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Microstructure and Strength Properties of Sustainable Concrete Using Effective Microorganisms as a Self-Curing Agent

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Abstract: In practical applications, problems related to proper curing arise for inclined structural elements, especially in skyscrapers, wherein concrete is very thick. To overcome this problem, the implementation of self-curing technology using varieties of smart materials has become significant. Based on these factors, this study determined the impact of effective microorganisms (EMs) as a new self-curing agent on the microstructures and strength properties of sustainable concrete. Five concrete mixtures were prepared with various EM content (5, 10, 15, 20, and 25%) as water replacement under air-curing condition. The workability of the concretes was found to increase with the increase in EM contents from 0 to 25%. In addition, concrete designed with 10% of EM achieved the highest compressive strength (42 MPa) after 28 days of aging as opposed to the control specimen (35 MPa). The microstructures of the concrete made with 10% of EM revealed very a compact network, fewer voids, and formulation of dense C-S-H gel. Based on the results, the proposed EM may be implemented as a self-curing agent to achieve high-performance sustainable concretes beneficial for the construction sectors.

Keywords: self-curing concrete; EM; strength development; microstructures

1. Introduction

Since its introduction, concrete has remained one of the primary construction materials and is widely exploited worldwide. Concrete is the soul of infrastructure for modern construction industries, which demand almost twelve billion tons of it annually [1–3]. Over the decades, concrete has been modified with different types of materials to improve its properties, which can be useful for various applications in the construction sectors [4–6]. In recent times, several self-curing systems have been developed. Two types of system have commonly been used in concrete technology. In the first one, water reservoir is placed within the concrete for its release to cure which in turn hardens the concrete, maintains a satisfactory moisture content and temperature [7], and increases the water retention capacity [8] called internal curing [9] that do not require any external water for curing after placing the concrete [10]. In the second system, a chemical admixture is used to retain the water and reduce the evaporation from concrete, this is called autogenous curing [11]. Initially, this type of concrete was called water-entrained concrete, and used lightweight aggregates [12,13]. Lately, these self-curing systems have generated renewed interest in the field of concrete engineering [14]. According to the ACI 308 committee, "internal curing refers to the process by which the hydration of cement occurs because of the availability of additional internal water that is not a part of the mixing water" [15].

Curing is the most important factor for meeting the life-cycle requirements of concretes [16,17] and maintaining the hydration as it hardens or sets [18]. Even if a mix contains enough water for hydration, the initial water-to-cement (*w*/*c*) ratio can be reduced due to any loss of moisture from concrete, leading to less cement hydration. Consequently, concrete develops unsatisfactory properties, such as poor compressive strength (CS), high porosity, and numerous porous zones around the aggregates [10]. Normal concrete contains enough water for the hydration of cementitious material, but not enough for proper curing due to moisture loss [19]. In short, the curing process strongly influences the properties of hardened concrete: proper curing has been shown to increase durability, strength, volume stability, abrasion resistance, impermeability, and resistance to freezing and thawing, as well as reducing shrinkage-related problems [20–22]. The traditional curing techniques are often ineffective in concrete that is composed of a low water/binder ratio. In dense concrete, water from outside cannot reach into the concretes' interior to complete the hydration reaction [23]. Furthermore, a continuous curing is difficult when concrete is used in heights, vertical members, sloped roofs, pavement, and in the thicker structures of tall buildings [24].

The first group of EMs were developed in 1980 and were distributed in the form of liquid concentrates. These EMs were generated using a normal fermentation procedure, not genetically engineered or chemically metabolized. An EM is a mixture of different advantageous microorganisms that occur naturally or are contained in foods. However, EMs can be made by cultivating over 80 different types of microorganisms in vats. These microorganisms can be derived from 10 genera belonging to 5 diverse families, including *Lactobacillus plantarum*, *Lactobacillus casei*, and *Streptoccus lactis* (lactic acid bacteria); *Rhodopseudomonas palustrus* and *Rhodobacter spaeroides* (photosynthetic bacteria); *Saccharomyces cerevisiae* and *Candida utilis* (yeasts); *Streptomyces albus* and *Streptomyces griseus* (actinomycetes); and *Aspergillus oryzae*, *Penicillium* sp., and *Mucor hiemalis* (fermenting fungi) [25–27]. They are nongenetic modifications of organisms, not pathogenic, not harmful, and not chemically synthesized. When an EM is introduced into the natural environment, the effects of individual microorganisms are greatly manifested. The innovations of EMs include the development, deployment, management, and re-establishment in an ecosystem or network of massive populations of valuable micro-organisms.

As EMs are a natural and organic invention, they can benefit humanity in many ways. EMs show very interesting properties, and because of that, their uses are now extending into different fields of research. In addition, they are marketed ambitiously as a way to fix many problems worldwide [28]. In recent years, microbial mineral precipitations that emerge from the metabolic activities of some specific microorganisms in concrete have been used to improve the overall performance of concrete. This has since become an active research area [29]. The main purpose of introducing EM in concrete is to produce a new breed of concrete that uses living things with enhanced properties. Better understanding of the impact of bacterial activity on the hydration, chemical phase, and microstructure of cement has enabled researchers to further explore the application of microorganisms in concrete [30]. EM-activated concrete structures are already used in hospitals, schools, apartments, residences, and so forth. A notable attribute of these concrete structures is their glossy appearance, which makes them aesthetically attractive [31].

The bacteria have been used in concrete [32] to generate an enzyme called urease that catalysed urea ($CO(NH_2)_2$) into ammonium ions (NH_4^+) and carbonate radicals (CO_3^{2-}). Through the chemical reactions, 1 mol of urea underwent intracellular hydrolyses to 1 mol of carbonate and 1 mol of ammonia, following Equation (1). Then, the carbamate was hydrolysed spontaneously to form one extra mole of ammonia and carbonic acid via Equation (2). These products later formed 1 mol of bi-carbonate (HCO^{3-}) and 2 mol of ammonium (NH_4^+) and hydroxide (OH^{1-}) ions according to Equations (3) and (4), respectively. The process described by Equation (5) was responsible for the enhancement of pH, drifting the bicarbonate in equilibrium to form the carbonate ions. It was shown that [33] the bacteria can accept cations (with Ca^{2+} deposited on the surface of cell-wall) from the surroundings because of their negatively charged cell walls. Then, Ca^{2+} can react with CO_3^{2-} and led to precipitate $CaCO_3$ at the cell wall surfaces, offering an active nucleation site following Equations (6) and (7). Using this mechanism of localized precipitation of $CaCO_3$ via the bacterial strains the pores and cracked surfaces can be filled, achieving a higher strength performance of the specimens.

$$CO(NH_2)_2 + H_2O \rightarrow NH_2COOH + NH_3 \quad (1)$$

$$NH_2COOH + H_2O \rightarrow NH_3 + H_2CO_3 \quad (2)$$

$$H_2CO_3 + H_2O \rightarrow HCO^{3-} + H^{1+} \quad (3)$$

$$2NH_3 + 2H_2O \rightarrow 2NH_4^+ + 2OH^{1-} \quad (4)$$

$$HCO^{3-} + H^{1+} + 2NH_4^+ + 2OH^{1-} \rightarrow CO_3^{2-} + 2NH_4^+ + 2H_2O \quad (5)$$

$$Ca^{2+} + Cell \rightarrow Cell\text{-}Ca^{2+} \quad (6)$$

$$Cell\text{-}Ca^{2+} + CO_3^{2-} \rightarrow Cell\text{-}CaCO_3 \quad (7)$$

The addition of 3% of EM in the concrete mix Isa and Garba [25] was observed to enhance its compressive strength (CS) by 14% at 28 days of curing age. Further, it was argued that the observed improvement in the CS of concrete at all ages were mainly because of the Bacillus subtilis bacteria presence in the local EM and partial filling of the pores. However, in reality the growth of bacteria needs time and, thus, a considerable early effect in the concrete cannot be evident without urea hydrolyses [34]. In fact, bacteria without its nutrients in the EM cannot participate in improving the concrete's strength [29]. Sato and Higa demonstrated [31] that concrete prepared with the addition of EM has 10 to 15% higher CS than the control specimen. In another study, 5% of EM was used as a partial replacement of water, wherein the water-cured sample showed an optimum CS of 43.17 MP, approximately 143.90% higher than the normal concrete (without EM addition) [35]. At an early age (1 day), concrete containing 5% of EM gained about 54% of its final strength, suggesting the benefits of EM to gain early strength in concrete. In addition, the splitting tensile strength (STS) and flexural strength (FS) of concrete were also enhanced. The self-compacting concrete incorporated with EM [36] at various levels (5%, 10%, and 15%) as replacement of water was shown to achieve a significant CS enhancement. Despite all these developments in EM incorporated concrete the large-scale production of eco-friendly and sustainable self-curing concretes remains deficient.

Considering the immense advantages of EMs, it is realized that such eco-friendly materials can be used to enhance the performance of self-curing concrete. The chemical and physical properties of EMs make them useful to apply in concrete as self-curing agent. However, no study was carried out to evaluate the self-curing characteristics of EM. In this view, we evaluate the microstructures and strength properties of some sustainable concrete mixes prepared with the incorporation of EM as self-curing agent. The main objective of this study is to determine the physical and chemical properties of EM together with the workability performance, mechanical strength, and microstructure characteristics of the proposed self-curing concrete mixes.

2. Methodology

2.1. Materials

The starting materials were ordinary Portland cement (OPC) as a binder, river sand as a fine aggregate, crushed stones as a coarse aggregate, and water. In addition, liquid solution of EM was used as a self-curing agent. OPC was collected from a distinct source (Tasek Corporation Berhad, Malaysia) and kept in the sealed container to prevent the contaminations. It conformed to the ASTM C150 requirements for type I of cement. It was mainly composed of CaO and SiO_2 (Table 1) and used as a main resource for calcium oxide (CaO). EM was procured from Peladang Johor Bahru (Malaysia) in a sealed container (5 kg) that was cultivated locally (Kota Tinggi, Malaysia). Then, EM was activated by the addition of water and blackstrap molasses mixture as its main source of nutrients. The obtained mixture was named as EM-activated solution (EM-AS). The EM solution was comprised of Bacillus subtilis and lactic acid bacteria (Figure 1). The activated solution was made (with pH less than 4) via the fermentation for 7–10 days without any oxygen supply. The existence of EM in the solution and its solubility in water were examined before their implementation into the concrete mixes. Due to the presence of lactic acid in EM it is important to control the solution pH, higher pH can be detrimental to the microorganisms' growths. Therefore, the pH of EM-AS was kept below 4 via the anaerobic fermentation for 7–10 days and then mixed in the concrete. The final mixture was comprised of water (90%), EM-1 (5%), and molasses (5%). Water was replaced by adding 5, 10, 15, 20, and 25% of EM in the solution and at each time pH value was measured. Ordinary tap water was used for the concrete mix preparation, curing, and tests.

Table 1. Chemical compositions of OPC obtained from XRF analyses.

Raw Materials	Elements (Weight%)								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	LOT
OPC	20.4	5.2	4.2	62.4	1.6	0.01	0.2	2.1	2.4

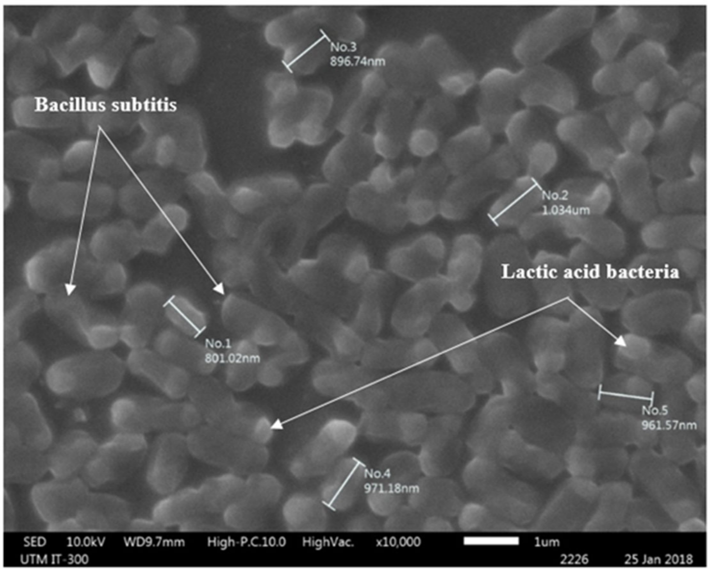
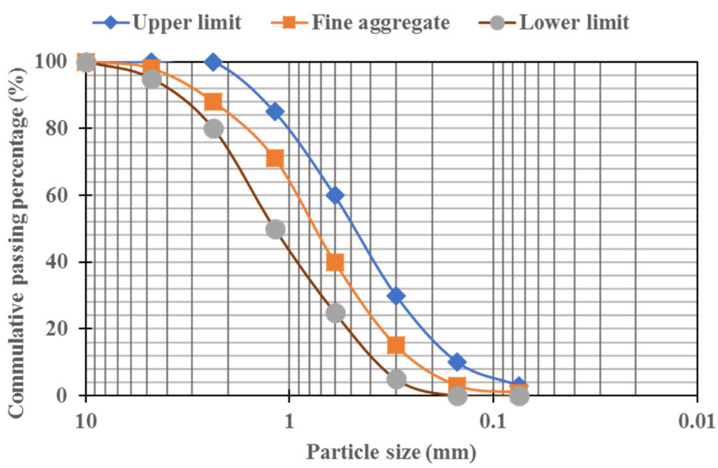


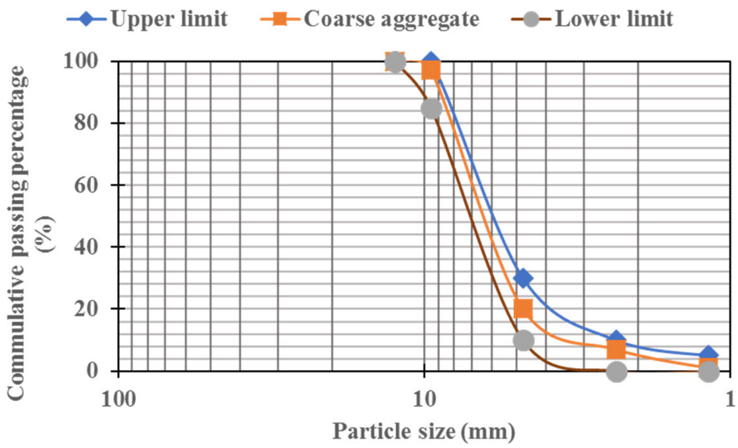
Figure 1. SEM image of the *Bacillus subtilis* and lactic acid bacteria.

