The hydration effect on palm oil fuel ash concrete containing eggshell powder

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Abstract. Hydration reaction is an exothermic reaction, where heat is released during the hardening process of concrete until it reached stable low energy state. The hydration temperature describes the hardening behaviour of concrete. This paper presents the hydration effect on Palm Oil Fuel Ash (POFA) concrete containing Eggshell Powder (ESP)—combination of silica and calcium oxide (SiO₂-CaO) that has becoming a new trend to improve catalyst characteristic of ESP in POFA concrete. In this study, ESP was combined between two types of POFA: Ground and Unground POFA (GPOFA and UPOFA). Initially, the optimum mix proportion was determined by two parameters: workability and compressive strength of POFA-ESP concrete. Higher compressive strength with sufficient workability of concrete was prioritized as optimum mix proportion. Overall, the compressive strength of concrete showed increment; GPOFA-ESP concrete gained superior compressive strength as compared to normal concrete (NC) and UPOFA-ESP concrete. The desired optimum mix proportion of the samples were investigated further under hydration reaction test. The overall hydration temperature for GPOFA-ESP concrete was slightly higher as compared to NC and UPOFA-ESP concrete. All in all, ESP has potential to act as a low catalyst in POFA concrete.

1.0 Introduction

Originally, concrete mixture is based on three elements: cement—basically from Ordinary Portland Cement (OPC), aggregates (course and fine aggregates), and water. Concrete emitted carbon dioxide (CO₂) produced from cement and CO₂ is one of the global warming causes. In order to reduce the amount of CO₂, the cement content has to be reduced. So, year by year, cement manufacturers came out with ideas of introducing concrete supplementary to replace cement. One of the potential cement replacement used in industry is Palm Oil Fuel Ash (POFA). POFA is rich in silica content (SiO₂) [1], which helps in reducing the hydration temperature of concrete mix [2]. However, POFA is pozzolanic materials. It has low energy stage of hydration temperature reflects to the maturity stage of the concrete, which attributes to later strength development [2]. Therefore, in order to boost up the hydration temperature, any catalyst or accelerator is needed in a concrete mix. Since silica and calcium oxide (SiO₂-CaO) have become a new trend in biocomposites, in which CaO acts as a catalyst [3], it has create interest and motivation to
researchers to formulate a concrete containing pozzolanic materials with any calcium based materials. The eggshell wastes are one of the green materials and abundantly disposed. It is rich in CaO by having 96-97% calcium carbonate (CaCO$_3$) [4]. This percentage makes the eggshell—that can be turned into eggshell powder (ESP)—as a promising material that can increase the calcium oxide content in the concrete mix [5] [6][7][8].

This paper presents, the combination of POFA and ESP to produce POFA-ESP concrete. These combination materials were inspired from the combination of SiO$_2$-CaO. However, POFA can be divided into two categories: Ground and Unground POFA (GPOFA and UPOFA). The objectives of this study are: (1) to check the chemical composition of POFA and ESP under x-ray fluorescence (XRF); (2) to determine the optimum mix proportion of concrete containing POFA and ESP—two parameters involved: workability and compressive strength of the concrete; (3) to investigate the hydration effect of concrete containing POFA and ESP—limited to the desired optimum mix proportion of the concrete only.

2.0 Experimental Program

2.1 Materials

Palm Oil Fuel Ash (POFA) was obtained from the palm oil industry located at the southern state of Malaysia. The palm endocarp and mesocarp are undergoing a combustion process at high temperature of about 800 °C to 1000 °C to produce POFA. In this study, the POFA was divided into two categories: Ground POFA (GPOFA) and Unground POFA (UPOFA). POFA was sieved to pass through 300 µm to obtain UPOFA. A laboratory grinding mill with rod bar was used to obtain homogenize size of GPOFA.

Eggshell wastes were collected at the frozen food industry at the southern state of Malaysia. The eggshell wastes were first dried under the sun before crushed by using industrial food grinder. After that, the crushed eggshells were washed by using tap water. The clean eggshells were then filled into trays and dried again in the oven. The temperature of the oven was set at 100 °C ± 5 °C 24 hours. After 24 hours, the eggshells were taken out and left to cool for 1 hour. The crushed and dried eggshells were finally ground by using industrial grinder to obtain the fine eggshell powder (ESP).

2.2 X-Ray Fluorescence (XRF)

XRF is accurate analytical equipment to check the chemical composition of materials. The POFA (UPOFA and GPOFA) and ESP were through the oven dried process about 100°C ± 5°C for 24 hours. This process aim to eliminate the excessive moisture. The sample was mixed with the wax based CH$_2$ with the ratio 4:1 in order to produce in pallet form. Then, the pallet sample was pressed by using press machine at the pressure reached approximately 15 x 104 N. The samples were stored in desiccator with silica gel until it will be tested under XRF equipment.

