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Influence of palm oil fuel ash on behaviour of green high-performance fine-grained cement mortar

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Abstract. In the recent years, the use of agricultural waste in green cement mortar and concrete production has attracted considerable attention because of potential saving in the large areas of landfills and potential enhancement on the performance of mortar. In this research, microparticles of palm oil fuel ash (POFA) obtained from a multi-stage thermal and mechanical treatment processes of raw POFA originating from palm oil mill was utilized as a pozzolanic material to produce high-performance cement mortar (HPCM). POFA was used as a partial replacement material to ordinary Portland cement (OPC) at replacement levels of 0, 5, 10, 15, 20, 25, 30, 35, 40% by volume. Sand with particle size smaller than 300 μm was used to enhance the performance of the HPCM. The HPCM mixes were tested for workability, compressive strength, ultrasonic pulse velocity (UPV), porosity and absorption. The results portray that the incorporation of micro POFA in HPCMs led to a slight reduction in the compressive strength. At 40% replacement level, the compressive strength was 87.4 MPa at 28 days which is suitable for many high strength applications. Although adding POFA to the cement mixtures harmed the absorption and porosity, those properties were very low at 3.4% and 11.5% respectively at a 40% POFA replacement ratio and after 28 days of curing. The HPCM mixtures containing POFA exhibited greater increase in strength and UPV as well as greater reduction in absorption and porosity than the control OPC mortar from 7 to 28 days of curing age, as a result of the pozzolanic reaction of POFA. Micro POFA with finely graded sand resulted in a dense and high strength cement mortar due to the pozzolanic reaction and increased packing effect. Therefore, it is demonstrated that the POFA could be used with high replacement ratios as a pozzolanic material to produce HPCM.

Keywords: cement; high-performance cement mortar; mortar; palm oil fuel ash; POFA; sand

1. Introduction

In the second half of the twentieth century, a dramatic increase in Portland cement production was reported (CEMBUREAU 2020) due to escalating global demand. Cement is the most widely produced building and construction material with the current worldwide demand amounting to nearly 4 billion tons per year (Nagaratnam *et al*. 2019, Ting *et al*. 2020, United States Geological

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Survey 2021). The growth in cement demand is expected to increase further by 2030 due to significant economic growth (Wan Hassan *et al*. 2020). Based on the available statistics, 5-8% of global carbon dioxide (CO_2) was related to the manufacturing of cement (Agensi Inovasi Malaysia (AIM) 2013, Sumesh *et al*. 2019, World Business Council for Sustainable Development 2016). Fig. 1 shows the increasing trend in CO_2 emissions alongside with the rising in cement production.

Based on the report by the Environmental Protection Agency (EPA 2020), the pollution caused by greenhouse gases emission is counted as one of the main threats to our environment. In 2018, CO_2 constituted 81% of the total greenhouse gases. Portland cement manufacturing process requires a great deal of energy, and consequently, massive amounts of greenhouse gases are released to the atmosphere (Rukzon and Chindaprasirt 2009a, Madlool *et al*. 2013, Celik *et al*. 2014, Hamada *et al*. 2019). A well-known fact is that approximately 900 kg of CO_2 is emitted into the atmosphere due to the production of every ton of cement, which results in devastating effects on the atmosphere and depletes our natural resources (Benhelal *et al*. 2013, Marthong 2019, Hamada *et al*. 2021). The negative consequences of cement production extend further by threatening the whole ecological system and hindering our hopes for a sustainable future. For the reasons mentioned earlier, the International Energy Agency (IEA) raised the alarms on the impacts of the cement industry on the earth. These issues have motivated the international research community to look for eco-friendly alternatives for the cement to preserve our planet (Mijarsh *et al*. 2020, 2021).

Furthermore, greener production is an important topic worldwide. To attain cleaner production, industries should reduce solid waste generation, and the natural resources should be utilized conservatively (Zeyad *et al*. 2017, Mohammadhosseini *et al*. 2018). As part of the efforts to solve or alleviate the problem, many researchers have explored partial replacements of ordinary Portland Cement (OPC) with supplementary cementitious materials (SCMs) which are mostly pozzolanic including solid wastes generated by the agricultural industry (Al-Kutti *et al*. 2018, Alsubari *et al*. 2018, Mohammadhosseini *et al*. 2020). In fact, the utilization of industrial by-products has reshaped the construction industry (Zeyad *et al*. 2021a, b, c). The usage of SCMs such as palm oil fuel ash (POFA) has been associated with solutions to this environmental concern in addition to improvements in the mechanical properties of concrete (Kroehong *et al*. 2011, Usman *et al*. 2017).

