



Compressive strength and microstructure properties of modified concrete incorporated effective microorganism and fly ash

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ABSTRACT

With the growing global demands for more sustainable and environmental-friendly construction materials with lesser carbon emissions, some concrete mixtures were modified by incorporating an optimum ratio of effective microorganisms (EMs) and fly ash (FA) and the novel concrete product mixtures were served as an alternative for the ordinary Portland cement (OPC). In this paper, the compressive strength performance, and microstructures of the designed modified concrete materials were evaluated. Four different ratios of EM and FA were selected and evaluated before determining the optimum composition for the conventional OPC. Besides, EMs solution with a ratio of 5, 10, 15, and 20% was added to replace the water content in the OPC. From the study, the addition of EMs and FA components were found to improve the strength and microstructure features of the proposed concretes. Under the presence of FA and 10% EMs, the initial compressive strength (CS) of the proposed concrete was found to be improved by 30%, and the microstructure was observed to be enhanced considerably due to the replacement of OPC with FA and EMs contents. The detailed microstructures analyses (using XRD, EDX, SEM, TGA and DTG) of the FA and EM incorporated concretes displayed the formation of high amount of the $\text{Ca}(\text{OH})_2$ and C-S-H gel, reduction in the pores, improvement in the strength performance and enhancement in the structure morphology. From this study, the concrete materials prepared from FA and 10% EM solution can be regarded as an environmental-friendly alternative with fewer contributions toward global warming. As concrete is the main building material used extensively in many construction industries, the present disclosure may alleviate the FA-waste-based landfill requirement considerably.

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1. Introduction

Green concretes emerged as prospective alternative to conventional concrete wherein diverse waste materials have been converted as valuable spin-offs [1–6]. Nowadays, EM solutions have been presented as a novel materials based bio-concrete and its applications can be used in numerous construction industries [7,8]. Many previous researchers have demonstrated the application of EMs as an effective biological strategy in developing and designing novel self-healing concrete products [9,10]. The EMs

are available abundantly in the natural environment like soil, water and oil reservoirs, acidic hot springs, and wastewater effluents discharged from different industries. It can be served as a potential candidate for the development of novel self-healing bio-concrete products [11,12]. As a whole, EM solutions can be categorized into three different classes of bacteria, fungi, and viruses. Among the EM solution, some special bacterial strains possess the ability to precipitate specific chemical or elemental compounds, which could be beneficial for the development of self-healing bio-concrete products. Furthermore, EM solution can be added into the bio-concrete products by directly incorporate into the pristine concretes, incorporating it as spores, immobilizing it onto a silica gel or activated carbon, encapsulating it, or through vascular

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networks followed by a chemical route for a homogenous microorganism distribution [9,13].

According to Isa et al. [14], the addition of 3% EMs was found to improve the CS performance of concrete materials by more than 14% after 28 days. Furthermore, the concrete pores were partially filled with *Bacillus subtilis* bacterial strains, which were derived from the locally produced EMs. The bacterial strains were introduced into the concrete pores to reduce the pore sizes and promote the CS performance. On the contrary, it was claimed that the time-consuming bacterial growth process did not offer a significant early effect on the concrete materials due to absence of urea hydrolysis [15]. Furthermore, it was also reported that the bacteria did not improve the concrete strength due to the absence of food for the EMs [16]. In another study of Sato, Higa [17], the CS of the concrete materials was reported to be increased by 10% to 15% in contrast to the control samples [18]. 5% EMs was applied to replace the water content in the concrete material and was found to be optimum in improving the concrete strength. The CS of the EMs-based concrete was determined as 43.17 MPa, which was 143.90% higher than that of the pristine concrete. Furthermore, the EM-based concrete demonstrated a final strength of 54% on the first day, which highlighted its potential application when early concrete strength is required. Besides, the EMs-based concrete demonstrated a better tensile and flexural strength. In the same vein, Rizwan et al. [19] designed an EMs-based concrete with a self-compacting feature by replacing the water content with 5%, 10%, and 15% of EMs. It was demonstrated that the presence of EM solution could affect the viscosity of plastic, delay the setting times of plastic, alter the hydration kinetics, disrupt the volume stability, decrease the water absorption ability of the self-compacting paste system under hardened condition, and lastly improve the CS of the EMs-based concrete. With its improved workability, CS, and durability performance, the modified concrete can be applied for several structural applications in many construction industries. With the numerous benefits demonstrated by the EMs-based concretes, this study aimed to design and develop a novel sustainable concrete product with enhanced early age strength by blending EMs and FA into the conventional concretes. In addition, the effects of different EMs and FA content on the mechanical and microstructure properties were assessed.

2. Methodology

2.1. Materials

Different resource materials like cement (OPC), wastes fly ash (FA), and EM solution were used to compose the modified concretes. OPC was obtained from a local cement manufacturer and used as the main source based hydration process. Waste material (FA) was acquired from the south Malaysia, and utilized as the primary source of aluminum silicate (Al_2SiO_3) without any purifica-

Table 1
Effect of water content on EM pH, viscosity and surface tension properties.