2.3 Mix Proportions

The mix proportion of GPOFA-ESP and UPOFA-ESP were designed. In this study, the combination of GPOFA-ESP and UPOFA-ESP replacement were limited to 0%, 5%, 10%, 15%, and 20% of total 455 kg/m$^3$ of OPC. The fine and coarse aggregates were fixed at 932 kg/m$^3$ and 763 kg/m$^3$, respectively with the water cement ratio 0.55. Quality of concrete was monitored through trial mix and the workability of fresh concrete was tested under targeted 60–180 mm slump. At this stage, approximately 11 design mixes were designed for concrete grade of 30 MPa at 28 days. The notation of mix designed for GPOFA-ESP concrete and UPOFA-ESP concrete is presented in Table 1 and Table 2.
Table 1. Notation and percent replacement of GPOFA-ESP.

| Concrete Type | Mix Design | GPOFA %, (kg/m³) | ESP %, (kg/m³) | Notation |
|---------------|------------|------------------|----------------|----------|
| GPOFA-ESP     | 0, (0)     | 20, (91)         | G1             |
| GPOFA-ESP     | 5, (22.75) | 15, (68.25)      | G2             |
| GPOFA-ESP     | 10, (45.5) | 10, (45.5)       | G3             |
| GPOFA-ESP     | 15, (68.25)| 5, (22.75)       | G4             |
| GPOFA-ESP     | 20, (91)   | 0, (0)           | G5             |
| Normal Concrete | -         | -                | NC             |

Table 2. Notation and percent replacement of UPOFA-ESP.

| Concrete Type | Mix Design | UPOFA %, (kg/m³) | ESP %, (kg/m³) | Notation |
|---------------|------------|------------------|----------------|----------|
| UPOFA-ESP     | 0, (0)     | 20, (91)         | U1             |
| UPOFA-ESP     | 5, (22.75) | 15, (68.25)      | U2             |
| UPOFA-ESP     | 10, (45.5) | 10, (45.5)       | U3             |
| UPOFA-ESP     | 15, (68.25)| 5, (22.75)       | U4             |
| UPOFA-ESP     | 20, (91)   | 0, (0)           | U5             |
| Normal Concrete | -         | -                | NC             |

2.4 Workability test
The workability of the fresh concrete was conducted according to ASTM C143 [9], the concrete workability was tested. To get the slump, the difference between the height of the mould and that of the highest point of specimen was being measured. The targeted slump for this study was between 60–180 mm. The test is very important because different materials of GPOFA and UPOFA were involved in this study.

2.5 Compression test
A total of 132 concrete including normal concrete cube with dimensions of 100 mm × 100 mm × 100 mm were tested under compression test in order to determine the compressive strength for each of the mix proportion strictly according to the BS 1881-108 [10]. The test was conducted on concrete cube specimens at curing age of 1, 14, 28, and 56 days by using universal testing machine with machine capacity of 2000 kN and with a loading rate of 6 kN/s.

2.6 Hydration test
The testing was carried out by setting the thermocouple (type K) with the fresh concrete in a concrete cube and fixed in simple cubical plywood box. The cubical plywood box was insulated by polystyrene in each side of the box’s walls to avoid influenced from surrounding temperature. The cubical concrete samples, each from the optimum mix proportions obtained, were casted in a mould and placed in the ready mould inside the box. Thermocouple was connected to the data logger; then, the thermocouple was attached quarter depth to each of the test specimen to record the hydration activity. The hydration is set for 30 hours duration with the interval of 10 minutes. The testing was conducted by referring from previous testing procedures [2][11]. Hydration reaction was observed to record the amount of heat released throughout the process of concrete hardening. Hydration reaction test for this study was conducted on the optimum mix proportion of GPOFA-ESP concrete and UPOFA-ESP concrete including with NC.
3.0 Results and discussion

3.1 Chemical Composition

Table 3 below shows the chemical composition of POFA and ESP. Both materials of GPOFA and UPOFA are from the similar source. So, it have been categorized as POFA. As can been seen, POFA was moderately rich in pozzolan of SiO₂ with 53.30%. While, ESP has superior CaO content about 98.00%. The combination of high SiO₂ – CaO could provide high potentially to produce moderate strength concrete. Similar finding have been observed in other studies [3].

| Chemical Composition (%) | POFA | ESP |
|--------------------------|------|-----|
| SiO₂                     | 53.30| 0.05|
| MgO                      | 4.10 | 1.12|
| CaO                      | 9.20 | 98.00|
| K₂O                      | 6.10 | 0.11|
| Fe₂O₃                    | 1.90 | 0.02|
| ZnO                      | -    | 0.01|
| Al₂O₃                    | 1.90 | 0.05|
| P₂O₅                     | 2.40 | 0.10|
| SO₃                      | -    | 0.49|

