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Original Article

Evaluation of Synergic Effect of Graphene Oxide Nanosheets (GO) and Ground Granulated Blast Furnace Slag (GGBS) on the Durability Characteristics of Cement Mortars

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Abstract - *Nanomaterials addition in cement-based materials has shown enhanced properties related to strength and durability. The mechanical and durability performance of cement-based materials is greatly influenced by the addition of Ground Granulated Blast Furnace Slag(GGBS), which is effective as an alternative construction material. This study reports the investigation on the inclusion addition of Graphene Oxide nanosheet (GO) and Ground Granulated Blast Furnace Slag (GGBS) on the durability performance of cement mortar samples and comparison with reference samples. With 0.08 % GO and 30% GGBS with cement, strength loss, mass variation and visual deterioration of cement mortar samples immersed in two different concentrations of 5% and 10% sodium sulphate solution were evaluated after 90 and 180 days. The addition of GO and GGBS showed remarkable improvement in deterioration to sulphate attack with enhanced resistance to strength and to mass variation, which is confirmed by a visual deterioration of samples presented in this work. Results show that the composite mixture of GO and GGBS provided effective resistance against sulphate attack compared to reference/ control samples. The synergic effect of GO and GGBS proved effective in enhancing the durability performance of cement-based materials against sulphate attack.*

Keywords - *Durability, Sulphate attack, Graphene Oxide, GGBS, Cement mortar*.

1. Introduction

The durability of Reinforced Concrete (RC) structures under aggressive exposure and extreme climatic conditions has become a matter of great concern to the construction industry worldwide [1]. Concrete is more prone to deteriorate due to the aggressive environmental conditions in the atmospheric and splash zone due to chloride content in water, sulphate attacks, abrasion erosion caused by slit, sand and gravel, wetting and drying cycles and due to presence of waste and chemicals in water [2]. The utilization of Supplementary Cementitious Materials (SCM) like Rice Husk Ash (RHA), Silica Fume (SF), Ground Granulated Blast Slag (GGBS), Palm Oil Fuel Ash (POFA), Eggshell Powder (ESP), and Fly Ash (FA) has a positive influence on the properties of concrete [3, 4]. A large amount of waste is generated due to industrial activities, which leads to various ecological issues. Numerous research studies have extensively documented the positive impact of alternative binder systems, such as Metakaolin (MK), Ground Granulated Blast Furnace Slag (GGBS), Fly Ash (FA), Condensed Silica Fume (CSF), for partial replacement of cement [5-11]. As one of the important factors affecting the structural durability of cementitious materials, sulphate erosion not only reduces the service life of the material but also poses a threat to the structural safety of the project [12]. The study on HVFA concrete mixes provided enhanced resistance compared to Portland cement concrete. The HVFA 80% cement replacement performed better in acid media, while the HVFA concrete mix with 65% cement replacement was superior in sulphate media [13]. As Ultra-High-Performance Concrete (UHPC) shows significant advantages compared to conventional concrete, but its use is limited due to high cost and design procedure, a comprehensive investigation of the durability characteristics of UHPC is essential to provide fundamental information for material testing requirements and procedures and expand its practical applications [14].

Nanomaterials are now being applied and developed in the realm of materials, where they have shown strong filling effects on composite materials that significantly enhance the integrity of composite materials [15]. Nanoparticles are reactive because of their large surface area and small particle size, and that will reinforce the layer transition between mixed cement paste to increase strength and decrease permeability [16]. In the past decades, carbon nanomaterials have been successfully used to enhance cementitious materials properties., e.g., carbon nanotubes, carbon nanofibers, and graphene [17]. Nanomaterials act as the seed for nucleation, which leads to dense and less porous CSH-hydrated products [18]. As a result of the addition of GO at an optimum dosage [19]. GO as the nanomaterial fills pores and cracks in the cement matrix, making the matrix more compact and restraining the initiation and propagation of microcracks [20]. Using GO-Silane composite emulsion, a water absorption upto 40% was achieved compared to concrete coating with silane [21]. It was demonstrated that, at best, GO coating of concrete surface can reduce water absorption and capillary absorption of concrete by about 40% and 57%, respectively [22]. With recent technological advances, adding nanomaterials as a reinforcement material in concrete has gained immense attention [23].