In the mix design, the natural siliceous river sand was utilized as fine aggregates, wherein the sand was cleaned as per ASTM C117 requirements to eliminate silts and impurities. Next, it was oven-dried (60 °C) for a day for the moisture control. The air-dried crushed granites with the maximum grain size of 10 mm, specific gravity of 2.7, and 0.5% of water was used as coarse aggregates in all the mixes. Care was taken to ensure that the aggregates are free from organic matters, such as dry mud, leaves, and other harmful materials. The physical properties of the fine and coarse aggregates (such as density, specific gravity, degree of voids, evaporable moisture content, and water absorption) were recorded according to the method described in ASTM C128 and ASTM C127, respectively. These parameters fulfilled the conditions needed to produce the normal concrete and the highest allowed limit required by ASTM C33 stipulation. The percentage passing the 600 μm sieve was also found as 40% to be used in the mix design calculations. The bulk density of the oven-dried fine aggregates was 1730 kg/m^3. The specific gravity on the saturated surface dry (SSD) condition (2.70) was slightly lower than that of the coarse aggregates. Thus, the fine aggregates were somewhat lighter than the coarse aggregates. However, the difference between the specific gravity of the fine and coarse aggregates was very insignificant. A significant disparity in the specific gravity of the fine and coarse aggregates can cause their enhanced segregations within the concrete matrix [37]. The amount of net evaporable moisture in both coarse and fine aggregates was 0.1% which was mainly due to their identical drying process. In addition, the fine aggregates showed a water absorption value of 0.7%.

Figure 2 shows the grading outcomes of fine and coarse aggregates obtained from the sieve analysis. The grading of different sizes of the fine aggregates (Figure 2a) occurred within the upper and lower limits specified by ASTM C33. The nominal highest size, fineness modulus, and specific gravity of the fine aggregates were 4.75, 2.3, and 2.6 mm, respectively. Generally, the well-graded aggregates showed less water demand and fewer segregation tendencies, thus responsible for the overall properties enhancement of the proposed concrete mixes. Figure 2b displays the results obtained from sieve analyses and grading of the coarse aggregates which showed various sizes (mostly in the range of coarser sieve sizes from 10 to 4.75 mm) consistent with the highest and lowest limit set by the ASTM C33.



(**a**)



(**b**)

Figure 2. Particle size distribution of the aggregates (**a**) fine and (**b**) coarse.

2.2. Mixes Design and Specimens' Preparation

On the trail mixtures level, the effect of molasses on compressive strength was evaluated. For this purpose, the molasses (5% from EM weight) was used to replace the water, as shown in Table 2. A total of five concrete mixes were designed with various level of EM as water replacement. Water was replaced by 5, 10, 15, 20, and 25 weight% of EM and the mixture with 0% of EM was adopted as the control sample (Table 2). In this study, the traditional concretes with water and air curing were considered as control samples. The produced concrete specimens were characterized to determine their workability, strength, and microstructure properties. Preparation of materials, batching, mixing, and samples were completed according to BS 1881-125. For the hardened properties testing, the concrete specimens were prepared by mixing, casting, and curing. However, for the fresh properties, the specimens were tested immediately after the mixing. Before adding the materials, the drum mixer was washed and dried within one day to ensure that no extra water and previous residues are present. Next, half of the total aggregates were added in the mixer, followed by the addition of a full volume of fine aggregates. The remaining volume of the coarse aggregates was added in the drum mixer. After adding fine and coarse aggregates, the mixer was started and run for 30 s. The mixing process was continued for another 15 s and, meanwhile, a half volume of water was added in the mixer. Later, the mixing was continued for 2 to 3 min followed by covering with plastic sheet for 5 to 15 min. Thereafter, the total amount of cement volume was added on the aggregates. The drum containing the mixture was allowed to run for 30 s followed by the inclusion of the remaining volume of water and further mixing of all aggregates for another 2 to 3 min. After the mixing was completed, the concrete mixture was discharged onto a clean non-absorbent surface and turned it over using a hand tool to ensure the uniformity of the specimen before sampling.

Moulds in the form of cube of size (100 mm × 100 mm × 100 mm), cylinder of size (100 mm × 200 mm), and prism of size (100 mm × 100 mm × 500 mm) were used to prepare the test specimens according to BS 1881-108. The resultant concrete mixes were poured in the moulds with three layers and each layer was compacted with the vibration table to remove the air voids. Eventually, the top surface of the moulds was smoothened using plasterer's float and excess concrete from the sides of mould was removed. The curing process started after keeping the specimen in the moulds for 24 h to harden. After 24 h, each mould was open and then the specimen was cured in the air. The control sample (0% of EM) was cured in water (by dipping in a water tank) and air (inside the laboratory without sun and rain).

Table 2. Details of the normal and optimized self-curing concrete mixtures.

Mix Code	Cement	Water	Molasses *	EM Content **	Fine Aggregates	Coarse Aggregates	Curing Regime
C0EM	455	250	0	0	875	815	Water Air
C0.25-5M	455	249.94	0.625	0	875	815	Air
C0.5-10M	455	247.75	1.25	0	875	815	Air
C0.75-15M	455	248.12	1.875	0	875	815	Air
C1.0-20M	455	247.5	2.5	0	875	815	Air
C1.25-25M	455	246.87	3.125	0	875	815	Air
C5EM	455	237.5	0	12.5	875	815	Air
C10EM	455	225	0	25	875	815	Air
C15EM	455	212.5	0	37.5	875	815	Air
C20EM	455	200	0	50	875	815	Air
C25EM	455	187.5	0	62.5	875	815	Air

* molasses only; ** EM solution containing 5% molasses.

2.3. Tests for Characterizations

The physical appearance and homogeneity of EM and the solution containing 10% and 25% of EM as water replacement were recorded. The colours of the original materials and after mixing with water were evidenced by pouring them in a simple glass cylinder. A pH meter (Mi 151pH) was used to record the pH value of EM. First, the pH meter was calibrated using distilled water (pH 7). Then, the pH meter rod was inserted in the glass cylinder (for 24 h) filled with the test liquid to obtain a constant pH value (Figure 3a). The viscosity values of water, EM, and water replaced with various EM contents (5 to 25%) were determined according to ASTM D455, wherein a digital Brookfield viscometer was used (Figure 3b). For the measurement of viscosity, about 50 mL of each liquid was taken. The surface tension of water (measured according to ASTM D971), EM, and water replaced with various EM contents (5 to 25%) were measured for which known value of the liquid density was used. The surface tension values of these liquids were measured by a tensiometer (Kruss Germany EasyDyne model) wherein about 100 mL of each liquid sample was taken.

(**a**) (**b**)

Figure 3. The equipment and setup for measuring the (**a**) pH value and (**b**) viscosity.

The slump test was used to determine the concretes workability performance, providing their easy handling in the fresh condition with consistency. The studied concretes target slump values without the addition of superplasticizer (SP) were ranged from 60 to 180 mm. The slump test was conducted by following BS 1881-102. The fresh concrete from tray was poured in the mould (in three layers) which was firmly held from all sides. After pouring the first layer, it was tamped with 25 stocks using a taming rod. Enough precaution was taken so that the taming was not forcedly performed when it was released freely from the top of the mould. This process was repeated for each layer poured in the mould. The excess concrete was poured in the last layer to obtain a smooth surface on the top of the mould. Upon achieving a smooth surface, the excess concrete on the top of the mould and tray were removed. After this process, the mould was carefully removed vertically from the concrete mix within 5 to 10 s. Next, the mould was kept vertically inverted on the tray and then the measurement was taken using a ruler. The result of the slump was defined as difference in the height (in mm) from the top of the mould to the top of the concrete specimen.

The CS, ultrasonic pulse velocity (UPV), STS, and FS tests were carried out after adequate curing at 3, 7, 28, 56, and 90 days. After various curing ages for each mixture, three samples were tested, and the average value was adopted. The CS test was performed following BS 1881-116 and BS 1881-15. During the CS test, the specimens were duly prepared and positioned accurately between the upper and the lower metal bearing plates as specified by the relevant standard. A load at a constant rate (5 kN/s) was applied up to the failure of the specimen. Since the machine has inbuilt configurations, the CS was generated automatically based on the assigned specimen dimensions. The crystallographic structures, elemental compositions, and thermal behaviour of the specimens were examined by a scanning electron microscope attached with an energy dispersive X-ray spectrometer (SEM-EDX), X-ray diffraction (XRD), and thermogravimetric and differential thermal analyser (TGA-DTG). The concrete specimens obtained after different tests were crushed and then immediately immersed in the containers filled with acetone. The samples were then oven-dried for 24 h at 105 °C to stop the hydration process. Further, the samples were ground to finer particles as per the requirement of different international standards. The SEM-EDX analyses of the concrete specimens provided the information of microstructures, interfacial transition zones, hydration mechanism, and elemental composition. Each batch of specimens was tested for early (7 days) and late ages (28 days) of curing. Before testing, these specimens were coated with gold to attain the desired surface structure.

3. Results and Discussion

3.1. Physical Properties and Homogeneity of EM Solution

Figure 4 shows the physical appearance and homogeneity of EM solutions with respect to water (Figure 4a). The colour of EM was dark brown in its original state (Figure 4b). This physical characterization was mainly performed to understand their potential for the implementation as a self-curing agent in the concrete technology. The physical appearance of EM and its solubility in water in original form was evaluated to know their behaviour in fresh and hardened concrete in terms of colour changes as desirable for practical building applications. The colour was changed from dark brown to dark yellowish when 10% of water was replaced with EM (Figure 4c). The dark brown appearance was altered to light brown when 25% of water was replaced with EM (Figure 4d). Thus, with the increase in water replacement percentage with EM the mixed solution appeared darker. It was found that (Figure 4) EM was completely soluble in water, making the resultant solution homogeneous and uniform. It was also noticed that although EM was composed of solid particles, but they were not traceable visually in the mixed solution (Figure 4). A similar observation regarding the brown colour of EM was made by Zakaria and Gairola [28].

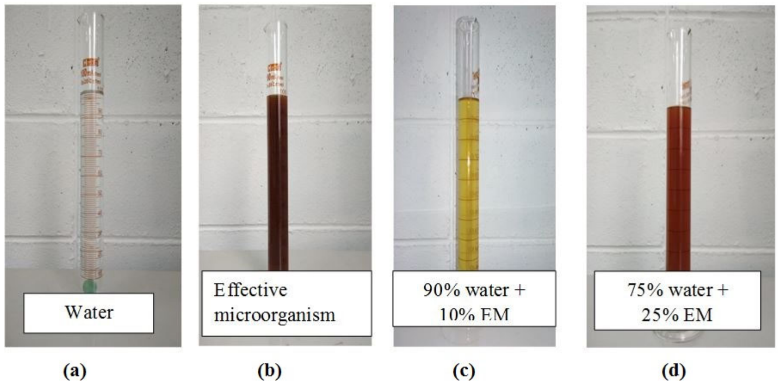


Figure 4. Physical appearance and homogeneity of solution made with EM of (**a**) 0%, (**b**) 100%, (**c**) 10%, and (**d**) 25%.

3.2. Density

Figure 5 displays the measured density of pure water, pure EM, and EM–water solutions. The density of all materials was measured at an ambient temperature of 25 ± 1 °C which was the same temperature that the concrete work was carried out in the laboratory. The density of original EM and water were correspondingly 980 kg/m^3 and 1000 kg/m^3. However, the density of the EM–water mixture containing 5 to 25% of EM as water replacement was higher than pure EM. The density of water–EM solution was decreased with the increase in EM in the solution. The mixture prepared with 25% of EM revealed a density value (990 kg/m^3) lower than pure water. The density of liquid did not significantly affect the density of the concrete mixes because 60–70% volume of the mixes was occupied by fine and coarse aggregates. According to ASTM C1602, the density of mixing water should be less than 1010 kg/m^3.

