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Eggshell as a partial cement replacement in concrete development

Yeong Yu Tan
Master Candidate, Faculty of Civil Engineering and Earth Resources,
Universiti Malaysia Pahang, Pahang, Malaysia

Shu Ing Doh
Senior Lecturer, Faculty of Civil Engineering and Earth Resources,
Universiti Malaysia Pahang, Pahang, Malaysia (corresponding author:
dohsi@ump.edu.my)

Siew Choo Chin
Senior Lecturer, Center of Excellence for Advanced Research in Fluid Flow,
Universiti Malaysia Pahang, Pahang, Malaysia; Faculty of Civil Engineering
and Earth Resources, Universiti Malaysia Pahang, Pahang, Malaysia

Research on the reuse of waste materials in the concrete industry has been quite intensive in the past decade. The objective of this research is to identify the performance of oven-dried eggshell powder as a partial cement replacement in the production of concrete under both water-cured and air-cured regimes. Eggshell powder of various amounts, namely 5%, 10%, 15% and 20% by volume, was added as a replacement for ordinary Portland cement. The results showed that water-cured eggshell concrete greatly improved the compressive and flexural strength of concrete, by up to 51·1% and 57·8%, respectively. The rate of water absorption of eggshell concrete was reduced by approximately 50%, as eggshell powder filled up the existing voids, making it more impermeable. However, the compressive strength of the eggshell concrete decreases gradually when the amount of eggshell powder increased, during immersion in acid and alkali solutions, because eggshell contains a high amount of calcium, which reacts readily with acid and alkali solutions. As the eggshell content increases, the solution reacts with the paste so the bonding of the paste reduces, and therefore the strength also reduces. The reduction of compressive strength during immersion in sulphuric solution and sodium sulphate solution was 27·5% and 31·2%, respectively, when 20% eggshell powder was used to replace cement. It can be concluded that the optimum percentage of oven-dried eggshell powder as a partial cement replacement is 15%.

Introduction

Concrete materials are extensively used in the construction industries. One of the essential ingredients, ordinary Portland cement (OPC), is generally expensive and yields carbon dioxide emissions during production. Approximately 900 kg of carbon dioxide greenhouse gases is produced to make 1 t of cement. As a result, the social and environmental issues of sustainability and energy conservation are encouraging the cement industry to lower and partially replace its cement production with supplementary cementing materials. Nowadays, various waste materials are being studied and utilized as raw materials in concrete production. Recycled materials have been added to concrete to reduce the amount of post-consumer waste and industrial byproducts entering landfills (Naik, 2002). Eggshell is a solid waste generated from chick hatcheries, bakeries and fast-food restaurants (Sivakumar and Mahendran, 2014). In Malaysia, it is reported that approximately 36·5 million eggs are produced daily, and this figure is expected to increase by 3–5% in 2018 (Stock Hut, 2015). In fact, for every billion eggs produced, 6600 t of high-grade lime powder could be produced (Duncan and Allison, 2015). As an alternative, eggshell could be used as a partial or total substitute for natural mined limestone. Moreover, the main component of eggshell is calcium carbonate, which is very similar to cement (Okonkwo *et al.*, 2012). Thus, in our ever-increasing efforts to convert waste into wealth, the efficacy of putting eggshells to beneficial use becomes an idea worth embracing.

Few attempts have been made in the past by researchers to utilize eggshell powder. The common salt in eggshell has been used to stabilize lateritic soil in the subgrade during roadworks (Amu and Salami, 2010). It was additionally found that, while eggshell powder possesses low binding properties, it significantly improved the strength of the subgrade soil (Olarewaju *et al.*, 2011). Furthermore, eggshell may be a good accelerator because it provides additional calcium oxide, which is responsible for expediting both the initial and final setting of concrete (Mtallib and Rabiu, 2009). During prolonged periods of heavy rain, construction works may frequently be interrupted and it is thus desirable to minimize the length of the setting time of the stabilized matrix as much as possible.

This study focuses on the performance of concrete using eggshell powder as a partial cement replacement at volumes of 5%, 10%, 15% and 20%. The analysis of eggshell concrete was divided into three categories: the characteristics of eggshell powder, the mechanical properties of eggshell concrete, and the performance of eggshell concrete in terms of durability.

