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Effect of Sulfate Attack on Fly Ash / Slag Geopolymer Mortar

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Abstract: This paper aims to investigate the behavior of fly ash/slag geopolymer mortar (FA/S-GM) exposed to sulfate attack. Four FA/S-GM mixtures with different slag replacement levels (0 - 20% by mass of FA) were prepared. Four more ordinary Portland cement/slag mortar (OPC/S-M) mixtures with the same slag replacement levels were prepared as control specimens. All mortar specimens were immersed in 5% sodium sulfate solution for 52 weeks. Compressive strength, Expansion, weight gain, x-ray diffraction (XRD), and scan electron microscopy (SEM) approaches were conducted throughout this research. The mechanical and physical properties of FA/S-GM were superior to those of OPC/S-M, revealing excellent sulfate resistance. After 52 weeks of sulfate exposure, the compressive strength of FA/S-GM increased while the compressive strength of OPC/S-M decreased when compared to their pre-sulfate strength. FA/S-GM and OPC/S-M had optimal slag contents of 20% and 10%, respectively. XRD and SEM analyses revealed no ettringite or gypsum in FA/S-GM.

Keywords: Geopolymer; Expansion; Compressive Strength; Slag; Flay Ash.

1 Introduction

The cement industry is considered a process that consumes raw materials and energy because it requires large amounts of limestone, clay, and fuel. The annual global cement production of 1.6 billion tones accounts for approximately 5-8% of the amount of carbon dioxide released into the atmosphere [1-2]. The increasing consumption of natural resources has become a major environmental challenge confronting today's researchers, prompting concrete technologists to search for substitutes to the widely used Portland cement. Undoubtedly, the inability to recycle agricultural waste and industrial by-products has become a major environmental concern. Many researchers have used cement replacement materials in concrete such as silica fume, fly ash, rice husk ash, and slag among others as an attempts to address the above mentioned challenges [3-8]. However, total cement replacement is a more sustainable option.

Geopolymer technology has the potential to completely replace cement in concrete. Geopolymer concrete is produced by activating reactive silica and alumina-rich source materials in an alkaline solution commonly composed of sodium hydroxide and sodium silicate mixture [9]. Numerous studies conducted on geoplymers have proven that they are a promising generation of materials for use in the concrete industry [9-12]. Most common aluminosilicates available are fly ash, slag, rice husk ash, and metakaolin. However, simply categorizing published research on geopolymer composites reveals that the majority of efforts are directed towards using fly ash and slag as precursors [13].

Repairing and strengthening concrete structures after they deteriorate is a real challenge due to insufficient funds and lack of appropriate repair strategies. As a result, investigating the various deterioration mechanisms of concrete is critical. Sulfate attack is a major concern for the durability of concrete. In ordinary Portland cement (OPC) concrete, sulfates react with cement matrix including calcium hydroxide and calcium silicate hydrate forming aggressive products such as ettringite and gypsum, which cause directly or indirectly structural damage [14].

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The degradation process of geopolymer concrete exposed to sulfate environment is still not fully understood and needs further investigation in the future. Some authors observed that geopolymers performed better than conventional concrete, and they attributed this to factors such as: lower amounts of calcium oxides in the structure of geopolymers [15-18], further geopolymerization in the presence of sulfate solution [19, 20], formation of new crystals in exposed samples' structures [21], and the cross-linked structure of the alumino-silicate gel. Other authors, however, reported an adverse effect on the mechanical behavior of geopolymer samples when exposed to sulfates. [22-23].

Based on the foregoing, further research into the degradation process of both FA/S-GM and OPC/S-M when exposed to 5% sulfate solution is required. An extensive experimental program was established and conducted to accomplish the following specific goals:

1. To assess the physical properties (length change and weight change) of FA/S-GM and OPC/S-M when exposed to 5% sulfate solution.
2. To investigate the compressive strength development of FA/S-GM and OPC/S-M immersed in 5% sulfate solution.
3. To determine the optimal slag content in FA/S-GM and OPC/S-M.

2. EXPERIMENTAL

2. 1. Materials

Ordinary Portland cement (CEM I, 52.5 grade) with a surface area of 3,700 cm^2/g and complying with ES: 4756-1 [24] was used. Slag and class F fly ash were used according to ASTM C989-18a and ASTM C618-19, respectively [25-26]. Table 1 presents the chemical composition of OPC, slag, and fly ash. Particle size distribution of fly ash and slag is presented in Fig. 1. X-ray diffraction (XRD) spectra of fly ash and slag is illustrated in Fig. 2. Sand of a specific gravity of 2.65 and a fineness modulus of 2.75 and complying with ASTM C33 [27] was used. The grading of sand is depicted in Fig. 3. Sodium silicate (specific gravity = 1.49, Na_2O = 15.6% and SiO_2 = 29.6 %) and sodium hydroxide of 99% purity were used to prepare the alkaline activated solution. Sodium sulfate (Na_2SO_4) of 99.9% purity was used as a source of sulfate ions. To avoid errors when preparing the various aqueous solutions, sodium sulfate was stored in dry air before use.

