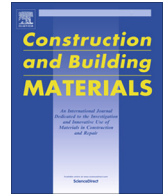




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Durability performance of modified concrete incorporating fly ash and effective microorganism

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HIGHLIGHTS

- Effective microorganism and fly ash included modified concrete was produced.
- Achieved concretes showed strong resistance against sulfuric acid and sulphate attack.
- The modified concrete revealed enhanced durability and high strength.

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ABSTRACT

Environmentally sustainable construction materials with reduced carbon footprint have ever-growing demand worldwide. Based on this factor, some modified cement concrete mixtures incorporated with optimum ratio of fly ash (FA) and effective microorganism (EM) were produced. The strength performance, water absorption, resistance to aggressive environments (sulfuric acid and sulphate) and microstructures of the proposed concretes was evaluated. Four different ratios of FA replacing ordinary Portland cement (OPC) were used to select the optimum composition. The water content was replaced with the EM solution of 5, 10, 15, and 20%. The FA and EM incorporating OPC in the concrete matrix were found to enhance the mechanical and durability characteristics of the modified concretes. The early compressive strength of proposed concrete was enhanced over 30% and the durability was improved against the harsh environment due to the incorporation of OPC with 10% of FA and EM. The optimum concrete obtained with the FA and EM of 10% was asserted to be environmentally beneficial towards less global warming.

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1. Introduction

Rapid deterioration of conventional concrete in aggressive environments has been one of the major problems in the concrete industry. The aggressive environments cause an expansion and cracks in the OPC based concretes. Occasionally, the cracks in the concretes may result in severe structural disorder and eventual failure [1,2]. Thus, continual efforts have been made to enhance the durability of concretes by order of magnitude via improvement

of smart construction engineering materials so called self-healing types [3]. To overcome this limitation, the construction sectors have been gradually accepting the usage of supplement cementitious materials (SCM) such as., fly ash (FA), silica fume (SF), ground blast furnace slag (GBFS), palm oil fuel ash (POFA), wastes tile ceramic particles (WCP), limestone fines (LF), etc., as a blend to cement for reducing the excessive use of OPC.

At present, the traditional cements are usually made by combining the FA or GBFS with the OPC, wherein the C₃S level is in the range of 55–60% [4]. The FA is manufactured as the spinoff due to coal burning in the thermal power plants. The prospective usage of the FA as an effective SCM in the concrete became a practice since the 20th century [5]. Several studies were made to deter-

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mine the overall characteristics of FA included concretes [6]. It was demonstrated that FA with the pozzolanic characteristics are highly active to improve the overall traits of the concretes. However, the inconsistent nature of its chemical and physical behaviour have limited the full adaptation of the FA [7]. Moreover, the early stage strength development of FA-based concretes was shown to be slower compared to OPC-based concretes. Also, the FA included concretes show slower cement hydration process and higher permeability, which results in reducing the early compressive strength of proposed concrete. Despite these advantages of the FA included concretes is seldom used in the civil engineering purposes. This may be due to its drawbacks related to the very slow early age strengths development. Thus, an improvement in the early age strengths development of the FA-based cement concretes is essential for widespread structural usages. To speed up the early age strength development of the FA-based cement concretes several strategies have been adopted. For instance, variation in the finer FA particles size proportion was shown to accelerate the strengths development of the mortar faster compared to those prepared with coarse FA particles [8]. In addition, Kipkemboi et al. [9] shown an increase in the early strengths and thermal cracking resistance and reduction in the drying shrinkages cracking trend of the FA-based cement concretes with the increase in C_3S contents.

Lately, the effective microorganisms (EMs) have been introduced as a novel self-healing and bio-concrete material in the construction industries [10]. For designing the self-healing concretes, the researchers have classified the implementation of the EM as an efficient biological strategy [11,12]. These EMs are abundant and can easily be available from the soil, water and oil reservoirs, acidic hot springs and wastewater effluents from various industries. Therefore, these effective microorganisms can be considered as the potential candidate to design the self-healing concretes [13,14]. The EMs can be grouped into three classes such as the bacteria, fungi, and viruses. Amongst these EMs, some special bacterial strains have the capacity to precipitate certain chemical compounds or elements beneficial for the biological self-healing and bio-concrete design. In addition, these EMs can be incorporated into the self-healing bio-concrete via varieties of methods. These methods include the direct inclusion of the microbial broth into the fresh concretes; incorporation as spores, immobilized form onto the silica gel or activated carbon, encapsulated form, or by the vascular networks following the chemical route for the microorganisms homogeneous distribution [11,15].

Isa et al. [16] reported that the concrete compressive strength at 28 days of age has enhanced by more than 14% with the inclusion 3% of EM in the concrete matrix. It was assumed that the improvement in the compressive strength at all the testing ages were due to the presence of the *Bacillus subtilis* bacterial strains in the locally produced EM wherein the pores in the concrete was partially filled with the introduction of the bacteria. This material development could reduce the pores, thereby leading to an increase in the compressive strength. It was also argued that the bacterial growth takes time and it cannot give significant early effect in the concrete without urea hydrolysis [17]. Bacteria without its food in the EMs cannot contribute to the strength improvement [18]. Sato, Higa [19] showed that by using EM one can increase the compressive strength by 10 to 15% compared to the control samples [20]. Use of 5% EM as partial replacement of the water was found to be optimum to get higher strength compared to the normal concretes without EM. The use of 5% EM could yield a compressive strength of 43.17 MPa, which was 42.9% higher than the sample prepared without EM. At age of 1 day, the 5% EM-based concrete achieved 54% of its final strength, thus EM can also be used where early strength is required. It also showed improvement in the tensile and flexural strengths. Rizwan et al. [21] used the EM-based self-compacting concrete, where the concrete mixtures

were designed with 5, 10, and 15% of EM as water replacement. The results showed that the presence of EM could modify the plastic viscosity, delayed setting times, hydration kinetics, volume stability and reduced the water absorption of the self-compacting paste systems in hardened state and enhanced the compressive strength of self-compacting paste systems. Based on previous studies [22–25], it has been found that the EM have the ability to enhance the corrosion resistance. In short, the EMs-based concretes have found some structural applications in the construction sectors due to the enhanced workability, strength and improved durability of the concretes.

Considering the substantial benefits of EM included concrete, the present work was aimed to develop some sustainable modified concrete mixtures incorporating EM and FA to improve their early age strength. The effects of various contents of EM and FA on the compressive strength and durable properties were investigated. Qualities such as the early and late compressive strength, water absorption, acid and sulphate resistance were evaluated. Results were analysed, interpreted, discussed and validated regarding the eco-friendliness and sustainable traits of these modified concretes.

2. EM, hazardous classification and its impact

To achieve a clean environment, it is very important to produce non-toxic materials using non-polluted natural resources. Effective microorganisms (EMs) are a consortium of beneficial microorganisms [26]. These microorganisms change the balance from degeneration to regeneration. EM technology is widely used in agriculture, production of health drink, concrete industry, waste water treatment, general health and others, etc. The EMs fermented extract was claimed to possess strong anti-oxidation property [27,28]. As EM technology is non-toxic and cheap [29], its applications in agricultural sector can be integrated with standard agricultural fertilizers which reduces also the usage of other chemicals. This technology has another desirable effect, i.e., it increases significantly the organic matter, nitrogen, phosphate and potassium content of wastes which can convert them into a byproduct that can be utilized as an ecofriendly soil fertilizer, municipal waste treatment and activated solution in concrete production. To produce healthy drinking water, Kannahi and Dhivya [28] reported that the inclusion of 2% EM along with 5% herbal extracts could generate a herbal healthy drink that contains more antioxidant property. Based upon the above-mentioned literature studies, it is clear that the EMs are non-toxic environmental friendly materials and their incorporation in FA-OPC matrix will be safe and will not have any side effects or hazardous to humans as well as the environment.

3. Methodology

3.1. Raw materials compositions and characterizations

The resource materials including the OPC, FA and EM were utilized to compose the modified concretes. The OPC satisfying the requirement of the ASTM C150 for cement Type I was used. The OPC was obtained from local cement producer in Malaysia and utilized as the primary source of the calcium oxide (CaO). The FA, as the primary source of the aluminium silicate was acquired from Tanjung Bin power station (Johor, Malaysia) and utilized without any further purification. The chemical compositions of the OPC and FA were determined using the X-ray fluorescence spectroscopy (XRF) as shown in Table 1. The amount of calcium oxides and aluminium-silica oxides was 88% in OPC, and 91.2% in FA, respectively. An appreciably high level of the CaO (about 62.4%) was traced in the OPC and very low amount (5.16%) in FA. The amount

Table 1
Chemical compositions and physical properties of OPC and FA.

Chemical compositions of OPC and FA (weight%)									
Materials	Elements (weight%)								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	LOT
OPC	20.4	5.2	4.19	62.4	1.55	0.01	0.14	2.11	2.36
FA	57.20	28.8	3.67	5.16	1.48	0.94	0.08	0.10	0.12
Physical properties of OPC and FA binder									
Properties	OPC			FA			Permissible limits		Relevant standard
Specific gravity	3.15			2.20			3.10–3.25		ASTM C33
Color	Dark Grey			Grey			–		–
% Passing through 45 µm wet sieve	90			100			≥ 34		ASTM C430

of Al₂O₃, SiO₂ and CaO could significantly affect the production of the concrete via the creation of the C-(A)-S-H gels as a share of the hydration process. The K₂O level was below 1% for all the examined samples. The level of iron oxide (Fe₂O₃) in the OPC and FA was correspondingly very high (4.19%) and low (3.67%), respectively. Table 1 shows the specific gravity, color and the fineness values of the OPC and FA obtained from the characterizations. The specific gravity of the OPC and FA was 3.15 and 2.20, respectively. The color of OPC was dark grey while FA revealed grey appearance. According to the ASTM C430, the percentage passing through 45 µm (No. 325) wet sieve for OPC and FA were 90.0% and 100%, respectively.