Type of solution	pH	Viscosity, mPas	Surface tension, mN/m
H ₂ O	6.7	0.95	66
EM solution	3.5	1.5	36
EM 5%	6.4	1.0	58
EM 10%	6.3	1.05	55
EM 15%	5.7	1.1	52
EM 20%	5.2	1.2	45
EM 25%	4.0	1.3	40

tion. X-ray fluorescence spectroscopy (XRF) was used to analyze the OPC and FA' chemical compositions. The quantities of Al_2SiO_3 in OPC and FA were determined as 88% and 91.2%, respectively. On the contrary, OPC exhibited a higher CaO content of 62.4% than FA (5.16%). For the production of concrete, the formation of dense gels during the hydration reaction can be affected significantly by the quantity of aluminum (Al), silicate (SiO), and CaO. Besides, it was found out that all three samples possessed a K_2O content of less than 1%. On the other hand, the iron oxide (Fe_2O_3) content was higher in OPC (4.19%) as compared to FA (3.67%).

5 kg of EMs with type EM-1 was collected from Peladang, which was located at Johor Bahru, Malaysia. The collected EMs were stored in an air-tight container. Initially, the EMs were activated by adding a mixture of water and blackstrap molasses, which served as the primary food source for the microorganisms. The activation process involved anaerobic fermentation for 7 to 10 days in order to achieve a pH level of less than 4. The resulting mixture was named EM activated solution (EM-AS). Following that, the EM-1 solution was prepared by mixing 90% water, 5% EM-1, and 5% molasses before used to prepare the concrete. The physical appearance and solubility of the EMs before and after mixed with water were compared to ascertain the color differences between fresh and hardened concrete. It was revealed that the initial color of the pristine EMs was dark brown and changed to dark yellowish when 10% of water was added. The color of EMs subsequently changed to light brown when 25% of water was added to replace the EMs. Overall, uniformity was observed in all mixed solution.

The pH of EM solution was measured using pH meter (Mi 151pH). According to ASTM D455, the viscosity of EM solution was determined using digital Brookfield viscometer. The surface tension of solution was measured according ASTM D971 using tension-meter (Kruss Germany easdyne model). The surface tension of a solution plays a key role in affecting the properties of fresh and hardened concretes. Table 1 shows the surface tension of water, EMs, and mixed solution with 5, 10, 15, 20, and 25% water weight. From Table 1, it was observed that the surface tension of the original EM solution was 36 mN/m, which was lower than the surface tension of water (66 mN/m). On the other hand, the surface tensions of the mixed solution with 5, 10, 15, 20, and 25% water weight were 58 mN/m, 55 mN/m, 52 mN/m, 45 mN/m,

Table 2
Proposed FA mix design with various FA and EM solution content.

Proposed concrete mix	Binder, kg/m ³		Solution, kg/m ³		Aggregates, kg/m ³	
	OPC	FA	Water	EM solution	River sand	Crushed stone
M1-OPC	450	0	247.5	0	875	815
M2-10%FA	405	45	247.5	0	875	815
M3-20%FA	360	90	247.5	0	875	815
M4-30%FA	315	135	247.5	0	875	815
M5-40%FA	270	180	247.5	0	875	815
M6-5%EM	450	0	237.5	12.5	875	815
M7-10%EM	450	0	225	25	875	815
M8-15%EM	450	0	212.5	37.5	875	815
M9-20%EM	450	0	200	50	875	815
M10-10%(FA-EM)	405	45	225	10	875	815

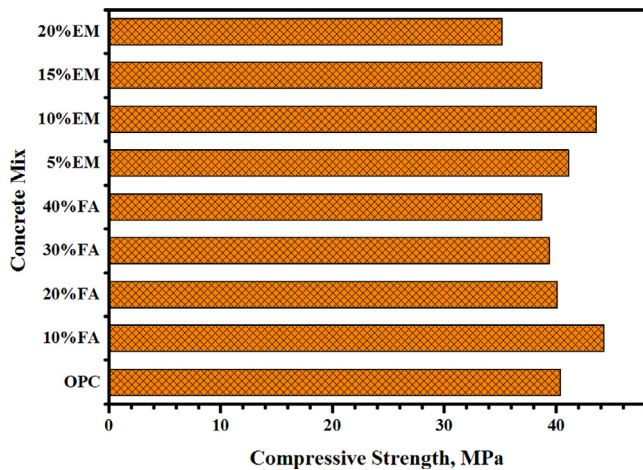


Fig. 1. Compressive strength of modified concrete at 28 days of curing age.