Table 1 Chemical composition of OPC, fly ash, and slag.

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
OPC	21.7	4.53	4.61	61.6	2.38	0.36	0.44	1.8
Fly ash	58.1	26.7	6.25	3.55	0.51	0.38	1.10	0.41
Slag	37.1	9.51	0.54	43.2	6.89	0.52	0.53	0.1

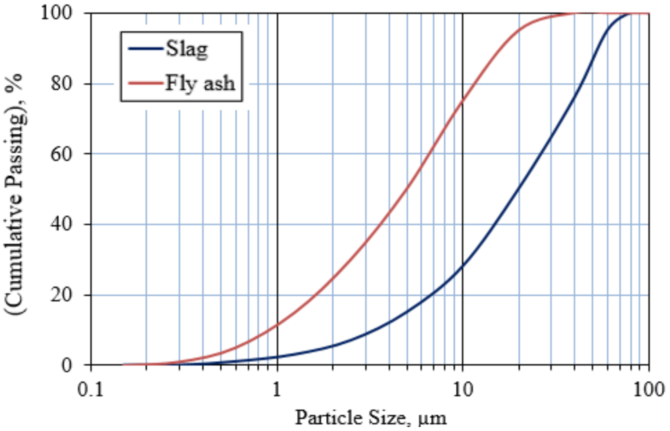

Fig. 1 Particle size distribution of fly ash and slag.

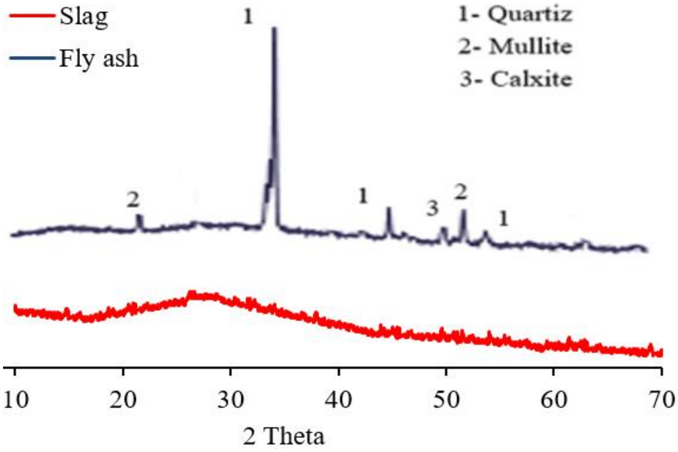

Fig. 2 XRD patterns of fly ash and slag.

2. 2. Mortar mix proportion

Four FA/S-GM mixtures with four slag replacement levels, 0 %, 10 %, 15 %, and 20% by mass of FA were prepared. Four more ordinary Portland cement/slag mortar (OPC/S-M) mixtures with the same slag replacement levels were prepared as control specimens. For FA/S-GM mixtures, a constant alkaline activator to total binder ratio of 0.4 was used. For OPC/S-M mixtures, a constant water to binder ratio of 0.4 was used. A constant binder to sand ratio of 0.5 was considered in all mixtures. The proportions for FA/S-GM and OPC/S-M mixtures are shown in Table 2.

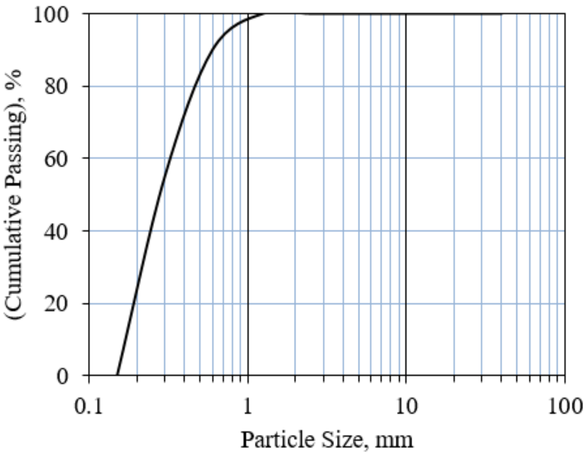

Fig. 3 Particle size distribution of sand.

