Palm Oil Fuel Ash and Mussel Shell Powder as Supplementary Cementitious Materials in Non-Load Concrete Brick

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Abstract. The pozzolanic properties of the industrial by-product palm oil fuel ash (POFA) has given this material a unique characteristic that can be used as a cement substitute. Meanwhile, the abundance of waste seashells which contain an enormous amount of calcium oxide (CaO) available around the world has led this material to be another potential waste material as substitutes for conventional materials in concrete. The percentage amount of POFA used as a replacement was constant at 20% from the amount of cement. On the other hand, mussel shell powder (MSP) was replaced by 2%, 4%, 6% and 8% with the decrement of 2% of OPC used for each of the design. The physical and chemical properties of the materials and the mechanical properties of concrete brick were identified for 7, 14 dan 28 days. The inclusion of POFA and MSP in the concrete brick mix in this study reduced the compressive strength of the concrete in the early days and enhanced at the late stages. Apart from that, the compressive strength of the concrete brick mixtures decreased along with the increasing percentage of MSP as the increment content of CaO from MSP reduced the hydration process. The optimum percentage of MSP replacement in this study is 4% with 20% replacement of POFA and 76% OPC. The application of these materials as supplementary cementitious materials in the construction industry, particularly in producing non-load concrete brick will simultaneously encourage reducing potential pollutions and promote sustainability.

1. Introduction
The cement industry is currently facing several challenges including the increasing demands of cement production and the necessity of diminishing energy consumption during the production of Portland cement clinker. However, the biggest challenge faced in this industry is the environmental concerns regarding greenhouse gas emissions. The cement industry is responsible for 5% to 8% of CO2 emission in the world [1-3] and approximately 2% of the world energy consumption is attributed to the process of cement production [3,4]. These challenges have driven the cement industry to continuously expand and improve its cement formulations and production methods and meet the sustainable development goal. This includes globally reduce the consumption of non-renewable material and maintaining natural resources concepts of recycling and sustainability.

The pozzolanic properties of by-product palm oil fuel ash (POFA) has given this material a unique characteristic that can be used as a cement substitute in the mortar and concrete mixes [5]. As one of the largest producers of palm oil with 28% of world palm oil production and 33% of world exports [6], Malaysia has continuously increased its production, lead to the excessive amount of POFA produced...
and hence, create a large environmental-load [7]. In order to minimize the environmental load caused by this by-product, numerous investigations have been carried out to study the potential of POFA as fertilizer for agricultural purposes [8]. However, the insufficient nutrients required for fertilizers causes POFA to be mostly dumped in an open field close to palm oil mills, creates a lot of environmental pollutions and attracts criticism due to its carcinogenic and bioaccumulative effects [9-11]. To reduce the potential pollutions and health hazards caused by this agricultural by-product, as well as to reduce the production cost of concrete, POFA often being utilized as one of the supplementary cementitious materials in the concrete mix. The high silica oxide content in POFA which met the pozzolanic properties criteria has increased the potential of this agricultural waste to be utilized as cement replacement or as filler to produce a durable and strong concrete [12,13].

Meanwhile, the abundance of waste seashells available around the world has led this material to be another potential waste material as replacements for the existing conventional materials in concrete. As mentioned by [14,15], the largest producer of shellfish in the world, China, has disposed of about 10 million tonnes of waste seashells including oyster, clam, scallop, and mussel shells in landfills every year. However, only a small fraction has been reused for other purposes such as fertilizers and handicrafts due to the limitation on the amount that can be used, the problem of soil solidification and economic problems [15,16]. The remaining large quantity of waste seashells generated hence has created problems with illegal dumping into public waters and reclaimed land [15]. The waste seashells can cause foul odours or the microbial decomposition of salts into gases, such as hydrogen sulfide (H₂S), ammonia (NH₃) and amines if left untreated for over a long period [15,17]. According to [3], the uses of waste seashell in the construction sector vary from the replacement of natural aggregates in producing concretes and mortars to adding filler in the manufacturing of Portland cement. Many previous studies have shown that the chemical compositions of seashells include more than 90% calcium carbonate (CaCO₃) [16-21]. This composition is similar to the content of CaCO₃ in the limestone dust used in Portland cement production [22]. Hence, seashell can be considered as a prospective substitute for conventional limestone in the cement industry.