External Sulphate Attack (ESA) is a complex degradation process that can compromise the long-term durability of cement-based materials in contact with sulphate-rich environments [24]. Due to high surface area and filling ability, nano fine particles can improve the properties of concrete. There are scopes for using newly developed nanomaterials because nanotechnology in cement-based materials is still in the research stage [25]. Graphene oxide has a large specific surface area with higher aspect ratios [26-28]. The inclusion of nanomaterials in concrete has a positive effect in terms of promoting its mechanical strength and durability performance, as well as resulting in energy savings due to reduced cement consumption in concrete production [29]. The chemical attack is initiated with the physical diffusion of sulphate ions along with a series of chemical reactions. This subsequently leads to formations of gypsum and/or ettringite, both of which expand in volume to densify the concrete microstructure temporarily [30].

However, the deterioration mechanisms in detail and the combined effect of GO and GGBS have not been studied in sulphate media for cement-based materials. Also, the combined effect of GO and GGBS on cement-based materials is not clearly understood. Hence an attempt is made to study the combined effect of GO and GGBS to study the durability performance of cement-based materials and to understand the mechanism of deterioration under sulphate medium. This study aims to investigate the resistance to sulphate attack of three mixes: GO, GO-GGBS and a reference mix. In the present study, three different mixes were exposed to sulphate immersion over 90 and 180 days. With 5% and 10% sodium sulphate concentrations, the samples were monitored and evaluated based on compressive strength loss, mass variation and visual deterioration.

2. Experimental Investigation

Figure 1 presents a flowchart of the experimental procedure adopted in the present study. The study involves the preparation of three different mixes of cement mortars designated as SM1, SM2 and SM3, as described in the previous section. SM1 samples are prepared with 0.08% GO and 0% GGBS, SM2 samples are prepared with 0.08% GO and 30% GGBS, SM3 is a plain cement mortar sample prepared with 0% GO and 0% GGBS. The prepared samples were subjected to normal curing in water for 28 days. After curing in water, all samples were subjected to sulphate attack by immersing the prepared and cured samples in sodium sulphate solution with two different concentrations of 5% and 10% each separately for a period of 90 and 180 days. The loss in strength, mass variation, and visual deterioration of the samples were monitored and evaluated at 90 and 180 days of exposure to sulphate solution. The test results after immersion in sulphate resistance of all the mixes were analyzed at 90 and 180 days of exposure to sulphate solution under complete immersion conditions. The experimental results after 90 and 180 days of sulphate exposure are then compared with samples without any additives and analyzed.

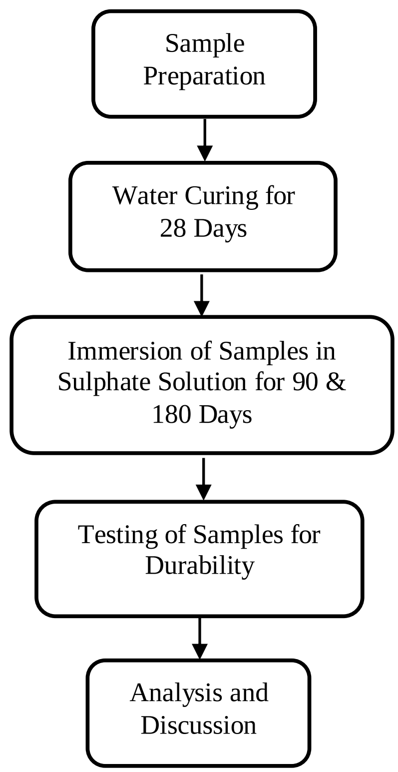

Fig. 1 Flow chart of the experimental procedure

2.1. Constituent Materials

Materials used in the present investigation are OPC 53 grade cement with a specific gravity of 3.15, fineness of 1.3%, standard consistency of 30%, and initial and final setting times are 40 and 430 minutes, respectively. Tests on the physical properties of cement are carried out in accordance with the Indian standards confirming IS-12269:1987. Naturally occurring river sand with a fineness modulus of 2.4 and specific gravity of 2.7, confirming zone II. Tests on fine aggregates were carried out in accordance with IS: 650-1966 and IS: 2386-1968. Ground Granulated Blast Furnace Slag (GGBS) obtained as a by-product from the manufacturing of iron, off-white in colour, with a fineness of 386 m^2/kg, has been used as a partial replacement with the cement in the present investigation. Graphene Oxide nanosheet (GO) dispersed in an aqueous solution of dark grey, with purity greater than 99%, thickness 0.8-2nm, and surface area of 110-250m^2/g has been used in the present study. Ordinary potable water has been used for mixing and curing purposes in the present work.