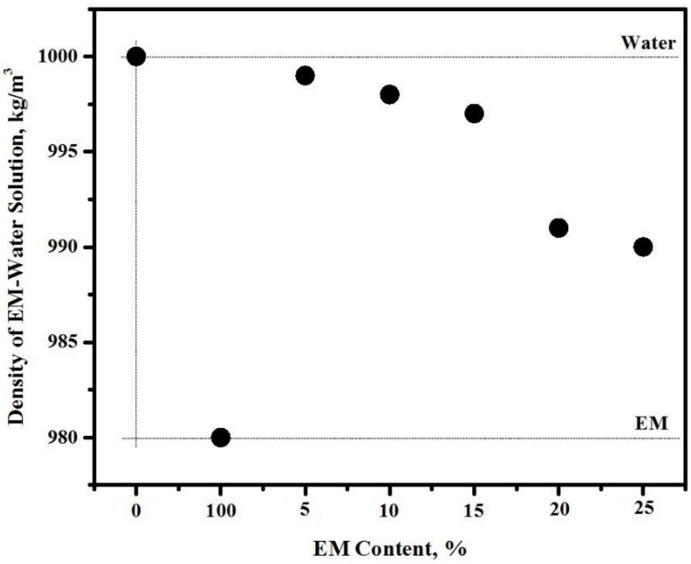


Figure 5. Density of EM, water, and EM–water solution.

3.3. pH Values

Figure 6 shows the pH values of EM, water, and water–EM solutions. The pH value of tap water and EM was 6.7 and 3.5, respectively. The pH value of the mixture containing 5 and 10% of EM as water replacement was correspondingly 6.4 and 6.3 with agreed with other reports Isa and Garba [25], and Andrew and Syahrizal [35]. The pH value of the mixture prepared with 15, 20, and 25% of EM were found to be 5.7, 5.2, and 4, respectively.

An overview of recent literatures about EM indicated that for fresh EM and EM solution the pH value must be in the range of 3.2–3.5 and above 3.5, respectively [38]. According to Cabrera and Galvín [39], the pH value of water can significantly affect the fresh and hardened characteristics of concrete, impacting the hydration reaction processes at early ages and, thus, determines the final durability performance. In this research, tap water and solution composed of water and EM were used to prepare the concrete mixes. According to BS EN 1008, the pH value of mixing water should be greater than 4. In addition, water used for the mixing and curing purposes must be very clean and devoid of various contaminants, such as oil, acid, alkali, salt, sugar, organic components, or other substances that may be detrimental to the concrete mixes or reinforced steel.

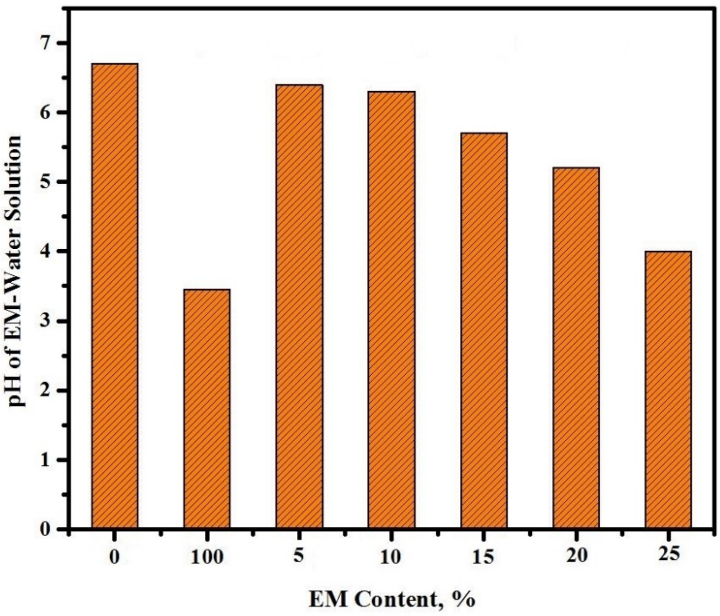


Figure 6. pH values of the EM, water, and EM–water mixture used to design the specimens.

3.4. Viscosity

Figure 7 depicts the viscosity values of water, EM, and water–EM solutions. The viscosity of original EM (1.44 mPas) was higher than pure water (0.95 mPas). The viscosity of water–EM solutions containing 5, 10, 15, 20, and 25% of EM as water substitution was correspondingly 1, 1.05, 1.10, 1.20, and 1.25 mPas which were slightly higher than that of water. According to Wang and Li [40], viscosity of mixed water can considerably affect the fresh and harden characteristics of concretes, enabling their fabrication with adequate cohesiveness, reduced bleeding, segregations, and settlements, thus improving the durability, strength, and workability performance of concretes [41]. To produce the concrete mixes, they used low viscosity due to very slight difference in the viscosity between water and EM–water mixture. The increase in the viscosity of EM was mainly due to the inclusion of molasses during EM solution preparation.

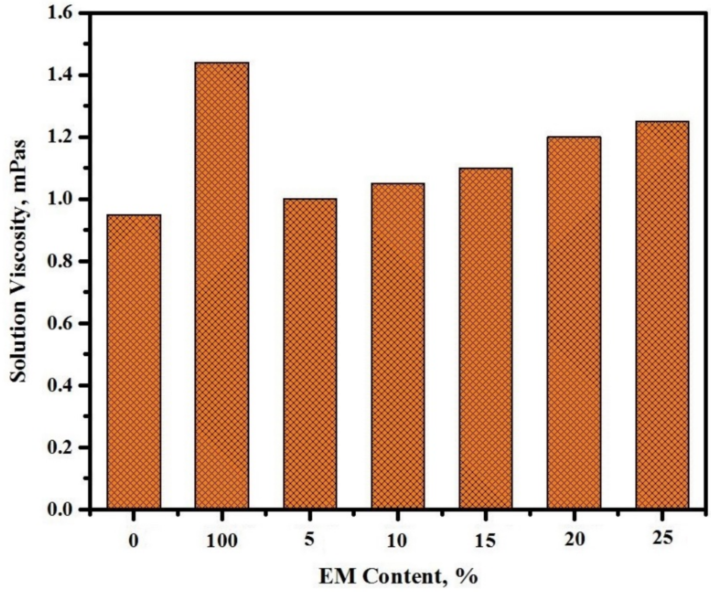


Figure 7. Viscosity of pure water, EM and EM-water mixture used to design the specimens.

3.5. Surface Tension

Figure 8 shows the surface tension values of water, EM, and water-replaced EM solutions. The fresh and hardened characteristics of the proposed concretes were decided by these surface tension values. The original EM showed a surface tension of 35.6 mN/m which was lower than water surface tension (66 mN/M). The surface tension of solution prepared with 5 to 25% of water replaced with EM was in the range of 58.3 to 39.9 nM/m, respectively, and lower than that of water. The surface tension being the measure of the tangential force per unit velocity gradient that acts at the interface between two phases wherein the force becomes attractive when the boundary is between a liquid and a solid or between a liquid and a gas, such as air. In other words, it is the elastic tendencies of the fluids surface that enable obtaining the lowest feasible surface areas. It is a necessary factor for the capillarity action that appears when fluids are discharged and come in contact with the porous rocks/minerals. It is worth mentioning that the interfacial tension is to some extent analogous to the surface tension in terms of the involved cohesive force. Nonetheless, the major forces responsible for the interfacial tensions are the adhesive forces (tensions) between the fluid state of one substance and fluid or solid state of other. In brief, the surface tension of various components played a vital role in the properties of concretes.

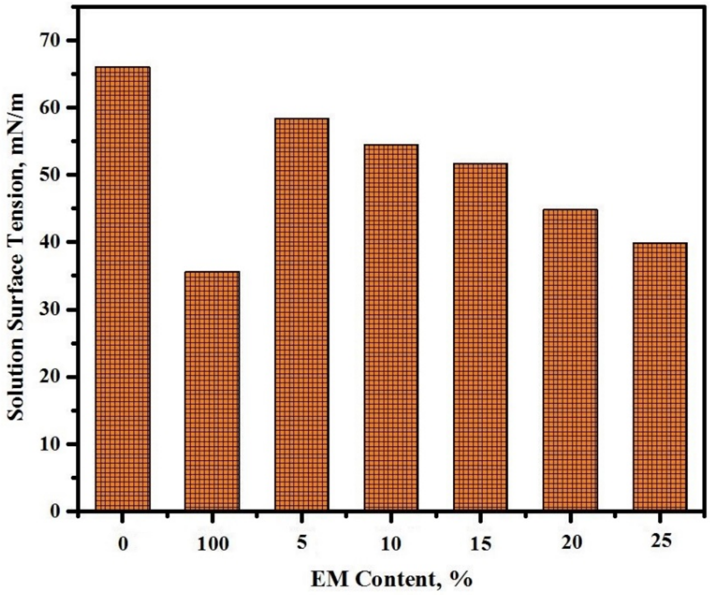


Figure 8. Surface tension of pure water, EM, and EM–water mixture used to design the specimens.

3.6. Quality Index

Table 3 presents the quality indices of EM and solution of water with EM. According to ASTM C1602/C1602M standard, the normal tap water was used for producing and curing the concrete mixes. The drinking water was free from any contaminants and did not cause any unfavourable influence in terms of the colour or odour changes of the concrete mixes. According to the standards, all parameters involving the quality indices of water, EM and 10% water substituted with EM solution were below the allowed limits. The water quality was vital since the presence of impurities might have interfered with the setting times of the cement, negatively affecting the strength properties of the concrete or causing a stain on its surface, leading to the corrosion of the steel reinforcement. Therefore, the appropriateness of the water quality required for both mixing and curing was ensured [42].

Table 3. Quality indices of various constituents used for the mixes design.

Quality	Water	EM	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	10,000–50,000	
Total dissolved solids	17.55	5824	1092	mg/L	≤50,000	ASTM C1602
Salinity	0.01	5	0.85	ppt		

3.7. Workability

Figure 9 shows the measured slump values of various concrete mixes. The slump values of the mixes prepared with EM as water replacement were monotonically increased with increase in EM contents. The slump value of the control sample was 70 mm. The slump values of the specimens containing 5, 10, 15, 20, and 25% of EM as a replacement of water were 80, 100, 150, 180, and 220 mm, respectively.

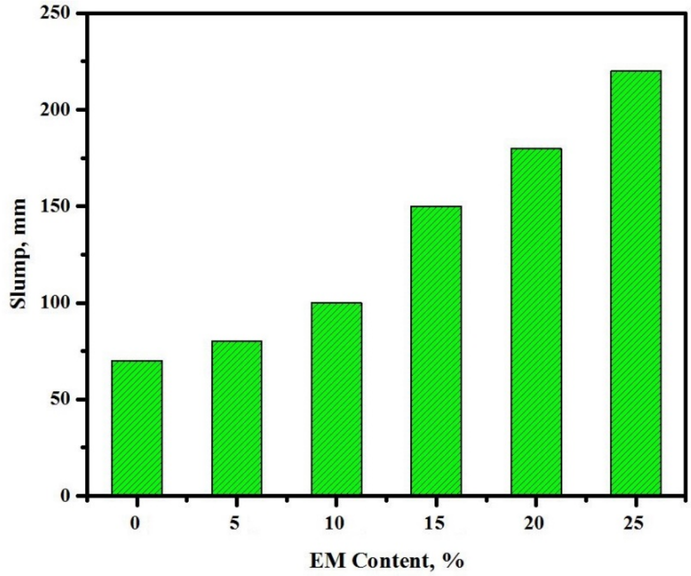
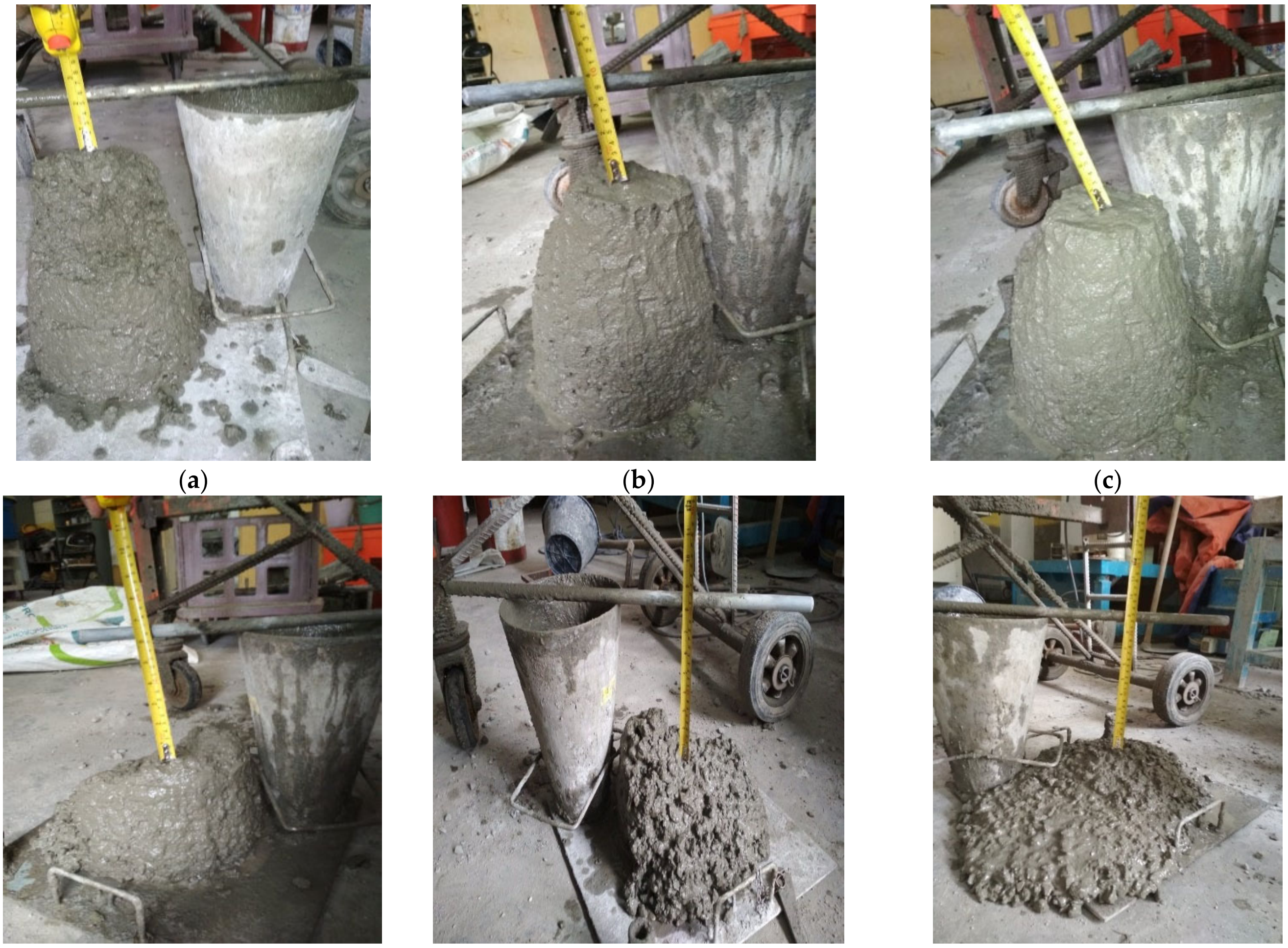