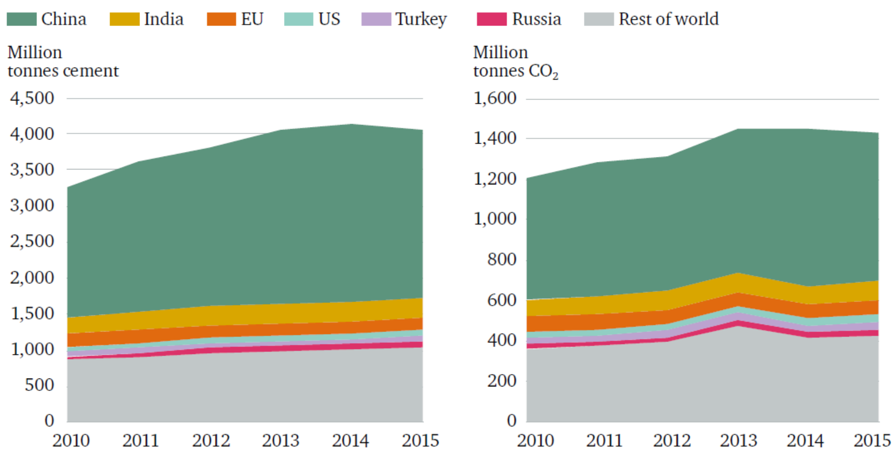


Fig. 1 Global cement production and CO_2 emissions (Lehne and Preston 2018)

Studies on the potential application of agricultural by-products as construction materials are currently among the trending research topics, mostly in countries where those materials are plentiful (Chindaprasirt *et al*. 2008, Rukzon and Chindaprasirt 2009b). A significant amount of solid wastes is generated by agriculture related industries, which mostly end up in the environment for easy disposal. The negligence in the disposal of solid wastes can result in acute land, water, and air pollution problems, and therefore has to have proper disposal planning or recycled into useful products. POFA is a solid waste usually produced from the combustion of palm kernel shells, empty fruit bunches, and palm fibres for electricity generation in the palm oil mills (Alsubari *et al*. 2018, Tasnim *et al*. 2018, Muthusamy *et al*. 2019). The usage of small-sized particles of POFA in the preparation of ultrafine POFA is a well-known technique to enhance its properties as a SCM. While a large amount of POFA is associated with a reduction in early strength development, the pozzolanic activity can be improved with further grinding and proper heat treatment (Altwair *et al*. 2013, Hamada *et al*. 2020b). By utilizing these abundant waste materials as a partial replacement of the cement, the construction industry could lessen its demand of the scarce natural resources (Rukzon and Chindaprasirt 2009a, Homwuttiwong *et al*. 2012, Shaladi *et al*. 2019).

POFA is a promising pozzolan and a few regions of the world have this type of waste material. The primary oxide composition of POFA is SiO_2 (42-66%), which is considered as the primary ingredient of pozzolanic materials (Chindaprasirt *et al*. 2008). Grinding POFA to nanoparticles leads to improvement in the pozzolanic activity (Hamada *et al*. 2020a, b, c, Zeyad *et al*. 2018). The finer particle size creates the filler effect which is defined as the suitable and appropriate arrangement of microparticles that work to fill the voids between matrix layers and contribute towards increasing the compressive strength without chemical reaction. Based on previous researchers, POFA can be utilised as a supplement or alternative for ordinary Portland cement, leading to global sustainable development and reducing its impact on the environment (Hamada *et al*. 2020a, b, c, Wan Yusof *et al*. 2015).

Many researchers and previous studies have already investigated the impact of POFA on the physical and mechanical properties as well as performance of Portland cement mortar and concrete. However, other properties such as UPV, absorption and porosity of high-performance cement mortar (HPCM) containing POFA with different replacement levels have not been well established. Also, the use of highly fine sand in producing cement mortar was rarely discussed in the literature. A better understanding of how micro-POFA affects cement mortar performance could increase the popularity and use of POFA in the mortar, which could bring great environmental benefits by recycling solid wastes instead of disposing them in the landfills. Thus, the objective of this research is to evaluate the effect of micro POFA on the performance and characteristics of HPCM. The Portland cement in the HPCM mixtures was replaced with varying replacement levels of POFA up to high replacement level of 40% which will work to reduce CO_2 emissions due to cement manufacturing and contributes towards safer and sustainable disposal of the polluting ash residue of the palm oil industry, the POFA. In this paper, POFA was enhanced with a new treatment method which is different from the previous studies. The new treatment process was aimed to reduce the grain size and increase the fineness of POFA, and then used with fine aggregates with particle size of smaller than 300 μm. The cement was replaced with the micro-POFA at replacement levels of 0-40%. The compressive strength, UPV, absorption, and porosity of the resulting HPCM mixtures were studied.