2.2. Mixing Procedure

A pan mixer was used for the mixing of cement mortar. Fine aggregates were added and mixed for 2 mins, followed by the addition of cement and the GGBS and mixed for a further 2–3 mins. The GO in dispersed form was weighed and added to the calculated quantity of mixing water and thoroughly mixed by stirring. The size of moulds used for sample preparation was 70.6 mm × 70.6 mm. The specimens were kept at 23^0C room temperature for 24 hours before demoulding.

After demoulding, samples were cured in water for 28 days. The samples are then subjected to immersion in sulphate solution for 90 and 180 days for monitoring and evaluating durability performance in terms of strength loss, mass variation and visual deterioration.

2.3. Mix Design

Figure 2 presents the flowchart of mix design adopted for the present study for durability investigation. Three different cement mortar mixes were prepared in the present investigation using GGBS and GO. The cement mortar mix designated with mix ID SM 1 contains 0.08% GO and 0% designated with mix ID SM 1 contains 0.08% GO and 0% GGBS, SM 2 contains 0.08% GO and 30% GGBS, while SM 3 contains 0% GO and 0% GGBS which is a reference cement mortar mix.

The water binder ratio of the three mixes was kept constant at 0.45, and the cement-to-sand proportion was also kept constant at 1:3 for all the mixes. The prepared samples were kept in complete immersion in 5% and 10% sulphate solution using sodium sulphate for 90 and 180 days. Table 1 Presents the detailed mixture proportions of the three mixes, SM1, SM2 and SM3.