Table 2: Mix proportions for OPC/S-M and FA/S-GM

Mix No.	Mix Code	OPC (%)	FA (%)	S (%)	Note
1	100% OPC/0% S	100	0	0	OPC /S-M
2	90% OPC/10% S	90	0	10	
3	85% OPC/15% S	85	0	15	
4	80% OPC/20% S	80	0	20	
5	100% FA/0% S	0	100	0	FA/S -GM
6	90% FA/10% S	0	90	10	
7	85% FA/15% S	0	85	15	
8	80% FA/20% S	0	80	20	

2. 3. Preparation of alkaline activators

Sodium hydroxide was first mixed with a specific amount of water to produce a sodium hydroxide solution with the desired molarity (12 M), then allowed to cool for 24 hours at room temperature before casting. Sodium silicate solution and sodium hydroxide solution were mixed two hours before mixing with the other mortar ingredients [28].

2. 4. Mixing, casting, and curing

The FA/S-GM mixtures were manually mixed until homogenous mixtures were achieved. Before adding the activator solution, sand was thoroughly mixed with the binders (fly ash and slag). The prepared activator solution was then gradually added and mixed for an additional 5 minutes until a consistent mixture was achieved. On the other hand, sand, slag, OPC, and water were mixed together in the same laboratory pan to make OPC/S-M.

Three cubical specimens of 50x50x50 mm from each mortar mix were prepared for compressive strength test. Prismatic specimens of 25 × 25 × 285 mm from each mortar mix were manufactured for both expansion and weight gain measurements. All FA/S-GM specimens were de-moulded three days after casting to ensure proper polymerization [29], and then cured at room temperature in ambient conditions (23 ± 5 °C; 60% ± 5% RH) for 28 days. All OPC/S-M specimens were removed from the mould after 24 hours of casting and then water cured in a controlled temperature of 23 ± 5 °C for 28 days.

2. 5. Test techniques and procedures

The experimental program was designed to investigate the sulfate induced degradation of FA/S-GM and OPC/S-M. The sulfate resistance of mortars was studied by immersing of the specimens in 5% Na_2SO_4 solution for up to 52 weeks. Throughout the exposure period, the Na_2SO_4 solution was renewed monthly. The expansion of mortar was regularly measured every two weeks in accordance with ASTM C157 [30]. The weight gain was measured for all samples used for expansion measurements. The compressive strength of mortars after exposure periods of 0, 8, 16, 26, and 52 weeks, was determined according to ASTM C109 / C109M [31]. The microstructure of mortar specimens was examined using a benchtop SEM (Gemini SEM 300, Zeiss, Germany).

3. RESULTS AND DISCUSSION

3.1. Compressive Strength

Figs. 4 and 5 present the compressive strength development of FA/S-GM and OPC/S-M exposed to 5% Na_2SO_4 solution, respectively. As shown in Fig.4, the compressive strength of all FA/S-GM specimens immersed in 5% Na_2SO_4 solution increased with time when compared to their pre-sulfate strengths. During the first two months of immersion, strength increased at a faster rate than after. At 2 months of exposure, the compressive strength of 100%FA/0%S, 90%FA/10%S, 85%FA/15%S, and 80%FA/20%S specimens increased by 16, 19, 21, and 18 %, respectively, compared to their pre-sulfate strengths. The increase in compressive strength indicates that FA/S-GM performed well when exposed to sodium sulfate. The presence of neutralized cross-linked aluminosilicate structure, the continued geopolymerization by the Na+ from Na_2SO_4 solution, and the low calcium content of the precursors could all contribute to the geopolymer's good performance in the presence of an aggressive sulfate environment [15-20]. On the other hand, the observed slowness of strength increase after two months could be attributed to alkaline ion migration from the geopolymer to the solution, which changes the geopolymer's structure and adversely affects the mechanical behavior [21-22]. Furthermore, Fig. 4 demonstrates that increasing slag content significantly improves the compressive strength of FA/S-GM. The optimal slag content in FA/S-GM exposed to 5% Na_2SO_4 solution is 20% by mass of fly ash.

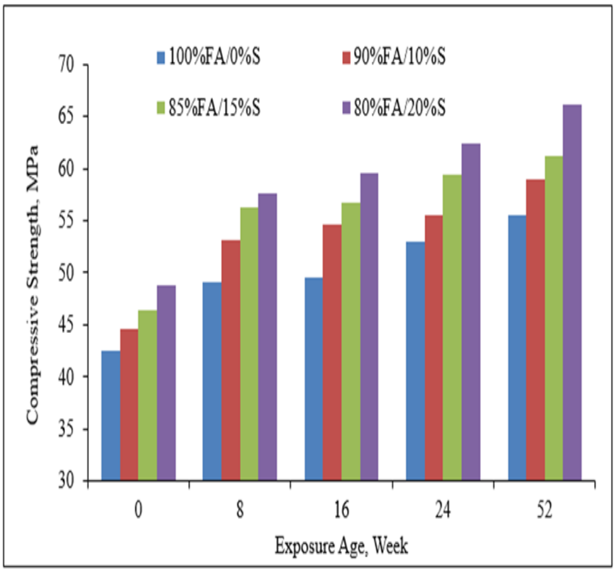

Fig. 4. Strength development of FA/S-GM exposed to 5% Na_2SO_4.