Experimental investigation

Materials

The eggshells were obtained from Eggtech Manufacturing Sdn Bhd, located at Puncak Alam, Malaysia. The eggshells were obtained from the factory in pre-compacted and semi-crushed condition. They were packed in 8 l plastic bags, as


(a) (b)

Figure 1. Eggshell powder

Table 1. Mix proportions for concrete with different amounts of eggshell powder replacement

Materials: kg/m^3	C	WO 5%/AO 5%	WO 10%/AO 10%	WO 15%/AO 15%	WO 20%/AO 20%
Cement	400	380	360	340	320
Coarse aggregate	1120				
Fine aggregates	690				
Eggshell: %	—	5	10	15	20
Water/cement ratio	0·45				

Note: A, air curing; C, control specimens; O, oven-dried eggshell powder; W, water curing

shown in Figure 1(a). The eggshells were immediately washed with tap water after retrieval from the factory. Then the washed eggshells were oven-dried at 105°C for 24 h. Finally, the eggshells were ground into powder using a grinding machine. Only eggshell powder with particles smaller than 60 μm, as illustrated in Figure 1(b), was used in the research. Concrete cubes 100 × 100 × 100 mm and beams 100 × 100 × 500 mm were used as the test specimens for this research. Details of the proportions of the concrete mixture are tabulated in Table 1. The fraction of eggshell powder replacement was 5%, 10%, 15% and 20% by volume. All of the concrete cubes were demolded after 24 h and underwent water curing and air curing. On each test date, the samples were removed from the water bath and the surface allowed to surface dry prior to the conduct of the analysis.

Methodology

X-ray fluorescence (XRF) analysis

XRF analysis was performed using a Bruker S8 TIGER spectrometer to analyse the chemical composition of the eggshell powder.

X-ray diffraction (XRD) analysis

XRD analysis of the eggshell samples was recorded using Philips X'Pert Pro Diffraction with copper (Cu)-Kα radiation at an operating voltage of 45 kV, a current of 40 mA and a scanning speed of 0·02°/min. The measurement of the pattern was made at the diffraction angle of $2\theta/\theta$ ranging between 3° and 80°. XRD was used to identify the crystalline structure and qualitative composition of the eggshell powders after grinding.

Microstructure characterization

Microstructural analysis of the eggshell concrete specimens was conducted using a scanning electron microscope (SEM), in this instance the Hitachi TM 3030 Plus. The samples were mounted on a small stub using double-sided carbon tape.

Mechanical properties analysis

The compression strength test was carried out using a MATEST S.p.A. Treviolo testing machine with a loading rate of 2·4 kN/s, while the flexural strength test employed a U-test machine with a loading rate of 0·5 kN/s. The samples were tested at room temperature after 1, 7, 28, 56 and 90 d of full water curing. Both compressive and flexural strength tests consisted of three specimens at each composition and were averaged respectively. Subsequently, a compression test was performed according to BS 1881: Part 116 (BSI, 1983a), while a flexural strength test was performed according to BS 1881: Part 112 (BSI, 1983b).

Durability properties analysis

Durability analysis for the specimens involved water absorption, acid attack and sulfate resistance. The concrete cubes were selected to identify the durability performance, including mechanical properties and mass change. A water absorption test was performed to identify the rate of water absorption in the concrete specimens. The concrete cube specimens were dried in the oven at 105·5°C for 24 h. The initial weight of the specimens was measured. Then the specimens were submerged in the water before the surface was wiped dry and the weight of the specimens finally taken. The concrete cubes were repeatedly measured at 1, 5, 10, 15, 30, 60, 120, 180, 1440, 2880 and 4320 min. The testing procedure was conducted according to BS 1881: Part 122 (BSI, 2011).

All the concrete cubes were demolded after 24 h before subjected to full water curing and air curing for 28 d prior to immersing them in a 5% sodium sulfuric acid solution for 1800 h and a 5% sodium sulfate solution for 50 weeks respectively. The durability of both ordinary concrete and eggshell concrete mixes was determined by measuring the mass loss and reduction of compressive strength.

Results and discussions

Chemical composition

The chemical composition of eggshell powder and OPC is shown in Table 2. The major component in the oven-dried eggshell powder is calcium oxide, which represents 62·35%. Calcium oxide is one of the key elements required for strength development during the hydration process. It is for this reason that oven-dried eggshell powder may be used as partial cement replacement.