2. State of the art

2.1 Palm oil

In tropical countries such as Malaysia and Indonesia, palm oil is one of the main export commodities, and this is because it has broad industrial applications, especially in food and biofuel production. The production of palm oil has witnessed a significant rise during the last five years. From 1995 till 2015, the produced palm oil increased by four times, and the same rate of increase is forecasted to happen by2050 (McCarthy 2020). Fig. 2 indicates the continuous increase in the production of palm oil during the last five years. Considering how ubiquitous palm oil has become, statistics from the United States Department of Agriculture (2020) says that more than three-quarters of the palm oil production happens in two countries only, namely Indonesia and Malaysia. Fig. 3 shows the production rate and amount of palm oil in 2020. Indonesia alone hosts the production of 58 per cent of the global supply or 43.5 million tons, while Malaysia produced 19.9 million tons or 26 per cent.

Extraction of the oil from fresh oil palm fruits requires separation of the oil from the fruit bunches before further processing (Yin *et al*. 2008, Huda *et al*. 2017, Samadhi *et al*. 2020). The ensuing empty fruit bunches, kernel shells and fibres generated are usually utilized in the palm oil mills as boiler fuel to catalyse the combustion, produce steam, and generate electricity essential in palm oil extraction. The remnant ash residue remains in the boiler from the combustion of the waste materials is called the palm oil ash (POA) which is lately more commonly known as palm oil fuel ash (POFA) and has to be appropriately disposed. Nowadays, the norm in the industries is to reuse the ash resulting from the combustion to promote sustainable development by recycling of solid wastes.

An example of such an effort in Malaysia is the usage of POFA in agriculture as fertilizers. Nevertheless, this does not come without presenting a new environmental problem, which is the difficulty of monitoring its use and having the possibility of significantly altering the physicochemical properties of the existing soil. To this date, numerous efforts have been made to study the feasibility of reusing POFA as opposed to agricultural applications and landfill disposal. These studies focused on the application of POFA and its applicability as a partial replacement to

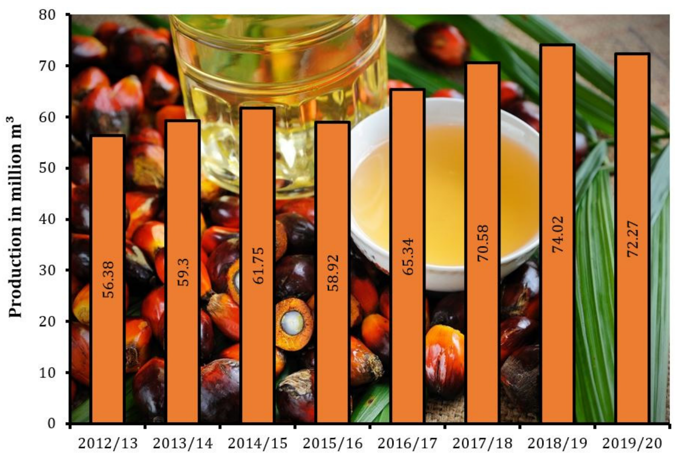


Fig. 2 Palm oil production worldwide in millions of metric tons (Statista 2020)

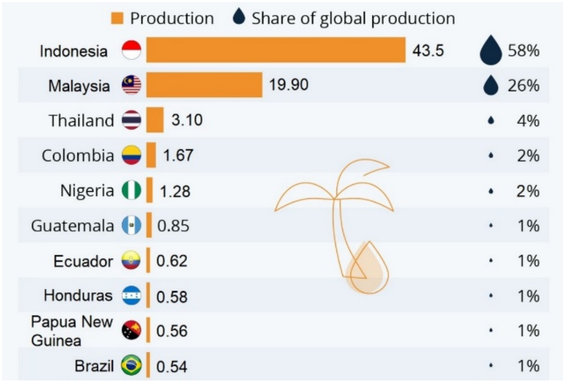


Fig. 3 Amount and production rate of palm oil in the most productive countries for 2020 (in gigatons)

Portland cement and as an additive to Portland cement mortar or concrete (Mujedu *et al*. 2020, Alabi and Mahachi 2020, Altwair *et al*. 2012, Zeyad 2013, 2016, 2021a, b, c).

2.2 Palm oil fuel ash (*POFA*)

In the process of palm oil manufacturing, one of the residues or a significant by-product which is produced is called POFA. The chemical compositions of POFA closely approximate those of Portland cement with different proportions such as Al_2O_3, SiO_2, CaO, Fe_2O_3, and MgO (Salami *et al*. 2018). It is a by-product that remains a solid waste after extracting palm oil, including the burning of palm oil fibre, kernel shells, and empty fruit bunches to produce essential electrical energy via steam boiler for the extraction process (Mujedu *et al*. 2021). Physically, they have a grey colour, an irregular shape, a lightweight, and a porous characteristic (Vijaya *et al*. 2008). While the disposal of POFA was always through landfilling, the recent spike in the volumes of these solid wastes has burdened the authorities. The improper disposal of these deposits, specifically industrial and agricultural solid wastes, has caused an environmental dilemma. The disposal of POFA without proper treatment can cause various health hazards to human beings. Since the demand of palm oil products is escalating year after year, the amount of POFA also increases proportionally. These wastes could cause various threats to humans as well as the environment, consequently causing harm to humans and financial losses (Mohammed *et al*. 2014, Muthusamy *et al*. 2019, Ting *et al*. 2020). Oil palm ash has been utilized as novel adsorbent, crude fertilizer, blended cement, and pozzolanic supplementary cementitious material for concrete (Samadhi *et al*. 2020).