The effect of sulfate on OPC/S-M followed a different pattern than that observed in FA/S-GM. As illustrated in Fig. 5, the compressive strength of all OPC/S-M specimens exposed to 5% Na_2SO_4 solution increased up to 24 weeks and then decreased. The increase in strength of OPC/S-M stored in 5% Na_2SO_4 solution may be attributed to the production of ettringite which contribute to the pore size reduction [32], but after 24 weeks, the volume of such crystals increases and create internal tensile stresses, resulting in a decrease in compressive strength [33]. The compressive strength of OPC/10% slag samples continued higher than that of pure OPC samples up to 52 weeks. The compressive strength of OPC/15% slag and OPC/20% slag mortars was lower than that of pure OPC mortar for the first 16 weeks, then increased but stayed lower than that of OPC/10% slag mortar. The optimal slag content in OPC/S-M exposed to 5% Na_2SO_4 solution is 10% by mass of OPC.

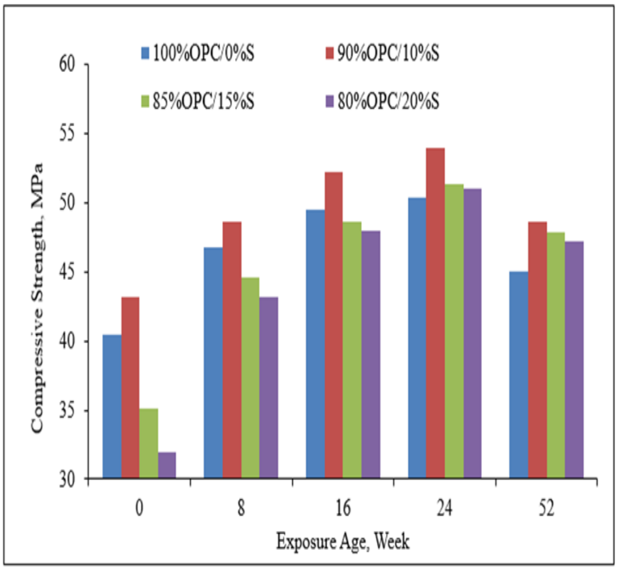

Fig. 5. Strength development of OPC/S-M exposed to 5% Na_2SO_4.

3.2. Weight Change

Figs. 6 and 7 illustrate the weight change measurements of OPC/S-M and FA/S-GM exposed to 5% Na_2SO_4 solution, respectively. From Fig.6, it can be observed that all OPC/S-M specimens immersed in 5% Na_2SO_4 solution achieved weight gain when compared to their pre-sulfate weights. The weight gain of all OPC/S-M specimens increases with increasing the exposure period especially for pure OPC specimens. The rate of weight gain was faster during the first month of immersion than after this period. The increase in weight gain of all OPC/S-M specimens could be due to the production of ettringite and gypsum as a result of chemical reactions between OPC hydration products and sulfate ions [14]. Compared to pure OPC specimens, replacing 10% OPC with slag showed a significant reduction in weight gain. The weight gain of 0 %, 10 %, 15 %, and 20% slag specimens after a month of exposure was approximately 2, 1.8, 2.2, and 2.4%, respectively. The decrease in the weight gain of 10% slag specimens could be attributed to the pozzolanic reaction of slag with the calcium hydroxide (CH) produced from cement hydration, which reduces the CH available to react with sulfate ions. The weight gain of 15% slag and 20% slag samples continued to be higher than that of pure OPC samples for 24 and 28 weeks, respectively, and then decreased but stayed higher than that of 10% slag specimen. These results are consistent with the compressive strength findings, which indicated that the optimal slag content in OPC/S-M exposed to sulfates is 10% by mass of OPC.

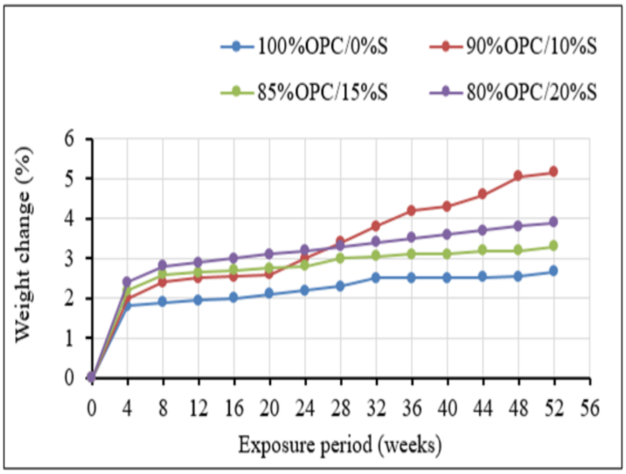

Fig. 6. Weight change of OPC/S-M exposed to 5% Na_2SO_4.