Table 2. Percentage of oxide groups contained in cement and oven-dried eggshell powder

Oxide group	Cement	Oven-dried eggshell powder: %
Calcium oxide	60·10	62·35
Sulfur trioxide	2·50	1·32
Iron oxide	4·10	0·63
Silicon dioxide	21·80	0·61
Magnesium oxide	0·50	0·36
Potassium oxide	0·25	0·22
Aluminium oxide	6·66	0·07

XRD results

The XRD patterns of the eggshell powder are shown in Figure 2. During the production of the eggshell powder, the majority of the membrane was removed since it is considered as an amorphous group that is not detected during XRD analysis (Pliya and Cree, 2015). The characteristics of the crystalline XRD peaks showed that the major component in eggshell powder is calcium carbonate ($CaCO_3$) in the form of calcite. Calcite was reported to be the most thermodynamically stable component under ambient conditions (Schultz *et al.*, 2013). The XRD peaks for the calcite appeared at approximately 2θ angles on 23·64°, 29·1°, 36·54°, 39·5°, 44·1°, 47·1°, 49·44°, 57·2°, 62·5°, 70·1°, 72·24° and 77·8°, with a major peak at 29·1°. The intensities of the calcite peaks were comparatively indistinguishable with no other crystalline phase formed, as a previous investigation has similarly shown (Intharapat *et al.*, 2013).

Slump result

A slump test was carried out to determine the workability of the concrete prior to casting. The results of the slump test are illustrated in Figure 3. Based on the slump result, it can be suggested that all the slumps can be categorized as true slump ranging between 70 and 84 mm. In addition, all the slump results fall into the acceptable slump, which is 75 ± 25 mm. Thus, the eggshell powder does not absorb water excessively.

Compressive strength

The compressive strength performance of eggshell concrete is illustrated in Figures 4 and 5. The continuous water supply to the concrete specimens allows a better hydration process and pozzolanic reaction to take place, resulting in a larger amount of calcium silicate hydrate (C–S–H) gel filling the voids, thus ultimately increasing concrete strength (Khairunisa *et al.*, 2015a).

It is interesting to note that eggshell concrete exhibits high early compressive strength compared with the control specimen

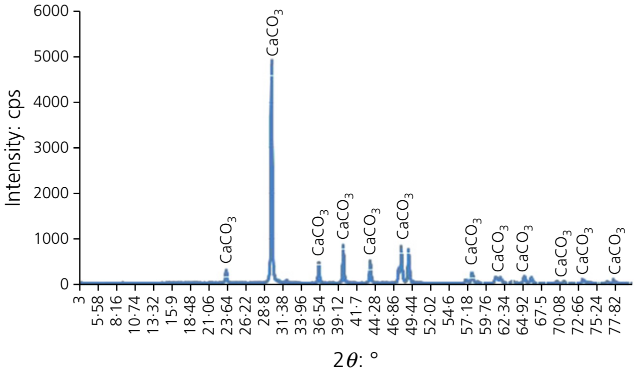

Figure 2. XRD pattern of eggshell powder

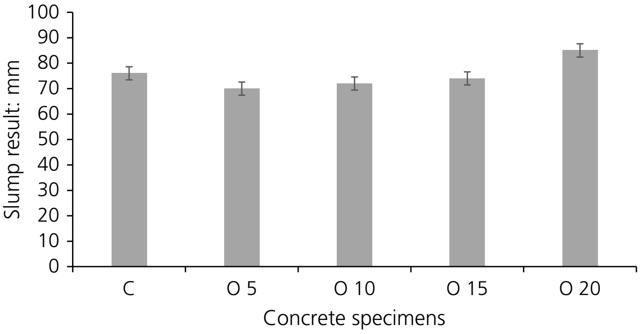

Figure 3. Slump result for eggshell concrete specimens. C, control; O, oven-dried eggshell

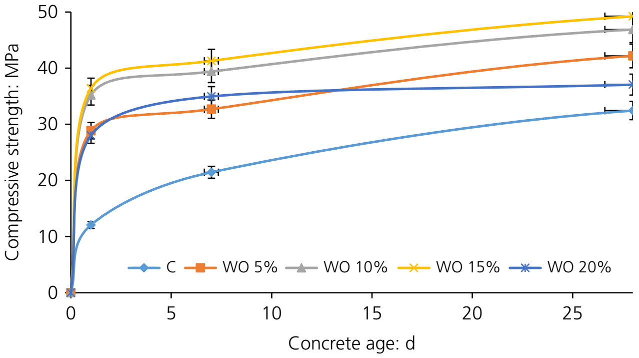

Figure 4. Compressive strength results for concrete specimens produced using full water-curing. C, control, WO, water-cured, oven-dried eggshell