3.2 Workability

Figure 1 and Figure 2 show the height of slump for concrete of GPOFA-ESP and UPOFA-ESP, respectively. From the figures, overall GPOFA-ESP concrete within the aforementioned range (60–180 mm) except several UPOFA-ESP concrete lies out of the slump’s range. The workability of GPOFA-ESP concrete increased when the percentage of 5% GPOFA with 15% ESP (G2) was added. Meanwhile, the workability gradually decreased after increasing the percentage of 10% GPOFA with 10% ESP (G3). Comparison of GPOFA-ESP to UPOFA-ESP concrete showed that overall, UPOFA-ESP concrete seems sticky in physical, and had poor and lower workability compared to NC. For UPOFA-ESP concrete, the workability obviously decreased after increasing the percentage of 10% UPOFA with 10% ESP (U3). This increasing might be caused by the natural open cellulose structure found in UPOFA. The structure tends to absorb more water during the production of the concrete and attributed to the poor bonding between materials [13].

![Figure 1](image-url)  
**Figure 1.** Slump of GPOFA-ESP concrete and normal concrete.
3.3 Compressive strength

Figure 3 shows the strength development for different types of GPOFA concrete at different curing age. Overall, the pattern of the compressive strength shows that the strength increase with the increment of concrete’s curing age. However, GPOFA-ESP concrete had superior compressive strength as compared to UPOFA-ESP and NC. For 56 days of curing age, the compressive strength of concrete was also gained from 1 day of curing age and continued even after 28 days. The optimum compressive strength belongs to G3 (10% GPOFA with 10% ESP) with approximately 40 MPa (about 3 times higher than NC). Figure 4 shows the strength development of different type of UPOFA concrete at different curing age. When the concrete was utilized with UPOFA-ESP, there is no significant compressive strength development. However, the compressive strength was increased by increasing the concrete’s curing age. This phenomenon occurred because, as filler, UPOFA and ESP are insignificant to give reaction to the concrete. The optimum mix proportion of UPOFA-ESP was U2 (5% UPOFA with 15% ESP) with the compressive strength approximately about 30 MPa. This compressive strength is also similar with the targeted strength of NC. The optimum mix proportion of concrete was determined by considering two factors: workability and compressive strength of the concrete.

Figure 3. Strength development for different types of GPOFA-ESP and normal concrete at different curing age.
3.4 Hydration behaviour

Overall, the graph demonstrated that the hydration temperature of concrete mixes had gradually increased with the increasing of recording time and decreased approximately after 5 hours. Concrete containing GPOFA-ESP showed a slightly higher hydration reaction as compared to others. Meanwhile, concrete containing UPOFA-ESP had almost similar behaviour of hydration reaction with NC. Previous author stated that the total temperature rise during a concrete hardening could be reduced by using POFA as cement replacement regardless of the fineness of POFA compared to NC [12]. Similar agreement from another study claimed that the reasons of reduction in hydration temperature in POFA concrete were caused by the reduction of cement percentage in the mix and the pozzolanic behaviour of POFA itself [2]. However, in contrast from this finding, the concrete containing GPOFA-ESP had slightly higher temperature rise during hydration process compared to NC and UPOFA-ESP concrete because the catalyst activity contributed by ESP affected from increasing hydration reaction. It was found that the ESP seems to be a compatible material as a low catalyst or booster if combined with GPOFA in concrete. This finding can be supported by the compressive strength of G1 and U1 (solely 20% ESP), in which the compressive strength insignificantly increased. However, when the material combined with GPOFA and ESP, the compressive strength was slightly increased.
4.0 Conclusion
This study presents the hydration effect on concrete containing POFA and ESP and the conclusion were made based on the designed objectives as shown below:

i. Palm oil fuel ash (POFA) was moderately rich in pozzolan of silica dioxide (SiO$_2$) content whereas ESP has superior calcium oxide (CaO) content

ii. The GPOFA-ESP concrete had higher compressive strength as compared to UPOFA-ESP and NC. For GPOFA-ESP concrete, the optimum mix proportion was G3 (10% GPOFA with 10% ESP) with the compressive strength approximately about 40 MPa. Meanwhile, UPOFA-ESP concrete showed that the optimum mix proportion was U2 (5% UPOFA with 15% ESP) with the compressive strength approximately about 30 MPa.

iii. In hydration reaction of concrete, GPOFA took place to give later strength to the concrete, while ESP took place as a low catalyst to boost-up the hydration temperature. Therefore, combination of both materials seem to be compatible as cement replacement that contribute dual function; ESP acting as catalyst in GPOFA concrete.

5.0 References
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