The sulfate impact on the weight gain of FA/S-GM followed a different pattern than that observed in OPC/S-M. From Figs. 6 and 7, it is clear that the weight gain of all OPC/S-M specimens is greater than that of all FA/S-GM specimens. As illustrated in Fig. 7, the weight gain of all FA/S-GM exposed to 5% Na_2SO_4 solution increased with time. This increase may be attributed to the samples' absorption of exposed liquid [34]. The weight gain of FA/S-GM decreases with increasing slag content. The weight gain of 0 %, 10 %, 15 %, and 20% slag specimens was approximately 2.36%, 1.7%, 1.3%, and 0.9%, respectively. The weight gain measurements agree with the compressive strength results which indicated that the optimal slag content in FA/S-GM exposed to 5% Na_2SO_4 solution is 20% by mass of fly ash.

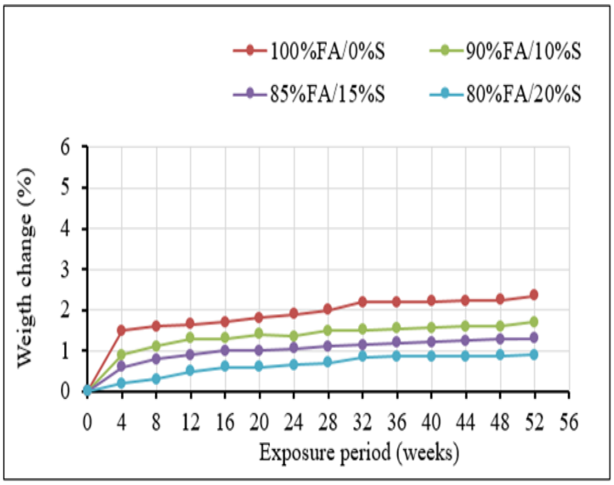

Fig. 7. Weight change of FA/S-GM exposed to 5% Na_2SO_4.

3.3. Length Change

Figs. 8 and 9 illustrate the length change measurements of FA/S-GM and OPC/S-M exposed to 5% Na_2SO_4 solution, respectively. It can be observed from Fig. 8 that all FA/S-GM samples expanded very slowly up to 52 weeks. After a week of immersion, expansion of 0 %, 10 %, 15 %, and 20% slag specimens were 0.015, 0.01, 0.0.007 and 0.006%, respectively. The 52 weeks expansion of 0 %, 10 %, 15 %, and 20% slag specimens were 0.044, 0.033, 0.027 and 0.02%, respectively. It is clear that the expansion decreases with increasing slag content. The 52 weeks expansion of 10 %, 15 % and 20 % slag samples decreased by approximately 25%, 37%, and 55%, respectively compared to pure fly ash geopolymer specimens. Replacing 20% fly ash with slag exhibited the highest decrease (55%) in expansion of fly ash/slag specimens compared to 0% slag specimen. Furthermore, all FA/S-GM specimens expanded significantly less than the ACI allowable limit (0.10% at 1 year and 0.05% at 6 months) [35].

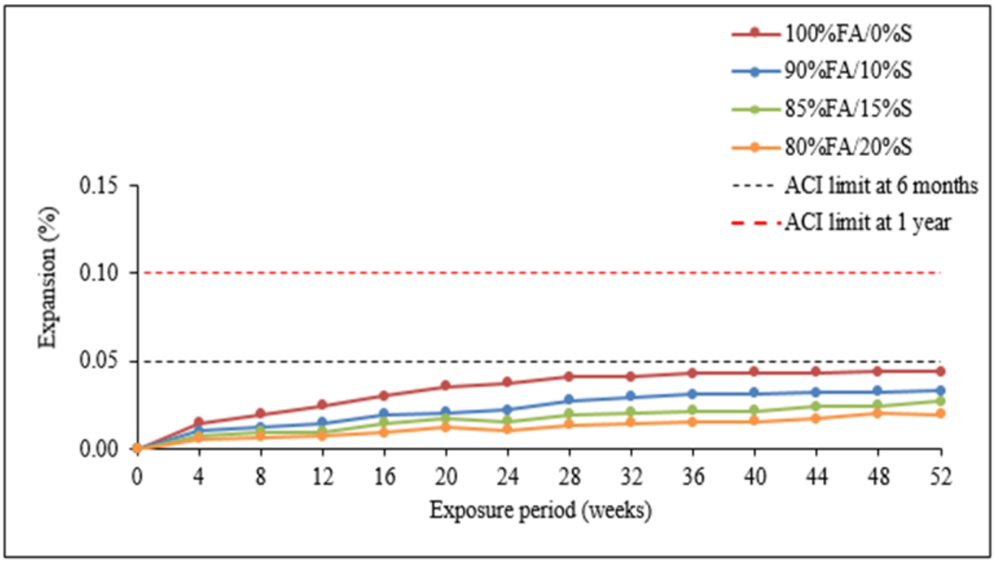

Fig. 8. Expansion FA/S-GM exposed to 5% Na_2SO_4.