Figure 9. Slump values of the proposed concrete mixes.

Figure 10 displays the slump appearance of the concrete mixes prepared with various percentages EM (0 to 25%). It was also noticed that control mixture and the mixes designed with 5 and 10% of EM achieved a true slump (Figure 10a–c). Figure 10d,e show the shear slump of the specimen prepared with 15 and 20% of EM as water replacement, respectively. The shear slump of the specimen containing 25% of EM as a water replacement was collapsed (Figure 10f), which was mainly attributed to the attachment of some particles with the aggregates and free movement of some mixing water particles due to lower surface tension between water molecules. Consequently, the workability of the concrete mix was improved with the increase in water replacement by EM solution. In addition, the bleeding in the concrete occurred when 25% of water was replaced by EM (Figure 10f). Li and Miao [43] stated that a reasonable increase in the viscosity of mixing water can reduce the workability of concrete, but a little increase in the viscosity of liquid may not affect the slump of workability. It is worth noting that the EM solution contained 5% of molasses of its total weight wherein its presence was responsible for the improvement in the workability of concrete. The results are in agreement with previous studies regarding the addition of molasses in concrete [44,45].

It is known that a major fraction of molasses is composed of alkaline hydrolyses products, such as inverted sugar, that contain free carboxylic groups which, in turn, act as plasticizers/water-reduction agents in the cement paste. The pyrolysis of sucrose may lead to the creation of another product that contains high molecular weight particles as much as 13200, thus acting as plasticizer/water-reduction agents. This reduction in water content in the cement matrix can significantly affect the structures of molasses at higher dosages [46]. It was shown that the presence of sugar in cane-derived molasses can delay the C_3S initial hydration and expedite C_3A hydration and, thus, the mixing water was not perfectly achieved. As a result, a large amount of mixing water remained as free-flowing water on the paste, leading to better workability. Furthermore, an improvement in the workability of concretes at higher contents of molasses was reported Jumadurdiyev and Ozkul [47] and Weifeng and Suhua [48] which was ascribed to the participation as surface-active agents, thus increasing the free water level in the cement paste. In short, the requirement of water at higher dosages of molasses was lower, thus an increased amount of molasses at a fixed value of the water to cement ratio caused a significant workability enhancement.



(a) (b) (c) (d) (e) (f)

Figure 10. Slump appearance of the concrete mixes with EM of (**a**) 0%, (**b**) 5%, (**c**) 10%, (**d**) 15%, (**e**) 20%, and (**f**) 25%.

3.8. pH Value of EM-Concrete

Figure 11 displays the pH values of the proposed concretes containing different amounts of EM as substitute of water. The pH values of the concretes were decreased with the increase in EM contents, thus affecting the hardened properties of the self-curing concrete (EMC). The pH value of the fresh control concrete was 13, which is in agreement with other studies [49]. Concretes containing 5, 10, and 15% of EM showed a pH value of 12 and those prepared with 20 and 25% of EM showed a pH value of 11 which was lower than the control mixture. Furthermore, this low pH value also affected the early and late age properties of the proposed concrete mixes. Thus, it was essential to investigate the pH value of the fresh concrete. The pH level of pore fluids in OPC-based concretes is mostly controlled by the contents of alkali metals, the addition of supplementary cementitious materials and curing conditions. The mechanism behind the observed lowering of pH values of the proposed concrete mixes due to the addition of EM as a self-curing agent in place of water can mainly be due to two reasons. Generally, the pH of fresh concrete is around 13. The observed reduction in the pH value of the fresh concrete due to the substitution of water by EM was mainly due to lower pH value of EM. Another reason may be the existence of molasses that slowed down the hydration reaction at an early age. Yadav and Das [50] acknowledged that [46] the pH value of concrete between 10 to 13 has insignificant effect on the concrete properties. So, the results obtained in this study are in the safety limit of concrete usage in the construction sectors.

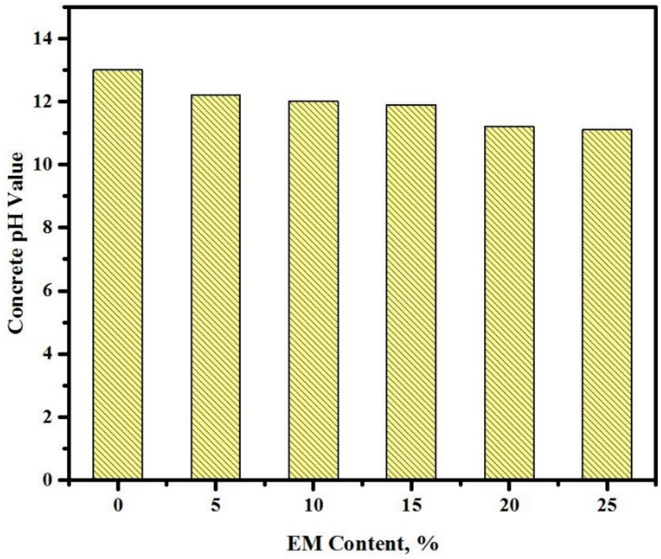


Figure 11. pH values of the concrete mixes.

3.9. Compressive Strength

The effect of molasses on compressive strength (CS) development of designed concrete evaluated at 28 days of curing age and the obtained results illustrated in Table 4. The results show that replacing water by up to 10% with molasses slightly improved the strength. It was observed that there was a trend of CS to increase from 35.8 to 35.9, 36.1, 36.4, and 36.7 MPa with increasing molasses content from 0 to 0.5, 0.75, 1.0, and 1.25% as water replacement. It can be said that molasses contributed to reduce the pore diameter of the paste. The decreased pore diameter is favourable to the strength development of concretes [51,52]. It was found that the enhancement on CS after 28 days of curing age less than 2.5% compared to the control sample. Based on the obtained results, it is clear that using only molasses is not suitable as a self-curing agent.

Table 5 shows the CS of the concrete mixtures prepared with 0, 5, 10, 15, 20, and 25% of EM at various curing ages. CS being the main attribute of concrete, it is often used to predict its quality. It shows the load bearing capacity of concrete at a certain load, after which concrete undergoes a failure mode. The results showed that the CS values of the concrete mixes were significantly influenced by the curing regime. For the control sample, the CS values were dropped from 25.4 to 44.1 MPa (cured in water) and 23.1 to 36.5 MPa (cured in air) when curing ages were changed from 3–90 days in each regime. At 3 days of age, the CS of the proposed concrete containing 0 to 25% of EM and cured in air condition showed a loss by 9.1 to 42.1%, respectively, compared to the one cured in water. The CS of all specimens containing EM (at 3 days of age and cured in air) was lower than control sample (23.1 MPa); whereas at the same concrete age for specimens cured at air, the loss on CS for mixtures containing 5, 10, 15, 20, and 25% was noted 10.7, 5.1, 16.6, 23.5, and 35.9%, respectively, which was lower than control mixture (23.1 MPa).

Table 4. Effect of molasses content on concrete CS values at 28 days curing age.

Mix Code	C0EM	C0EM	C5M	C10M	C15M	C20M	C25M
Curing regime	Water	Air	Air	Air	Air	Air	Air
CS values (MPa)	43.3	35.8	35.6	35.9	36.1	36.4	36.7

The observed reduction in the CS values (at early ages) of the concretes prepared with EM were mainly due to the presence of molasses in EM that slowed down the hydration process, reduced the C-S-H gel formation and did not fill the voids. According to Weifeng and Suhua [48], the obtained retarded hydration can be ascribed to the various factors. First, the molasses adsorption on the hydrating cement particle surface and/or hydration product. Second, the ions solubility enhancement emerged from the cement particles followed by their adsorption on Portlandite (CH) and hydrated (C-S-H), as shown in Equations (8) and (9), preventing their formation due to acidic nature of EM (pH value of 3.45). Additionally, initially hardened concretes showed lesser pH than C0W and C0A, implying their lower alkalinity at an early age. Lower alkalinity of the pore fluid had negative impacts on the cement hydration, thereby reducing concrete strength, especially at an early age. The lesser pH impeded the early development of cement paste microstructure and has a delayed effect on cement hydration, some mixing water molecules are adsorbed at the water–solid (air) interface and generate "organic molecular film" which causes a reduction in the interfacial energy of cement particles and hydrated products to impede the progression of the hydration reaction. It also reduces the alkali content and pH value in the pore solution, resulting in hydration retardation of tricalcium silicate. These early age (3 days) results agreed with the findings of Ali and Qureshi [45] wherein the CS values of the mixes containing molasses at all ages (except early ages) was found to improve.

$$Ca_2SiO_5 + H_2O > CaO\text{-}SiO_2\text{-}H_2O \quad (8)$$

$$Ca_3SiO_5 + H_2O > CaO\text{-}SiO_2\text{-}H_2O + Ca\,(OH)_2 \quad (9)$$

At 7, 28, 56, and 90 days of curing age, the concrete mixture containing 5% of EM achieved 20.1, 13.9, 14.6, and 14.2% strength enhancement, respectively, whereas, at the same curing ages, the concrete mixture containing 10% of EM gained 24.4, 19.5, 19.9, and 20.3% CS, respectively. At 7, 28, 56, and 90 days under air curing condition the specimen containing 15% of EM achieved a strength of 6.9, 0.8, 3.6, and 3.3%, respectively. However, the increase in the content of EM as water replacement up to 25% led to a reduction in the strength development of the concrete mixes. The enhancement in the CS values for the specimens' containing 5, 10, and 15% of EM was due to a stronger hydration reaction that could fill the voids in the concrete network with the generation of dense C-S-H gels. The low surface tension of EM mixing water was the other reason for the increase in CS that allowed creating tiny voids in the concrete matrix. Essentially, the reduced surface tension of the mixing water considerably influenced the interaction potential amid capillary walls, lowering the diameter of the capillary pores during drying. In addition, due to the decrease in the mixing water surface tension the molecules were easily adsorbed on the surface of the cement particles, thus reducing the surface energy to improve the dispersion, resulting in an increase in the innocuous pores thereby forming a dense internal structure.

The concrete mixes prepared with 5 and 10% of mixing water replacement by EM achieved optimum quality in air curing regime. It was demonstrated Tramontin and Onghero [53] that an admixture can reduce the water surface tension and efficiently perform by substituting some part of the mixing water, thus improving the concrete's performance. The other reason for the increase in CS for the concretes made with 5% and 10% of EM was due to the presence of molasses in the EM solution that retard the hydration process in the concrete matrix. Consequently, the delayed hydration process resulted in fewer products containing C-S-H gel and calcium hydroxide that are responsible for the early and later strength of concrete. Retardation in the hydration reaction significantly affected the CS of the concrete, reducing the CS at an early age and increasing at the late ages. According to Ali and Qureshi [44], the addition of a little quantity of molasses can increase the CS by 10–12%, but an increase in the amount of molasses beyond certain value can reduce the CS of concrete. The water-reducing effect of molasses present in EM enables to achieve denser and durable concrete. Improvement in the strength performance at smaller dosages of molasses can be attributed to the extended setting of the cement paste within the concrete matrix. The small amount of molasses can significantly improve the CS due to the reduction in porosity and size of the voids [48]. Rashid and Tariq [46] claimed that the incorporation of molasses in the concrete mixes can positively affect the concrete CS at both early and late ages due to a more uniform and denser structure after prolonged hardening.