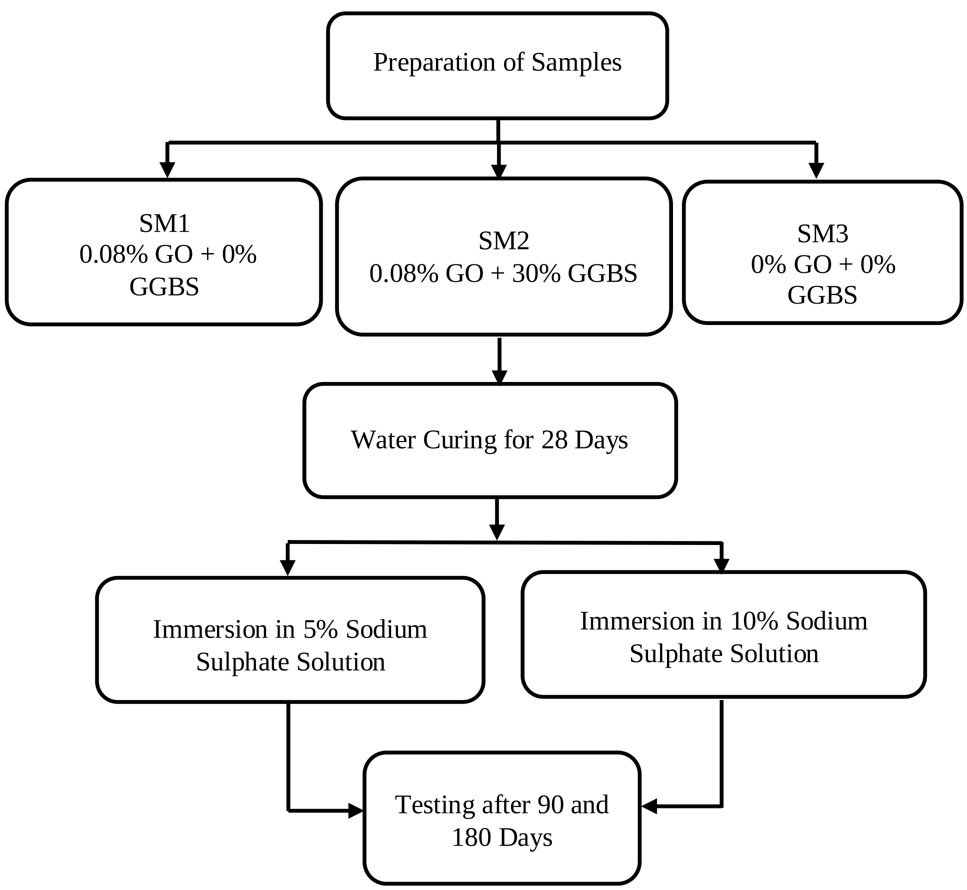

Fig. 2 Flowchart of mixed design

Table 1. Mix the proportion of ingredients

Mix ID	Concentration of GO and GGBS (weight %)	Water-Binder Ratio	Cement to Sand Proportion	$NaSo_4$ Concentration	Duration (days)	Test
SM1	0.08% GO and 0% GGBS	0.45	1:3	5% & 10%	90, 180	Strength loss, mass variation, and visual deterioration
SM2	0.08% GO and 30% GGBS	0.45	1:3	5% & 10%	90, 180	Strength loss, mass variation, and visual deterioration
SM3	0% GO and 0% GGBS	0.45	1:3	5% & 10%	90, 180	Strength loss, mass variation, visual deterioration

Note: SM 1: Sulphate Mix 1, SM 2: Sulphate Mix 2, SM 3: Sulphate Mix 3

2.4. Test Procedure

Tests on durability properties were carried out by preparing three different types of mixes. The mix SM1 contains 0.08% GO and 0% GGBS; mix SM2 was prepared with 0.08% GO and 30% GGBS, and SM3 with 0%GO+0%GGBS. All samples were cured in water for 28 days as per standard procedure before the samples were immersed in sulphate solution for 90 and 180 days. All samples were immersed in sodium sulphate with 5% and 10 % concentration separately. The strength loss was evaluated by subjecting the cement mortar samples to compressive strength testing under a loading rate of 35 MPa / min in accordance with IS 4031 PART 6 at 90 and 180 days. The strength loss of the samples was evaluated in percentage by comparing with the strength of samples subjected to normal water curing. The variation in the mass of all the samples was monitored and evaluated based on the loss in mass of the samples under sulphate attack exposure at 90 and 180 days and compared with the samples cured under plain water. Further, the visual deterioration of the samples subjected to sulphate attack was examined based on their physical deterioration appearance at 90 and 180 days. The test results on above mentioned parameters are presented in the next section.

3. Results and Analysis

The results of the experiments conducted on the various samples are analyzed and presented in this section.

3.1. Strength Loss

The strength loss of three different types of mixes, SM1(0.08%GO+0% GGBS), SM2 (0.08% GO + 30% GGBS), and SM3 (0%GO+0%GGBS), were evaluated at 90 and 180 days after immersion in 5 % and 10% sodium sulphate solution. All samples were cured in water for 28 days as per standard procedure before the samples were immersed in sodium sulphate solution. The loss in compressive strength was evaluated for all mixes SM1, SM2 and SM3, compared and analysed. The test results of strength loss subjected to sulphate attack of three mixes, SM1, SM2, and SM3, are presented in Table 2.

Table 2. Strength loss of samples subjected to sulphate attack

Mix ID	Average Strength Loss (%)			
	90 Days		180 Days	
	5% Na_2So_4	10% Na_2So_4	5% Na_2So_4	10% Na_2So_4
SM1	2.87	3.4	4.2	6.1
SM2	2.23	2.8	3.3	5.6
SM3	7.2	9.4	12.3	15.3

Figure 3(a) Presents the strength loss of three samples, SM1, SM2 and SM3, subjected to sulphate attack for 90 days at 5% and 10% Na_2So_4. From the results, it can be observed that SM1 showed a loss of strength by 2.87%, Mix SM 2 showed a strength loss of 2.23%, and Mix SM 3 showed a strength loss of 7.2% at 90 days at 5% sodium sulphate concentration. The loss in strength for 90 days at 10% sodium sulphate concentration was 3.4%, 2.8% and 9.4% by SM1, SM2 and SM3 samples, respectively.