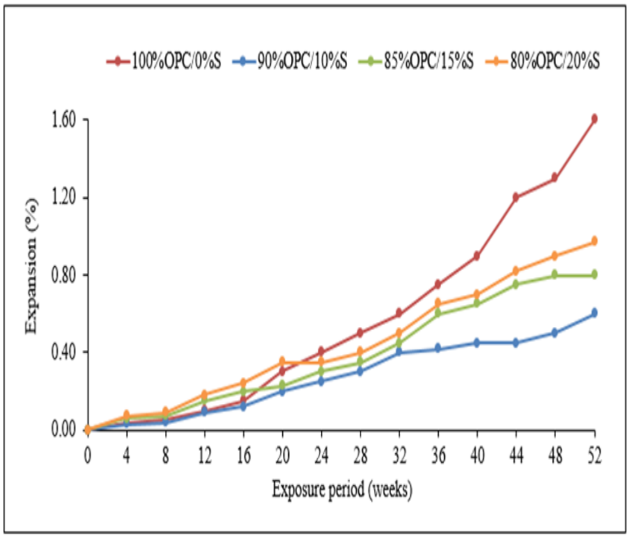

Fig. 9. Expansion OPC/S-M exposed to 5% Na_2SO_4.

The effect of sulfate on the expansion of OPC/S-M followed a different pattern than that observed in FA/S-GM. From Fig. 9, it is clear that expansion of all OPC/S-M specimens is greater than that of all FA/S-GM specimens. From Figs. 8 and 9, it is obvious that all OPC/S-M specimens expand at a faster rate than FA/S-GM specimens, particularly pure OPC specimens. As illustrated in Fig. 9, the expansion all OPC/S-M specimens exposed to 5% Na_2SO_4 solution exceeded the acceptable limits of ACI guidelines (0.05% at 6 months and 0.10% at one year). The 24 weeks expansion of 0 %, 10 %, 15 %, and 20% slag specimens was approximately 0.4 %, 0.25 %, 0.3 %, and 0.35% respectively. The 52 weeks expansion of 0 %, 10 %, 15 %, and 20% slag specimens was approximately 1.6 %, 0.6 %, 0.8 %, and 0.97% respectively. Replacing 10 % OPC with slag produced the smallest expansion compared to other specimens. The expansion of 15% slag and 20% slag samples continued to be greater when compared to pure OPC specimens for 20 and 24 weeks, respectively, and then decreased but stayed higher than that of 10% slag sample. These results are consistent with the compressive strength and weight change findings, which indicated that the optimal slag content in OPC/S-M exposed to sulfates is 10% by mass of OPC.

The expansion and weight gain tests produced high levels of agreement. As a result, an attempt was made to coordinate the results of the two tests, as depicted in Fig. 10. As illustrated in Fig. 10, there is a logical, direct relationship between length and weight change for pure OPC mortar and pure fly ash geopolymer specimens.

3.4 XRD analysis

XRD analysis was performed on OPC/S-M and FA/S-GM exposed to 5% Na_2SO_4 solution. The measured XRD patterns of OPC/S-M and FA/S-GM are shown in Figs. 11 and 12, respectively. XRD patterns of OPC/S-M presented in Fig. 11 revealed the formation of expansive products such as ettringite and gypsum. As shown in Fig. 11, the pure OPC sample had the strongest ettringite and gypsum peaks, while the 10% slag sample had the lowest ettringite and gypsum peaks. Furthermore, the ettringite and gypsum peaks are more distinct in 15% slag and 20% slag samples than in 10% slag samples. These findings are consistent with the weight change and length change measurements of OPC/S-M shown in Figs. 6 and 9, respectively, which indicated that pure OPC samples exhibited the greatest weight gain and expansion, while 10% slag specimens demonstrated superior physical behavior against sulfate. FA/S-GM samples showed a different XRD patterns as shown in Fig. 12. No evidence of ettringite or gypsum in FA/S-GM.