However, there is inadequate literature available to conclude the effect of using POFA and waste seashell as cement replacements on its workability. Therefore, in this study, the effect of POFA and a small fraction of mussel shell powder (MSP) on concrete properties were explored under several proportions and curing time in developing concrete to be applied for non-structural building element, particularly as a non-loading brick.

2. Research Method

2.1. Materials

With a specific gravity of 2.983, Ordinary Portland Cement (OPC), a type of cement that has been produced worldwide was used in this study. The sand with a specific gravity of 2.7 also was used as fine aggregate with particle size ranged 0.3 and 4.75 mm. To prepare the concrete brick mix, tap water was used and taken from the concrete laboratory. The cementitious materials as substitution used in this study were POFA and mussel shells.

2.1.1. POFA. POFA is a solid waste product obtained from the burning palm oil husk or fibre in a form of ash. The combustion usually takes place in a palm oil mill boiler. With a planted area of 332.34 hectares, Keck Seng Oil Palm Estate, a plantation under Keck Seng (M) Berhad company located in Masai, Johor has been selected as the source of by-product POFA in this study. It is then was air-dried for 24 hours to reduce the moisture. Then, POFA was oven-dried at a temperature of 105 – 110°C for another 24 hours. Initially, POFA obtained from the mill was coarser than cement size, hence it was sieved at the size of 90 µm for the purpose of supplementary cementitious material.

2.1.2. Mussel Shells. Perna viridis (family: Mytilidae) is known as the Asian green mussel, Philippine green mussel or green-lipped mussel in certain parts of the world [23]. According to [24], Johor has
been the largest producer of *P. viridis* mussels in Malaysia and majorly harvested at the adjacent to the Pasir Gudang Seaport and industrial zones, such as Kampung Pasir Puteh [23,25]. Due to the abundance of *P. viridis* mussels in this area, Perkampungan Orang Asli Kampung Pasir Puteh has been selected as the sampling collection site in this study. *P. viridis* mussel is a wedge-shaped shell that can be found in saltwater and freshwater locale. This mussel is either wild-caught or farmed to meet the demand of the market that requires its flesh as a food source. However, mussel shells do not have any direct application and unrecyclable, hence often ended up in landfill or dumped in the sea. The collected mussel wastes were washed and cleaned by scrubbing off any debris such as barnacles, sand and mud spot that attached to the shells. Fully cleaned shells were then dried in the oven at 105 – 110°C for 2 hours. The oven-dried shells were crushed and finely ground before being sieved at the size of 90 µm, equivalent to cement particle size. Finally, the mussel shell powder (MSP) underwent a calcination process at a temperature of 800°C for 2 hours [26] (figure 1).

**Figure 1:** Preparation of MSP

2.2. **Mix Design**

The brick moulds were designed based on the standard brick size of 215 mm x 102.5 mm x 65 mm (length x depth x height) [27]. The total volume of each brick is 0.00158 m³ including 10% of wastage consideration. The design of the non-load bearing brick was based on JKR standard specification (JKR 20800-0183-14) with a ratio of 1: 6 (cement to sand) [27]. The mix proportion was prepared based on the proportion of cement replacement with POFA and MSP (Table 1). A water/binder ratio of 0.5 was used in all mix designs. Table 1 shows the percentage and weight of materials used as partial OPC replacement. The percentage amount of POFA used to replace cement was set constant at 20% from the amount of cement, while the amount of MSP increased by 2% with the decrease of 2% of OPC used for each design. As mentioned by [28], the amount of POFA may be used up to 20% without any
considerable reduction in the workability. To assess the viability of the result, control specimens with 100% OPC also had been prepared. All prepared samples were tested for compressive strength on day-7, day-14, and day-28 of room temperature.