From Figure 3(b), it can be observed that after 180 days of exposure to sodium sulphate attack, samples SM1, SM2, and SM3 exhibited 4.2%, 3.3% and 12.3 % strength loss at 5% concentration, respectively. At 10% sodium sulphate concentration, SM1 showed 6.1 %, SM2 showed 5.6%, and SM3 showed 15.3 % strength loss at 180 days of exposure. From the experiment it can be understood that mix SM2, which contains 0.08% GO and 30% GGBS, exhibited better resistance to compressive strength loss compared to the other two mixes. The mix SM 1 with 0.08% GO and 0 % GGBS also displayed enhanced resistance to strength loss in comparison with SM3(0%GO and 0% GGBS). The mix SM3, which is a plain cement mortar without GO and GGBS, exhibited less resistance against sulphate attack in terms of compressive strength loss. Better resistance to strength loss in mixed SM2 may be due to the densification of the micropore's reduced permeability due to the combined effect of GO and GGBS.

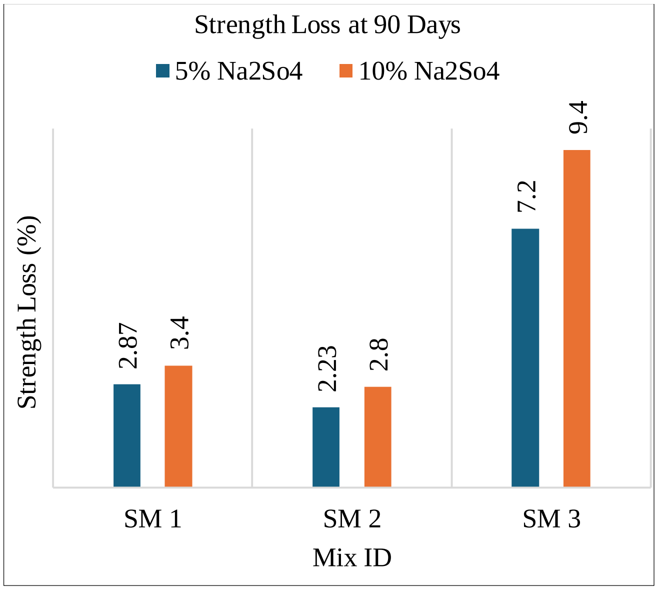

Fig. 3(a) Strength loss of samples subjected to sulphate attack at 90 days

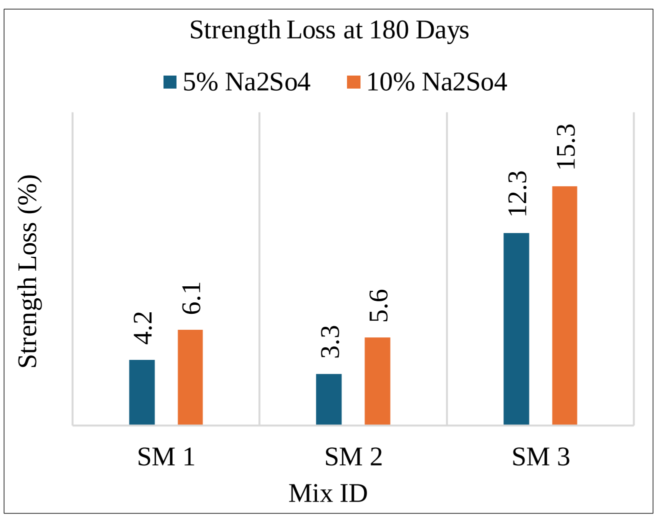

Fig. 3(b) Strength loss of samples subjected to sulphate attack at 180 days

3.2. Mass Variation

The mass variation of three different types of mixes, SM1 (0.08%GO+0% GGBS), SM2 (0.08% GO + 30% GGBS), and SM3 (0%GO+0%GGBS), were evaluated at 90 and 180 days of exposure to sulphate attack. All samples are cured in water for 28 days as per standard procedure before the samples are immersed in 5% and 10% Sodium sulphate solution for 90 and 180 days.

The mass variation of the samples subjected to sulphate attack was examined by weighing the mass loss after 90 and 180 days of exposure to sulphate attack. The test results of mass variation subjected to sulphate attack of three mixes, SM1, SM2 and SM3, are presented in Table 3.