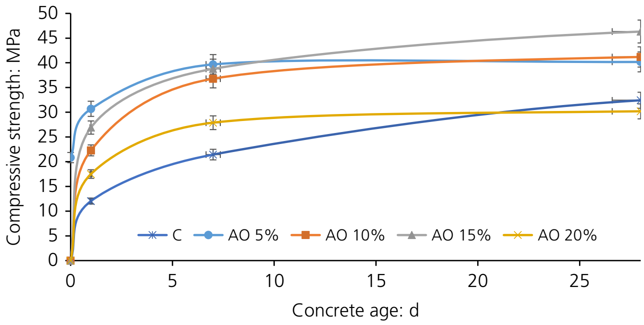

Figure 5. Compressive strength results for concrete specimens produced using air-curing. AO, air-cured, oven-dried eggshell; C, control

at the early stage of curing. Partial replacement of cement with eggshell powder improved the reaction between silica from the cement and calcium oxide from the eggshell powder, a byproduct of the cement hydration process in the continuous presence of moisture leading to the formation of the secondary C–S–H gel. In addition, the eggshell powder also acted as a filler by filling up the existing voids, which made the internal structure of the concrete more packed and led to the development of higher compressive strength. WO 15% had the highest compressive strength among all the concrete specimens, at 48·36 MPa at the age of 28 d.

Air-cured concrete cube specimens exhibited lower compressive strength than fully water-cured concrete cube specimens, as shown in Figures 3 and 4. Evidently, the concrete cube cured within an air-curing environment depended only on the concrete moisture itself for the hydration process as well as pozzolanic activity. Insufficient moisture can retard the formation of primary and secondary C–S–H gel during the hydration process and pozzolanic activity. Thus, the compressive strength for air-cured concrete specimens was lower. The optimum for air-cured concrete was AO 15%, which reached 45·1 MPa at the concrete age of 28 d. Clearly, the inadequate supply of moisture had greatly affected the compressive strength development of the eggshell concrete. The compressive strength performance of eggshell concrete is illustrated in Figures 4 and 5. The continuous water supply to the concrete specimens allowed a better hydration process and pozzolanic reaction to occur, resulting in the formation of a larger amounts of C–S–H gel to fill the voids, which in turn eventually increased concrete strength (Khairunisa *et al.*, 2015a, 2015b).

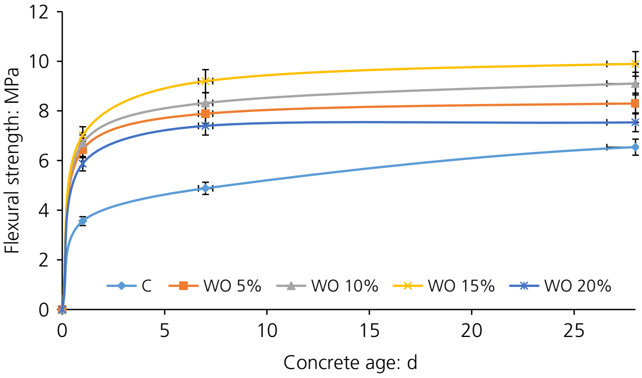

Figure 6. Flexural strength results for concrete specimens produced using full water-curing. C, control; WO, water-cured, oven-dried eggshell

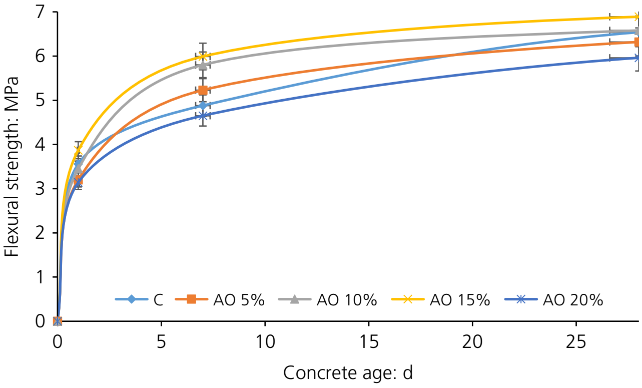

Figure 7. Flexural strength results for concrete specimens produced using air-curing. AO, air-cured, oven-dried eggshell; C, control

Flexural strength

Figures 6 and 7 show the effect of the curing regime on the flexural strength of eggshell concrete beams. Evidently, water curing is the most suitable type of curing in order to obtain a better flexural strength of the eggshell concrete beams compared to the air-curing method. The satisfactory performance of the water-cured eggshell concrete is due to the improvement in the pore structure; the concrete may have lower porosity as a result of the high degree to which the hydration process takes place without any moisture being evaporated into the air. In addition, calcium hydroxide contributes slightly to the strength and impermeability of the concrete because it reduces the total pore volume by converting some of the liquid water into solid form (Neville, 2011). As can be seen in Figures 6 and 7, it is significant that eggshell concrete for both different curing methods had high early flexural strength development compared to the control specimens. The importance of the presence of water in the curing process has been highlighted by previous researchers (James *et al.*, 2011; Popovics, 1986).