The effective microorganism (type EM-1) of 5 kg was collected from Peladang (Johor Bahru, Malaysia) in the air-tight container. XRF analysis was carried out to evaluate the chemical elements by the percentage of mass in the EM-1. The EM-1 mainly contained Magnesium (0.114%), Aluminium (0.0316%), Silicon (0.0546%), Phosphorus (0.0233%), Potassium (0.767%), Calcium (0.981%), Zirconium (0.0148%), Ruthenium (0.214%), Rhodium (97.5%), and Promethium (0.0125%). The Mg, Al, Si, K, and Ca can be beneficial for the concrete strength development at the early and later age. The EM-1 enclosed maximum percentage of Rh by mass because it mostly exists in the river sand or agriculture soil. In addition, the EM-1 was contained in the form of soil. The element Rhodium is helpful to protect the concrete from the corrosion. These elemental analyses are explained later in the context of microstructures test on the optimum solution. First, the EM-1 was activated by adding it to a mixture of water and blackstrap molasses (its primary food source) where the resultant mixture is called the EM activated solution (EM-AS). The activation required anaerobic fermentation for 7 to 10 days to achieve pH less than 4. Next, the EM-AS was used for the concrete preparation. The mixture consisted of 90% of water, 5% of EM-1 and 5% of molasses. The physical presence of EM and solubility, when mixed with water and in its original form was carried out. The main purpose of this investigation was to visualize the physical appearance (color) of the fresh and hardened concrete. The color of the EM was dark brown in its original state but after replacing with 10% of water its color was changes to dark yellowish. Furthermore, when 25% of water was replaced with EM, the color was transformed from dark brown to light brown color.

Table 2 displays the effect of EM replaced by water in pH of the prepared solution. The pH value of the tap water was 6.7 compared to pH 3.5 of the EM. The pH value of the solution tends to decrease with the increase in EM content and the replacement with 5, 10, 15, 20, and 25% of EM produced the pH value of 6.4, 6.3, 5.7, 5.2, and 4.0, respectively. Similar results were also reported by Isa, Garba [16], Andrew, Syahrizal [20]. The pH of the fresh EM included solution was above 3.5 [30]. Cabrera, Galvín [31] a high and low value of pH of water can affect the fresh and hardened

Table 2
pH, viscosity and surface tension of EM-water solution.

Solution	pH	Viscosity, mPas	Surface tension, mN/m
Water	6.7	0.95	66
EM-AS (90%H ₂ O, 5% EM-1 and 5% molasses)	3.45	1.44	35.6
95% water + 5%EM-AS	6.4	1.0	58.3
90% water + 10%EM-AS	6.3	1.05	54.5
85% water + 15%EM-AS	5.7	1.1	51.7
80% water + 20%EM-AS	5.2	1.2	44.8
75% water + 25%EM-AS	4.0	1.25	39.9

properties of the concretes. In addition, it plays a vital role in the hydration process in the early age of the concrete and durability of the concrete in the later periods. The viscosity value of water, EM and EM solutions are shown in Table 2. The viscosity of the EM in its original state was higher than the viscosity of water where viscosity of the EM and water was discerned to be 1.44 mPas and 0.95 mPas, respectively. However, the replacement of water by 5, 10, 15, 20 and 25% to the EM produced the corresponding viscosity of 1, 1.05, 1.10, 1.20, and 1.25 mPa which are slightly higher than the viscosity of water. Wang and Li [32] stated that the viscosity of mixing water also have an effect on the fresh and hardened properties of the concrete. It can enable the production of fluid concrete having sufficient cohesiveness to reduce the bleeding, segregation, and settlement. It can also improve the workability of the concrete [33]. In this work, the effect of viscosity was low due to the absence of any differentiation between the viscosity of water and EM-water solution used to modify the concrete characteristics. The increase in the viscosity of the EM was due to the addition of the molasses at the time of formation of EM solution.

The surface tension of mixing water played a critical role in terms of the fresh and hardened properties of the produced concretes. Table 2 illustrates the surface tension of water, effective microorganism and 5, 10, 15, 20 and 25% water substituted EM solution. The surface tension of the EM in its original form is 35.6 mN/m which was lower than water surface tension (66 mN/m). The mixed solution of 5, 10, 15, 20 and 25% water replaced EM achieved the corresponding surface tension of 58.3, 54.5, 51.7, 44.8, and 39.9 mN/m which are lower than the surface tension of the original mixing water in the concrete. The surface tension of a liquid characterizes the attractive force that acts at the interface between two phases such as the liquid–solid or liquid–vapour or liquid–gas (–air). It refers to the tendency of the fluid surface elasticity that enables the fluid to attain the minimum probable surface area. It is a vital factor in the process of capillary action that happens when fluids are released and become in contact with the porous rocks or minerals. The interfacial tension is almost analogous to the surface tension with respect to the involvement of the

cohesive forces. In the former one the adhesive force (tension) between the liquid state of one interface and solid phase of another is involved. In the current study, the surface tension played a critical role for the achieved concretes properties.

The normal drinkable tap water in compliance with the ASTM C1602 specification was used to produce and cure the concrete mixtures. The water did not contain any impurity or unwanted components that could produce detrimental effects on the colour or odour of the products. The composition of the water, EM and mixed solution of water with EM were analysed in the Department of Environmental Engineering (Universiti Teknologi Malaysia) and the results of the quality indices are enlisted in Table 3. All the parameters related to the quality of water, EM and 10% water replaced EM solution were investigated and found to be lower than that of the allowed limits specified by the standard. The physical quality of the water was significant because any impurity might have interfered with the setting of the cement. In addition, it might have negatively affected the strength properties of the concrete or caused staining of its surface, leading to the corrosion of the steel reinforcement. Therefore, the quality of the water for both mixing and curing purposes was considered [34].

3.2. Mix design

The Mix design of the concrete is very important in order to produce a batch of concrete with desirable properties. The parameters like the compressive strength and workability can be altered through the design process. Table 4 shows the concrete mix proportions and other mixes. The mix proportions were designed according to the method referred in the Marsh (2007) guide which is also known as Department of Environment (DOE) method. It was then optimized to get the desired characteristic strength of 30 MPa at 28 days with 15 mm slump for the control mix. The cement in the control mix was subsequently replaced by the FA at 10, 20, 30, and 40% by weight. The main focus of the study was to investigate the effect of EM on the concrete properties (10% of water ratio). In order to develop a stronger correlation, the replacement of the FA was selected from 10% and onwards for the workability and other tests. The proportion of the mix was created accordingly to reveal the effect of FA on the concrete properties. A water-to-cementitious material ratio of 0.55 was used for all the mixes (Table 4). The mix proportions of FA were cast separately with different amounts. The cubes were cured for 7 days before being tested for the compressive strength to determine the optimum percentage of FA admixtures suitable for the next stage of specimens' preparation.

3.3. Test methods

The workability of the as-prepared concretes was evaluated to find the influence of the OPC substitution by the FA and EM in accordance to the ASTM C230. All the prepared concretes were examined for their fresh state performances including flow diameters measurements. Compression strength tests were performed on

three samples at ages of 3, 7, 28, and 56 days in accordance to ASTM C109 [50]. The water absorption testing was performed following the ASTM C 642 [51]. Every specimen of size 100 mm × 100 mm × 100 mm was fully submerged in the water (at 27 °C) for one day following the maturing. Each sample was fully immersed in water and then hung for weighing (Ws). After the saturation, the specimens were dried at 105 °C for one day using a ventilated oven and then weighed (Wd). The water absorption rate was evaluated by taking the average of 3 specimens. The water absorption (%) of the mix designs were calculated via:

$$\text{Water absorption (\%)} = [(W_s - W_d)/W_d] \times 100 \quad (1)$$

where Ws and Wd are the respective saturated and oven-dried weight of the concrete (in gram).

The impact of the sulphuric acid (H₂SO₄) on the concrete specimen was tested by crumbling the binder paste solution. The H₂SO₄ solution (10%) was prepared using the deionised water. For every concrete, 6 mixtures each of 28 days of age were weighed and then immersed into the acid solution for one year. The acid solution was altered in every 2 months in order to retain the fixed pH level during the experimental period. Next, the samples were checked after 180 days and 1 year before the performance evaluations concerning the microstructural changes, weight losses and remaining strengths based on the ASTM C267 specification [35]. The sulphate attacks in the concrete were caused mainly by the penetration of the sulphate ions (SO₄)²⁻. The sulphate attack enabled decomposition of the concrete produced different concentrations of Mg, Ca or Na cations in the acid solution. The test results of the concrete for sulphuric acid and magnesium sulphate solution provided their resistance against aggressive environments.

To access the microstructural properties of modified cement exposed to acid and sulphate attack, several tests such as, X-ray diffraction (XRD) and Scanning electron microscopy (SEM) were adopted. The XRD test is a rapid and simple technique for non-destructive characterization of crystalline materials. It provides information especially on structures, phases, preferred crystal orientations, and structural parameter. After 365 days of exposure period to acid and sulphate solution, cement paste powders were scanned in the 2-theta range of 5 to 90° at the step size of 0.02°. To analyse XRD data, the MDI Jade software version 6.5 and Match software version 3.10.2.173 were utilized to verify the glassy nature of the specimens. SEM with high magnifications was used to examine the tested specimens' surface morphology. First, the modified cement samples were collected from the specimens tested for compressive strength at 365 days of exposure period. Each sample was then sowed on to the double cellophane sheets followed by attaching them on the coin. For SEM imaging, each sample was placed in the sample holder (brass stub type) and dried for 5 min using IR radiations before using a Blazer sputter coater to cover with gold. The resultant patterns were monitored using 20 kV with 1000 × magnification. Significant morphology images were captured immediately after selecting the reasonably high image magnifications.