Table 1: Quantity (kg/m³) and percentage (%) of material proportions used in brick samples

| Sample | Cement/material proportion | Sand |
|--------|-----------------------------|------|
|        | % | kg/m³ | % | kg/m³ | % | kg/m³ | % | kg/m³ |
| M1     | 100 | 426.68 | 0 | 0 | 0 | 0 | 2317 |
| M2     | 78 | 332.81 | 20 | 68.75 | 2 | 6.08 | 2317 |
| M3     | 76 | 324.28 | 20 | 68.75 | 4 | 12.16 | 2317 |
| M4     | 74 | 315.75 | 20 | 68.75 | 6 | 18.25 | 2317 |
| M5     | 72 | 307.21 | 20 | 68.75 | 8 | 24.33 | 2317 |
| M6     | 70 | 298.68 | 20 | 68.75 | 10 | 30.41 | 2317 |

2.3. Characterization of Physical Properties and Chemical Compositions

2.3.1. Particle Density. Particle density is a density of solid particles, expressed in units of grams per cubic centimetre (g/cm³). The particle density of OPC, POFA and MSP were determined according to BS EN ISO 17892-12:2018 [29]. The determination of density was done by a small pycnometer method whereas the material was prepared for three sets to get the average value of density. All data were tabulated and calculated based on BS EN ISO 17892-12:2018 [29].

2.3.2. Fineness. The fineness of POFA is important as it can be used as a supplement or replaces the cement. This method is required to check and control any presence of coarse POFA particles that may affect the mixing process. The amount of SiO₂, the important chemical composition in concrete, increases with the increase in fineness of POFA [28]. During the test, the fineness of POFA was measured by sieving it on a standard sieve size of 90 µm, and the proportion of POFA of which the grain sizes are larger than the specified sieve size was then determined.

The fineness of MSP is also important as it affects hydration rate and sequentially, the strength of the concrete. A satisfactory value of fineness indicates whether green mussel shells can be adopted as a partial replacement for cement. This method is required to determine any presence of coarse green mussel shells particles that may affect the mixing process and indirectly controls the quality of the production process. The fineness of green mussel shells was measured and determined by the same procedures as POFA. According to BS EN 196-6:2018, the value of fineness is equivalent or should not exceed 10% [30].

2.3.3. Chemical Composition. X-Ray Fluorescence (XRF) provides an insight into chemical properties of POFA and MSP by estimating the proportion of each element accurately, whereby the elemental ratios acquired is converted into weight fractions of each oxide present in these two materials. The chemical composition determined from the procedures designates the suitability and stability of POFA and MSP, whether both can act as well as cement.

2.4. Mechanical Properties of Concrete Containing POFA and MSP

In this study, the samples of 215 mm x 102.5 mm x 65 mm in sizes were tested for compressive strength test, ultrasonic pulse velocity (UPV) test and rebound hammer test. All samples including the control mix were tested on day-7, day-14 and day-28 after curing.

2.4.1. Compressive Strength Test. Compressive strength is considered as the utmost necessary engineering property among other mechanical properties to determine the capacity of the concrete structure to resist failure under compression.
2.4.2. **UPV Test.** UPV test is conducted to determine the quality of the concrete by analyzing its uniformity, cracks, voids, defects, and cavities [31,32] and is assessed by determining the velocity of an ultrasonic pulse penetrating through the inner concrete structure. A low UPV value implies the presence of voids inside the concrete structure, which influence the compressive strength and quality of concrete as cited by [33].

2.4.3. **Rebound Hammer Test.** Rebound hammer is another non-destructive measurement that considered a reliable method in identifying deficiencies in concrete. It mainly provides an in-situ rapid indication of elastic properties and strength, mainly surface hardness and penetration resistance.

3. **Results and discussion**

3.1. **Physical Properties and Chemical Composition**

The physical properties and chemical composition of OPC, and the substitute materials, POFA and MSP are presented in table 2 and table 3 respectively.

3.1.1. **Particle Density.** The physical properties of POFA are strongly influenced by the burning environment, particularly the burning temperature [34]. The colour of POFA collected was dark grey to black due to unburned carbon content left at relatively low burning temperature. Based on the results obtained, the specific gravity of OPC and POFA used in the study was found to be 2.983 and 2.403 respectively as tabulated in table 2. Thus, it can be seen that the specific gravity of POFA was 19.4% lower and lighter than OPC. The grinding process has increased the specific gravity of POFA because a decrease in porosity would result in a reduction in particle size.