2.3 Previous studies on the use of POFA in cement mortar

Currently, various types of pozzolans are used worldwide, typically as SCMs to reduce the usage of Portland cement while at the same time to improve the properties and performance of mortar and concrete. It is widely believed that the utilization of pozzolan in the production of concrete, mortar, and paste contributes to desirable advantages for example in terms of compressive strength improvement in concrete or mortar as a result of the filler effect and pozzolanic reaction (Hamada *et al*. 2020a, b, c, Jaturapitakkul *et al*. 2011). However, it is complicated to determine how much the compressive strength is affected by each of these effects.

ASTM C618-19 (2019) requires that not more than 34% of the pozzolan particles' total weight should retain on a 45-µm sieve. Moreover, it also stipulates that the strength activity index on the 7th or the 28th days should be more than 75% of the control mortar strength. Despite that, ASTM C618 does not specify the amount of contribution of the pozzolanic reaction and the filler effect to the mortar's compressive strength.

The usage of solid waste materials from the palm oil industry comes with great benefits to the construction industry and many of its sectors. Palm oil clinker is a secondary output material obtained from the palm oil processing stages. Funnelling this material into the construction industry is a practical action to promote sustainability in terms of resolving waste disposal issues. The environmental pollution resulting from poor management of solid waste systems can be reduced significantly (Kanadasan and Abdul Razak 2015). The first study on using POFA in concrete commenced in 1990 when it was found that POFA is a suitable pozzolanic material due to its chemical activity, making the concrete denser, and giving a higher hydration rate (Wan Hassan *et al*. 2020). The research was then followed using POFA in cement composites such as cement mortar and paste. This section of the study provides a brief summary of previous studies on the use of POFA as a partial cement replacement in mortar. Table 1 shows the material and the replacement ratios for each research work from the previous studies. Table 2 illustrates the characteristics or properties studied in each research work. Most of the previous studies discussed the effect of partial replacement of cement by POFA on the performance of architecture or ordinary mortar with a compressive strength lower than 40 MPa. A few researchers evaluated the behaviour of HPCM containing POFA. As mentioned earlier, the aim of this study is to improve HPCM with a high replacement ratio of POFA to cement of up to 40% but without inducing adverse effects on the mechanical characteristics of the resulting HPCM mixes.

Chindaprasirt *et al*. (2008) pointed out that partial replacement of OPC with POFA contributes to a drastic improvement in durability properties such as resistance to chloride ion penetration. Besides the highly effective nature of POFA as a pozzolan, its incorporation requires more superplasticizer to maintain the mortar flow. Jaturapitakkul *et al*. (2011) stated that the compressive strength of mortar containing 40% POFA as replacement to cement changed from 4.5 MPa to 22.5 MPa at 7 days and 90 days, respectively. This observation indicates that while the pozzolanic reaction of POFA was low at the early age 7 days, it increased tremendously at the later age of 90 days. Furthermore, the pozzolanic reaction of POFA improved with the increase in the age of the mortar, cement replacement rate, and particle fineness.

Kroehong *et al*. (2011) mentioned that the compressive strengths of cement pastes containing coarse POFA matched those of the control OPC paste. Additionally, blending finely ground POFA with cement paste resulted in a mix that gave higher compressive strength compared to cement paste containing coarse POFA. Blended cement pastes that included 20% of finely ground POFA had the lowest total porosity. The incorporation of POFA with high fineness lowered the average pore size of blended cement paste and the critical pore size as opposed to that with coarser POFA. Kanadasan and Abdul Razak (2015) stated that replacing cement in the mortar by 50% of POFA gave specimens with a compressive strength that is nearly 70% of the control specimens. Nevertheless, these specimens provided a 60% structural efficiency and reduced the cost by 41% compared to the control specimen.