Table 3
Quality indices of various constituents of EM solution.

Quality	Water	Effective microorganism	10% EM + 90% water	Units	Allowable limit	Standards
Dissolved oxygen (DO)	7.35	2.33	0.6	mg/L	≤ 7.8	ASTM D888
Electrical conductivity	26.6	8831	900	µs/cm	1000–2000	
Electrical resistivity	37,525	113.24	707.16	Ω·cm	10000–50000	
Total dissolved solids	17.55	5824	1092	mg/L	≤50000	ASTM C1602
Salinity	0.01	5	0.85	ppt		

Table 4
Mix design ratios of the prepared concrete.

Mix	Binder, kg/m ³		Solution, kg/m ³		Aggregates, kg/m ³	
	OPC	FA	Water	EM-AS	Sand	Gravel
OPC	450	0	250	0	875	815
FA10	405	45	250	0	875	815
FA20	360	90	250	0	875	815
FA30	315	135	250	0	875	815
FA40	270	180	250	0	875	815
EM5	450	0	237.5	12.5	875	815
EM10	450	0	225	25	875	815
EM15	450	0	212.5	37.5	875	815
EM20	450	0	200	50	875	815
FAEM10	405	45	225	25	875	815

4. Results and discussion

4.1. Compressive strength

Fig. 1 displays the compressive strength of the trial mixtures including the conventional concrete (OPC), FA replacing OPC concrete and EM concrete. All the concrete specimens were tested at the 7 and 28 days of curing age to select the optimum FA and EM for designing the high performance concrete. In this word, the OPC-based concrete specimens presented the strength of 33.9 and 40.4 MPa at the 7 and 28 days of curing and adopted as the control sample. For the specimens prepared with FA as OPC replacement, the 7 and 28 days' strength showed an increasing trend with the increase of FA contents from 0 to 10%. However, with the increase of FA content the strength of the concretes were dropped. For 7 days cured tested specimens, the strength was increased to 34.7 MPa with 10% of FA replacement and the value of the compressive strength tend to decrease with the values of 32.3, 31.4, and 31.1 MPa for FA contents of 20, 30, and 40%, respectively. Similar trend was observed with specimens tested at 28 days of age, where the strength was reduced from 44.3 MPa to 40.1, 39.4, and 38.7 MPa with the corresponding increase in the FA level from 10% to 20, 30 and 40%. Among these four levels of the OPC replacement, the 10% of FA was considered as the optimum ratio for the second stage of experiment. Many studies [36–40] reported the inclusion of FA in concrete matrix as OPC replacement and led to increase SiO₂ and Al₂O₃ levels which are mainly responsible for the hydration process. Young et al. [41] and Sathyan et al. [42] observed that the ions transportation and gel generation in cement

matrix can highly be influenced by the FA incorporation, resulting variations in electrochemical performance and impedance spectral profile. The replacement of OPC by 10% of FA increased the hydration process and enhanced the hydration degree of alite (Ca₃SiO₅), belite (Ca₂SiO₄) and tricalcium aluminate (Ca₃Al₂O₄) [43,44]. The increment in compressive strength of the specimens prepared with 10% FA was mainly ascribed to the formation of extra calcium silicate hydrate (C-S-H) derived from alite and belite [45] - secondary to extra C-S-H gel derived from FA [46].

For the concrete specimens containing the EM, the replacement of water by 5 and 10% of EM led to enhance the compressive strength for both the ages. However, the increasing level of replacement to 15 and 20% affected negatively the strength development, lowering the strength compared to the control specimen. At the 7 days of age, the tested specimens showed a gain in the strength from 33.9 MPa to 34.9 and 36.7 MPa with the corresponding increase in the EM level from 0% to 5 and 10%. Unlike, the strength dropped to 33.2 and 28.9 MPa with increasing replacement level of EM to 15 and 20%, respectively. Similar trend was found for the samples tested at age of 28 days, in which the increasing levels of EM from 10% to 15% and 20% led to reduce the corresponding strength from 43.6 MPa to 38.7 MPa and 35.2 MPa, respectively. The reduction in early age compressive strength of concrete specimens prepared with 15 and 20% of EM could be ascribed to the lower hydration due to the inability of molasses which do not fill the pores.[47]. It was also noticed that the initial hardened concrete had lower pH value compared to the control concrete, implying lower alkalinity of the concrete in the early age. According to Sun, Yu [48], the lower alkalinity of the pore fluid have negative effects on the cement hydration, thus leading to the reduction of the strength of the concrete especially at early ages. Zhan and He [49] argued that the lower pH can hinder the early evolution of the microstructures in the cement pastes, thereby delaying the cement hydration reaction.

The high performance of the concrete specimens prepared with 5 and 10% of EM than the control sample was mainly because of the higher hydration that filled the concrete pores with C-(A)-S-H gels. Other reason for the increase in the compressive strength was due to the lower surface tension of the mixing water, this property enabled the water to produce smaller pores when mixed in the concrete. Qin, Hao [50] reported that by reducing the surface tension of water, the interaction potential energy between the capillary walls can be influence, enabling a reduction in the capillary pores diameter at the microscale during the drying process. According to Dang, Qian [51], the reasonable reduction in the surface tension of the mixing water adsorbed on the cement particles' surface could lower the surface energy and improve the dispersability. This in turn, caused an improvement of the innocuous voids/pores, and thus forming a dense matrix structures. Ngene, Olofinnade [52] a reduction in the surface tension of the mixing water could increase the strength by 25–30% relative to the con-

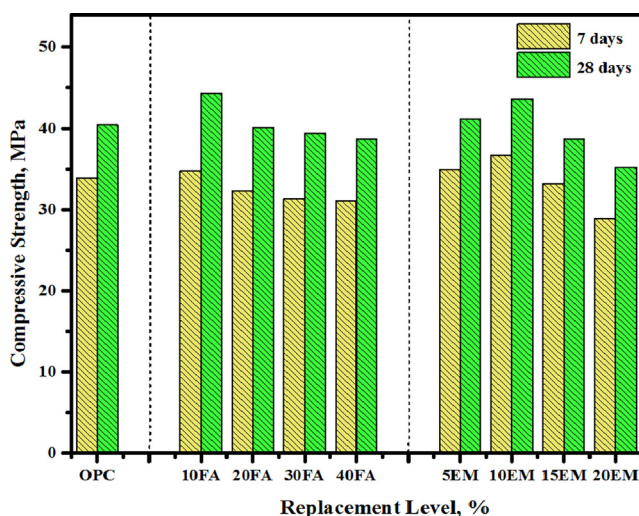


Fig. 1. Compressive strength development of concrete containing FA and EM.

ventional concrete. Tramontin Souza, Onghero [53] the admixture reducing the surface tension of the water can perform efficiently by replacing it with water than using extra mixing water. The researcher used an admixture that reduced the surface tension of the extra mixing water.

Fig. 2 displays the compressive strength of the modified concrete prepared with FA and EM. The strength of the concretes at 3, 7, 28, and 56 days of age was measured to determine the effect of FA and EM on the early and late strength performance. Clearly, the strength of the concrete specimens with the incorporation of the FA and EM as OPC replacement was significantly improved. At early age (3 days), the inclusion FA and EM in the OPC matrix led to enhance the strength value of the concrete to 30.5 MPa compared to 23.2 and 23.7 MPa obtained with OPC and 10% FA. Likewise, the specimens prepared with FA and EM together achieved the highest strength (42.49 MPa) among the other specimens prepared with only OPC (33.9 MPa) and FA as OPC replacement (34.8 MPa). For the specimens tested at 28 days of age, the strength was increased from 40.4 MPa to 44.3 and 50.3 MPa with the inclusion of 10% of FA and EM, respectively. Similar trend was observed for the specimens tested after 56 days of curing age and the specimens prepared with 10% of FA and EM. The OPC modified materials achieved the strength of 54.4 MPa compared to 44.4 MPa (OPC control sample) and 49.3 MPa (10% of FA as OPC replacement). Nedunuri et al. [54] reported that the replacement of OPC by the FA resulted in a decrease of the capillary pores and increase in the gel pores. The observed increase in the gel pores caused the generation of the extra C-S-H through the pozzolanic reactions. This in turn enhanced the C-S-H contents, thereby contributing to the improvement in the compressive strength. Fig. 3.

Fig. 4 illustrates the compressive strength increment percentage as a function of FA and EM content. The results of tested specimens prepared with 10% of FA as OPC replacement showed low rate of strength development at the early age (3 and 7 days). However, the percentage of the strength was sharply increased after the 28 and 56 days of curing age. Compared to the OPC control specimens, the specimen containing 10% of FA revealed an increase of 2.0, 2.4, 9.7, and 12.2% after 3, 7, 28 and 56 days, respectively. This finding is consistent with characteristics of the pozzolanic materials that have significant effects on the hydration process and strength development at late age of specimens [55]. Fraay et al. [56] reported that the pozzolanic reaction can occur in the concrete specimens only after 7 days. Unlike, the inclusion of EM in the concrete matrix presented the high performance in the early age and

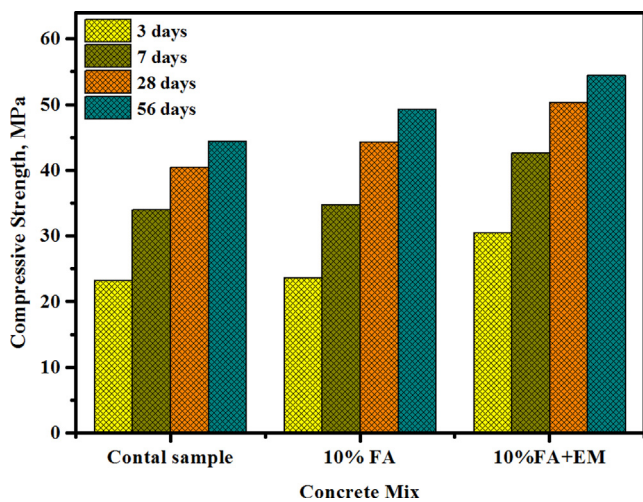


Fig. 2. Effect of EM incorporating FA on OPC concrete's strength development.