On the other hand, mussel shells are almost always in the form of a wedge or symmetrical shell. The shells are usually much longer and wider as shown in figure 1. As presented in table 3, the colour of MSP turned grey after the calcination process. The result obtained shows that the specific gravity of MSP used in the study is 2.126, 28.7% lower and lighter than that of OPC. This convinces that the MSP is comparable to the specific gravity of OPC. They can thus be combined to produce another material such as blended cement.

3.1.2. **Fineness.** Fineness is an essential asset for the hydration and pozzolanic reaction for rapid development of the brick strength. Due to this, the materials used must adhere to cement fineness in order to produce uniform hydration distribution and strength growth [35]. Table 2 depicts the fineness of OPC, POFA and MSP used in this study. On this matter, the OPC used in this research has a small grain size fineness of approximately 99.72% with respect to the 90 μm sieve. This result was supported by [36] which reported that POFA was much finer than cement as a measure in a specific surface area. Meanwhile, the particle size of MSP was marginally larger than cement at 99.50% referring to the percentage passing on the 90 μm sieve.

| Material | Density (kg/m³) | Specific gravity | Fineness (%) | Colour       |
|----------|-----------------|------------------|--------------|-------------|
| OPC      | 2983            | 2.983            | 0.28         | Grey        |
| POFA     | 2403            | 2.403            | 0.08         | Dark grey/black |
| MSP      | 2126            | 2.126            | 0.5          | Grey        |

3.1.3. **Chemical properties.** X-Ray Fluorescence (XRF) test provides insight into chemical compositions determined from the procedures designates the suitability and stability of POFA and MSP, whether both can act as well as OPC. The chemical analysis of OPC, POFA and MSP used in this study are shown in Table 4. The chemical analysis revealed that POFA consisted of SiO₂, Al₂O₃, Fe₂O₃ less than 70% and high in calcium (>5% calcium oxide, CaO) which may be classified as Class C pozzolan.
as indicated in ASTM C618 [37]. Based on table 4, all these ashes have an almost similar chemical composition and complimenting one another. The distinction in the chemical composition of POFA was usually due to the burning situation and the source of materials. This outcome was comparable to [38] who found that a pozzolanic material had little or no cementing properties. However, when it is at a fine size particle, with the presence of moisture, it can react with calcium hydroxide at ordinary temperatures to provide the cementing properties. Thus, the brick produced from the partial replacement of cement with POFA has reaction by silicate, SiO₂, from POFA and slaked lime, Ca(OH)₂, from OPC to form calcium silicate hydrate (C-S-H) which is responsible for the compressive strength.

The temperature of the waste green mussel shell preparation may decide its crystallinity level, amorphosity and compound nature [39,40]. As revealed in table 4, the results of the XRF test indicate MSP mainly consists of CaO with several minor percentages of other chemical compositions. The descriptions coincided with the findings of the [16-21]. Through the calcination process, CaCO₃ can be converted into CaO. The percentage of CaO depends on the method of cleaning and calcination temperature that could increase both pH and the nature of compound especially calcite or CaO peaks [39,41]. Besides, the presence of silica in the mussel shell is within the optimum acceptable condition or recommended range by ASTM C618 [37], and not exceeding 3.0%. This indicates their propensity to be pozzolanic and could also be a potential source for replacement of conventional quarried limestone that can found in the OPC. These findings also align with the findings reported by other researchers [3,20,29] as well.