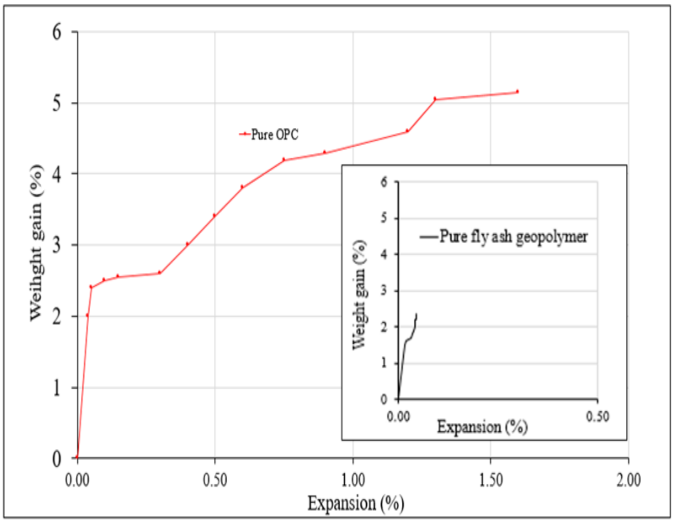

Fig. 10. Relationship between % of expansion and weight gain of pure OPC mortar and pure fly ash geopolymer specimens.

3.5 Scanning electron microscopy

Figs. 13 and 14 present the microscopic images 80% FA / 20% slag geopolymer mortar and 80% OPC / 20% slag mortar, respectively. The SEM specimens was collected from the outer surface of the mortar samples after 52 weeks of immersion in 5% sodium sulfate solution. As can be seen from Figs. 13 and 14, the microstructure of geopolymer mortar is denser than that of conventional OPC/slag mortar. SEM observation of geopolymer mortar specimen revealed no significant signs of interconnected pores, cracking or large crystals after 52 weeks exposure to the sodium sulfate solution. The microscopic image OPC/slag mortar specimen, on the other hand, revealed some pores and few needles of ettringite. It should be noted that the SEM observations are consistent with XRD results.

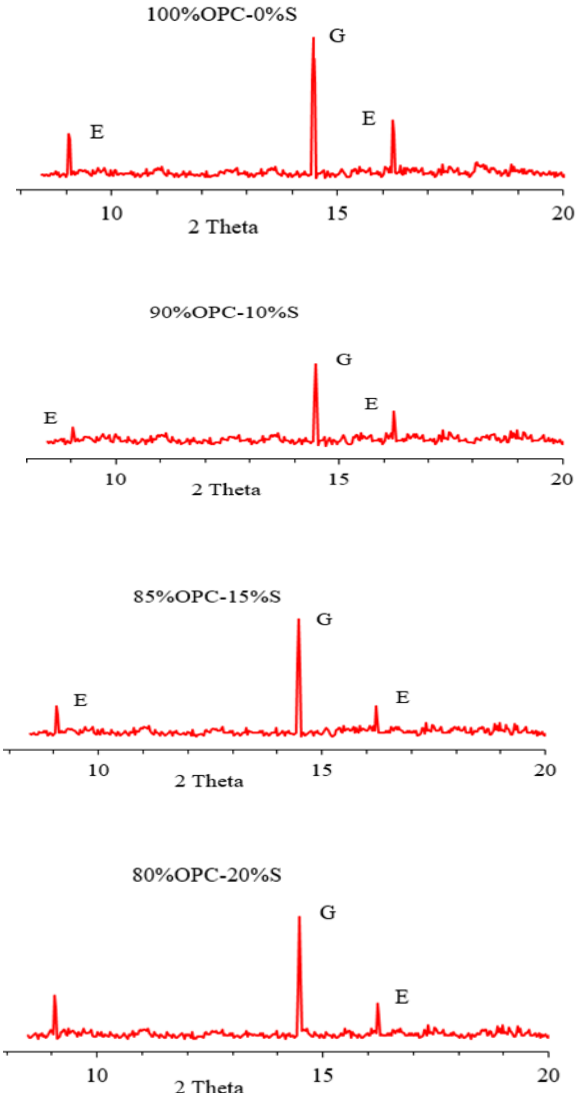

Fig. 11 XRD pattern of OPC/S-M after immersion 5% Na_2SO_4 for 52 weeks. (E: Ettringite and G: Gypsum)