Table 3. Mass variation of the samples subjected to sulphate attack

Mix ID	Average Mass Variation (%)			
	90 Days		180 Days	
	5% Na_2So_4	10% Na_2So_4	5% Na_2So_4	10% Na_2So_4
SM1	1.87	2.5	3.1	4.8
SM2	1.18	1.6	2.3	3.6
SM3	5.23	6.7	7.32	8.5

Figure 4(a) presents the mass variation of three samples, SM1, SM2, and SM3, subjected to sulphate attack for 90 days of exposure to sodium sulphate at 5% and 10%. From the results, it can be observed that SM1 shows a mass variation of 1.87%, SM2 shows a mass variation of 1.18%, and SM3 shows a mass variation of 5.23% at 5%. Further, 2.5%, 1.6% and 6.7% mass variation were observed when SM1, SM2 and SM3, respectively, were exposed to sulphate attack at 10% concentration.

Figure 4(b) presents a mass variation of samples SM1, SM2 and SM3 subjected to sulphate attack at 180 days. At 5% concentration for 180 days, mass variations of 3.1%, 2.3%, and 7.32% can be observed for SM1, SM2 and SM3 respectively. Later, when exposed to 10 % sulphate concentration, mass variations of 4.8 %, 3.6 % and 8.5 % were observed for samples SM1, SM2 and SM3, respectively, at 180 days. Both SM 1 and SM 2 show better resistance to mass variation compared to SM 3. Enhanced resistance in the mass variation may be due to better bonding of the constituents in the cement mortar samples due to the pozzolanic effect of GGBS and the reinforcing effect of GO.

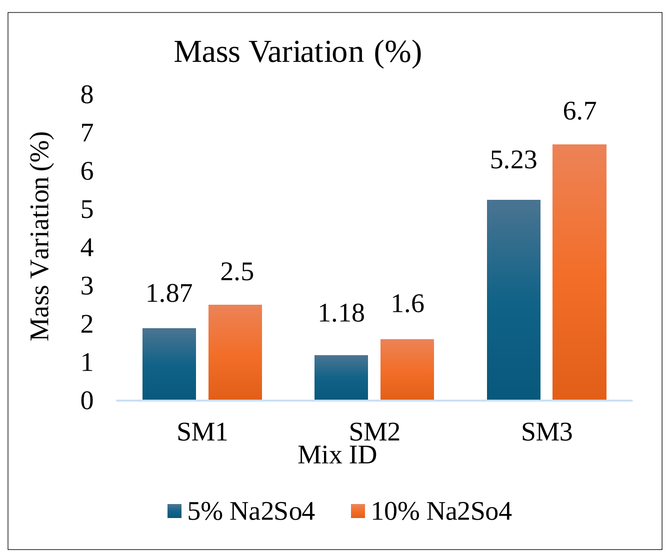

Fig. 4(a) Mass variation of samples subjected to sulphate attack at 90 days

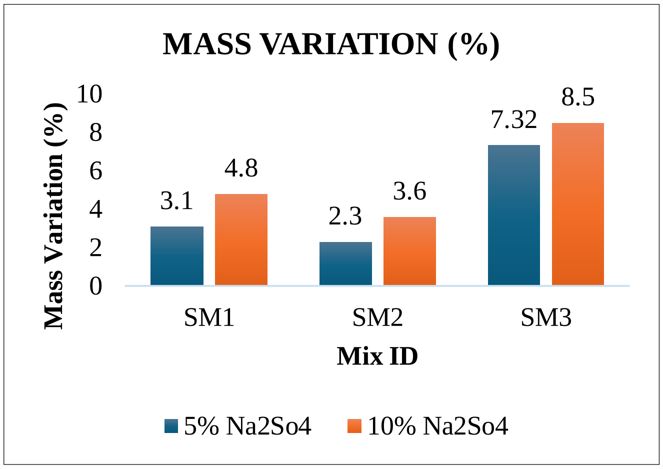

Fig. 4(b) Mass variation of samples subjected to sulphate attack at 180 days

3.3. Visual Deterioration

The physical appearance of three different types of mixes SM1(0.08%GO+0% GGBS), SM2 (0.08% GO + 30% GGBS), SM3 (0%GO+0%GGBS) were evaluated at 90 and 180 days at 5% and 10% sulphate concentration. All samples were cured in water for 28 days as per standard procedure before the samples were immersed in Sodium Sulphate solution for 90 and 180 days. The visual deterioration of the samples subjected to sulphate attack for 90 and 180 days was examined and compared. Figure 5 presents the visual deterioration of three samples, SM1, SM2 and SM3 subjected to sulphate attack at 90 days with 5% sulphate concentration.