Application of water curing to the eggshell concrete beams greatly enhanced its flexural strength properties, as illustrated in Figure 6. WO 15% had the highest early strength of 7·8 MPa when the concrete was at the age of 1 d because the replacement of 15% of eggshell powder had increased the amount of calcium hydroxide and allowed the percentage of formation of C–S–H gel to increase at an early stage of the hydration process (Fadzil *et al.*, 2015). At the age of 28 d, WO 15% exhibits the highest flexural strength of 10·1 MPa, which is 57·8% higher than that of the control beam, 6·4 MPa. Thus, the eggshell powder seems to have greatly improved the flexural strength of concrete with a partial cement replacement of up to 15% under a water-curing regime. Although the flexural strength of the concrete beam reduced when the replacement was 20%, it was still 6·3% higher than the control beam, at 6·8 MPa, at an age of 28 d. The optimum volume of eggshell powder as the partial cement replacement under water-cured conditions is 15%.

The performance of the air-cured concrete beam specimens was clearly lower than the water-cured specimens. The decline in flexural strength of air-cured eggshell concrete AO 5%, AO 10%, AO 15% and AO 20% compared to the water-cured specimens was 19·7%, 20·1%, 20·8%, and 26·51% for WO 5%, WO 10%, WO 15% and WO 20% respectively. The non-availability of water for air-cured concrete specimens retards the hydration process and pozzolanic reaction, leading to a lesser amount of C–S–H gel and thereby causing the concrete beams to possess lower flexural strength compared to the water-cured specimens (Isaia *et al.*, 2016). Clearly, the inadequate supply of moisture reduces the flexural strength development of the eggshell concrete. The reduction in strength may be due to the lower availability of the cementing properties found in the Portland cement (Yan, 2010). According to Figure 8, 5–10% is the desirable percentage to use as partial cement replacement. The eggshell powder is generally satisfactory for use as a partial cement replacement when not exceeding this range.

Water absorption

The results of the rate of water absorption for water-cured specimens and air-cured specimens are shown in Figures 9 and 10 respectively. Referring to Figure 9, we can see that the permeability of the eggshell concrete for water-cured concrete specimens was greatly reduced. The rate of penetration measured by the rate of water absorption for water-cured specimens was reduced by up to 75%. Thus, eggshell powder may be good filler for concrete as it provides extra calcium to produce more secondary C–S–H gel (Doh *et al.*, 2014). As a

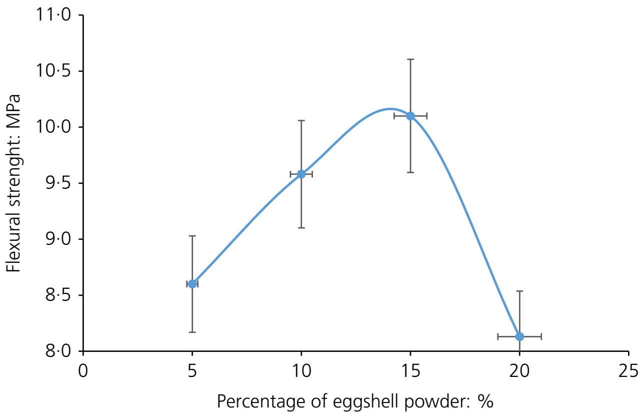

Figure 8. Change in flexural strength with different percentages of eggshell powder replacement

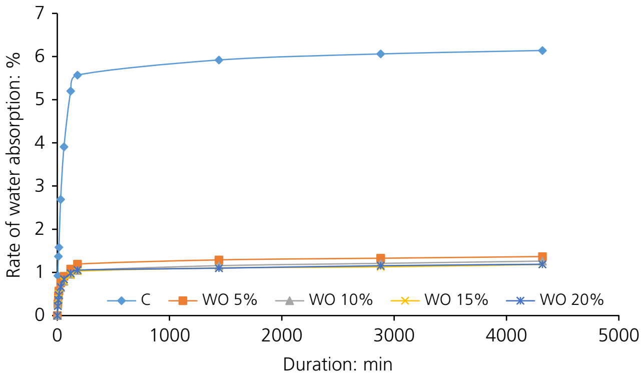

Figure 9. Rate of water absorption for water-cured concrete specimens. C, control, WO, water-cured, oven-dried eggshell