It is well known that the bacteria growth is significantly influenced by pH of concrete. As shown in Table 5, the pH of normal concrete was 13 and this value decreased with increasing EM content. It was observed with increasing level of EM as water replacement from 0% to 10% and 25% the pH value decreased from 13 to 11 and 10, respectively. Extreme pH affects the structure of all macromolecules. The hydrogen bonds holding together strands of DNA break up at high pH. To keep bacterial viability during unfavourable conditions in the concrete matrix, protection of bacterial cells is necessary. As this experimental focusing on self-curing concrete and improve the early strength development (below 28 days), keeping the bacteria viable for a long time was not considered. The essential step that enables the self-curing effect of bacteria-based concrete is reduce the surface tension and water loss with increase the bacterial production of carbonate ions which are transferred from cell to environment. This step also immediately provides an increase in system alkalinity [54]. However, this step is not necessarily the most important role that bacteria can play in $CaCO_3$ formation. Bacterially induced carbonate precipitation (BICP) is, consequently, a result of metabolic activity, $CaCO_3$ nucleation, and crystal formation around individual bacterial cells. In addition to bacterial contributions to changes in ion concentration and pH value, active bacterial cells have the potential to be efficient nucleation centres during $CaCO_3$ formation and fulfill the process of strength enhancement.

Table 5. CS values of the concrete mixes at curing regime and ages.

Mix Code	Curing Regime	pH of Fresh Concrete	CS Values (MPa) at Various Curing Ages				
			3	7	28	56	90
C0EM	Water	13	25.4	32.1	43.3	43.8	44.1
C0EM	Air	13	23.1	30.3	35.8	36.2	36.5
C5EM	Air	11	20.6	36.4	40.8	41.5	41.7
C10EM	Air	11	21.9	37.7	42.8	43.4	43.9
C15EM	Air	11	19.2	32.4	36.1	37.5	37.7
C20EM	Air	10	17.6	25.1	31.5	32.3	32.8
C25EM	Air	10	14.7	20.3	29.1	30.3	30.6

3.10. Water Loss

Figure 12 shows the water loss of the control mixture and self-curing concretes prepared with 5 to 25% of EM as water replacement. The increase in the EM contents from 5 to 15% reduced the water loss from concrete compared to the control specimen. However, when the water replacement by EM percentage was increased from 20 and 25%, the water loss was also increased. The control mixture achieved 2% water loss at 6 days of air curing. The concrete mix made with 5% of EM as water replacement showed 1.27% of water loss at 6 days of air curing. Likewise, the specimen made with 10 to 25% of EM as water replacement showed a water loss of 1.4 to 2.95% at 6 days of air curing, respectively.

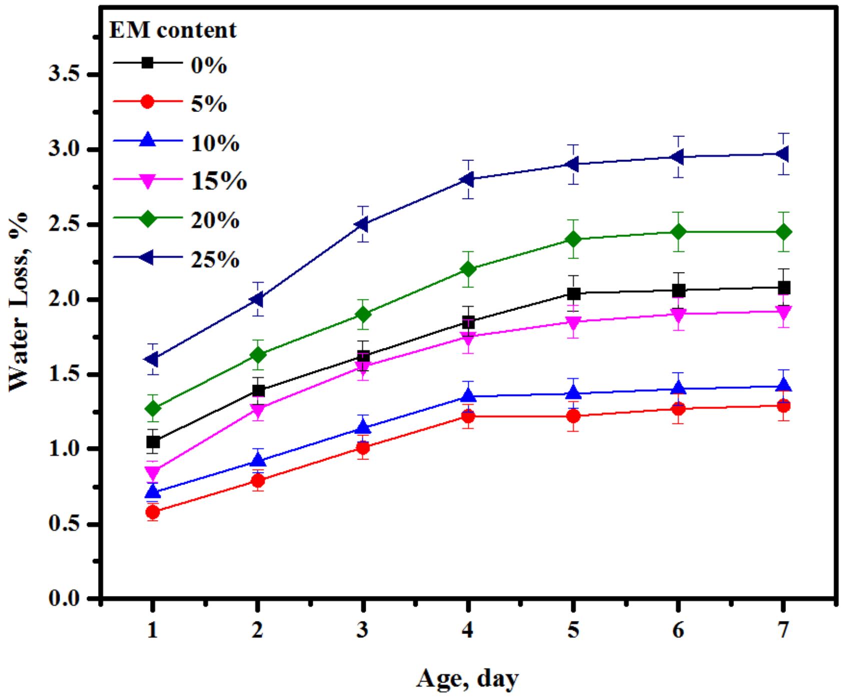


Figure 12. Effect of EM on water loss of the self-curing concretes.

The reduction in water loss from concrete was ascribed to the low surface tension of the mixing water. First, the universal mechanism that lowered the water surface tension also lowered the water drop size wherein the mixing water produced small voids in the concrete, making the connection of voids poor and, thus, reducing the capillary evaporation of water from concrete. According to Rongbing and Jian [55] and Dang and Shi [56], the capillary stress can be considered as the predominant mechanism. When pore water evaporates from the capillary pores in hardened concrete during drying, the tension in the liquid is transferred to the walls. The internal stress generated upon evaporation is proportional to the surface tension of the pore water solution. A higher stress between particle and liquid causes a higher rate of evaporation. Other processes for the reduction in water evaporation may be due to slowing down of the hydration reaction caused by the presence of molasses in EM that acts as a self-curing agent. The hydration of concrete is lower at a lower temperature. However, for the self-curing concrete mixtures containing EM up to 15% the observed increase in water loss can be attributed to the dispersion of liquid particles from the solid particles. The workability results (Section 3.7) of the proposed concrete mixes showed the occurrence of bleeding, wherein the water–cement paste flew on the sides during the pouring of concrete which was soft, indicating the presence of more water as the hydration reaction did not utilize much water, causing more evaporation of mixing water from concrete. Another reason for the high evaporation at this percentage was due to the presence of a high amount of molasses in the concrete which slowed down the hydration, making the availability of more mixing water and, thus, resulting in more evaporation of water from concrete. Consequently, the risk of more voids formation in this mixture was increased.

3.11. XRD Patterns

Figure 13 illustrates the XRD profiles of the self-cured concrete (at 7 days of air curing) prepared with 0 and 10% of EM as water replacement. The intensity of quartz peak (Q) at 20.1° and 26.9° for the specimen containing 10% of EM was decreased. The observed Ettringite (E) peak ($Ca6Al_2(SO_4)_3(OH)_{12}{\cdot}26H_2O$) at 35.1° for the concrete containing 10% of EM was replaced by the peak of Portlandite (P). The intensity of the Calcite ($CaCO_3$) peak

(C) at 37.2° was increased when the EM content was increased from 0 to 10%. The self-cured concrete (without EM solution) showed five peaks for Portlandite ($CaOH)_2$) at 18.2, 48.1, 55, 68.5, and 71° in addition to a weak calcite peak at 29°. The specimen prepared with 10% of EM showed an increase in the amount of Portland from 14.2 to 23.8% that increased the C-S-H gel product (Table 5). This can be attributed to the enhancement in the self-cured concrete properties especially increment of CS values from 32.1 MPa (C0EM) to 37.7 MPa (C10EM) at 7 days of age.

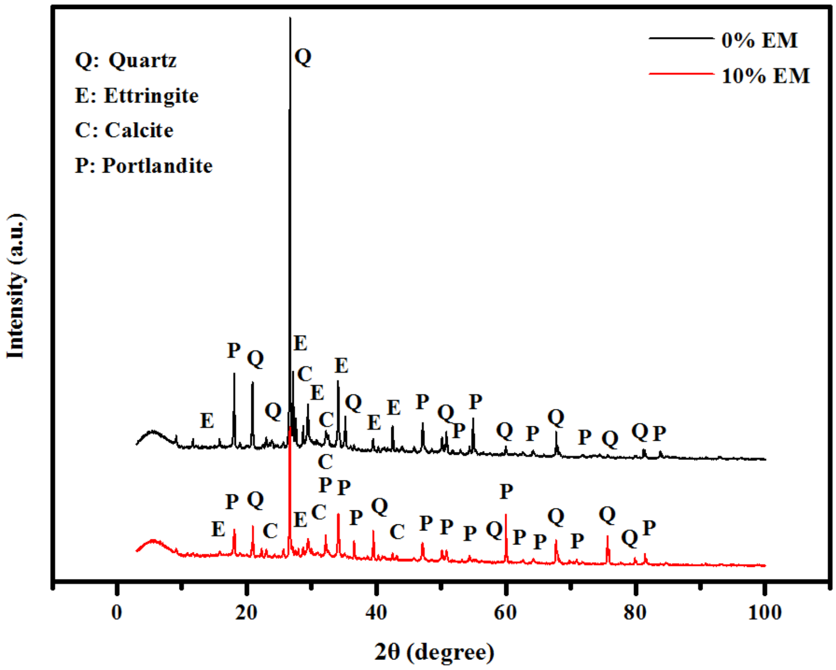


Figure 13. Effect of 10% of EM on the structures of self-cured concretes at 7 days of age.

Figure 14 presents the XRD patterns of self-cured concrete made with and without 10% of EM as water replacement at 28 days of air curing. The observed XRD peaks due to the crystalline quartz (SiO_2), Portlandite, Calcite, and Ettringite phases were derived from the OPC. The self-cured concrete containing 10% of EM solution showed an intense Portland peak compared to the control sample. In addition, the short-range ordering of the Ettringite showed a broad and diffuse halo at 10°. The Portlandite and Calcite peaks were observed around 18–65° for samples without EM. Concrete made with 10% of EM displayed an increase in the Portlandite peak intensity at 18 and 29°, indicating an increase in the crystalline phases. Additionally, the intensity of the calcite peak at 36° was enhanced due to the addition of EM. Table 6 shows the presence of each product, including quartz, Portland, Calcite, and Ettringite. It is known that an increase in the Portlandite and Calcite content in the concrete can enhance their CS values and microstructure. However, at 28 days, the addition of EM led to an increase in the amount of calcite from 13.9% (concrete without EM) to 15.4% (concrete with 10% of EM), improving the corresponding CS from 35 MPa to 42 MPa. The specimens prepared with 10% of EM showed an enhancement of the Portlandite product by 23.8% (for concrete with 10% of EM) compared to 14.2% (for concrete with 0% of EM) at 7 days of air curing and 24.5% (10% of EM) compared to 20.6% (0% of EM) at 28 days of age for air curing, respectively. Briefly, the XRD results revealed a significant effect of EM incorporation and curing regimes change on the C-S-H gel formation and CS development of the proposed self-curing concretes.

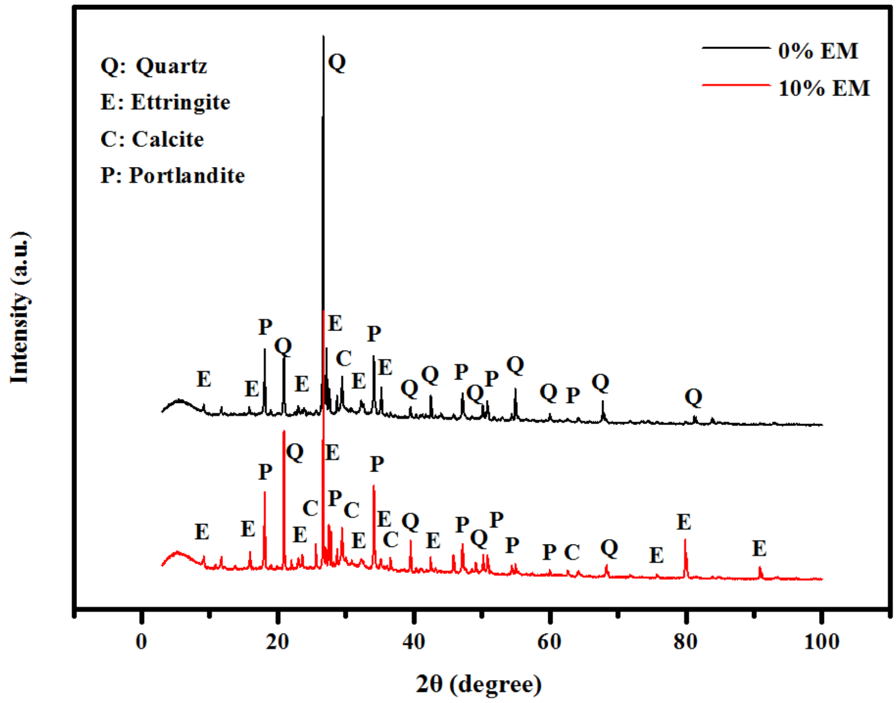


Figure 14. Effect of EM addition on the structures of self-cured concretes at 28 days of age.

Table 6. XRD analyses of self-cured concrete specimens.