Rajak *et al*. (2015) investigated the morphological properties of hardened cement pastes incorporating nano-POFA (nPOFA) having particle sizes between 20 nm and 90 nm. It was found that the nPOFA particles can drastically affect the pozzolanic reaction in the pastes due to greater available surface area for pozzolanic reaction. Also, Wi *et al*. (2018b) conducted a study on the use

Table 1 Cementitious composites as discussed in the literature

ID	Authors	Material	Type and Level of use
1	(Awal and Hussin 1997)	Cement mortar	CR by POFA 10%, 30%, 50%
2	(Chindaprasirt *et al*. 2008)	Cement mortar	CR by POFA 20%, 40%
3	(Rukzon and Chindaprasirt 2009a)	Cement mortar	CR by POFA 20%, 40%
4	(Kroehong *et al*. 2011)	Cement paste	CR by POFA 20%, 40%
5	(Jaturapitakkul *et al*. 2011)	Cement mortar	CR by POFA 10%-40%
6	(Wan Yusof *et al*. 2015)	Fine-grained mortar	CR by POFA 10%, 20%, 30%, 40%
7	(Kanadasan and Abdul Razak 2015)	SCM	CR by POFA 0%-50%
8	(Farzadnia *et al*. 2015)	Cement mortar	CB by POFA 10%, 30%
9	(Usman *et al*., 2017)	Cement mortar	CR by POFA 10% + 10% MK
10	(Huseien *et al*. 2018)	Alkali activated mortars	Fly Ash replacement by 10%, 20%, 30%, 40%, 50%
11	(Salami *et al*. 2018)	Alkali activated mortars	Mixture proportioning of POFA + additives
12	(Tasnim *et al*. 2018)	Cement paste	CR by POFA 10%, 20%, 30%
13	(Wi *et al*. 2018b)	Mortar and paste	CR 0-90%
14	(Wi *et al*. 2018a)	Cement mortar	CR by POFA 10%, 20%, 30%, 40%
15	(Sumesh *et al*. 2019)	HSCM	CR by POFA 10%, 20%, 30%
16	(Shaladi *et al*. 2019)	HSCM	CR by POFA 30%, 40%, 50%, 60%

HSCM: High Strength Cement Mortar SCM: Self Compacting Mortar
MK: Metakaolin CR: Cement Replacement

Table 2 Properties of cementitious composites containing POFA as discussed in the literature

Properties	Researchers by ID (Based on Table 1)
Workability	(7, 8, 10, 11, 15, 16)
Compressive strength	All except (1)
Flexural strength	(6, 7)
UPV	(7, 9)
Porosity	(4, 10, 14)
Absorption	(7)
Abrasion resistance	(10)
Freezing-thawing resistance	(10)
Drying shrinkage	(8, 10)
Sulphuric acid attack	(10)
Sulphate attack	(10)
Chloride penetration	(2)
Morphology and microstructure	(2-5, 7-13, 15, 16)
Thermogravimetric analysis	(4, 13, 15, 16)

of nPOFA in producing cement mortar. The authors confirmed that there was an improvement in compressive strength of mortar specimens containing nPOFA as a replacement to cement. Nonetheless, cement pastes containing higher than 40% of nPOFA experiences a reduction in its compressive strength. As curing age extends, the samples with nPOFA exhibit denser microstructure compared to the specimens without nPOFA because of the refinement in the microstructure.

Usman *et al*. (2017) investigated the behaviour cement mortar containing POFA and Metakaolin (MK) as partial replacement to cement. The authors found that the exposure of the mortar to elevated temperature together with the use of MK and POFA resulted in notable improvements in the compressive strength and microstructure of the mix when compared to a control OPC mortar. The authors confirmed that a mixture of the three components; viz cement, POFA, and MK, can add fire resistance properties to the specimens. Wi *et al*. (2018a) stated that the mortar containing 10% of POFA as a replacement to cement gives the optimum compressive strength, marking a 33% improvement after 28 days of curing over the plain OPC specimens. Increasing the amount of mPOFA any higher than this ratio causes reduction in the compressive strength. However, prolonged curing for samples greater than 28 days reduced the strength difference between the control specimens and POFA40 by 27%. This is because POFA40 particles are rich in Si, which reacts with $Ca(OH)_2$, which is produced as one of the products of cement hydration process, to create more quantity of C-S-H.

Sumesh *et al*. (2019) used two types of POFA as a substitute for cement in a mortar, the first called POFA and the second treated POFA (T-POFA) with a median particle size of 19.41 µm and 19.19 µm respectively. Replacing cement in the mortar with 30%, 20%, and 10% of T-POFA contributed to increase compressive strengths to 83.6, 88.4, and 101.4 MPa, respectively, which is very close to the 110 MPa obtained by the control sample at 90 days. Furthermore, the higher strength activity index produced by the T-POFA samples indicated a pozzolanic activity that is better than the normal POFA mortar. Generally, it is recommended to replace 10% of cement with T-POFA in the preparation of mortar mixture with a strength that ranges between 60 and 100 MPa.

Narong *et al*. (2018) used POFA as partial cement replacement in plastering work to solve electromagnetic interference issues. The authors confirmed that using 20% of POFA with 140 µm of POFA particle size and water immersion curing conditions achieves the optimal mixture. Shaladi *et al*. (2019) confirmed that using micro POFA as a substitution to cement to produce cement mortar with a 30% replacement ratio gives the highest compressive strength at 28 days of curing age, which is around 50 MPa.