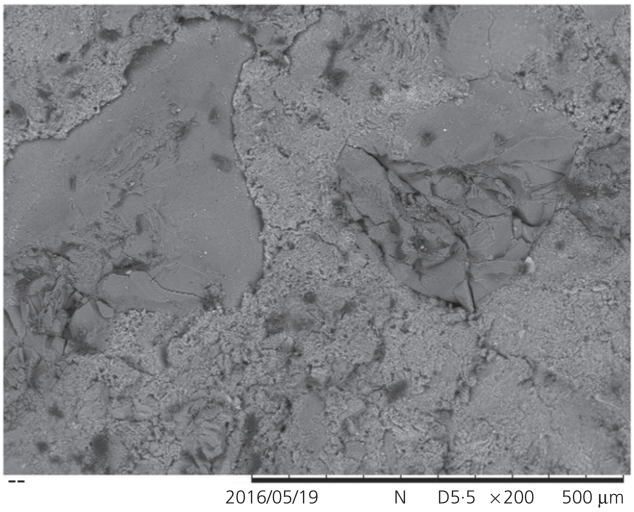

Figure 10. Rate of water absorption for air-cured concrete specimens. AO, air-cured, oven-dried eggshell; C, control

result, the gel can fill up the existing voids in the concrete, resulting in a lower rate of water absorption (Okonkwo *et al.*, 2012). Replacing OPC with eggshell powder may reduce the rate of water absorption due to its fineness and may chemically participate in the hydration process under desirable conditions (Zelic *et al.*, 2000).

From Figure 10 it can be seen that the rate of water absorption for air-cured concrete specimens is generally higher than in the water-cured concrete specimens. It is clear that C–S–H gel is a major strength-providing reaction product of cement hydration, as shown by compressive strength and flexural strength, while also acting as a porosity reducer resulting in concrete with a dense microstructure (Safiuddin *et al.*, 2007). Insufficient water supply during air curing greatly reduced the formation of the C–S–H gel, which would have otherwise been able to fill up the existing voids. Hence, the rate of water absorption for air-cured concrete specimens is higher than for the water-cured eggshell concrete specimens.

In order to investigate the influence of the curing on the microstructure of the concrete, the microstructure of concrete samples exposed to different curing conditions was also analyzed using SEM, and Figures 11 and 12 show the image of water-cured and air-cured specimens respectively. Based on Figure 12, it can be observed that the voids for the water-cured specimen are generally fewer than in the air-cured specimens, and as a result the rate of water absorption for the air-cured specimen is 57·1% higher than for the water-cured specimens. Generally, water-cured specimens produce a more compact microstructure compared to air-cured concrete specimens, resulting in a lower rate of water absorption (Zhang and Zong, 2014).

Acid attack

The durability of eggshell concrete under acid attack decreases as the percentage of the eggshell powder content in the mix increases, as illustrated in Figures 13 and 14. Control specimens produced from 100% OPC exhibit the lowest total strength reduction compared to other eggshell concrete mixes. Replacement volumes of up to 5% eggshell powder for both water-cured and air-cured specimens do not affect the specimens' behaviour in terms of compressive strength. The concrete specimens experienced gradual reduction in compressive