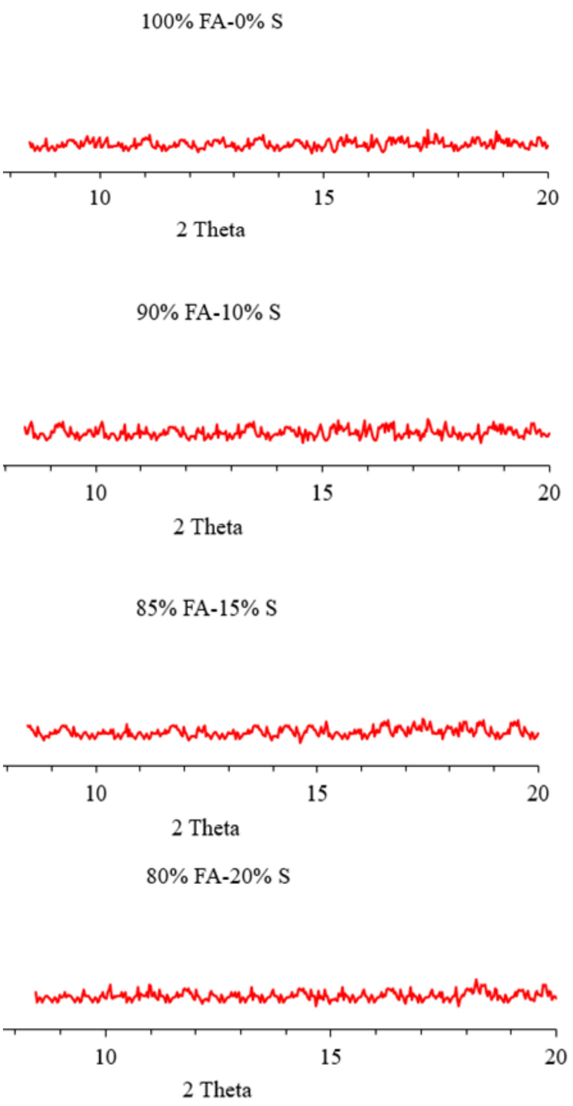

Fig. 12 XRD pattern of FA/S-GM after immersion 5% Na_2SO_4 for 52 weeks.

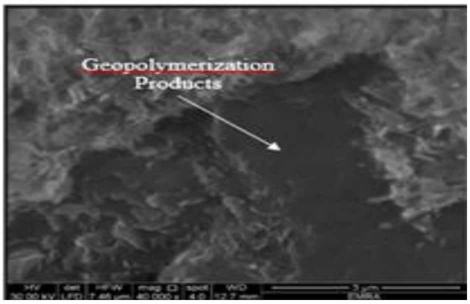

Fig. 13. SEM micrograph of 80% FA / 20% slag geopolymer mortar specimen after 52 weeks exposure to 5% sodium sulfate solution.

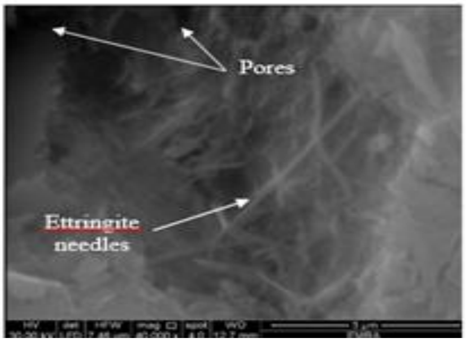

Fig. 14. SEM micrograph of 80% OPC / 20% slag mortar specimen after 52 weeks exposure to 5% sodium sulfate solution.

4. CONCLUSIONS

According to the experimental work carried out in this research, the main conclusions can be summarized as follow:

1. The mechanical and physical properties of FA/S-GM were superior to those of OPC/S-M, revealing excellent sulfate resistance.
2. The compressive strength of FA/S-GM specimens immersed in 5% Na_2SO_4 solution increased with time when compared to their pre-sulfate strengths, whereas the compressive strength of OPC/S-M specimens increased up to 24 weeks and then decreased.
3. In a 5% sulfate environment, OPC/S-M expanded and gained weight more quickly than FA/S-GM.
4. The optimal slag contents to be considered in FA/S-GM and OPC/S-M exposed to sulfate attack are 20% and 10%, respectively.
5. Pure OPC mortar showed the maximum degradation when compared to other mortars.
6. Based on XRD and SEM analysis, there is no evidence of ettringite or gypsum in FA/S-GM.
7. Reasonable correlations were established between expansion and weight gain of mortar.

5. Recommendations

Based on the findings of this research, several recommendations can be made to enhance further understanding and applications of fly ash based geopolymers exposed to severe conditions. These recommendations are as follows:

1. Future studies should investigate the effects of other deteriorating factors such as chloride attack, carbonation attack, freeze-thaw cycles, and alkali-silica reactions.
2. Future research work should be conducted on concrete specimens to investigate the difference in the behavior of both mortar and concrete exposed to the same aggressive environment.

List of abbreviations

FA/S-GM	Fly ash/slag geopolymer mortar
OPC/S-M	Ordinary Portland cement/slag mortar
XRD	X-ray diffraction
SEM	Scan electron microscopy
OPC	Ordinary Portland cement
ASTM	American society for testing and materials
FA	Fly ash
S	Slag

Declarations

Available of data and materials

Data available upon request

Competing interest

The author declare no conflict of interest.

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