume. The results show that the mixes exhibit decrease in density with the increase in SO_3 content in mortar mixes at all ages of the test; this can be attributed to the increasing the dispersal of SO_3 ions geopolymer structure caused disintegration and density loss. In advance time, the water absorption of all geopolymer mortars decreased, and in contrast, the density increased, where the lowest result of absorption and higher value density was 3.89% and 2280 kg/m^3, respectively, at the age of 90 days, this can be attributed to rising in pozzolanic reaction and developed the degree of geopolymerization.

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