

Original Research

CURING CONDITIONS IMPACT THE PROPERTIES OF A MODIFIED GEOPOLYMER MORTAR

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Abstract. The present work provides an experimental study for preparing eco-friendly geopolymer mortar based on ground granulated blasted furnace slags with good mechanical properties and durability. This work studied the effect of silica fume reactive powder as a replacement material for slag weight with constant weight ratios of 20% on geopolymer mortar's mechanical tests. The geopolymer mixtures were prepared with (15M) NaOH solution and a (2.5:1) ratio of (Na₂SiO₃/NaOH). The samples were cured at ambient conditions at different times of 7 and 28 days and a high temperature of 70°C in a furnace for three hours. It was found from the obtained results that silica fume played a vital role in improving all these properties by enhancing the degree of the silicate ion geopolymerization reaction in the alkaline solution. In addition, the curing process greatly impacted the geopolymers' features. The properties improve significantly with increasing the curing time and temperature, especially when cured at high temperatures, compared to ambient curing.

Keywords: *Geopolymer; mortar; compressive strength; silica fume*

1. Introduction

Cement production was estimated to reach about 4.2 billion tons in 2013 and is expected to increase to some 4 billion tons annually in 2050 [1, 2]. The cement industry emits a high amount of carbon dioxide (CO₂) into the atmosphere. Each ton of produced cement results in the release of approximately one ton of CO₂ gas [3].

CO₂ can be defined as a greenhouse gas that is primarily responsible for ongoing global warming. CO₂ gas is found to be responsible for (65%) of global environmental warming.

Moreover, the cement industry unintentionally contributes to a portion of global temperature change [4, 5]. Many efforts were made to limit the spread of global warming by developing new environmentally-friendly materials. One such attempt for producing mortar has been fractional replacement regarding the cement with supplementary materials like granulated blast furnace slag, fly ash, silica fume, metakaolin, and rice husk ash. Other projects include finding an alternative to cement as a binder [6, 7].

Geopolymer binder, an alternative to cement binder in the concrete industry, might effectively reduce CO₂ emissions to the atmosphere by up to 80% in terms of global warming [3, 8]. However, various studies indicated that the geopolymer mortar has superior mechanical characteristics, with shear and flexural strengths comparable to the Portland cement mortar [9]. In addition, the cost savings in the geopolymer's production might be achieved. Geopolymer binder is generally made by reacting aluminosilicate

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material with an alkaline solution [10, 11]. Joseph Davidovits claims the geopolymerization mechanism may be too similar to zeolite formation. He also mentioned that there are three stages to the process: dissolution, reorientation, and solidification. The stage of dissolution starts in the case when Si-Al from raw materials comes into contact with an alkaline solution, it forms Al and Si species, and after dissolution, Al and Si species diffuse into the oligomers during the reorientation stage. Condensation of oligomers in the aqueous phase results in creating fairly large networks leading to the formation of the gel. The solidification stage involves a reorganization process and rearranging of a continuous gel [3].

Many researchers have presented on the geopolymer mortar. Gorhan and Kurklu [12] examined the modifications generated by the geopolymerization on the characteristics of geopolymer mortars in varied NaOH alkaline solutions at different temperatures and during various curing durations. The molarity of solution impact on geopolymer concrete was investigated by Madheswaran et al. [13]. Three-, five-, and seven-molar sodium hydroxide solutions were used to make the various mixes cured at room temperature. The geopolymer samples were tested for compressive strength after seven and twenty-eight days.

Geopolymer mortar's strength was examined by [14]. The optimum mechanical qualities of the tensile compressive, and flexural strength were 12 molars at 28 days. In these measurements, the compressive strength was attained 1.25 times greater, the splitting tensile 1.18 times more, and the flexural strength 1.058 times more than other samples by thermal curing.

The impact of molarity on the compressive strengths of a geopolymer's fly ash mortar was examined by Budh and Warhade (2014) [15].

They determined that the compressive strength values have been enhanced by raising the NaOH content in the alkaline solution.

Chen et al. [16] studied geopolymer concrete using fly ash type C and substituting the fly ash with GGBS to investigate the influence of the slag to fly ash ratio on the structural characteristics of concrete. The findings demonstrated that increasing the amount of slag leads to higher compressive strength after 7 days.

Aliabdo et al. [17] investigated the influence of alkaline solutions' rest time on the compressive strength and workability of geopolymer concrete. The intermittent curing of fly ash-base geopolymers was examined by Ibrahim [18].

Zhang et al. [19] modified the ratio of Si-Al and the ratio of Na/Al on the phases composition, and mechanical characteristics of metakaolin-based geopolymers.