Chemical Compounds	EM (weight%) at Different Curing Ages			
	7 Days		28 Days	
	0	10	0	10
Quartz	69.3	50	53.7	48
Portlandite	14.2	23.8	20.6	24.5
Calcite	9.5	14.2	13.9	15.4
Ettringite	7	12	11.8	12

3.12. SEM Analyses

Figure 15 displays the SEM images of the self-cured concrete (at 3 days of air curing) prepared without EM (control sample) and with 10% of EM concrete. The microstructure of concrete made with 10% of EM showed partially reacted ecospheres, lower CS (21.9 MPa) and less C-S-H gel formulation compared to the control sample. In addition, the SEM morphology revealed more needle portlandite with some very interesting features, such as a large fraction of unreacted particles in the concrete matrix. Concrete prepared without EM showed a large quantity of calcite and C-S-H gel, indicating a higher CS of 23.1 MPa.

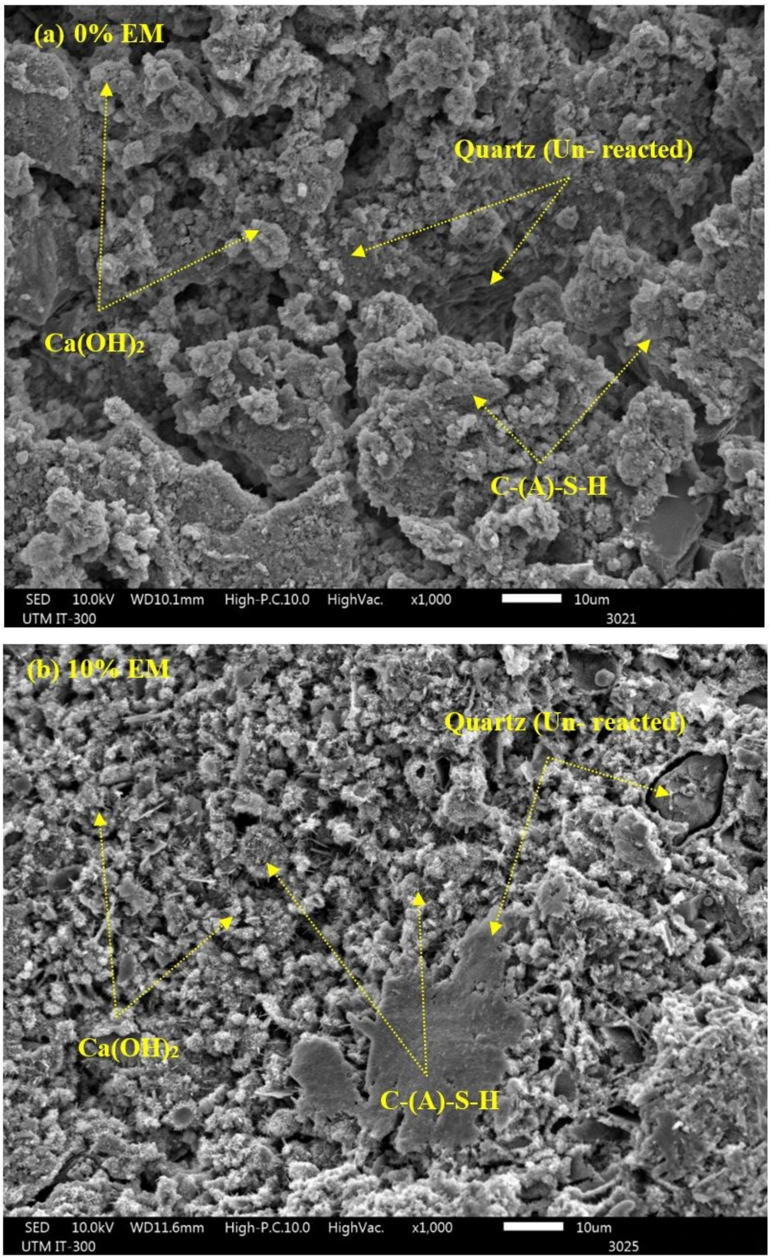


Figure 15. SEM images of self-cured concrete at 3 days prepared with EM of (**a**) 0% and (**b**) 10%.

Figure 16 shows the SEM images of the self-cured concrete made without EM and with 10% of EM after 7 days of air curing. In the microstructure of the control sample revealed partially reacted ecospheres as the main feature. However, the specimen containing 10% of EM showed more denser microstructures compared to the control sample due to the formation of much C-S-H gels. Furthermore, the SEM morphology of the concrete made with 10% of EM exhibited very little fraction of unreacted particles, high amount of Portlandite, and dense gel phase in the network matrix. With the addition of EM in the concrete, the hydration process and gel formulation was improved, leading to an enhancement in the CS (37.7 MPa) of the self-curing concrete compared to the control sample (30.3 MPa).

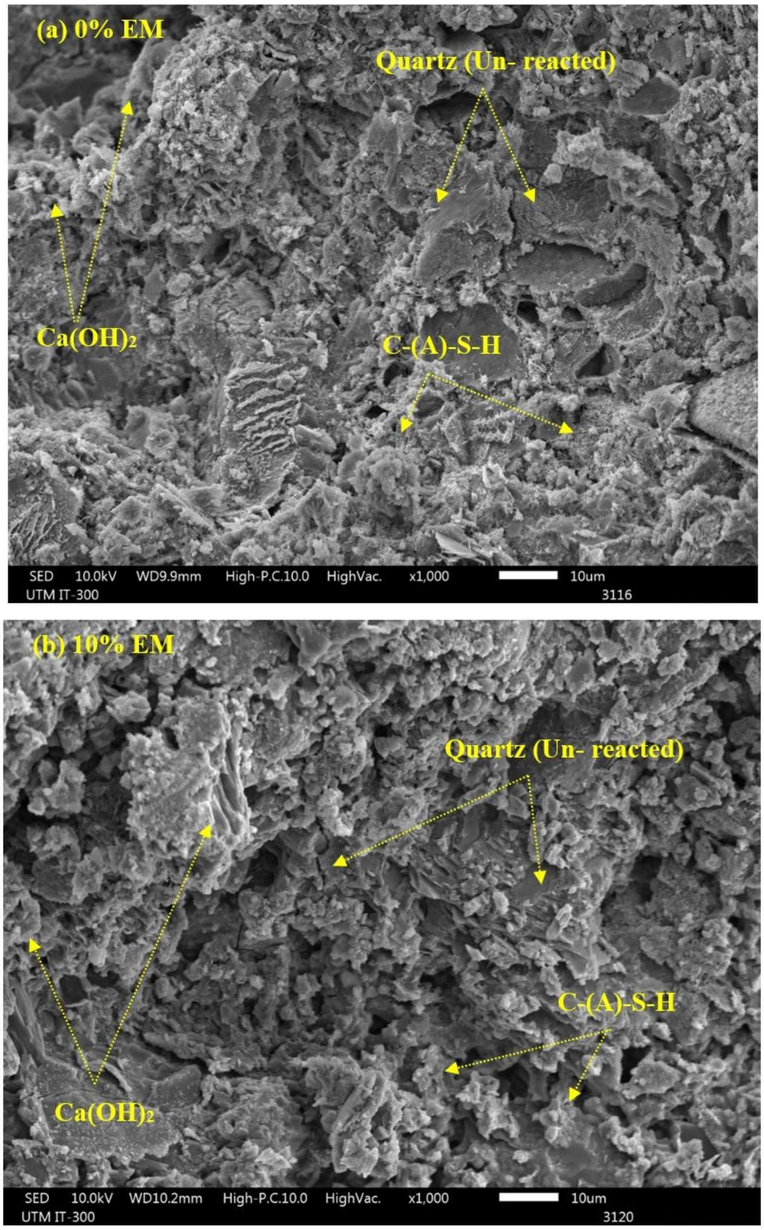


Figure 16. SEM images of self-cured concrete (at 7 days of age) containing (**a**) 0% and (**b**) 10% of EM.

Figure 17 illustrates the SEM images of the self-cured concrete (at 28 days of air curing) obtained without and with EM of 10% as water replacement. The self-cured concrete prepared with 10% of EM showed very dense surface morphology with little fraction of partly reacted and unreacted particles. The SEM microstructure of the control sample (0% of EM) displayed the existence of high amount of partially reacted and unreacted particles (Figure 17a) compared to the one made with 10% of EM (Figure 17b). The inclusion of EM was observed to cause less amount of unreacted silica formation, lower porosity, and enhanced morphology. Consequently, the CS of the proposed concrete containing 10% of EM was higher (42.8 MPa) than the control sample (35.8 MPa).

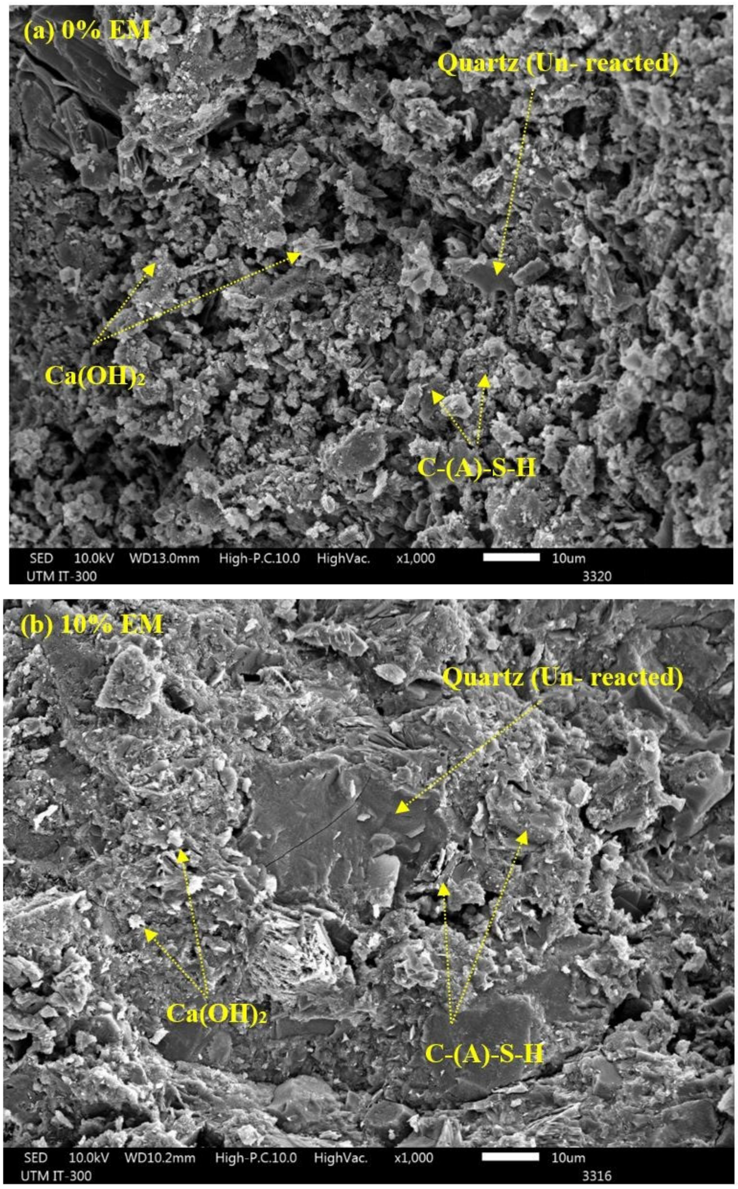


Figure 17. SEM images of self-cured concrete (at 28 days of age) containing (**a**) 0% and (**b**) 10% of EM.

3.13. EDX Analysis

Figure 18 presents EDX spectra and maps of the self-cured concrete (at 28 days of air curing) obtained with and without EM of 10% as water replacement. The EDX spectra detected the right elemental composition of the studied self-cured concrete. The results showed an increase in the ratio of CaO to SiO_2 from 4.31 to 4.52 due to the addition of 10% of EM in the concrete mixes. Furthermore, the ratio of SiO_2 to Al_2O_3 was dropped from 3.38 (for control specimen) to 2.75 (specimen containing 10% of EM). Compared to the control sample (with 0% of EM) the specimen made with 105 of EM showed relatively low amount of SiO_2/Al_2O_3, indicating the substitution of higher number of Al ions in the C-(A)-S-H chain. The observed increase in the CS of the concrete containing 10% of EM can be attributed to the increase in dissolved Al_2O_3 and CaO that led to the formulation of more gels compared to the control sample.