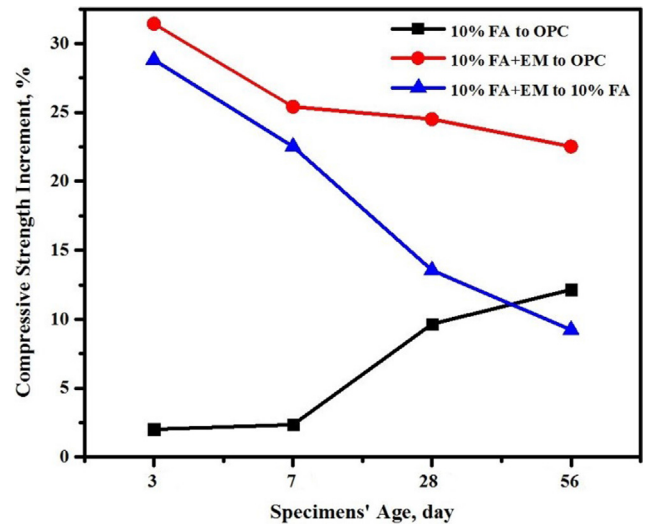


Fig. 3. FA and EM contents dependent compressive strength of OPC replaced concretes at different curing ages.

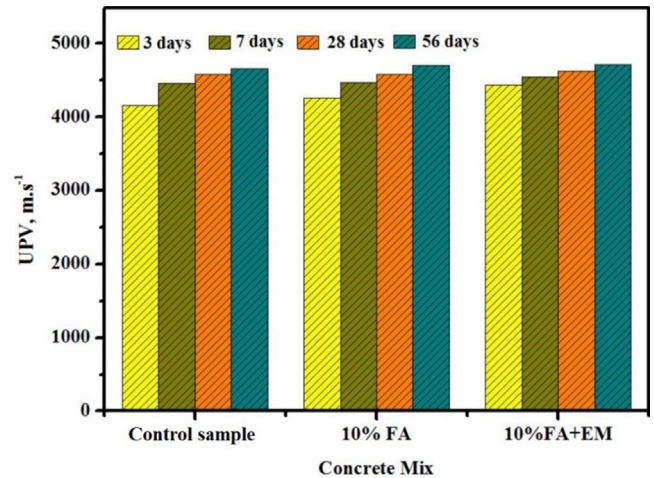


Fig. 4. UPV readings of different concrete mixtures.

significantly reduced with the increase in the curing time. Compared to the OPC specimens, the concretes prepared with the FA and EM achieved an enhanced compressive strength up to 31.4, 25.4, 24.5, and 22.3% after 3, 7, 28, and 56 days of curing, respectively. However, the results of the modified concrete with the FA and EM revealed an increase in the compressive strength by 28.8, 22.5, 13.6, and 9.2% at the curing ages of 3, 7, 28, and 56 days, respectively.

Fig. 4 shows the measured ultrasonic pulse velocity (UPV) of the specimens at the 3, 7, 28, and 56 days of age. The results indicated that the concretes structures were improved (total pores were reduced) due to the incorporation of the FA and EM. For all the prepared concrete specimens, the UPV readings were increased with the increase in the curing age and formation of the denser gels. The structure of FA and EM included specimens were more enhanced compared to the OPC specimens. This was attributed to the optimum content of the FA and EM that produced less number of voids/pores, thereby creating denser concrete so that shorter time was taken by the pulse to travel the distance. Consequently, both velocity and strength were increased. The values of the UPV obtained for all the concrete samples (≥ 4000 m/s) indicated their excellent qualities.

4.2. Water absorption

Fig. 5 displays the results of water absorption of the prepared concretes included with OPC, 10% of FA and EM tested at the ages of 28 and 56 days, where three specimens from each batch were tested and to get the average value. For the specimens tested after 28 day of curing age, it was found the value of water absorption dropped from 7.4% (OPC) to 7.2 and 6.9% with inclusion the 10% FA and EM respectively. Similar trend was observed for the specimens tested at 56 day of curing age and the water absorption value decreased from 7.3% for OPC specimens to 7.1 and 6.7% for FA and EM specimens, respectively. The low water loss of EM solution and pozzolanic reactivity of FA impact positively on hydration process and led to formulate denser gel which was reduce the pores in structure of specimens prepared with 10% FA and EM compared to OPC and specimens containing only 10% of FA. Li et al. [57] and Yu et al. [58] reported the reduce heat hydration and water loss in the OPC matrix that led to enhance the concrete durability by improve the structure and reduce the pores of specimens.

4.3. Acid attack

Fig. 6 shows the compressive strength loss percentage of OPC and OPC modified with FA and EM exposed to acid environment for 180 and 365 days. The durability of the modified concrete specimens containing FA and EM against the sulfuric acid attack were evaluated by measuring residual strength, weight loss, UPV, virtual inspection as well as the microstructure tests included XRD, SEM and EDS. For all concrete specimens, the loss on durability was increased with increasing the exposure time to sulfuric acid solution. The concrete specimens tested after 180 days of the exposure to the acid solution presented an improvement in the concrete durability and the percentage of the strength loss was dropped from 81.3% (OPC) to 69.8 and 67.6% with inclusion of the FA and EM, respectively. Similar trend was obtained for specimens tested after 365 days and the loss of the strength was decreased from 93.7% (OPC) to 84.2 and 83.1% for FA and EM concrete specimens, respectively.

Due to the pozzolanic reactions with FA, the portlandite ($\text{Ca}(\text{OH})_2$) was formed via the hydration of the cements where extra cement hydrates including the C-S-H gels were generated. Consequently, the strength and durability of the products were improved due to the densification of the hardened specimen. The inner por-

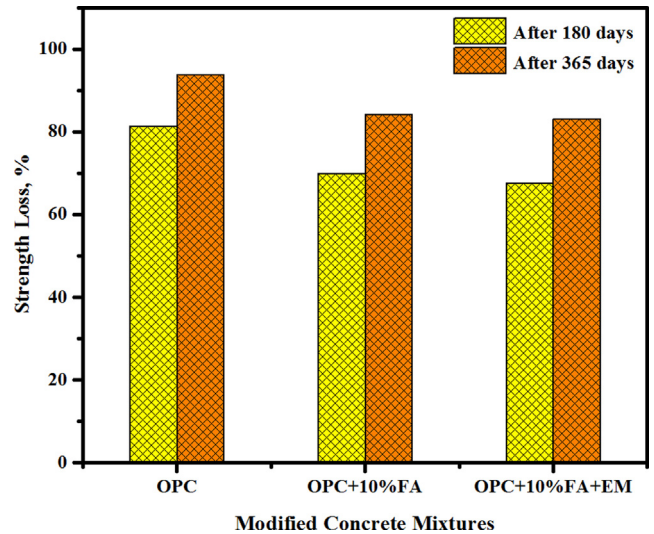


Fig. 6. Strength loss of prepared concrete mixtures exposed to 10% H_2SO_4 solution.

tion of the concretes being highly alkaline in nature the FA constituents (SiO_2 and Al_2O_3) were easily dissolved, which further reacted with the calcium present in the cement and triggered pozzolanic reaction [2,4]. The pozzolanic activity was appreciably influenced by the chemical properties and disordered glassy phases of the FA [22]. The glassy nature of the FA plays a significant role to determine its hydraulic attributes [29]. According to Sakai et al. [4], the pozzolanic activities of the FA can be enhanced by increasing its amorphosity. Therefore, the quantity of the glass phases of the FA must be evaluated.

Fig. 7 shows the weight loss percent of the proposed concretes subjected to the acid environment for 180 and 365 days. The increase in immersion time affected negatively the weight loss percentage and deteriorated the specimens' strengths. The specimens investigated after 180 days of immersion in the solution displayed loss in strength of 52.8% for the OPC specimen and the percentage dropped to 43.6 and 41.9% with the inclusion of the FA and EM in the OPC concrete matrix, respectively. Likewise, the highest deterioration tested after 365 days of exposure was observed with OPC specimens which was recorded 73.2% of weight loss and this percentage decreased to 64.4 and 61.1% with inclusion of the FA and EM in the OPC matrix, respectively.

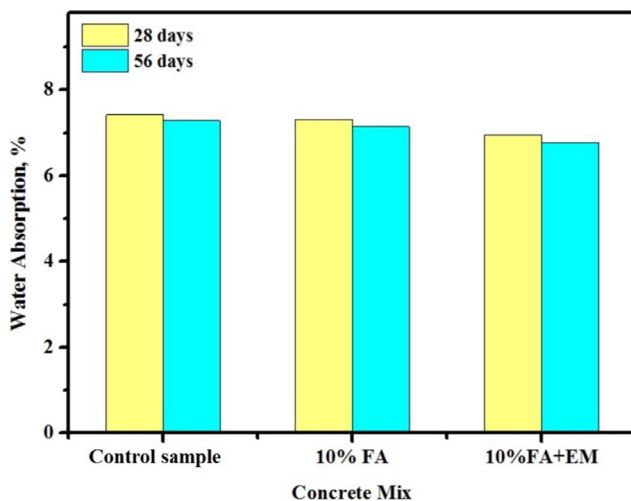


Fig. 5. Effect of FA and EM on water absorption properties of concretes.