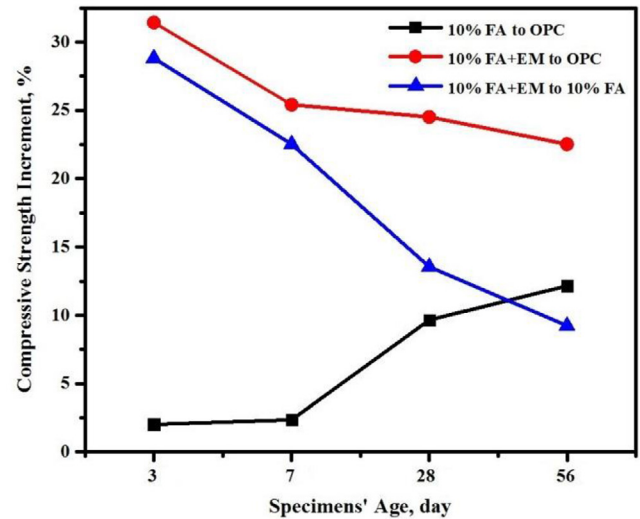


Fig. 3. Effect of FA and EM solution on CS increment percentage of various ages.

m, and 40 mN/m, respectively, which was lower than that of fresh-water. The surface tension of a liquid is defined as the attractive force that acts at the interface between two different phases like liquid–solid, liquid–vapor, and liquid–gas. The interfacial tension was nearly similar to the surface tension in respect to the cohesive forces.

It is important to consider the mixing design of the concrete in order to produce proposed concrete products with desirable properties. The parameters such as compressive strength (CS) can be changed through the design process. The proportions of the proposed concrete mixture illustrated in Table 2. In this study, the design of the mix proportions was adopted from the guide proposed by Marsh. The control mix was optimized to obtain the

desired compressive strength of 30 MPa after 28 days of curing age. Next, the OPC in the control mix was replaced successively by 10, 20, 30, and 40 wt% of FA. The main emphasis of this paper was to assess the effect of EM solution on the properties of modified concrete (10% of water ratio). For a better correlation between the addition of EMs and the concrete properties, FA ratio with 10% and onwards was selected for the compressive strength and other tests. For all concrete mixtures, water to binder materials ratio of 0.55 was applied. Other than the FA and cement proportion, all parameters were maintained constant to facilitate a better understanding of the direct effects of FA on the concrete properties. Different percentages of FA admixture was added to the concrete mix and replace the cement content. To obtain the optimum FA percentage, a total of 15 cubes with different percents of FA (10, 20, 30, and 40%) were prepared. The dimensions of the mold used were (100 × 100 × 100) mm. Lastly, the CS of the cubes were evaluated to select the FA' optimum percentage admixture for the next specimen preparation stage.

The CS tests were conducted following the ASTM C109, where three different samples were evaluated under the ages of 3, 7, 28, and 56 days. Each concrete specimen was accurately placed between the upper and lower metal bearing plates. Each specimen was subjected to a load of 2.5 kN/s. At the curing age of 28 days, the central portion of every specimen was taken and pulverized. The microstructures of the synthesized powders were examined via SEM, EDX, TGA, DTG, and XRD measurements. For the XRD analysis, the Match3 and MDI Jade software version 6.5 were used to verify the glassy nature of the specimens. The samples were scanned between the 2θ range of 5 and 90° with a step size of 0.02°. For the SEM imaging, each sample was placed in a brass stub type sample holder and dried for 5 min using the IR radiation and the samples were covered with gold by using a Blazer sputter coater. The resulting patterns were monitored using 20 kV under a magnification of 1000×.

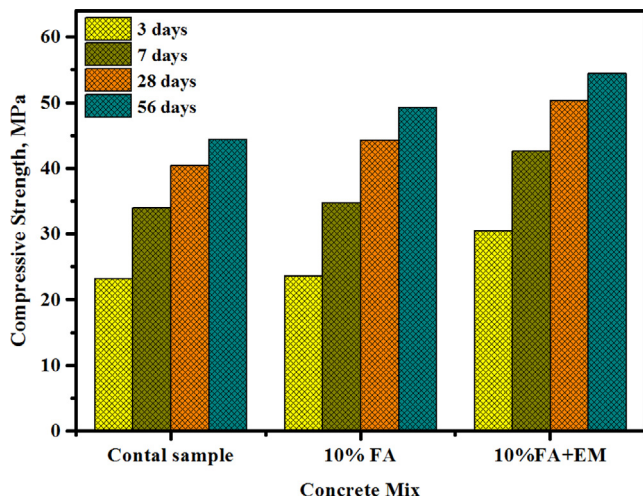


Fig. 2. Impact of FA and EM solution content on strength development of proposed concrete.

Table 3 Modified concrete water loss, internal temperature and pH at various curing age.

Mix	Water loss,%		Internal temperature, °C				pH			
	3 days	7 days	0.5 day	1 day	3 days	7 days	1 day	3 days	7 days	28 days
M1-OPC	1.5	1.9	35.1	32.3	31.1	29.6	12.9	13.1	13.2	13.2
M2-10%FA	1.3	1.6	34.9	32.1	30.8	29.3	12.6	12.8	12.9	13.2
M10-10%(FA-EM)	0.9	1.2	33.9	31.8	30.1	29.2	12.5	12.6	12.8	13.1

Table 4
The XRD peaks assignments for different samples.