Table 3: Chemical compositions of OPC, POFA and MSP

| Material | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | TiO₂ | K₂O | SO₃ | P₂O₅ |
|----------|------|-------|-------|-----|-----|------|------|-----|------|
| OPC      | 20.690 | 3.253 | 4.077 | 66.562 | 0.999 | 0.307 | 0.787 | 3.195 | 0.155 |
| POFA     | 64.542 | 10.343 | 4.956 | 20.066 | 8.001 | 0.345 | 31.161 | 19.729 | 5.192 |
| MSP      | 1.077 | - | 0.348 | 97.742 | - | 0.028 | 0.059 | - | 0.290 |

3.2. Mechanical Properties of Concrete Brick

3.2.1. Compressive Strength. The compressive strength of concrete brick samples for each mix at curing ages of 7, 14, and 28 days is shown in figure 2. The compressive strengths of day-7 for M2, M3, M4, M5 and M6 mixtures were recorded lower than the control mix M1 by approximately 29.2%, 27.1%, 60%, 64.3% and 50%, respectively. Day-14 shown increment for control mix M1, M2, M3, M4, M5 and M6 mixtures with 4.9, 3.5, 3.9, 2.4, 2.0 and 2.7 MPa. The compressive strength of the concrete bricks containing POFA and MSP continued to increase for M2, M3, M4, M5 and M6 to 3.6, 3.3, 3.2 and 3.1 MPa on the 28th day, as presented in figure 2. Based on the findings, all concrete brick mixtures shown improvement on day-28 compared to the condition on day-7 and day-14. The M3 mixture exceptionally showed a high compressive strength on day-28, gives 1.85% higher than the control mix M1 and has reached minimum permissible average compressive strength, 5.2 MPa, based on the JKR standard specification for brick [27]. This shows that the optimum percentage of MSP replacement in this study is 4% with 20% replacement of POFA and 76% OPC. This finding is parallel to that obtained by [42] that used ground cockle seashell as a partial cement replacement. The cement replacement level of MSP must not exceed 15% as suggested by [43].
The inclusion of POFA and MSP in the concrete brick mix reduced the compressive strength of the concrete at an early age and improved at the late age, and parallel with the findings recorded by [33] in examining nano-POFA and eggshell powder in concrete. According to [28], the ability of finer POFA particles to fill the void in the concrete structure might have increased the compressive strength at an early age. Meanwhile, at the later age, the presence of SiO2 in the POFA reacted with the CaO contained in the MSP, formed further calcium silicate hydrate (C-S-H) and advanced the interfacial bonding in the concrete and hence, increased the strength [28]. However, the compressive strengths of the concrete brick mixtures in this study were constantly declined as the percentage of MSP increased by 2% each. The increment content of CaO from MSP reduced the hydration process and hence decreased the strength of concrete [15,44]. This is agreed by [43] who studied cockle shell ash replacement for cement and filler in concrete. Similar findings were reported by [45] concerning the compressive strength obtained in the concrete mixtures when the percentage of waste materials replacement increased. The strength also decreased as a result of the reduced OPC content when the MSP was used as a cement replacement [15,45].

The main component of MSP is CaCO3, which has a similar composition as limestone, can be categorized as inert material in concrete. At a high temperature, the CaCO3 can be converted into CaO that is useful in enhancing the reactivity in the concrete with the presence of pozzolans [15], such as POFA in this study. Despite limited studies have been done on the combination of MSP along with pozzolanic material by the previous researchers, [46] found that the compressive strength of the concrete was increased when fine oyster shell powder was used as addition up to 20% in cement-fly ash brick mixtures. The presence of oyster shell powder in the cement-fly ash brick mixtures aided the pozzolanic reaction with fly ash as the calcium hydroxide concentration increased [15,46].

3.2.2. Ultrasonic Pulse Velocity. The day-7 UPV values recorded between 4528 and 4871 m/s range, while the UPV values for day-28 for all mixes were recorded between 5340 and 8590 m/s, which indicate good quality concrete. The wave velocity obtained for each mixture increased with age. From figure 3, the maximum UPV recorded was 8590 m/s for M3 at an age of 28 days. This high wave velocity obtained indicates a dense internal structure of the concrete brick. The replacement level of 20% of POFA and 4% of MSP might give a high pozzolanic reaction of fine POFA and MSP to improve the interfacial C-S-H bonds and fill the internal pores. Hence, increase the quality and strength of the concrete. Similar findings were reported by [47] using a reference sample of waste oyster shells as controlled low-strength materials. Based on figure 3, the overall trends associated with the wave velocity from the UPV test and compressive strength obtained in figure 2 was parallel in this study.
3.2.3. Rebound Hammer. As illustrated in figure 4, the early age of curing, RI value ranged between 27.8 and 41.3. The trends show a slight increment towards the late age in each of the mixtures, in line with the increase of compressive strength and UPV values as shown in previous figure 2. Although the RI values have not coincided with the overall trends of compressive strength and UPV values in all samples, however, the device is not expected to give an accurate measure of concrete compressive strength [48].