3. Experimental methodology

3.1 Materials

3.1.1 Cement

PANDA MS EN 197-1 CEM I 52.5N Portland cement was used as the main binder in this study. This type of cement was used to ensure the highest quality of mortar, as it is the highest grade available in the market in terms of compressive strength (52.5 N). The properties of the cement used and limits of specification are provided in Table 3.

Table 3 Cement properties

	Test		Average	Specification (BS EN 197-1 2011)
Physical tests	Normal consistency (%)		27.9	-
	Setting times:	Initial setting time (min)	98	Min. 45
		Final setting time (min)	252	Max. 360
	Soundness expansion (mm)		1	Max. 10
	Cement fineness (cm^2/gram)		4682	Min. 2600 (ASTM C150/C150M - 20 2020)
Compression test	**Age (Days)**		**Average (N/mm^2)**	**(BS EN 197-1 2011)**
	2		22.5	Min 20
	7		39.2	-
	14		47.7	-
	28		55.5	Min. 52.5

3.1.2 POFA

The POFA was collected from a palm oil mill located in Nibong Tebal, Penang, Malaysia. This mill provided the raw POFA in the form of ash residue of the combustion of empty fruit bunches, palm kernel shells and fibers to heat up boiler for electricity generation in the mill as mentioned previously. As the POFA is not protected and left exposed to weather near the palm oil mill, the POFA may be wet or saturated during delivery. The raw wet POFA (W-POFA) was first dried in an oven at 105 ± 5°C for 24 h. This was followed by sieving the W-POFA through a 300 µm sieve to remove coarser materials, debris and extraneous materials such as unburnt shells and fibres. After drying and sieving, the W-POFA turned into a dry state (D-POFA). The sieved D-POFA was then ground using a laboratory scale ball mill for 24 h to prepare it for the heat treatment, which follows, so that it is more efficient. The grinding process was very effective in reducing the size of the particles and homogenizing the powder. After grinding, the ground POFA (G-POFA) was produced. Then, the G-POFA underwent a heat treatment in a gas furnace with a temperature of 600°C for 1 hour to reduce or eliminate excess unburnt carbon, which could have potential negative effects on the pozzolanic properties. This procedure was previously followed and was noted to effectively expel the excessive unburnt carbon from the G-POFA. Subsequently, the heat-treated POFA (T-POFA) was subjected to further grinding for 24 hours using the same laboratory ball mill to obtain the ultrafine POFA (U-POFA). To ensure the quality, consistency, and uniformity of the resulting U-POFA that will be used in the mortar mixes, essential steps were taken to control all of the treatment procedures, which include the sizes of steel balls used, as well as the number of steel balls used in the ball mill during the grinding stages, duration of grinding, the amount of POFA added into the mill, mill rotation speed, and the thickness of POFA layer in the furnace at the time of heat treatment. The treatment method used is similar to the one adopted in a previous study (Mijarsh *et al*. 2021).

After grinding, the U-POFA particles' final shape was mostly crushed particles, irregular and angular in shape as shown by the SEM micrograph in Fig. 4. Fig. 5 illustrates the treatment process of POFA particles. The chemical and physical characteristics of the POFA are illustrated in