Index	OPC	10% FA	EM
Portlandite	14.2	11.2	23.2
Quartz	69.3	75.1	67.5
Calcite	9.5	13.6	8.1
Ettringite	7	0.20	1.2

3. Results and discussion

3.1. Compressive strength (CS)

The CS associated with the trial mixtures, including standard concrete, concrete with OPC substitution by FA, and EM concrete is shown in Fig. 1. To establish the ideal FA and EM for the preparation of concrete with high performance, every concrete sample was subjected to testing after 28 days of curing. The concrete samples with OPC content were chosen as the control samples, 40.4 MPa at 28 days. Meanwhile, in the case of samples in which OPC was substituted with FA, the rise in the proportion of FA from 0 to 10% caused an increase in CS at 28 days. However, additional FA increase led to a decrease in concrete CS. after 28 days of curing FA proportion of 10% was associated with CS of 44.3 MPa, but higher proportions of FA caused a decline in CS to 40.1 MPa (20% FA), 39.4 MPa (30% FA), and 38.7 MPa (40% FA). The CS increase in samples containing 10% FA was explained in term of the development of additional calcium silicate hydrate (C-S-H) [20–25]. In the case of concrete samples with EM content, CS was increased at 28 days when water was substituted with EM in proportion of 5 and 10%. By contrast, strength was lower than the control when substitution was elevated to 15 and 20%. At 28 days, strength increased to 40.4, 41.1, and 43.6 MPa for 0, 5, and 10% EM, respectively, before decreasing to 38.7 and 35.2 MPa for 15 and 20% EM, respectively. The optimum ratio of FA (10%) and EM (10%) are selected to design the mix of high performance concrete.

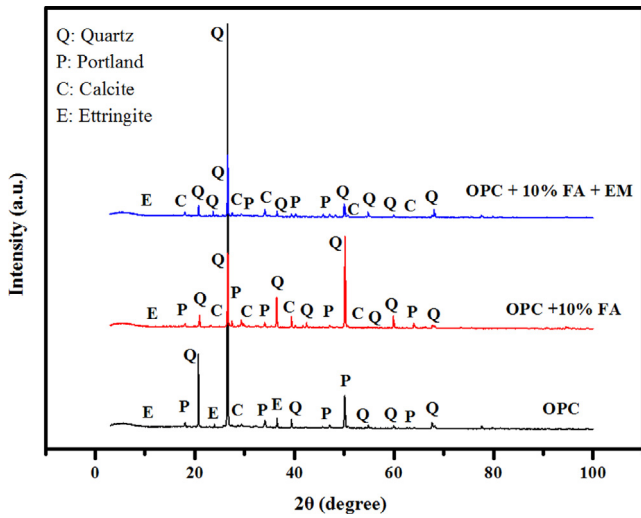


Fig. 4. Proposed concrete XRD analysis results.