![Figure 3: UPV test on concrete brick samples containing POFA and MSP](image)

3.3. Microstructural Properties of Concrete

3.3.1. Scanning Electron Microscopy (SEM) of POFA and MSP. Figure 5 shows the SEM images of POFA particles. The particles were irregular in shape and having porous surfaces texture. In addition, there was no agglomeration of POFA particles after heat treatment. The descriptions were in agreement with the results of [7] and [49]. The morphology of green mussel shell calcined at 800°C was analysed by SEM as shown in figure 6. The MSP revealed a typical layer. With the calcination temperature of

![Figure 4: Rebound hammer test on concrete brick samples containing POFA and MSP](image)
800 °C, natural shell microstructures were significantly showed layered to porous [50,51]. The calcined waste shells were irregular in shape, and some were attached as aggregates [50].

Figure 5: SEM images of POFA (2000x magnification)

Figure 6: SEM images of MSP (2000x magnification)

3.3.2. SEM of Brick. The SEM images of brick M3 (figure 7) were taken considering its highest strength based on the compression strength test result has been conducted and recorded in figure 2. The SEM shows a flaky yet compact surface of the brick sample with minimal voids. The materials were well combined and bonded together after 28 days and the mixture reached a high compressive strength for a non-load bearing brick.
4. Conclusion

Based on the assessed physical and chemical properties of OPC, POFA and MSP and mechanical properties of non-load bearing brick containing these materials as cement replacements, and the following conclusions were obtained.

- The specific gravity of POFA and MSP was 19.4% and 28.7% lower and lighter than cement. This convinces that these materials are comparable to the specific gravity of OPC and can be mixed to produce another substance such as blended cement.
- In comparison to OPC, the particle size of POFA was marginally finer than cement at 99.92% corresponding to the percentage passing on the 90 μm sieve. The main component of POFA is SiO₂.
- The result of the XRF test indicated that MSP mainly consists of CaO with minor percentages of other chemical components.
- The ability of finer POFA particles to fill the void increased the compressive strength in the concrete brick mix at an early age. Meanwhile, the formation of additional C-S-H bonds from the presence of SiO₂ in the POFA that reacts with the CaO contained in the MSP at the later stages enhanced the interfacial bonding in the concrete and hence, increased the strength.
- Except for M3, the compressive strength of the concrete brick mixtures decreased along with the increasing percentage of MSP. The increment content of CaO from MSP reduced the hydration process, thus decreased the strength of concrete.
- The UPV values obtained from the UPV test increased with age. The high pozzolanic reaction of fine POFA and MSP to improve the interfacial C-S-H bonds and fill the internal pores. Hence, increased the quality and strength of the concrete.
- The rebound hammer test showed a slight increment towards the late age of all mixtures.
- The optimum percentage of MSP replacement in this study is 4% with 20% replacement of POFA and 76% OPC.

As mentioned by many previous researchers, introducing POFA in the mix designs had extensive outcomes on concrete properties, exceptionally at a late age. Meanwhile, due to its filler effect, the MSP could add a positive influence but reduced the mechanical properties of the structure. A suitable portion of MSP, however, may improve the interfacial C-S-H bonds and increase the strength of the concrete. Based on the findings, it is certainly interesting to determine the effect of concrete brick on the insulation properties, especially on thermal and sound, for further research in the future. With the high production of POFA and abundance disposition of mussel shells are forecasted to increase in the future, the combine application of these two as supplementary cementitious materials in the construction industry, particularly in producing non-load concrete brick will reduce the environmental glitches and promote sustainability.
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