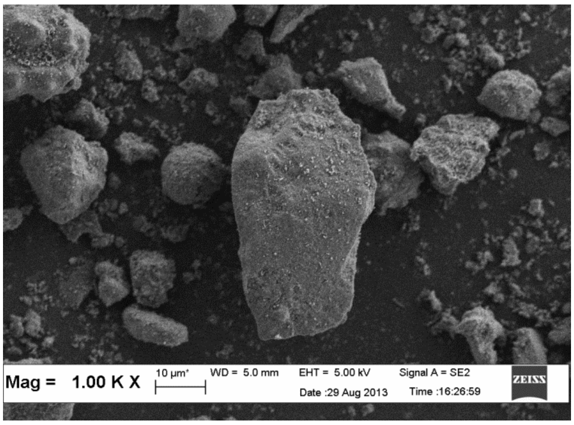


Fig. 4 TPOFA particle shape analysed using SEM technique

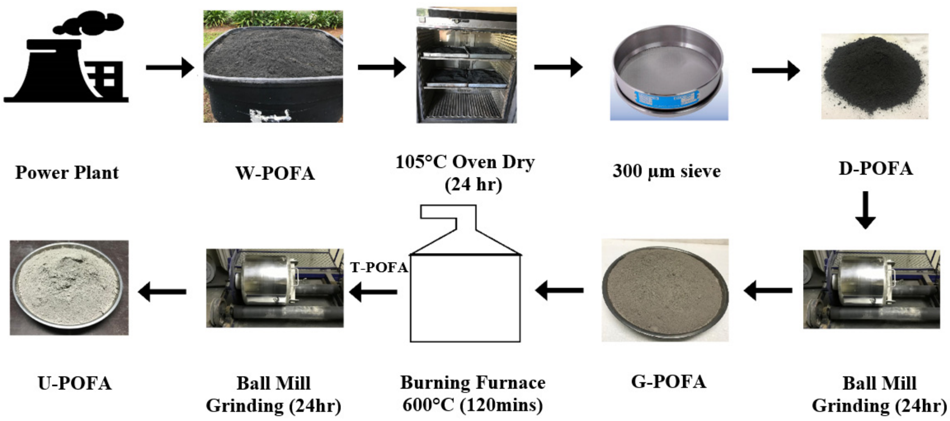


Fig. 5 Processes involved in the treatment of POFA

Tables 4 and 5, respectively. The specific surface area of the U-POFA is 1.77 m^2/g. The major oxides were SiO_2 and CaO, which represent 65.01% and 8.19%, respectively. Al_2O_3 was present in a smaller amount of 5.72%. The high CaO found in the POFA is mostly from fertilizer and lime.

3.1.3 Fine sand

In this study, natural siliceous sand was used as fine aggregates with a maximum size of 1.18 mm. The natural sand was sieved to obtain particle sizes ranging from 150 to 1,180 µm. The calculated fineness modulus and the measured specific gravity of the sand was 2.8 and 2.65, respectively.

3.1.4 Superplasticizer

A high-performance superplasticizer, Sika® ViscoCrete®-2044 was used. This type of admixture is particularly suitable for the production of cementitious composite mixtures that demand a high early strength development, excellent flowability and powerful water reduction, thus increasing density and reducing permeability towards water. The pH-value is between 3 and 5

Table 4 Chemical compositions of Portland cement and POFA

Comp	Cement	G-POFA	U-POFA
	Mass (%)	Wt. (%)	Wt. (%)
CaO	64.89	6.93	8.19
SiO_2	21.01	51.18	65.01
Al_2O_3	4.68	4.61	5.72
SO_3	3.66	0.36	0.33
Fe_2O_3	3.20	3.42	4.41
K_2O	1.17	5.52	6.48
MgO	0.81	4.02	4.58
TiO_2	0.22	0.19	0.25
MnO	0.19	0.093	0.11
Na_2O	0.086	0.056	0.07
P_2O_5	0.079	4.1	4.69
C	-	19.05	0.09
SiO_2+ SiO_2+ Al_2O_3	90.58	62.72	78.92
LOI	0.48	21.6	2.53

Table 5 Physical characteristics of Portland cement and POFA

Property	Cement	POFA	G-POFA	T-POFA	U-POFA
Specific gravity	3.16	2.42	2.51	2.56	2.66
Median particle size, d50 (μm)	6.79	15.76	2.45	2.99	2.06
Specific surface area (m^2/g)	0.785	0.435	1.694	1.438	1.775
Blaine fineness (m^2/g)	0.329	-	0.839	0.788	1.136

and the total chloride ion content is lower than 0.1% which follows ASTM C1017/C1017M-13e1, (2013) and ASTM C494/C494M-19 (2019).

3.2 Mix proportions and samples preparation

Table 6 illustrates the mix proportions for the mortar specimens. The mixture proportioning of ingredients were carried out based on the absolute volume method as per ACI 211.1-91 (Nataraja and Das 2010). The target compressive was set to be 90 MPa which is slightly higher than the compressive strength obtained in previous research works. Nine mixes were designed to achieve the objectives of this study including a reference mix with no POFA, and an additional eight mixes with POFA replacement levels of 5, 10, 15, 20, 25, 30, 35, 40% by volume. The control mix was referred as CM, whereas the mixes containing POFA as (PM) + (replacement level). The CM was designed to get about 90 N/mm^2 compressive strength at 28-day with flowability index of fresh mortar between 1.9 and 2. A sand/cement ratio of 1.5 was used in this current study, similar to a previous study (Mijarsh *et al*. 2015). A constant water/binder (w/b) ratio of 0.27 was used for all mixes. The superplasticizer (Sp) was added to all mortar mixtures by mixing it with water in order

Table 6 Mixture proportions (kg/m^3)

Mix	Cement	POFA	Fine Sand	Water	Sp
	kg	kg	kg	kg	kg
CM	867.68	0.0	1301.53	234.27	19.12
PM5	824.30	36.52	1301.53	234.27	18.94
PM10	780.92	73.04	1301.53	234.27	18.79
PM15	737.53	109.56	1301.53	234.27	18.64
PM20	694.15	146.08	1301.53	234.27	18.48
PM25	650.76	182.60	1301.53	234.27	18.33
PM30	607.38	219.12	1301.53	234.27	18.18
PM35	563.99	255.64	1301.53	234.27	18.03
PM40	520.16	292.16	1301.53	234.27	17.88