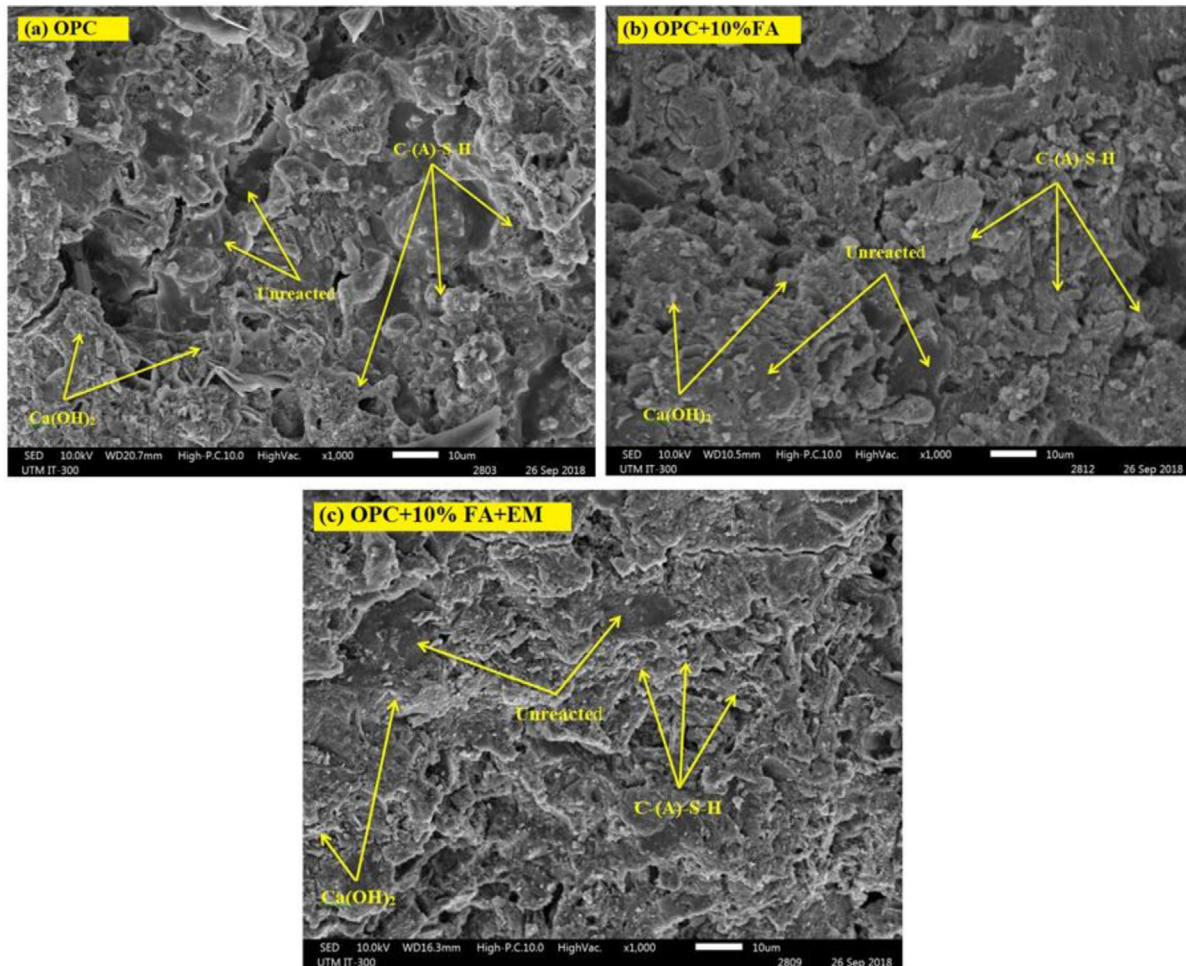


Fig. 5. SEM micrographs of the modified concretes.

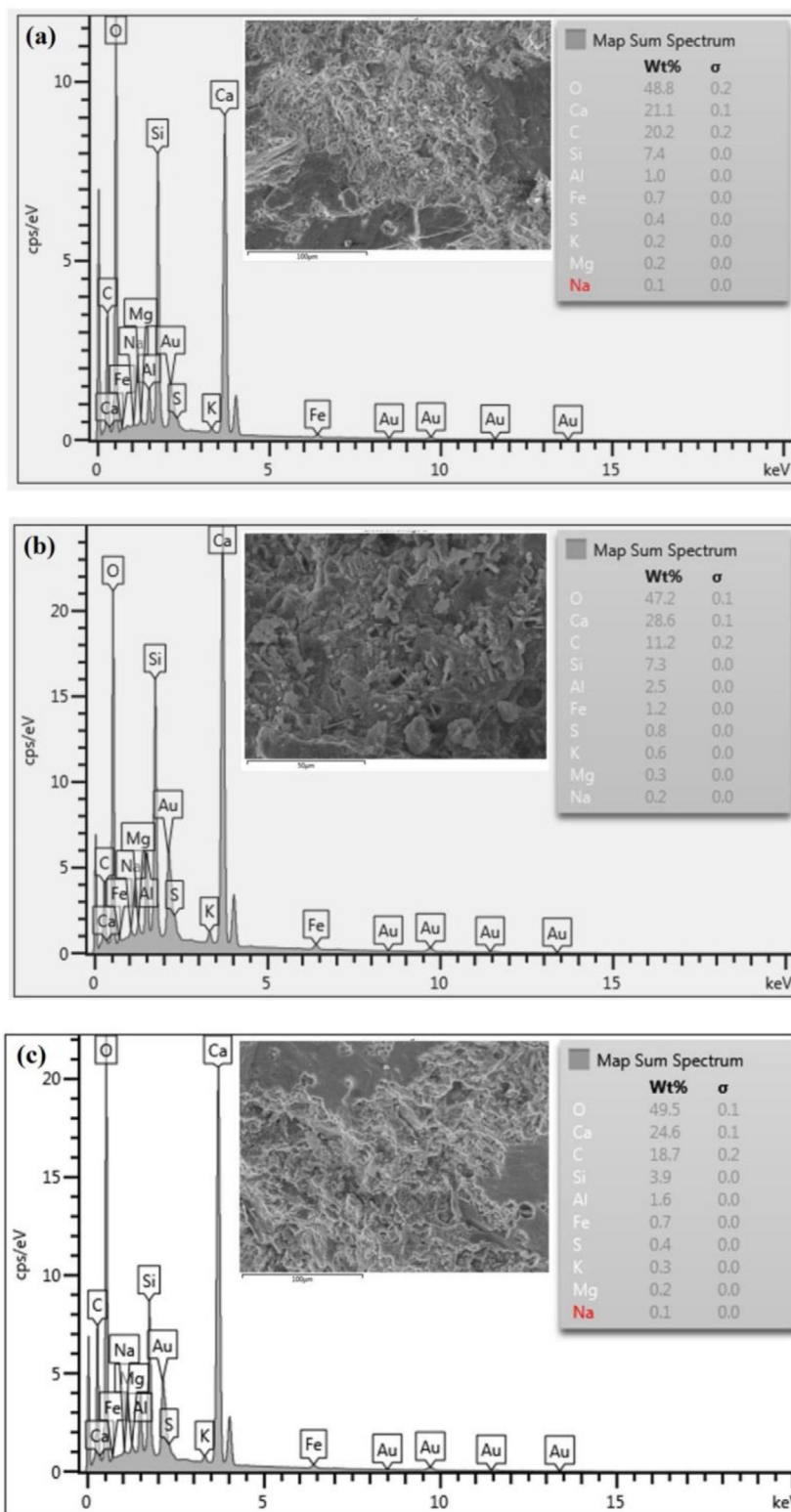


Fig. 6. EDX spectra of concretes made with (a) OPC (b) 10% of FA and (c) 10% of FA + EM.