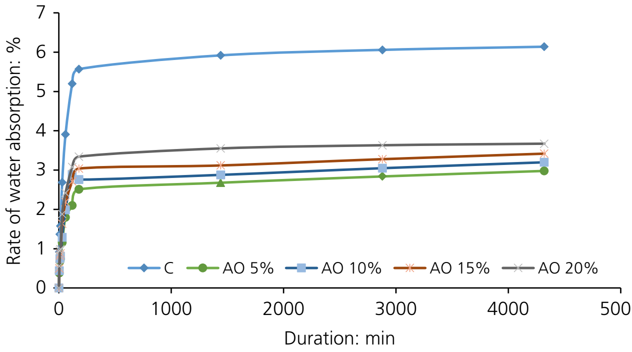

Figure 11. SEM image for water-cured concrete specimens

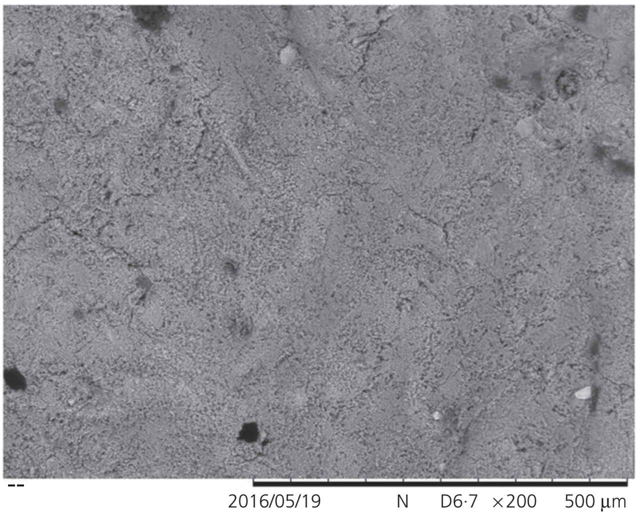

Figure 12. SEM image showing air-cured concrete specimens

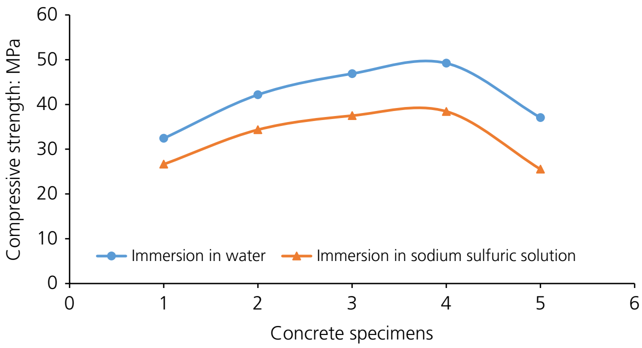

Figure 13. Effect of compressive strength for water-cured concrete specimens after submergence in sodium sulfuric solution

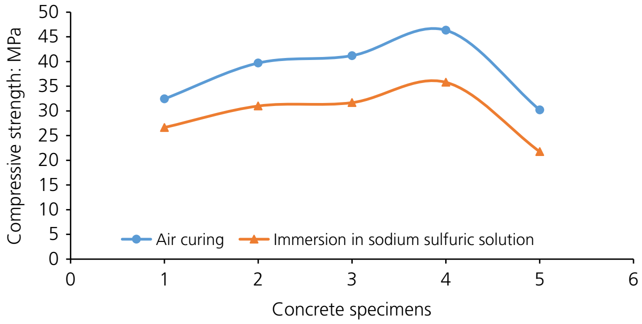

Figure 14. Effect of compressive strength for air-cured concrete specimens after submergence in sodium sulfuric solution

strength as the eggshell powder content increases for both curing regimes. Moreover, when the eggshell powder increases up to 20%, there is a significant reduction of the compressive strength for water-cured and air-cured specimens, of 31·1% and 36·4%, respectively.

With longer immersion periods, the sulfuric acid solution attack progresses from the concrete surface to the interior, resulting in a larger percentage of strength loss (Zivica and Bajza, 2001). Basically, the gradual reduction of the compressive strength that takes place as the amount of eggshell powder is incrementally increased occurs because the most vulnerable element to acid attack is calcium hydroxide ($Ca(OH)_2$) (Kong and Orbison, 1987). Since replacing cement with eggshell powder increases the percentage of calcium hydroxide, the durability of the eggshell concrete decreases as the amount of eggshell powder increases. Strength reduction and the severity of physical damage are higher for those specimens using eggshell powder as a partial cement replacement at volumes greater than 20%.

The reduction of compressive strength in air-cured concrete specimens is more severe than in water-cured eggshell concrete specimens. This is because the formation of the C–S–H gel is not completed during the hydration process, or the pozzolanic reaction, due to the insufficient amount of water available in the air-curing regime. Thus, the voids in the air-cured concrete are generally larger, allowing the sulfuric acid to react with the cement paste more easily. The compressive strength of AO 20% was reduced by up to 27·5% due to the significant reduction in silicon dioxide content, which is responsible for producing primary C–S–H gel during the early strength development (Khairunisa *et al.*, 2015a, 2015b).

Generally, it is not advisable to use eggshell powder as a partial cement replacement beyond 20% due to the adverse effect on the resistance of concrete to acid attack. The increased proportion of eggshell powder may reduce the silicon dioxide in OPC, thereby inhibiting the hydration process forming secondary C–S–H gel. As the result, unreacted eggshell powder is left in the concrete, which readily reacts with the acid solution (Karthik and Gandhimathi, 2015).

Sulfate attack

The results illustrated in Figures 15 and 16 show the changes to the compressive strength of eggshell concrete produced under both curing regimes after being immersed in sulfate solution for 50 weeks. Comparing the strength reduction under both curing regimes, eggshell concrete produced using the water-cured regime demonstrates a lower strength reduction throughout the whole experimental period. The continuous availability of moisture during water curing provided the conductive medium for an uninterrupted hydration process as well as for the pozzolanic reaction to take place, thus producing eggshell concrete with a denser internal structure that in turn enables it to exhibit better strength performance compared to air-cured eggshell concrete. The significance of water curing has been highlighted by Ozer and Ozkul (2004) who reported that sufficient water is important for strength development since it allows the hydration process and pozzolanic activity to take place. Thus, the compressive strength of the water-cured eggshell concrete specimens was generally much higher than that of the air-cured eggshell concrete specimens.