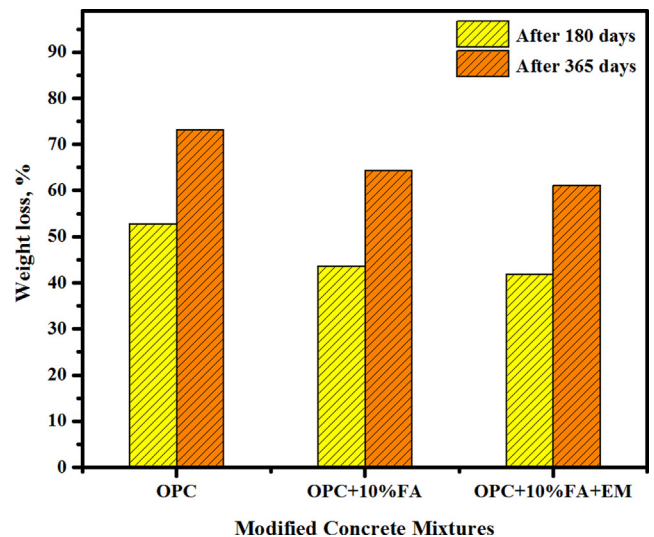


Fig. 7. Weight loss of prepared concrete mixtures exposed to 10% H_2SO_4 solution.

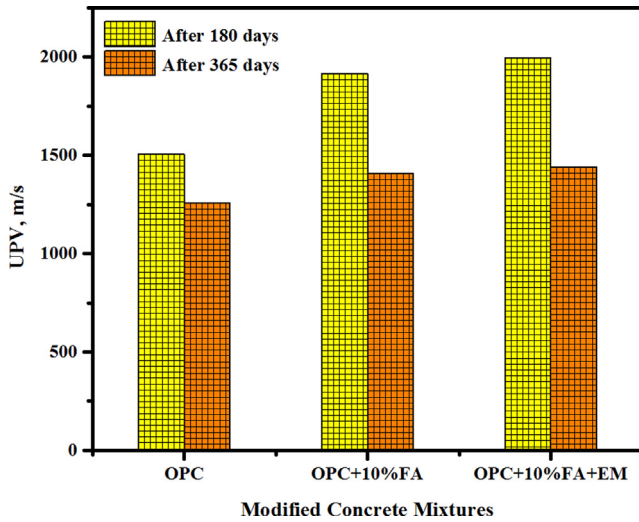


Fig. 8. UPV of prepared concrete mixtures exposed to 10% H₂SO₄ solution.

The UPV test was conducted on prepared concrete specimens after exposing them for 180 and 365 days in 10% sulfuric acid solution. The results are presented in Fig. 8. For all the specimens, the internal and external deterioration increased with increasing the period of exposure. The lower UPV reading for specimens tested after 180 days of exposure was recorded with OPC specimens and presented reading 1504 m/s compared to 1914 and 1995 m/s for FA and EM specimens, respectively. After 365 days of exposure period, the deterioration in specimens was increased and shown lower readings of UPV and recorded 1259 m/s, 1408 and 1439 m/s for OPC, FA and EM specimens, respectively. The reduction in UPV readings of 365 days' specimens compared to 180 days' specimens ascribed to increasing the internal cracks which led to more porous structure and show lower durability. Meanwhile, the inclusion of FA and EM effect positively to reduce the internal crack and show more stable gels in aggressive environment compared to OPC matrix. Upon exposing the prepared concrete to the acid envi-

ronment, the Ca(OH)₂ was reacted with the SO₄²⁻ ion and created CaSO₄·2H₂O, which expanded the concrete network and caused cracks in the specimens' interior (Fig. 8). Many reports [59–62] stated that an increase in the Si and Al level could lead to more stable (C-(A)-S-H) gels in which the lower amount of Ca(OH)₂ can reduce the gypsum formation, thereby improving the durability of the modified concretes. The observed loss in both strength and mass were due to diffusion of the acid molecules inside the concrete matrix and destruction of the cement gels binder. The produced Ca₂SO₄·2H₂O (gypsum which is soft and easily soluble) underwent reaction with the Ca(OH)₂ and created 3CaO·Al₂O₃·3CaSO₄·32H₂O (ettringite). However, in the presence of FA and EM, the content of unstable Ca was reduced in the matrix and restricted the gypsum formulation, resulting in a lower spalling and degradation of the concretes [63–65].

Fig. 9 illustrates the visual look of the concretes produced with the insertion of the FA and EM after 365 days of exposure to the acid media. The durability of the concretes was improved as shown by the reduced surface deterioration. The control specimen prepared with only OPC revealed wider cracks than those made with FA and EM. The deterioration of the concrete was slightly increased with the replacement of the OPC by 10% and inclusion of EM. Addition of FA and EM into the concrete matrix led to the generation of more stable gels which in turn enhanced the durability of the modified specimens and manifested low amount of cracks.

Fig. 10 shows the XRD patterns of the OPC, FA and EM concrete specimens after 365 days of exposure into 10% H₂SO₄ solution. The concrete specimens prepared with FA and EM exhibited the existence of the gypsum (C-S-H) where the intensity of the gypsum peak at 29.7° 2θ was dropped with the inclusion of the FA and EM in it. In addition, the gypsum peak intensity observed at 11.8°, 20.1°, and 23.8° was enhanced and became closer to the quartz at 27.6° and 27.9°. Bellmann and Stark [66] reported the occurrence of the quartz and gypsum peak at 27.6 and 27.9° was difficult to differentiate. The observed double peak structure upon closer inspection indicated the existence of the quartz and gypsum minerals in the concretes. In fact, the inclusion of the FA and EM into the OPC concrete matrix could limit the formation of the gypsum. This is evidenced from the peak appeared at 29.7°, indicating

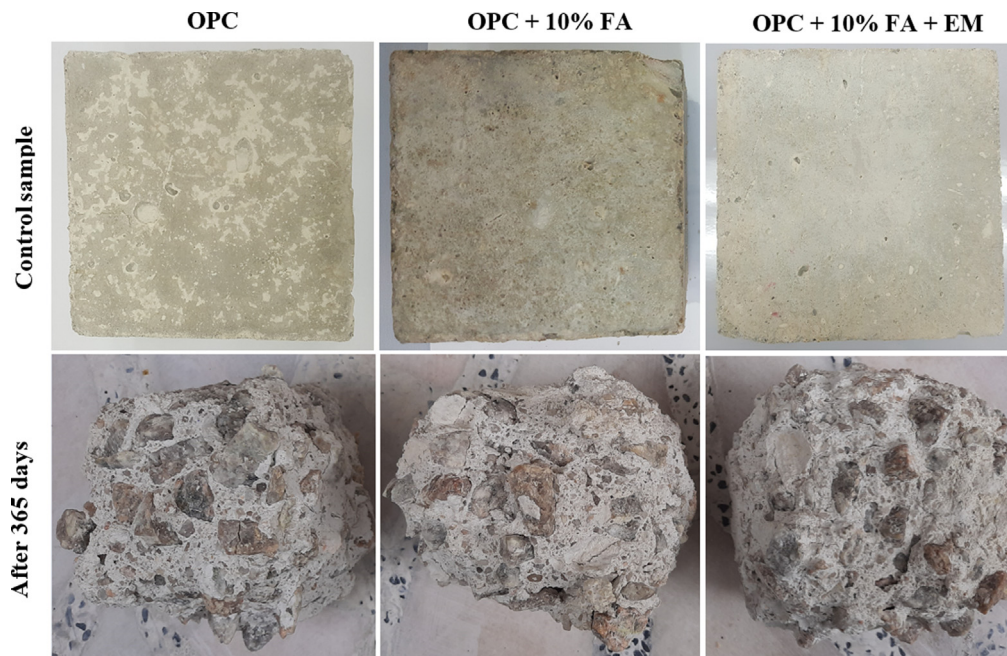


Fig. 9. Visual appearance of designed concrete mixtures exposed to 10% H₂SO₄ solution.

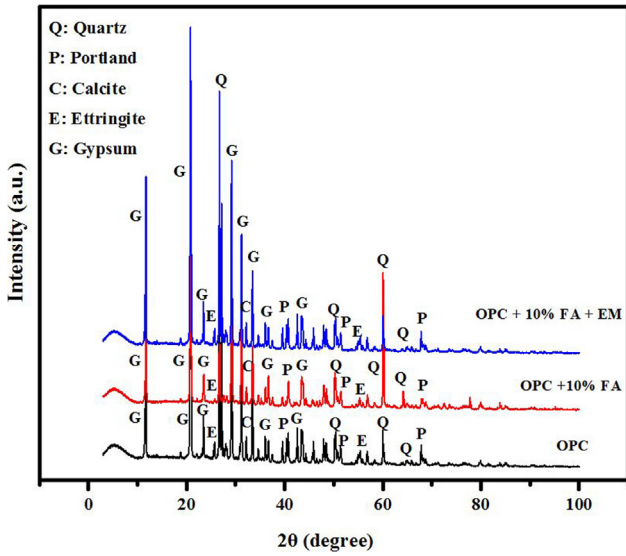


Fig. 10. XRD patterns of prepared concretes exposed to 10% H₂SO₄ solution.

an improvement in the durability and resistance of the specimen against acid attack. In short, it is asserted that the incorporation of the FA and EM into the concrete matrix can help to generate the excess C-(A)-S-H gels [67] which is prone to the sulfuric acid attacks.

It is well known that the SO₄²⁻ are unstable and have strong tendency to react with the Ca(OH)₂ present in the OPC to generate gypsum as given by Eq. (2). Then, the formed gypsum can undergo reaction with the calcium aluminate hydrate present in the OPC to form ettringite as given by Eq. (3), causing a large expansion of the concrete matrix and then failure via cracking. Additionally, the amount of damages due to the H₂SO₄ exposure is related to the acid accumulation in the environment and the duration of the exposures. Table 5 shows percentages of different components present in the concretes (obtained from the XRD analyses) after 365 days of immersion in 10% of H₂SO₄. Therefore, the formulation of gypsum (CaSO₄·2H₂O) in the OPC amounted to 24.1% compared to the respective 14.8 and 12.5% observed with the FA and EM concretes. Likewise, the ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26H₂O) amount trend to reduce from 33.3% with OPC specimens to 28.8 and 26.7% with FA and EM concrete specimens, respectively. This extra amount of gypsum and ettringite in the OPC led to high expansion in the paste matrix that created more cracks and showed higher deterioration in the acid environment compared to the specimen prepared with 10% FA and EM.