Zhang et al. [20] examined geopolymer mortars' physical, mechanical, durability, and microstructure characteristics.

They found that the geopolymer mortar had shown significant feasibility and the promised application as an environmentally friendly building material. It may eventually replace the regular cement mortar in many suitable applications.

Tippayasam et al. [21] developed a geopolymer mortar by combining metakaolin with several wastes, like fly ash, bagasse ash, and rice husk ash.

2. Experimental

2.1. Materials

2.1.1. Ground Granulate Blasts Furnaces Slag (GGBS)

GGBS is a by-product of the steel-making industry. It is often utilized in mortar and concrete production as a supplementary cementitious material (SCM) to partly replace conventional Portland cement (OPC).

Table 1 and Table 2 demonstrate the physical properties and chemical analysis of the slag used.

2.1.2. Silica Fume (SF)

Silica fumes (SF), a micro silica type, are made when high-purity quartz and coal are broken down in electric furnaces to make silicon and ferrosilicon alloys.

In this work, the silica fume was brought from Iraq Sika Company for the Building Materials as shown in Table 3.

Table 1. GGBS chemical analysis.

Oxide	Content (%)
SiO ₂	29.01
Al ₂ O ₃	14.99
Fe ₂ O ₃	0.53
SO ₃	1.13
MgO	8.60
CaO	39.23
LSF	0.39

Table 2. Physical properties of GGBS

Physical characteristics	Results
Physical Form	Powder
Surface Area, m ² /kg	5338
Specific Gravity	3.2
Color	Light Grey

Table 3. Silica fume specifications.

Parameters	Standard Value
Bulk Density	350-700 Kg/m ³
Percent Retained on 45 μm (325 sieves)	2.0% max
Loss of Ignition (LOI)	3% max
Moisture Content (H ₂ O)	1.0% max
Silicon Dioxide (SiO ₂)	96% min

2.1.3. Sodium Hydroxide (NaOH)

NaOH, about 98% purities, in the flake's forms are widely offered. The solids must be dissolved in distilled water to make solutions with the needed concentrations.

2.1.4. Sodium Silicate (Na₂SiO₃)

The concentration of Na₂SiO₃ depended on the ratio of H₂O, SiO₂, and Na₂O. Also, the sodium silicate in this work was made in the UAE.

2.1.5. Water

According to the Iraqi standards IQS No. 1703, distilled water was used to dissolve the caustic soda flakes, and provides a medium to facilitate the geopolymerization reaction.

2.1.6. Fine Aggregate (Sand)

This study used naturally fine aggregates with the largest size of 4.75mm from the Al-Ekhadir area and the Karbala Governorate. It required cleaning before use. Standard sand was used in this work depending on IQS (No45/1984) [22]. Table 4. shows the grading of fine aggregate.

Table 4. Grading of fine aggregate

Sieve size	Passing percent %
1.18 mm	100
0.600 mm	98.6
0.425 mm	74.1
0.300 mm	24.3
0.150 mm	0.12

2.1.7. Highly-Ranged Water Reduced Admixture

A high-range water reducer (type Master Glenium®54 super-plasticizer (SP)) was applied to improve the workability of the geopolymer mortar. It meets the ASTM C494 Type F specification [23].

2.2. Distinguishing of Mixtures

Two mix designs of the geopolymer mortar were used in this study. It was divided into two groups. Group I (Gcont.) represents the reference mixture of the geopolymer mortar. Group II (GD) represents the geopolymer mortar mixture with (20% wt.) of silica fume as a replacement material for slag weight.

Each mixture has been chosen after many trials to obtain the required properties. The alkaline liquid was prepared the day before it was to be used, and the raw materials (slag, fine aggregate, and the silica fume) were mixed manually for approximately (5-10) min, after which mixed alkaline liquid with the super-plasticizer was added to the dry ingredients and mixed again for (5) min.

To ensure the homogeneity of the mixture, the total time of the mixing process was approximately (10-15 min), and then geopolymer mortar was poured into steel molds according to the geometry and dimensions required for each test.

Before pouring it is covered via an extremely thin oil layer for preventing any potential adhesions between the mixture and the internal surfaces of the mold.

The specimens of group II replaced weight ratios (20%) of the weight of the slag with silica fume to study the influence of reactive powder on the microstructure, and physical and mechanical properties of geopolymer mortar.

The curing method in this work was done in two ways. The first was done at ambient temperature for (7 and 28) days. The other way was done by placing the samples in the furnace at an elevated temperature (70°C) for (3) hours, and then the samples were placed under direct sunlight.