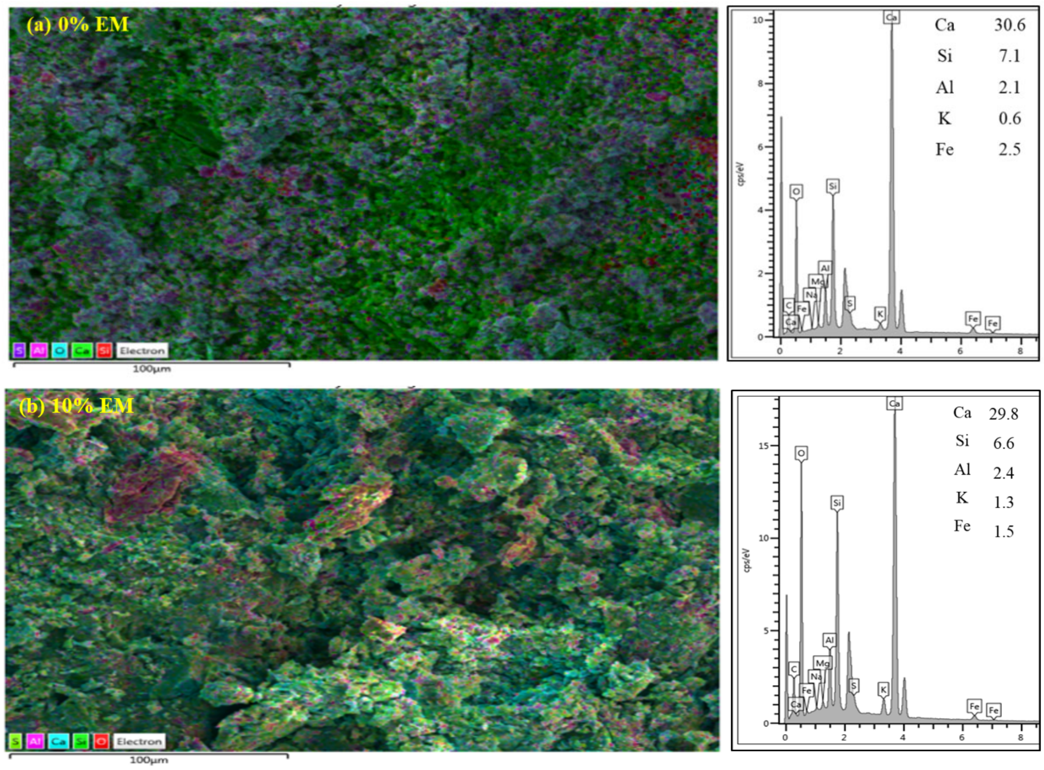


Figure 18. EDX images of the self-cured concrete (at 28 days of age) containing (**a**) 0% and (**b**) 10% of EM.

3.14. TGA and DTG Results

Figure 19 shows the TGA curves of the self-cured concrete of the self-cured concrete (at 28 days of air curing) made without and with EM of 10% as water replacement. Both curves indicated that the percentage of C-S-H gel in the specimen containing 10% of EM was higher (5.22%) than the one prepared without EM (4.34%). Further, the percentage of calcium hydroxide (Table 7) in the self-cured concrete made with 10% of EM was higher (0.33%) compared to the OPC specimen (0.28%). On top, the higher stability of the self-cured concrete made with 10% of EM was mainly due to the formation of high amount of C-S-H gel and $Ca(OH)_2$. These observations clearly verified the benefits of EM incorporation into the concrete mixes that led to an enhancement in its microstructure properties and CS from 35.8 to 42.8 MPa at 28 days of air curing. The TGA and DTG results determined the weight loss percentage of the proposed self-curing concrete mixes. The amount of calcium hydroxide (CH) and calcium silicate hydrate (C-S-H) present in the sample were calculated using Equations (10) and (11), respectively:

$$\text{CH (\%)} = \text{WL CH (\%)} \times [\text{MW CH/MW H}] \tag{10}$$

where WL(CH) is the weight loss due to CH dehydration, MW(CH) is the molecular weights of CH (74 g·mol^{-1}), and MW(H) is the molecular weight of water (18 g·mol^{-1}).

$$\text{C-S-H gel (\%)} = \text{Total LOI} - \text{LOI CH} - \text{LOI CC} \tag{11}$$

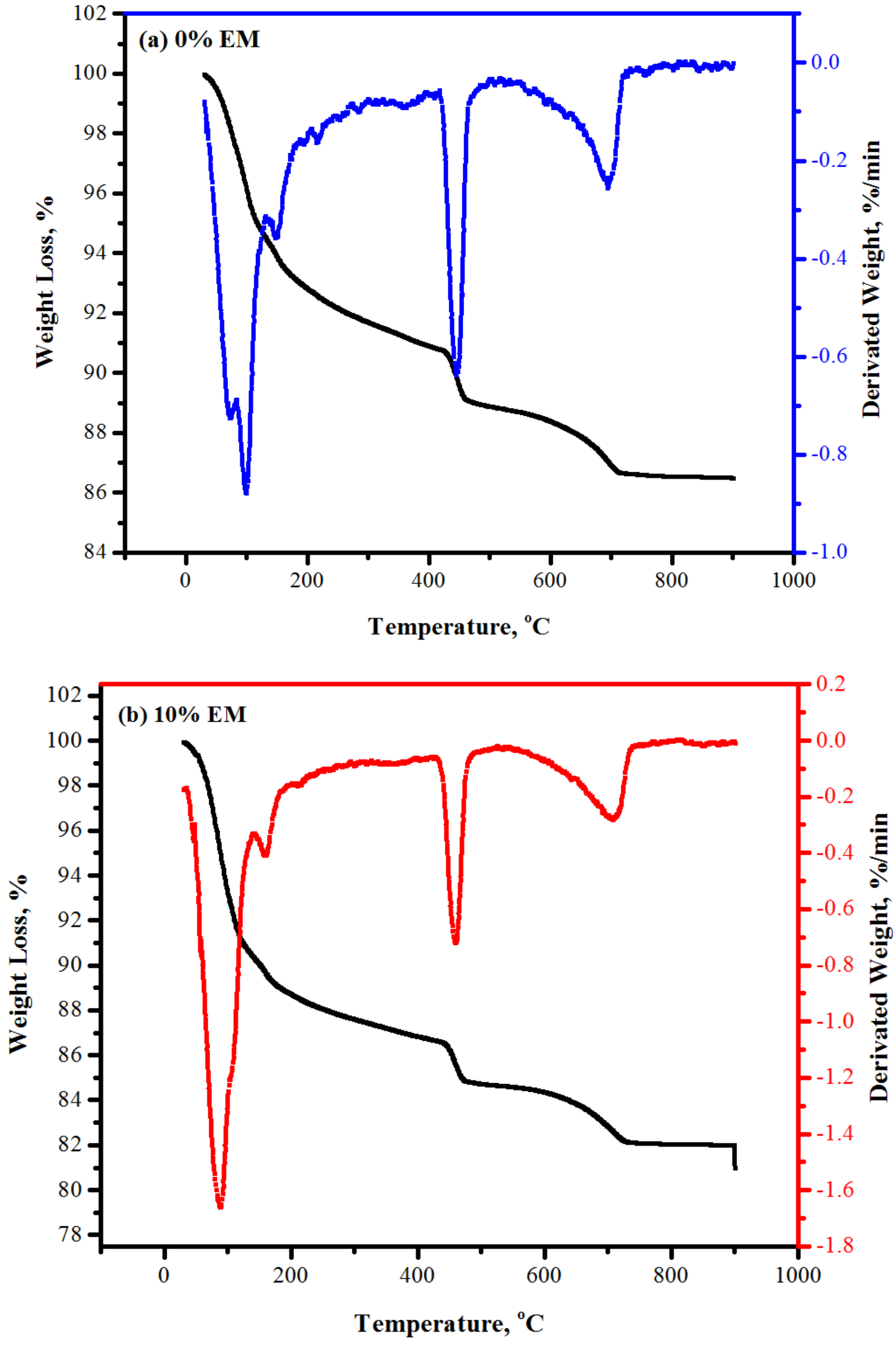


Figure 19. TGA and DTG curves of the self-cured concrete (at 28 days of age) containing (**a**) 0% and (**b**) 10% of EM.

Table 7. Amount (%) of C-S-H gel and $Ca(OH)_2$ present in the sample.

Chemical Compounds	3 Days		7 Days		28 Days	
	0% EM	10% EM	0% EM	10% EM	0% EM	10% EM
C-S-H gel	3.15	2.9	3.64	4.13	4.34	5.22
CH (Portlandite)	0.45	0.50	0.30	0.40	0.28	0.33

3.15. Ultrasonic Pulse Velocity

Figure 20 shows the curing age-dependent values of ultrasonic pulse velocity (UPV) of the self-curing concrete mixes prepared with various amounts of EM (from 0 at 25%) as water replacement. At 3 days of age, the control concrete mixture attained an UPV value of 3.4 km/s, whereas the mixes containing 5 to 25% of EM gained the corresponding UPV values in the range of 3.2 to 2.9 km/s which were lower than the one showed by the control specimen. At 7 days of curing age, the specimens made with 5 and 10% of EM attained the UPV values of 4 and 4.1 km/s, respectively, which were higher than the one attained by the control specimen (3.7 km/s). However, the specimen made with 15, 20, and 25% of EM achieved the corresponding UPV values of 3.7, 3.5, and 3.3 km/s. Specimen containing 15% of EM showed the same UPV value as the control sample. Conversely, the specimens made with 20 and 25% of EM displayed lower UPV values than the control sample. At 28 days of age, the specimen composed of 5 and 10% of EM reached the respective UPV values of 4.8 and 5.1 km/s which were again higher than the control specimen (4.4 km/s). Nevertheless, the mixes designed with 15, 20, and 25% of EM attained the corresponding UPV values of 4.3, 4, and 3.9 km/s. A sample containing 15% of EM had shown slightly lower UPV value than the control sample, but the sample composed of 20 and 25% of EM showed somewhat lower UPV values than the control specimen. At 56 and 90 days of age, the concrete mixes composed of 5 and 10% of EM achieved the respective UPV values of 4.85 and 5.15 km/s, wherein these values were higher than the control sample (4.45 km/s). However, the specimens made with 15, 20, and 25% of EM achieved the corresponding UPV values of 4.4, 4.1, and 3.9 km/s. Yet, again, the self-curing concrete made with 15% of EM showed slightly lower UPV value than the control specimen but specimen containing 20 and 25% of EM showed lower UPV values than the control specimen (0% of EM).

The UPV values are the indirect measure of the presence of internal voids in the concrete specimens. In addition, it indirectly determines the homogeneity of the concrete network structure. At 3 days of age, the observed reduction in the UPV values of all concrete mixtures containing EM compared to control specimen was mainly due to the presence of molasses in the network matrix that slowed down the hydration process, enabling the cement particles to form C-S-H gel, thus filling the voids. However, at a later age under both water and air curing, the specimen made with 5 and 10% of EM achieved higher UPV values compared to the control mixture, which was mainly due to the complete hydration, presence of C-S-H gel and filling of the voids. According to Akar and Canbaz [52], the addition of molasses can reduce the capillary voids in the concrete network, thus resulting in higher UPV value of the mixes. Ali and Qureshi [45] also claimed that the molasses addition can reduce the voids in the concrete network. In contrast, specimen prepared with 15% of EM behaved differently under both water and air curing compared to other specimens wherein the UPV values were somewhat lower in water curing and higher in air curing. This observation can be ascribed to the presence of curing by water that slowed down the hydration reaction compared to air curing. In addition, the specimen made with 20 and 25% of EM attained lower values of UPV compared to the control sample in all curing ages. This may be due to the occurrence of bleeding in the concrete matrix that disabled the formation of homogeneous structures. In addition, the slow hydration process of the proposed self-curing concrete mixes caused by the presence of molasses played a significant role on the UPV values attainment.

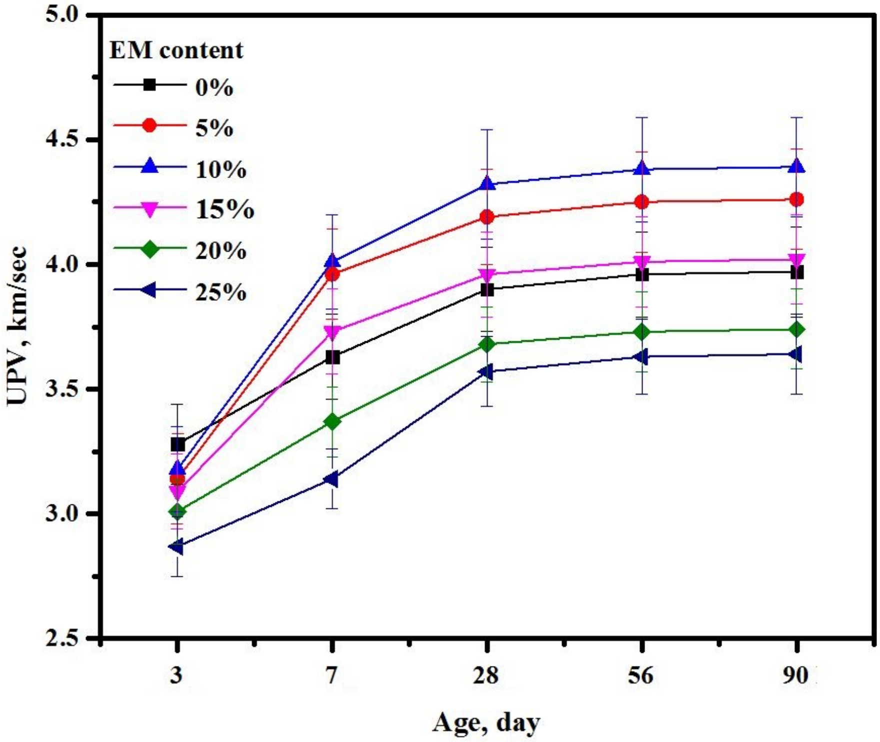


Figure 20. The curing age-dependent UPV values of the self-curing concrete specimens.