Fig. 2 shows the CS of the concrete specimens modified with FA and EMs. The influence of early strength performances by FA and EM solution content were investigated by measuring the CS of concrete specimens at 3, 7, 28, and 56 days. From the results, the concrete specimens with FA and EMs generally demonstrated a better CS performance than that of without FA and EMs. The OPC matrix

with blended FA and EMs was found to increase the CS performance up to 30.5 MPa at the early age of 3 days, which was significantly higher than the pristine OPC (23.2%) and 10% FA (23.7%). Besides, the FA and EMs-based specimens achieved the highest CS of 42.49 MPa as compared to pristine OPC (33.9 MPa) and FA (34.8 MPa). At the middle age of 28 days, the

Table 5
Elemental composition in the studied concretes obtained from the EDX spectral analyses.

Index	OPC	10% FA	EM
SiO ₂	7.4	7.3	3.9
CaO	21.1	28.6	24.6
Al ₂ O ₃	1.0	2.5	1.6
Fe ₂ O ₃	0.7	1.2	0.7
CaO:SiO ₂	2.85	3.92	6.31
SiO ₂ :Al ₂ O ₃	7.4	2.92	2.44

CS performances of 10% FA and EMs-based concrete specimen were found to be improved from 40.4 MPa to 44.3 MPa and 50.3 MPa, respectively. A similar trend was also observed in the concrete specimens prepared from 10% FA and EMs after 56 days of curing age. Besides, the OPC modified materials possessed a higher CS value than the pristine OPC (44.4 MPa) and 10% FA concrete specimen (49.3 MPa). When the OPC was replaced by FA, a decrease in the capillary pores and an increase in the dense of gels were also reported by Nedunuri et al. [26]. The latter phenomena could be attributed to the product of additional dense gel via pozzolanic reactions, which in turned enriched the C-S-H gel content and enhanced the porosity and CS performance.

Fig. 3 presents the effect of FA and EM content on the CS increment percentage. From the findings, a lower rate of strength development was observed from the concrete specimen with 10% FA at 28 and 56 days of curing age. For the specimen containing 10% FA, the rate of strength development was increased by 2.0, 2.4, 9.7, and 12.2 after 3, 7, 28, and 56 days, respectively. Such observation is in good agreement with the typical characteristics of the pozzolanic materials, which usually have significant impacts on the hydration and strength development stages during late age [27]. Furthermore, Fraay et al. [28] observed that the pozzolanic reaction in the concrete specimen only occurred after 7 days of curing age. However, the addition of EM into the concrete matrix was found to reduce considerably over the curing time despite a high CS performance was exhibited at an early age. As compared to the pristine OPC specimens, the concrete specimen with FA and EMs demonstrated enhanced CS performance up to 31.4, 25.4, 24.5, and 22.3% after 3, 7, 28, and 56 days of curing age, respectively.

It is well-known that the reduction of water loss in the concrete matrix can promote the formation of hydrated products such as Portlandite (Ca(OH)₂) and dense gels (C-(A)-S-H). As presented in Table 3 the addition of FA and EM reduced water loss from 1.5 to 1.3 and 0.9%, respectively. Several studies [29–32] have reported that the reduction of the water loss, as the result of a lower hydration heat and evaporation performance, could reduce the voids, capillary stress, and internal cracks that affect the strength development of the concrete positively. Furthermore, the addition of EM solution and FA into the OPC matrix could reduce the amount of hydration heat, which provides a proper environment to increase the OH⁻ contents and the pH up to 12.5, thus leading to the formation of more gel products (Table 4). These results clearly explained the benefits of EMs to improve the early CS.

3.2. X-ray diffraction patterns (XRD)

Fig. 4 presents the XRD results of the control sample (OPC), modified concrete with FA and EM solution at 28 days of age. From Fig. 6, the sharp crystalline peaks of the OPC and FA specimens can be assigned to quartz (SiO₂), Portlandite (Ca(OH)₂), calcite (CaCO₃), and Ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26H₂O) phases. Besides, a higher intensity of the quartz peaks can be observed when the

FA content was increased from 0% to 10%, wherein the additional quartz became non-reactive in the 10% of FA specimen as compared to that of without quartz. Such products were produced from the reaction between the amorphous FA and highly crystalline OPC phases. Conversely, the presence of the broad and diffuse background peaks at approximately 10° can be attributed to the presence of Ettringite with short-range ordering. Portland and calcite (CaCO₃) peaks can be observed at around 18–65° in the samples without EMs. As the FA content was increased to 10%, the Portland at 18–29° became more intense and the intensity of the calcite peak at 31° was enhanced due to the addition of the FA. On the other hand, the quartz peak intensity was found to be decreased at the expense of a higher Portland peak intensity, which was mainly due to the incorporation of the EMs in the concrete matrix. Besides, it was observed that the Portland peak at 18° was replaced by a calcite peak when the OPC was replaced by 10% of FA and mixed with EMs.