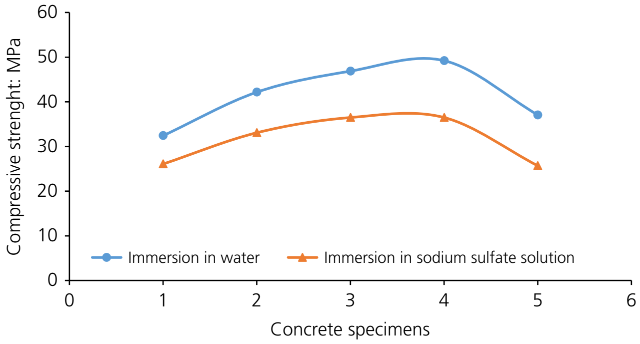

Figure 15. Effect of compressive strength for water-cured concrete specimens after submergence in sodium sulfate solution

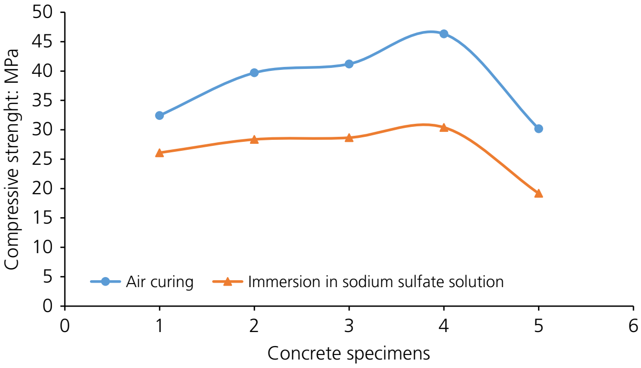

Figure 16. Effect of compressive strength for air-cured concrete specimens after submergence in sodium sulfate solution

There is a slightly higher compressive strength loss for WO 20% and AO 20%, up to 30·4% and 31·2%, respectively. This is because sodium sulfate is a salt known for being a very strong base, with a relatively high pH of 7·7. The formation of gypsum from the portlandite when under attack from sodium sulfate is presented in Equation 1. The formation of calcium sulfate, which precipitates and crystallizes in the cement paste microstructure, contributes to the expansion process. Thus, the loss of compressive strength in the concrete specimens during the sulfate attack is due to the loss of cohesion in the hydrated cement paste and of adhesion between it and the aggregate particles (Sumer, 2012). As the immersion continues, the sulfate ion concentration in the solution increases and reacts with the gypsum and monosulfate, resulting in the expansion, as shown in Equations 2 and 3. As the result, the ultimate disintegration of the C–S–H gel phase is delayed, causing the reduction in compressive strength.

Moreover, the compressive strength for both water-cured eggshell concrete and air-cured eggshell concrete reduces gradually when immersed in the sodium sulfate solution. The reduction in compressive strength is due to a saturation of calcium oxide and the smaller amount of silicon dioxide available to complete the hydration process and the pozzolanic reaction. Thus, the abundant availability of free calcium hydroxide in the concrete allows the sulfate ion to react and cause a high reduction in compressive strength with replacement volumes of up to 20% for both water-cured and air-cured regimes.

1. $$Ca(OH)_2 + Na_2SO_4.H_2O \rightarrow CaSO_4.H_2O + 2NaOH$$

2. $$C_3A.Ca(OH)_2.12H_2O + 3Na_2SO_4 + 2Ca(OH)_2 + 20H_2O \rightarrow C_3A.3CaSO_4.32H_2O + 6NaOH$$

3. $$C_3A.CaSO_4.12H_2O + 2NaSO_4 + 2Ca(OH)_2 + 20H_2O \rightarrow C_3A.3CaSO_4.32H_2O + 4NaOH$$

Source: Wojciech *et al.* (2014).

Conclusion

Based on the outcome of this study, the following conclusions can be drawn.

- The compressive strength and flexural strength of water-cured eggshell concrete is 6·7% and 3·2% higher than in the air-cured eggshell concrete, respectively. The eggshell concrete underwent an accelerated hydration process resulting in higher early strength. Based on this, eggshell concrete is suitable for use in precast industries since it has high early strength. The high early strength of eggshell concrete would be very beneficial to the expedition of the construction process as less time for strength development is required.
- The rate of water absorption for water-cured eggshell concrete was higher than in air-cured eggshell concrete. This was because the availability of moisture allowed the eggshell concrete to generate more C–S–H gel to fill up the existing voids and reduce the permeability of the concrete.
- The use of eggshell powder as a partial cement replacement decreased the durability of concrete. Thus, it is not recommended to use eggshell concrete in strong acid or sulfate environments.
- From the research, it was found that 15% of eggshell powder is the optimum percentage of partial cement replacement. Thus, eggshell powder could be potentially used in construction materials in the future as a way of reducing carbon dioxide emissions.

Acknowledgements

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