Mix proportions are shown in Tables 5 and 6.

Table 5. Mix proportions for cubic (5*5*5) geopolymer mortar.

Group	Slag (g)	Sand (g)	NaOH	Na ₂ SiO ₃	Silica fume (wt%)
Gcont	300	600	47.1	117.9	-
GD	240	600	47.1	117.9	20%

Table 6. Mix proportions for the prism (4*4*16) geopolymer mortar.

Group	Slag (g)	Sand (g)	NaOH	Na ₂ SiO ₃	Silica fume (wt%)
Gcont	572	1144	89.8	224.7	-
GD	457	1144	89.8	224.7	20%

3. Methods

3.1. Compressive Strength Test

The compressive strength test is determined according to ASTM: [C109-2 [24]. the values of compressive strength of every mix have been determined using an average value of three

50x50x50 mm cubic specimens. The samples were examined at the ages of 7 and 28 days and after furnace curing.

3.2. Flexural Strength Test

Flexural strength has been done according to ASTM C 293-03 [25]. An average of 3 prism specimens (160mm x 40mm x 40mm) were tested at 7 and 28-day ages and after the curing in the furnace to determine the flexural strength of each mixture according to the following equation.

$$R = \frac{3PL}{2bd^2} \quad (1)$$

Where:

- R: denotes the flexural quality (MPa),
- L: denotes the length of span (mm),
- P: signifies max. loads (N),
- d: denotes the average thickness (mm), and
- b: signifies the width of the specimens (mm).

3.3. Characterization Techniques

3.3.1. Scanning Electron Microscope

To investigate the microstructure of the samples and the effect of silica fume on the microstructure of prepared geopolymer mortar, SEM technique was used. High-resolution images with depth of field have been obtained at the fracture surface's mid-width using this approach.

4. Results and Discussion

4.1. Compressive Strength

The experimental results of this work, shown in Table 7, show that the compressive strength is significantly influenced by the curing conditions and silica fume concentration. The tests were carried out in three sample sets at different curing

times (7, 28 days), and at high temperatures (70°C).

It was observed that the compressive strength readings rise with the curing age. These values significantly rise when cured at high temperatures compared to ambient curing due to the formation of dense microstructure. The compressive strengths of geopolymers mortar improve with a rise in the proportion of GGBS substitution with silica fumes in the same context.

Introducing the silica fume powder causes more exothermic reactions with CaO in the GGBS resulting in increased compressive strength values. This reaction will improve the geopolymerization process by producing more N-(C)-A-S-H and C-S-H gelling, thus raising the strength of the hardened mixtures.

The excessive gel formation due to the silica fume will enhance the bonding between the constituents of geopolymer mortar, causing more compressive strength. In addition, the high alkaline solution molarity activated the precursors to generate more dissolved ion species, thus enhancing the geopolymer pastes geopolymerization.

Similarly, the compressive strength is closely related to the density of the material, as increasing the density raises the compressive strength of the material. Therefore, the compressive strengths increase due to silica fume's effect because it works to close the gaps and pores in the structure.

Finally, the compressive strength value when replaced (20%) of GGBS by silica fume is higher than GGBS-based geopolymer mortar in all mixtures. This has resulted from the fact that (SiO₂/Al₂O₃) ratio is constantly higher in this case since the increase in the alumina in the

source material causes the compressive strength values to decline.

Table 7. Results of Compressive Strength Test Mixtures (MPa).

Group	7-day	28-day	70°C
Gcont	43.2	50.1	56.28
GD	51.2	58.3	63.2

4.2. Flexural Strength

The flexural strength test was utilized to assess a material's capacity to withstand the deformations under load. The obtained data exhibited that the flexural strength rose with the curing time and temperature because of the geopolymer paste's exceptional adhesion to the fine aggregates. Also, it was noticed that the presence of silica fume leads to a slight increase in the flexural strength value occurring at 20%wt. The slight increase in flexural resistance at high levels of silica fume is due to the increase in the fragility and the hardness of geopolymer mortar.

Table 8. Flexural Strength (MPa) Results.

Group	7-day	28-day	70 C
Gcont	4.73	5.61	6.1
GD	5.03	5.69	6.55

4.3. SEM Characterization

SEM characterization was used to examine the microstructure of geopolymer samples and the influence of the silica fume on the geopolymer mortar microstructure. This approach offered high-resolution images with field depth at the fracture surface's mid-width.

Fig. 1 and Fig. 2 show the geopolymer mortar SEM images at 7 and 28-day curing time respectively. From the obtained images it was

observed that the microstructure of geopolymer mortar at 28-day curing was less pore and more regular than at 7-day curing.