Table 4 presents the XRD peak positions and assignments for the quartz, Portland, calcite, and Ettringite components present in each sample. From Table 8, the CS performance and microstructure of the concrete were improved with a higher loading of Portland and calcite. By replacing OPC with 10% of FA, the amount of calcite was found to be increased from 9.5% to 13.6%, and the CS performance was also improved from 44.4 MPa to 49.3 MPa. The specimens with 10% of FA and EM produced a higher amount of Portland products (23.2%) than the normal concrete (14.2%) and concrete with 10% FA (11.2%). An increase in the Portland product led to an enhancement in the strength from 44.4 and 49.3 MPa to 54.4 MPa. The XRD results demonstrated the prominent effects of EMs and FA on the formation of dense gel and the CS performance of the proposed concretes.

3.3. Scanning electronic image (SEM)

Fig. 5 shows the SEM micrographs of the concrete samples. From Fig. 5a, the microstructures of concrete samples with only OPC content (0% of FA and EM) showed partially reacted eco-spheres. On the other hand, a remarkable change in the microstructure can be observed when OPC was replaced by 10% of FA and EM. Generally, the microstructures of the OPC sample was less dense as compared to other specimens because the formation of C-S-H gel was reduced. When 10% of FA and EM were added to OPC, the microstructures of the concrete were enhanced with a much denser surface as shown in Fig. 5b and Fig. 5c. Samples with FA and FA/EM contents confirmed the presence of the gel structures. Besides, three main morphological features can be found and a very small amount of non-reacted particles was formed when FA and EM were added.

Fig. 6(a-c) illustrates the EDX spectra of the prepared concretes at 28 days of age. A high ratio of calcium to silica content (CaO: SiO₂ = 6.31) was obtained in the concrete specimen with EM. However, a reduction in the CaO content was observed when FA was added. The concrete made from pristine OPC and 10% of FA displayed a lower CaO: SiO₂ ratio of 2.85 and 3.92, respectively. Furthermore, the SiO₂:Al₂O₃ ratio was found to be increased from 2.44 to 2.92 when the EM content was reduced from 10% to 0%. This could be due to the effect of water substitution as compared to 7.4 for the normal concrete (Table 5). From the EDX spectra, the concrete specimen with EM content demonstrated a relatively lower SiO₂/Al₂O₃ ratio, which indicates that more Al ions were substituted in the C-(A)-S-H network. Besides, the CS performance was observed to be improved with a higher FA and EM contents from 0% to 10%. This can be explained by the increase of Al₂O₃ and CaO contents, and the decrease in the SiO₂ content, which led to the formation of more gel products as compared to the concrete material with only OPC.