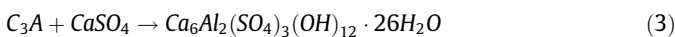
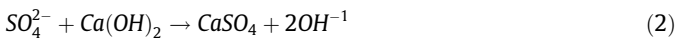


Fig. 11 presents the SEM images of OPC, FA and EM concrete specimens exposed to 10% sulfuric acid solution for 365 days. The SEM micrographs of the FA and EM concrete specimens revealed lower gypsum content as well as shown less deterioration

than the OPC control specimens. The SEM images of the normal concrete prepared with OPC (Fig. 11a) showed more cracks than the modified specimens containing 10% of FA and EM, thereby indicating the existence of the gypsum and ettringite. The observed homogeneously dense morphology in the EM matrix of the tested specimens indicated the presence of non-reacted and partly reacted silica (Fig. 11c). Conversely, the high magnification SEM image (Fig. 11a) showed the existence of several rod-like CaSO₄·H₂O crystallites (gypsum), supporting the XRD data [68,69]. The concrete incorporated with the FA and EM (Fig. 11b and c) revealed lower amount of gypsum and ettringite compared to the pure OPC concrete. The resistance against the acid attack was improved due to the incorporation of the FA and EM into all the studied mixes. This improvement could be due to the lower calcium hydroxide content in the FA and EM concrete, making the concrete less vulnerable to the acid attack.

Fig. 12 displays the EDX elemental maps of the prepared concrete exposed to 10% of H₂SO₄. Fig. 13 shows the detected trace elements (Wt%) in the prepared concrete exposed to 10% of H₂SO₄ obtained from the EDX spectral analyses. Significant alterations in the microstructures and elemental compositions were occurred when exposed to the acid environment which was due to the leaching effect of the calcium and Al. The EDx maps showed the presence of main elements (Ca, S, and Si) in the concrete, verifying the existence of the gypsum and supported the XRD outcome. The results showed the decreasing trend of the calcium content with the inclusion of the FA and EM, indicating the reduction in the g amount of gypsum and ettringite. The total amount of Ca and S (main chemical composition of gypsum and ettringite) was decreased (Fig. 13) from 97.6% for the sample prepared with OPC to 95.9 and 82.4% for those made with FA and EM, respectively. It was argued that the deterioration mechanism is in direct correlation with the gypsum and ettringite content, implying the lower deterioration of the concrete that contained FA and EM compared to the control specimen (OPC concrete). This finding agreed with the previous results [68,70,71] that showed the generation of more gypsum and ettringite with the increasing content of the calcium and thus low durable performance against the acid environments.

4.4. Sulphate attack

Fig. 14 depicts the strength loss of the prepared concretes after exposed to 10% of MgSO₄. Three batches of the concretes included with OPC, FA, and EM exposed to sulphate environment were tested. The strength performance of the specimens was evaluated after 180 and 365 days. With increasing exposure time, the deterioration and loss in strength for all specimens were increased. The concretes subjected to the MgSO₄ solution for 180 days showed a decrease in the strength loss from 22.9% with OPC specimens to 8.9 and 8.2% with FA and EM specimens, respectively. Similar trend was observed for specimens exposed for 365 days where the loss in strength was dropped from 59.4% for the OPC concrete to 28.1 and 27.8% with included FA and EM, respectively. This was due to the chemical binding of the FA and EM with Ca(OH)₂ and formation of the C-(A)-S-H gels, thereby making it unavailable to the gypsum, sulphate and ettringite and reducing the permeability of the spec-

Table 5
XRD peak analysis of concretes after 365 days of immersion in 10% H₂SO₄ solution.

Index	Amount, weight %				
	Gypsum	Ettringite	Portlandite	Quartz	Others
OPC concrete	24.1	33.3	13.8	27.7	1.1
10% FA concrete	14.8	28.8	21.2	33.3	1.9
10% FA and EM concrete	12.5	26.7	20.9	37.3	2.6

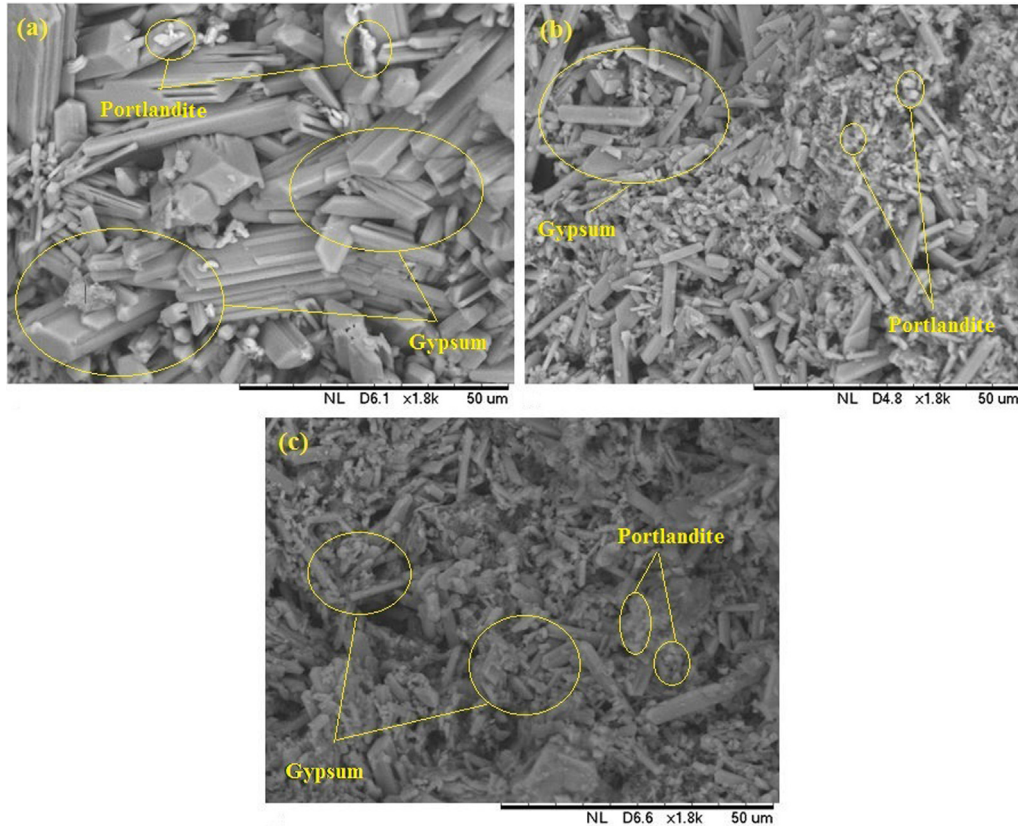


Fig. 11. SEM images of prepared concrete mixtures (a) OPC (b) 10% FA and (c) 10% FA + EM after exposure to 10% H₂SO₄ solution.

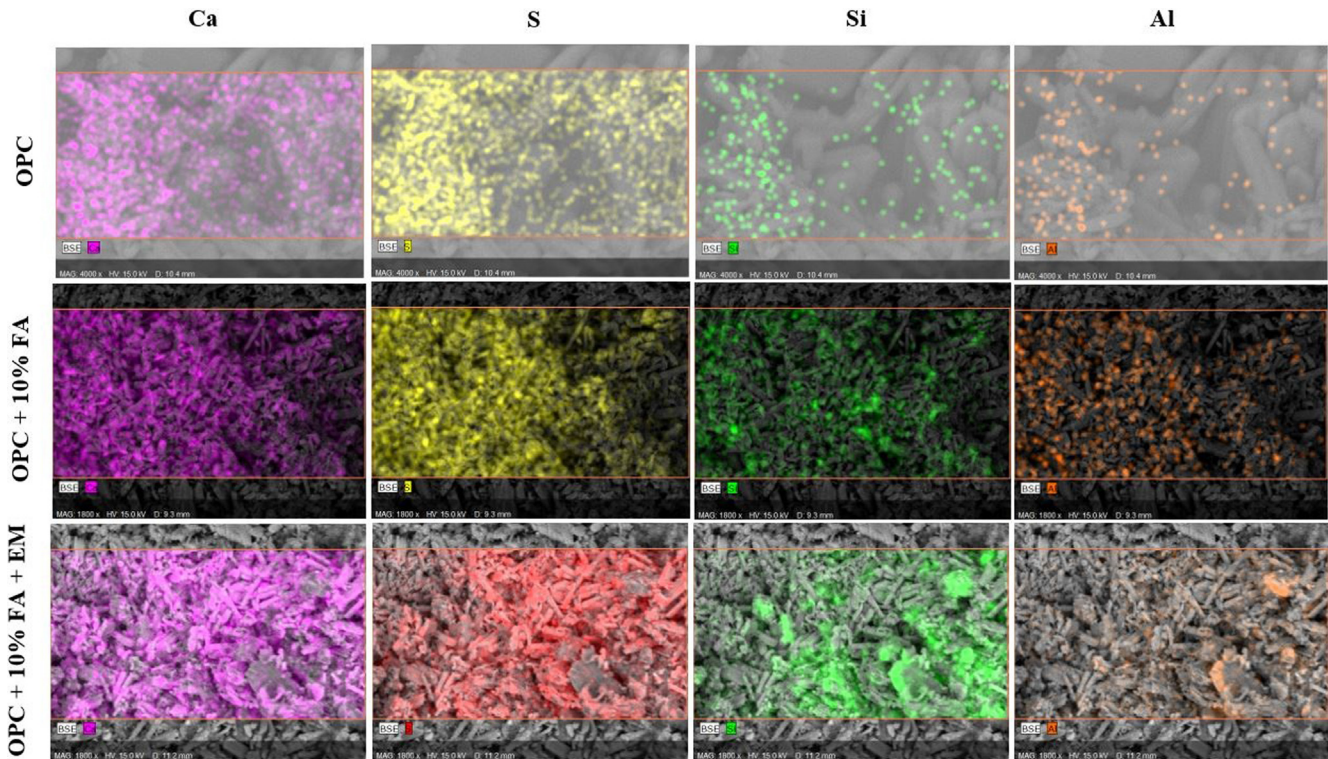


Fig. 12. EDX elemental maps of the prepared concretes exposed to 10% H₂SO₄ solution.