This fact is attributed to the amount of gel formation at 28-day curing because it needs more time to form at the end of the geopolymerization process.

The influence of silica fume addition on the microstructure of the prepared geopolymer mortar was shown in Fig. 3. It was noticed that using silica fume plays an important role in modifying the microstructure by filling the pores within the structure producing dense and homogenous microstructure. Similarly, adding silica fume enhances the process of geopolymerization by improving gel phase formation.

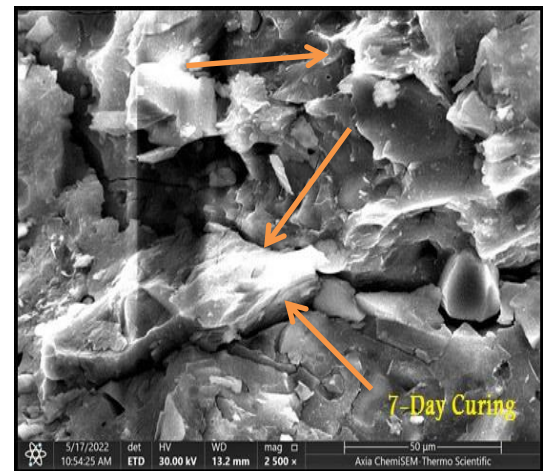


Figure 1. SEM images of the geopolymer mortar after 7-day curing.

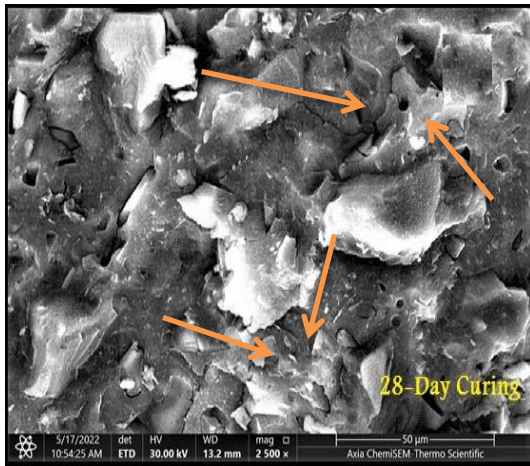


Figure 2. SEM images of geopolymer mortar after 28-day curing.

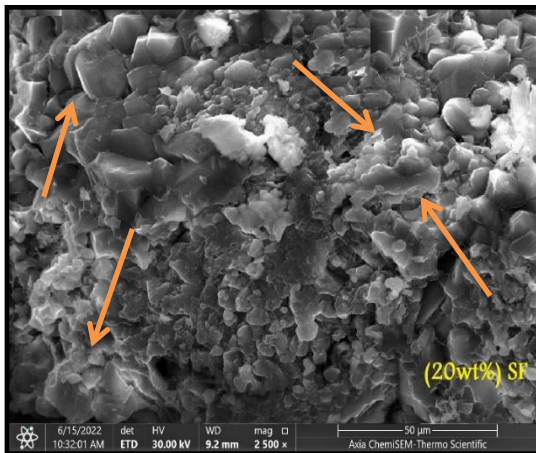


Figure 3. SEM images of geopolymer mortar containing 20% by weight of silica fume.

5. Conclusions

The obtained results show that using locally available waste materials such as GGBS as a geopolymer mortar source could be used to develop ecologically friendly, sustainable construction materials. In addition, the results show that using silica fume as a replacement material for slag's weight will accelerate the geopolymerization process of geopolymer mortar. It enhances the degree of silicate ion geopolymerization reaction in the alkaline solution. It also forms more calcium silicate hydrate N-(C)-A-S-H and C-S-H gels which enhances the geopolymer mortar's compressive strength.

The presence of silica fume improves the flexural resistance to some extent values because of the increase in fragility and the hardness of geopolymer mortar.

The impact of silica fume on the microstructure of the produced geopolymer mortar was also investigated. It was discovered that the silica fume addition was significantly affected on the microstructure.

It fills the pores and produces a fine and more homogenous microstructure. It also enhances the geopolymerization process by improving the gel phase formation.

The curing process is considered an essential factor in improving the properties of the geopolymer mortar.

The compressive strength of the geopolymer mortar was remarkably increased with the curing age where these values increased significantly at 28-day curing than 7-day curing. The properties improve significantly especially when the samples are cured at high temperatures compared to ambient curing.

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Author Contribution Statement

Author Hamza M. Kamal proposed the research problem. Author Rusul Jabbar did the experimental procedure and tests. Author Hamza M. Kamal interpreted and analyzed the obtained results.

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