3.16. Splitting Tensile Strength

Figure 21 illustrates the effect of EM on the STS of the self-curing concrete obtained at different air curing ages. At 3 days of age, the concrete mixtures made with 5 to 25% of EM gained the corresponding STS in the range of 15 to 43% that was lower than the control sample. At 7 days of age, the concrete mixtures made with 5 to 15% of EM achieved the corresponding STS in the range of 9 to 4% that was higher compared to the control mixture. However, with the increase in EM content from 20 to 25% the STS was increased from the attained 17 and 33%, respectively, that were again lower than the control specimen. At 28 days of curing age, specimens made with 5, 10, and 15% of EM achieved the STS of 19, 29, and 5%, respectively, that were higher compared to the control specimen. However, the mixes designed with 20 and 25% of EM attained the STS of 11 and 26%, respectively, which were lower than the control sample. At 56 and 90 days of curing age, similar trends in the STS values were observed, wherein an increase in EM content up to 15% led to a drop in the STS, thus displaying poorer performance of the self-curing specimen compared to the control sample.

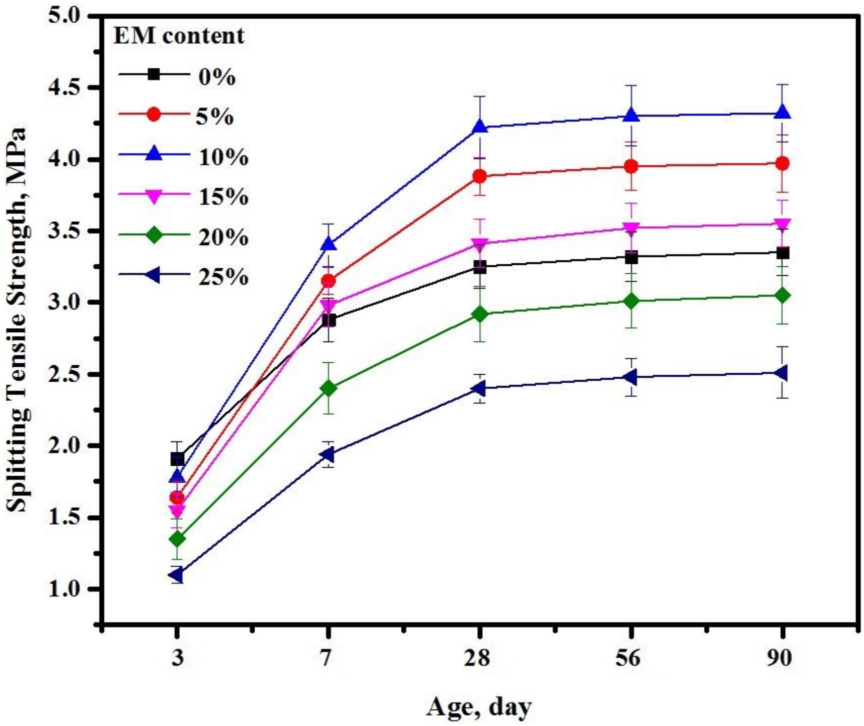


Figure 21. Effect of EM on the STS of the self-curing concrete at different air curing ages.

3.17. Flexural Strength

Figure 22 illustrates the effect of EM on the FS of the self-curing concrete obtained at different air curing ages. At 3 days of age, the concrete mixtures made with 5 to 25% of EM gained the corresponding FS in the range of 18 to 50% that were lower than the control specimen. At 7 days of curing age, the specimens made with 5, 10, and 15% of EM achieved the corresponding FS of 15, 25, and 3% that were higher compared to the control mixture. However, the specimen composed of 20 and 25% of EM attained the FS values of 28 and 44%, respectively, that were lower than the control sample. At 28 days of curing age, the mixes designed with 5, 10, and 15% of EM achieved the corresponding FS of 17, 27, and 4% that were higher than the control mix. Conversely, the specimens made with 20 and 25% of EM attained the FS of 13 and 22%, respectively, which were lower than the control mixture. Likewise, the results at 56 and 90 days of curing age the mixes designed with EM showed a drop in the strength performance with the increase in EM content up to 15% compared to the control sample.

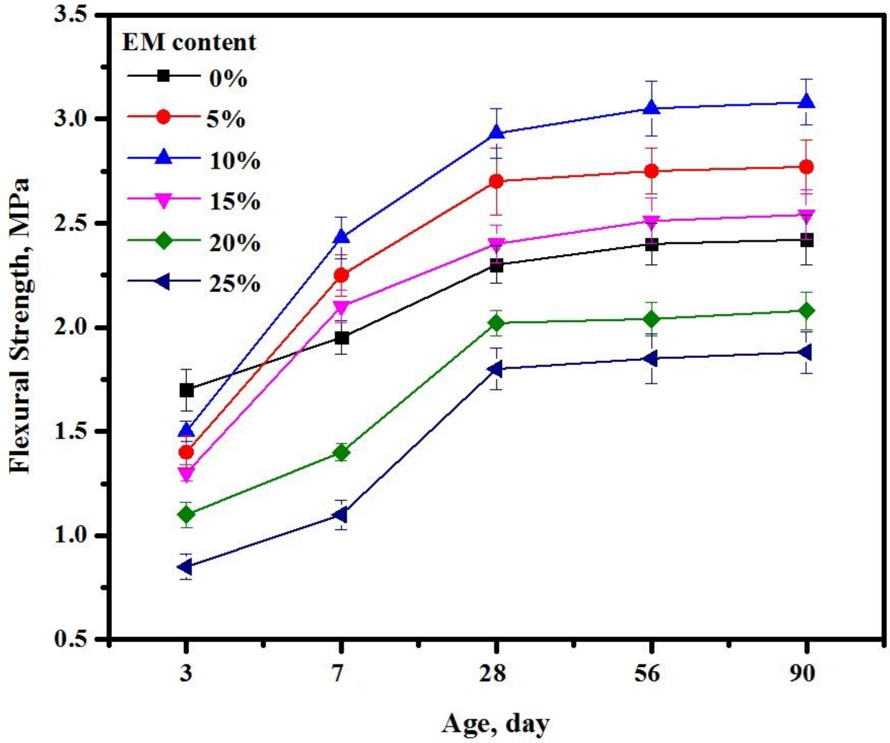


Figure 22. Effect of EM on the FS of the self-curing concrete at different air curing ages.

3.18. Correlation between Compressive, Splitting Tensile and Flexural Strengths

Figure 23 depicts the correlation between the CS and STS, as well as Cs and FS of the self-curing concrete at different curing ages. These relationships provided (obtained via the regression analysis of analytical models) a clear proof regarding the consequences of EM incorporation into the self-curing concrete mixes that could significantly affect their strength properties. These correlations seem to fit well with the power laws. The regression line approach was used for the overall data, where the CS was used as the predictor parameter whereas the STS and FS acted as the response parameters. A good relationship between the TS and CS of the proposed concrete mixes at all curing ages were observed. The coefficient of determination (R^2) for the CS-STS and CS-FS correlations were 0.97 and 0.96, respectively. These valued confirmed a good and reliable relationship between these mechanical strength values of the proposed self-curing concretes. This good relation among various mechanical properties clearly indicated the usefulness of effective microorganism inclusion into the self-curing concrete mixes required for diverse civil engineering construction applications. In addition, it suggested the possible way of customizing the mechanical properties of the self-curing concretes, such as CS, STS, and FS.

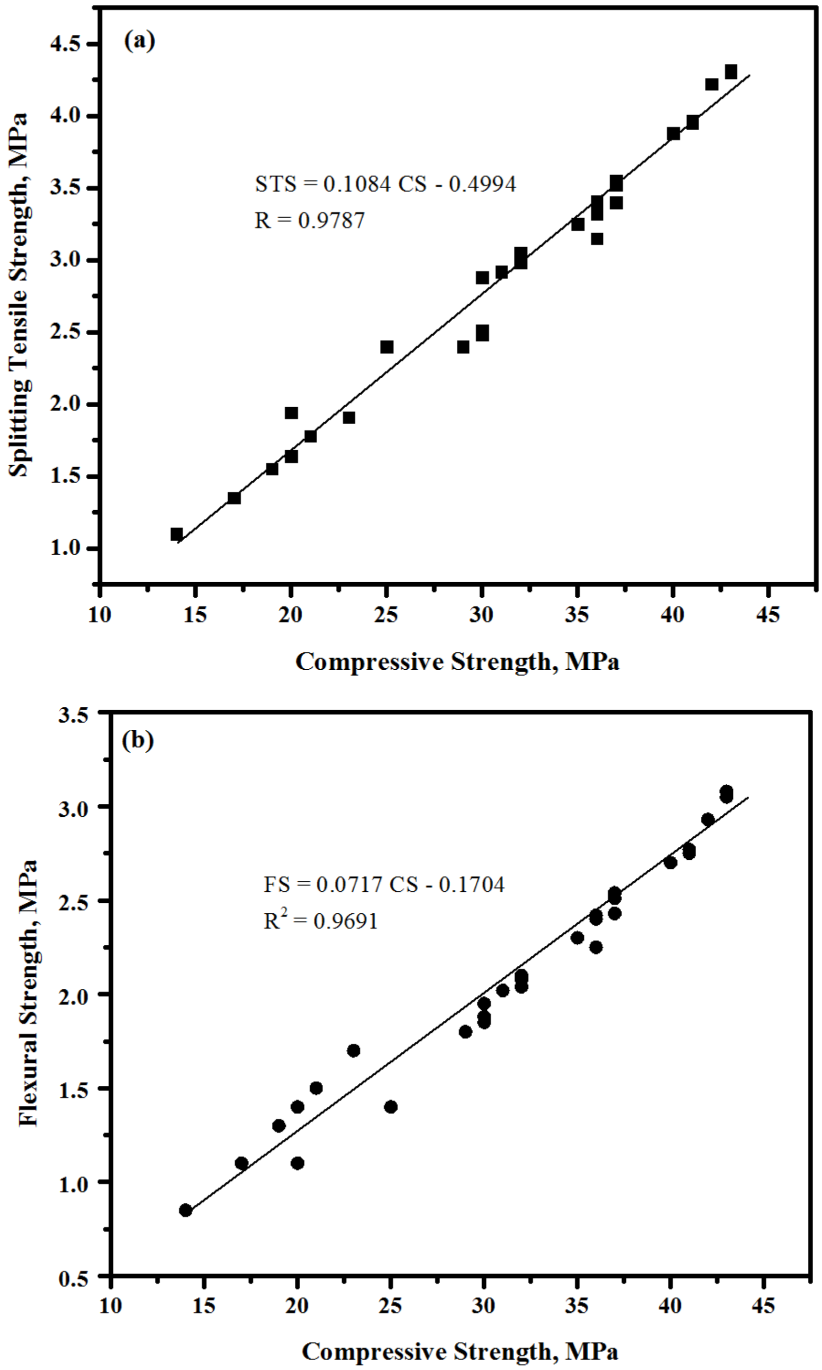


Figure 23. The correlation of CS with (**a**) STS, and (**b**) FS of the self-curing concrete at different curing ages.

4. Conclusions

This paper reports the use of EM as a self-curing agent to produce high-performance concrete. Based on the obtained experimental results and detail analyses the following conclusions can be made:

i The physical and chemical properties of EM, solution with 10% of EM as water replacement and pure water were examined in terms of surface tension, viscosity, and pH. These characteristics enabled us to understand the mechanism of concretes' strength improvement due to the inclusion of EM;

ii Workability of the fresh concrete mixes was improved with the increase in EM contents as water replacement wherein a maximum replacement of water by EM caused the shear and bleeding of the fresh concrete;
iii The pH values of the fresh concrete mixes made with EM were lowered compared to the control specimen;
iv The water loss of the mixes made with 5, 10, and 15% of EM was reduced compared to the control specimen. The minimum water loss was obtained for the self-curing concrete made with 10% of EM as water replacement;
v The effect of molasses on development of early strength of proposed were evaluated. It was found the compressive strength slightly increased which indicated to impossibility using molasses as self-curing agent;
vi In normal concrete, the EM solution performs very well as a self-curing agent. Concrete mixes containing EM solution under air curing showed comparable CS properties as that of normal concrete under water curing. The strength properties of all the proposed concrete mixes made with EM (except the one made with 10% of EM) displayed similar patterns at all curing ages. The specimen made with placed with 10% of EM achieved maximum CS under water and air curing after 7 days of age;
vii XRD, TGA, and DTG results indicated that the self-curing concrete made with 10% of EM have more amount of Portlandite, calcite, Ettringite and the lower amount of quartz than the control specimen;
viii SEM images displayed denser structure of the specimen composed of 10% of EM as water replacement compared to the one made without EM. Concretes containing EM as a self-curing agent showed the presence of a high amount of Portland, as well as C-S-H gel and a low amount of quartz;
ix Inclusion of 5 and 10% of EM in the proposed mix design significantly improved the STS and FS performance compared to the control specimen;
x A good correlation was observed between the CS, STS, and FS of the designed self-curing concretes;
xi The achieved enhanced microstructures and strength properties of the proposed self-curing concretes containing effective microorganism may be useful for the sustainable development in the construction industries.

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Abbreviations

EM	Effective microorganism
EM-AS	EM-activated solution
CS	Compressive strength
STS	Splitting tensile strength

FS	Flexural strength
C-S-H	Calcium silicate hydrate
CH	Portlandite ($Ca(OH)_2$)
CO_2	Carbon dioxide
OPC	Ordinary Portland cement
SSD	Saturated surface dry
XRD	X-ray diffraction
SEM	Scanning electronic image

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