imen. Consequently, less number of sulphate ions was penetrated into the FA and EM embedded concretes [64,72–75]. Several stud-

ies [76–78] have reported two types of sulphate attack including the reaction with the (i) alumina bearing hydration products

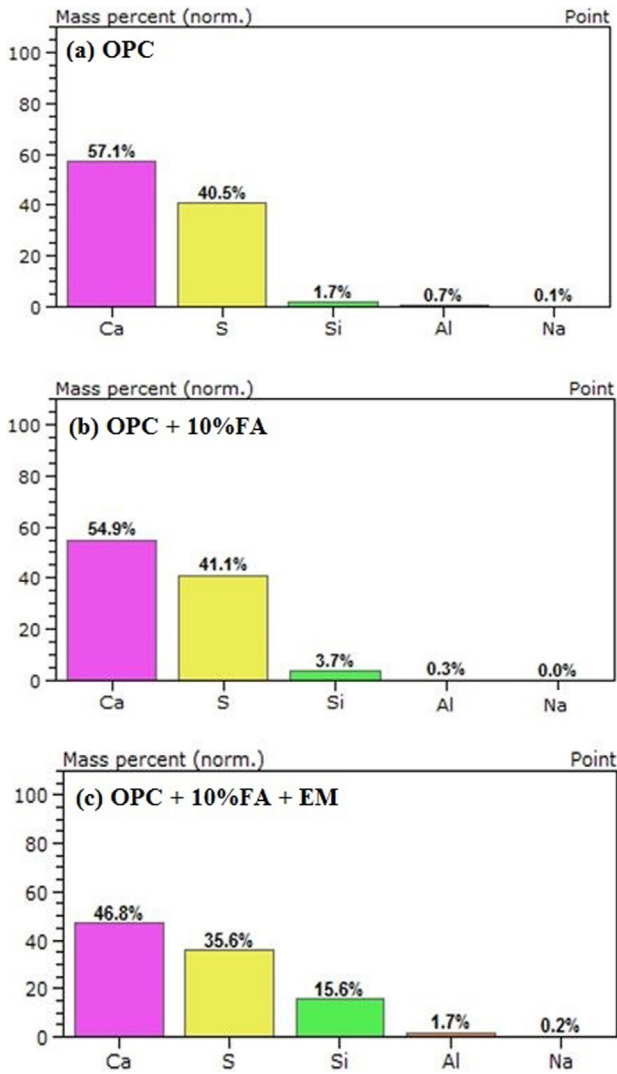


Fig. 13. Detected trace elements (Wt%) in the prepared concretes exposed to 10% H₂SO₄ solution obtained from EDX spectral analyses.

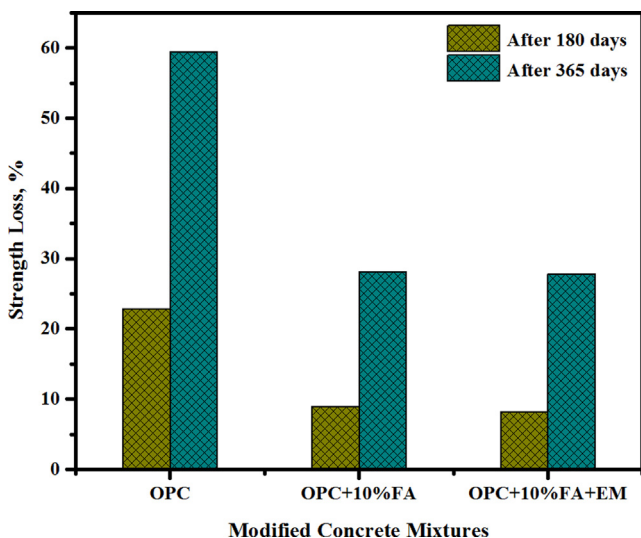


Fig. 14. Strength loss of prepared concrete mixtures after exposure to 10% MgSO₄ solution.

and/or non-hydrated tricalcium aluminate (C₃A) to produce ettringite and (ii) portlandite to produce gypsum. A reduction in the Ca (OH)₂ caused an enhancement in the durability of the concrete during sulphate attack and thus limiting the formation of the gypsum and secondary ettringite [76,79].

Fig. 15 shows the weight loss of the prepared concretes after exposed to 10% of MgSO₄. The weight loss of the concretes was assessed after 180 and 365 days of immersion into the sulphate solution. The inclusion of FA and EM in the OPC matrix showed positive effect and improved the durability performance. However, the deterioration and loss in the weight for all prepared specimens were found inversely proportional to the exposure period. After 180 days of exposure to solution, sharp deterioration and weight loss was evidenced that amounts to 4.9% (OPC) to 1.44 and 1.37% with FA and EM concrete specimens, respectively. The formulation of the gypsum and ettringite led to more expansion that negatively affected the bond zone between the paste and aggregates, resulting in a higher spalling and degradation of the concretes [63–65].

Fig. 16 shows the UPV of the prepared concretes after exposed to 10% of MgSO₄. The UPV of the concretes was measured after 180 and 365 days of immersion into the sulphate solution. Specimen added with FA and EM in the OPC concrete matrix showed an improvement in the concrete durability when exposed to the sulphate solution. The deterioration and internal cracks were reduced due to the inclusion of the FA and EM where the UPV was increased from 3871 and 3896 m/s compared to the 2955 m/s for OPC concrete tested after 180 days. Likewise, the internal deterioration trend was reduced due to the inclusion of the FA and EM in the concrete matrix after exposing in the solution for 365 days. The UPV value was increased from 2488 m/s to 3469 and 3488 m/s due to the addition of the FA and EM into the concrete matrix. Many studies [80–82] reported that the deterioration can cause strength loss, expansion, spalling of surface layers and eventual disintegration of the concrete. The sulphate attack was primarily ascribed to the generation of the ettringite and gypsum accompanied by the expansion and softening of the concretes.

Fig. 17 displays the surface deterioration of the OPC, FA and EM included concretes after 365 days of the exposure to the sulphate solution, where the OPC concrete clearly showed a physical degradation. Conversely, through close naked eye view the minimal physical alteration was observed in the FA and EM included concretes as than the control sample. The concretes made with the

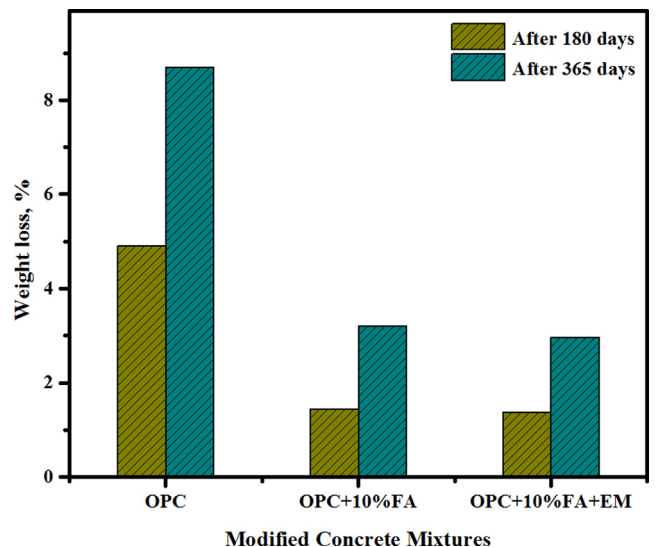


Fig. 15. Weight loss of prepared concrete mixtures after exposure to 10% MgSO₄ solution.

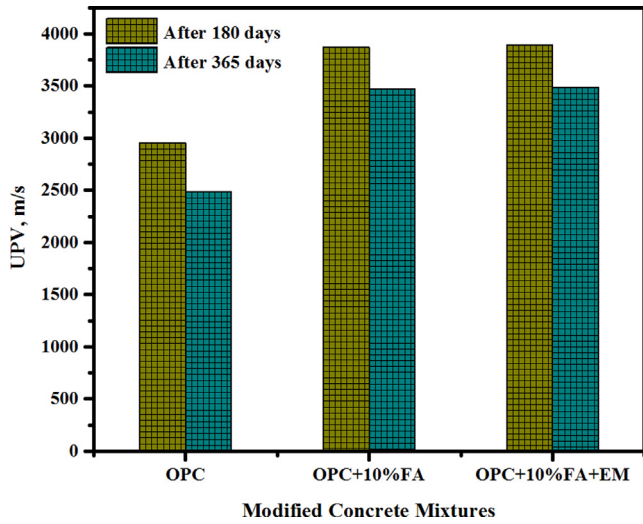


Fig. 16. UPV of prepared concretes exposed to 10% MgSO₄ solution.