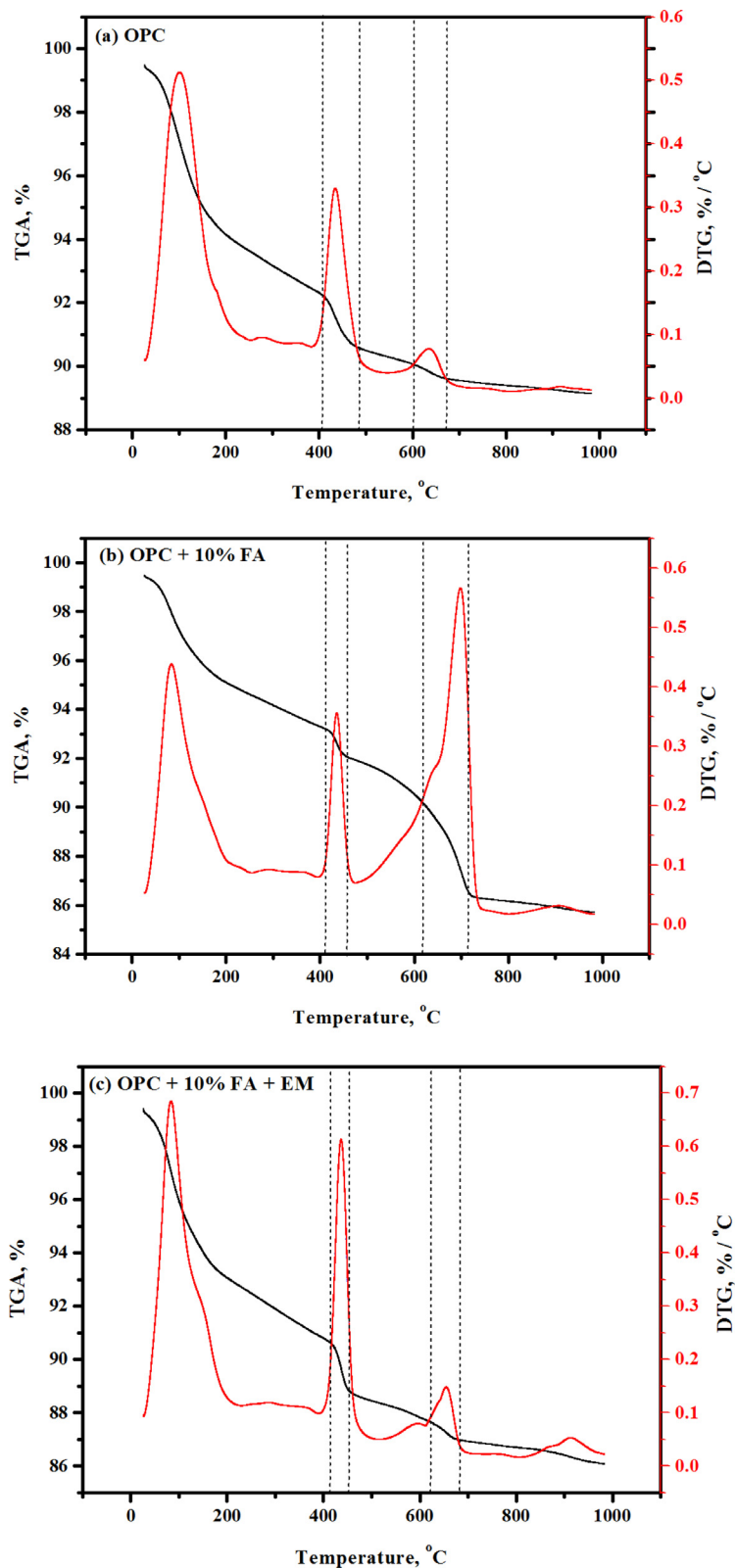


Fig. 7. The TGA and DTG curves of the concretes prepared with (a) OPC, (b) 10% of FA, and (c) 10% of FA + EM.

3.4. Thermal analysis (TGA and DTG)

Fig. 7 shows the TGA and DTG of the concrete specimens. The amount of $\text{Ca}(\text{OH})_2$ and calcium silicate hydrate present in the

sample was estimated by using equations (1) and (2), respectively. Generally, the OPC concretes possessed a lower weight loss (11.5%) as compared to 10% of FA and EMs (13.8%). From Table 7, the percentage of C-S-H gel in the EMs-based specimen (11.3%) was

Table 6
C-S-H gel and calcium hydroxide amount in different samples.

Index	OPC	10% FA	EM
C-S-H gel, %	8.85	9.03	11.3
Ca(OH) ₂ , %	6.57	4.27	7.39

significantly higher than the concretes prepared from OPC (8.85%) and 10% of FA (9.03%) as shown in Table 6. In addition, the percentage of Ca(OH)₂ was found to be the highest in the specimen made from EMs (7.39%) as compared to OPC (6.57%) and FA (4.27%). The high stability of the EMs-based concretes specimen revealed a high quantity of the C-S-H gels and Ca(OH)₂, which indicates that the addition of EMs can improve the microstructural characteristics and CS performances of modified concrete.

$$C - S - H_{gel}(\%) = TotalLOI - LOICH - LOICC \quad (1)$$

where LOI(CH) is the dehydration of Ca(OH)₂ in the range of 400–550 °C, and LOI(CC) is the loss of CO₂ in the range of 600–750 °C.

$$CH(\%) = WLCH(\%) \times [MWCH/MWH] \quad (2)$$

where WL(CH) is the weight loss due to the dehydration of CH, MW (CH) and MW(H) are the corresponding molecular weights of the CH (74 g.mol⁻¹) and water (18 g.mol⁻¹).

4. Conclusions

In this study, the feasibility of producing sustainable concretes with enhanced performance was investigated by replacing the OPC with EMs and FA. From the preparation, characterization, and analysis of the modified concrete specimens, the following conclusions were obtained: (1) Blending of EMs and FA components into the cement concrete matrix improved the strength performance of the concrete specimens. The addition of the EMs lowered the release of hydration heat and water loss in the FA-OPC concrete, which improved the early CS performance up to 29%. From the XRD, SEM, and TGA results, the microstructure of the concrete specimens with 10% of FA and EMs was enhanced as compared to the OPC specimens. A modified concrete with superior resistance against aggressive environments was obtained by replacing the OPC with FA and EMs contents. The superior properties of the modified concrete can be explained by the formation of a high amount of dense gel products in the matrix, which contributed to its enhanced durability properties. Several microstructure results such as XRD, SEM and TGA also confirmed the microstructures of the concrete specimens and a small quantity of free Ca(OH)₂ can be found in the FA and EM-based specimens. This has restricted the formation of gypsum and Ettringite, which enhanced the durability properties of the concrete. As a whole, the applications of EMs in the synthesis of sustainable concrete materials could alleviate the dependency of the construction industry on the highly polluting OPC concretes and serve as a sustainable alternative with fewer carbon emissions and more environmentally friendly features.

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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