It can be observed that SM3 showed more deterioration to sulphate attack compared to SM1 and SM2. Further, when these samples were exposed to 10% sulphate concentration, a similar pattern of deterioration was observed at 90 days, as shown in Figure 6. Reference samples (SM3) showed the appearance of pores and the deposition of some greyish-white products on their surface. This may be due to the rapid reaction of hydration products with sulphate ions, which has resulted in the formation of a weak microstructure.

Upon exposure to sulphate attack for 180 days at 5% sulphate concentration, SM2 samples (with GGBS and GO) showed a better visible appearance compared to SM3 (reference samples). Also, SM1 samples exhibited better resistance than SM3 samples, as presented in Figure 7.

Similar results were obtained when exposed to 180 days at 10% sulphate concentration, as shown in Figure 8. The deposition of a more greyish-white product on the surface of samples can be seen in SM3. This whitish deposition may be due to the formation of ettringite during the chemical reaction with the products of hydration during immersion in sulphate solution due to sulphate attack. In mixes SM1 and SM2 a small amount of whitish appearance can be observed compared to SM3. This may be due to better resistance developed by SM1 and SM2 samples against sulphate attack due to the synergic effect of GO and GGBS, which has resulted in a better stable hydration product compared to samples without GO and GGBS.

Fig. 5 Visual deterioration of samples SM1, SM2 and SM3 after exposure to sulphate attack for 90 days at 5% sulphate concentration

Fig. 6 Visual deterioration of samples SM1, SM2 and SM3 after exposure to sulphate attack for 90 days at 10% sulphate concentration

Fig. 7 Visual deterioration of samples SM1, SM2 and SM3 after exposure to sulphate attack for 180 days at 5% sulphate concentration

Fig. 8 Visual deterioration of samples SM1, SM2 and SM3 after exposure to sulphate attack for 180 days at 10% sulphate concentration

4. Conclusion

The experimental investigation conducted on three different mixes, SM1, SM2 and SM3, under full immersion in sulphate solution with 5% and 10% concentration revealed that cement mortar samples with the addition of Graphene oxide nanosheets along with GGBS (SM2) exhibited good resistance to strength loss compared to reference samples) SM3(0% GO and 0% GGBS) at 90 and 180 days. At 180 days of sulphate attack, the average strength loss of 3.3% and 5.6% at 5% and 10 %, respectively, was observed in SM2**.** Further, the samples with only Graphene oxide nanosheet SM1 (0.08% GO and 0% GGBS) also showed better resistance against attack by sulphate ions at both 5% and 10% concentration for 90 and 180 days of exposure, compared to the plain samples (0%GO and 0% GGBS), with an average strength loss of 4.2% and 6.1% at 5% and 10 % respectively at 180 days. Reference samples (without GGBS and GO) showed poor resistance to sulphate attack with an average strength loss of 12.3 % and 15.3% at 5% and 10% concentration at 180 days. After investigation of samples for mass variation, both the mixes SM1 and SM2 exhibited better resistance in comparison to reference samples SM3 (reference sample). SM2 samples showed good resistance to deterioration to sulphate attack by 2.3% and 3.6% at 5% and 10% concentrations of sulphates at 180 days. The physical appearance of the samples subjected to sulphate attack showed deposition of whitish product on the surface of samples. The deposition may be due to the leaching of chemicals formed during reaction with sulphate ions during immersion in sulphate solution. Samples with GO and GGBS presented better resistance against mass variation and strength loss, which is confirmed by the visual appearance of samples which shows less deterioration of samples and less deposition of whitish product on the surface of samples. The visual appearance of reference samples exhibited more deterioration with whitish deposition compared to the other two samples (SM1 and SM2). Both mixes, SM1 and SM2, showed better performance in terms of visual deterioration compared to reference samples. The addition of GO and GGBS provides greater durability against sulphate attack exposures.

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