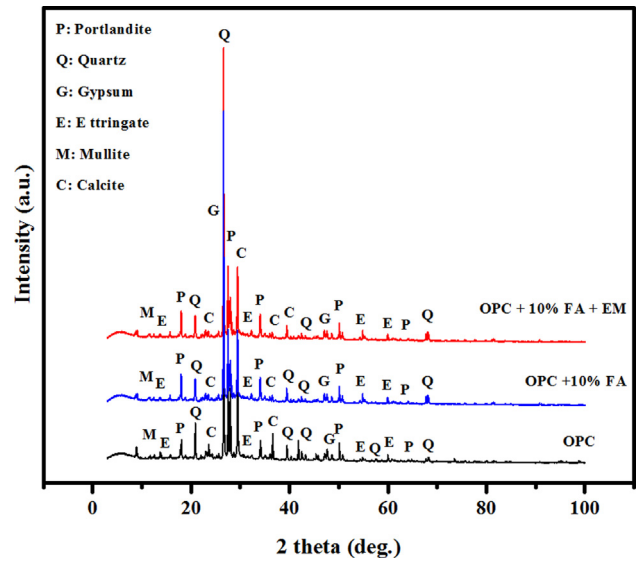


Fig. 18. XRD of prepared concretes exposed to 10% MgSO₄ solution.

inclusion of the FA and EM did not show any appreciable physical changes and retained the structural integrity largely except some minor erosion at the corners and nearby edges. The observed reduction in the Ca(OH)₂ for the FA and EM embedded and limiting the gypsum and secondary ettringite formation [76,79].

Fig. 18 illustrates the XRD pattern of the studied concretes after the exposure to the 10% of MgSO₄ solution for 365 days. The sulphate environment exposure led to the production of gypsum and ettringite with high intensity in the OPC specimens compared to those prepared with FA and EM. Meanwhile, the intensity of the peaks corresponding to the ettringite and gypsum were increased in the OPC specimen, indicating a better performance of the FA and EM included concrete against the sulphate environment. Furthermore, the XRD data revealed a steady increase in the presence of gypsum for the OPC specimen, which is also known to have expansive effect upon formation. Conversely, the XRD analyses

showed the minimal change in the presence of the ettringite or gypsum for the concrete prepared with FA and EM after immersion in the sulphate solution. This observation was ascribed to the lower amount of Ca(OH)₂ existence in the FA and EM included specimen that consumed by the hydration products to form an extra C-(A)-S-H gels, thus reducing the gypsum and ettringite formation.

Fig. 19 displays the SEM images of studied concretes after 365 days exposure to the sulphate solution. The results revealed the substantial structural deterioration of the OPC specimen and generation of high amount of gypsum and ettringite (Fig. 19a). However, the rate of deterioration of the FA and EM included specimen was significantly lower than the others. It was argued that the low amount of portlandite presence in the matrix containing FA and EM could restrict the gypsum and ettringite formation,

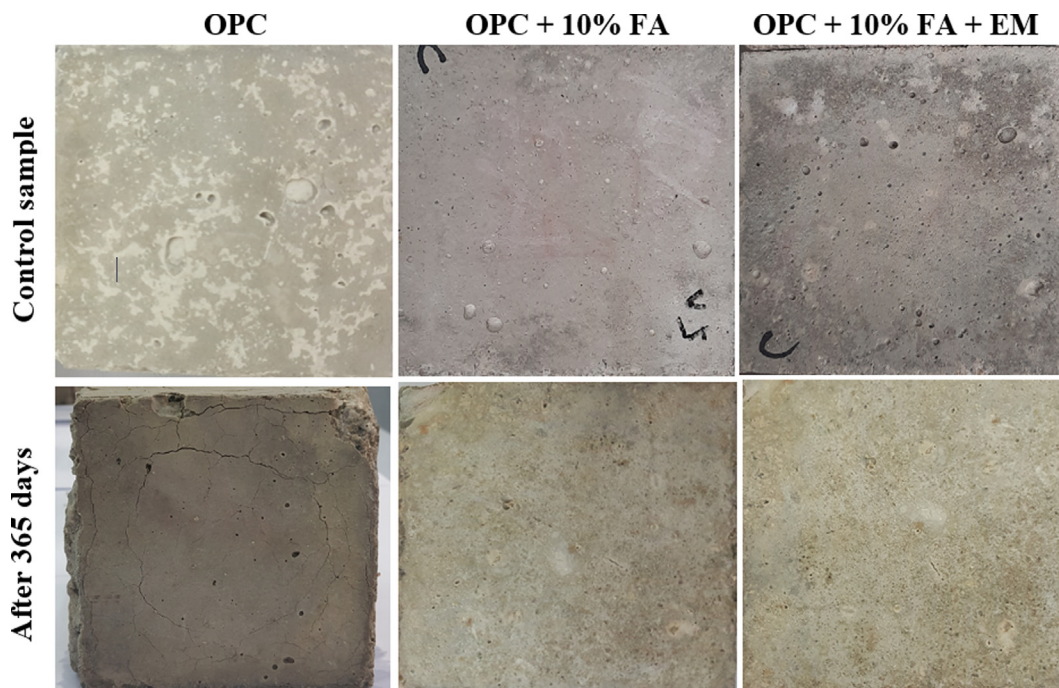


Fig. 17. Physical appearance of prepared concretes after exposure to 10% MgSO₄ solution.

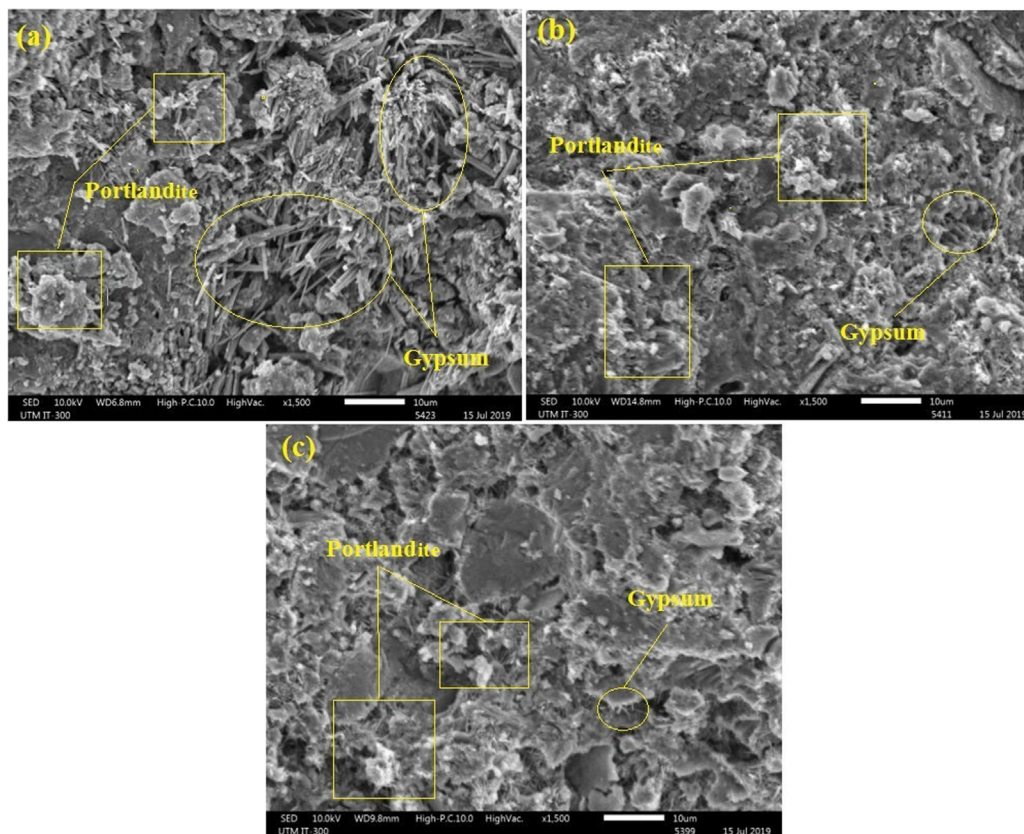


Fig. 19. SEM images of prepared concrete after exposure to 10% MgSO_4 solution (a) OPC (b) 10% FA and (c) 10% FA + EM.

thereby causing less structural deterioration (Fig. 19b and c). Similar trend was also reported by several researchers [76,79], where the deterioration degree was shown to be directly correlated with the gypsum and ettringite contents.

5. Conclusions

This study emphasized the feasibility of using EM with FA as OPC replacement to produce sustainable concretes with improved performance. The systematic studies for the specimen preparation, characterization and analyses of the experimental results allowed drawing the following conclusion:

1. Inclusion of the EM and FA in the OPC concrete matrix led to an improvement in the strength performance of the synthesized specimens.
2. The presence of EM helped to reduce the heat hydration and water loss of the FA-OPC concrete, thereby enhancing the early compressive strength up to 30%.
3. The replacement of the OPC by FA and EM yield modified concrete with high performance against the aggressive environments. The measured compressive strength, ultrasonic pulse velocity and weight loss of the studied modified concretes showed an enhancement in the resistance against sulfuric acid attack due to the inclusion of the FA and EM in the OPC concrete matrix. This outperforming nature of the modified concrete was ascribed to the formation of the high amount of stable gels (C-(A)-S-H), contributing to the enhanced durability.
4. The XRD, SEM and EDX analyses of the microstructures revealed the presence of low amount of free $\text{Ca}(\text{OH})_2$ in the FA and EM incorporated specimen, restricting the gypsum and ettringite formulation and improving the durability of the concrete.

5. The resistance of the concrete against sulphate attack was improved due to the inclusion of the FA and EM in the OPC concrete matrix.
6. It is established that the use of the EM in the construction industry may provide some sustainable materials with environmental friendliness, thereby lessening the dependence on the conventional OPC based polluting concretes.

CRediT authorship contribution statement

Ghasan Fahim Huseien: Methodology, Supervision, Writing - original draft. **Zahraa Hussein Joudah:** Supervision, Writing - review & editing. **Nur Hafizah A. Khalid:** Supervision, Writing - review & editing. **Abdul Rahman Mohd Sam:** Supervision, Writing - review & editing. **Mahmood Md. Tahir:** Supervision. **Nor Hasanah Abdul Shukor Lim:** Supervision. **Rayed Alyousef:** Supervision. **Jahangir Mirza:** Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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