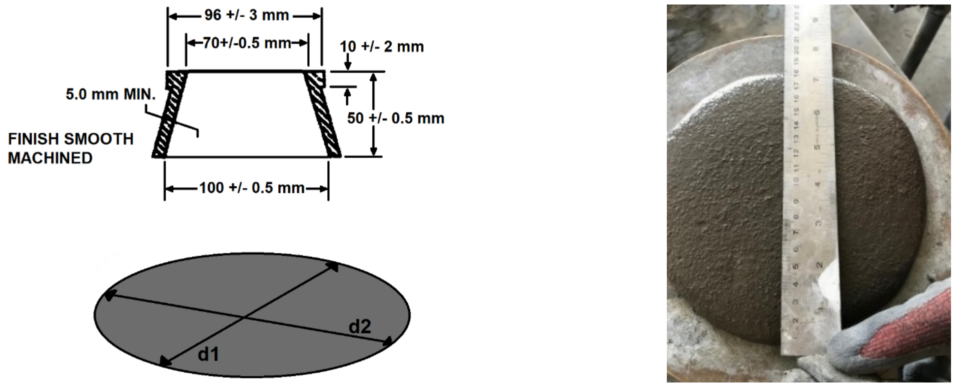
(a)

(b)

Fig. 6 (a) Mortar mixer; and (b) curing tank

to maintain the workability. The dose of Sp used (Sp/binder) was 2.2% by weight. For all mixtures, the fine aggregates, cement and POFA were accurately added into a mortar mixer machine shown in Fig. 6(a). The components were mixed for two minutes in dry condition, and then mixed with half of water for three more minutes. Finally, the Sp was mixed to the remaining half of the quantity of water and added to the mixer. The mixing was continued for three more minutes to reach the required consistency. Immediately after the mixing process, the mortar was cast into 50 × 50 × 50 mm oil smeared steel molds in two layers and each layer was compacted as prescribed by ASTMC109 (ASTM C109/C109M - 20b 2020).

After casting, all mortar specimens were cured in moist cabinets for 24 h with 20°C before demolding. After that, all mortar specimens were placed in a curing tank, which had a temperature of approximately 23°C. The curing condition via the laboratory curing tank was based on ASTM C511-19 (2019). Fig. 6(b) shows the samples in the curing tank.



(a) (b)

Fig. 7 (a) Flow table cone; and (b) flowability of mortar during the test

3.3 Experimental procedures

3.3.1 Flow table

The flow table is one of workability tests used for determining the consistency and flow of hydraulic cement mortars. This test was designed to determine the flow of mortars, either containing hydraulic cement or other cementitious materials. Although specifications for hydraulic cement does not include its flow, it is usually required in standard tests that the mortar has a water content that provides the desired flowability. This test was applied for fresh mixes at all POFA substitution levels. Figs. 7(a)-(b) show the flow table test for mortar containing POFA. This test was performed following the procedures stipulated in ASTM C1437-20 (2020) and ASTM C230/C230M - 20 (2020).

The maximum diameter can be determined after removing the slump cone, also the perpendicular diameter of spread is measured to calculate the flowability index (Γ). The following equation is to determine Γ

$$\Gamma = \frac{D_a^2 - D_0^2}{D_0^2} \tag{1}$$

where D_a is the mean value of both perpendicular diagonals of the formed circle or ellipsoid (d_1 and d_2) and D_0 is a constant number equal to 100 mm which is the bottom side diameter of the slump cone.

3.3.2 Compressive strength

This strength test allows the determination of the compressive strength of hardened cement paste, different mortar and concrete, where the results obtained may be then checked for the agreement with specifications or other studies. The test was performed according to ASTM C109 (ASTM C109/C109M - 20b 2020). Figs. 8(a)-(b) show the machine used for the compressive strength test in this study. The compressive strength was determined at 7, 14, and 28 days of samples age. For each mix, three cubes were tested for every curing age. The average compressive strength of the three samples was reported.

Fig. 8 Compressive strength test instrument (compression machine with 3000 kN capacity)

3.3.3 Ultrasonic pulse velocity

Non-destructive analyses were conducted with the aid of the UPV test on the high-performance cement mortar specimens with a direct contact method using direct transmission measurements. UPV measurements were conducted using a commercial non-destructive and portable UPV apparatus, TICO ultrasonic detector with a rate of pulsing around 3 p/s, resolution of 0.1 μs, measuring range 15 to 6550 μs, and scanning depth up to 3 m as shown in Figs. 9(a)-(b). This test was applied on 81 mortar cubes with dimensions of 50 mm for three curing ages (7, 14, 28 days). The average of UPV values obtained by testing three cubes was recorded and presented in this study. The test was performed according to ASTM C597-16, (2016). The quality of cement mortar can be investigated through the UPV test presented by Estévez *et al*. (2020) in Table 7. This test is normally used to determine the propagation of pulse velocity of internal longitudinal and inclined stress wave pulses in the mortar samples based on the following equation

$$V = \frac{L}{T} \tag{2}$$

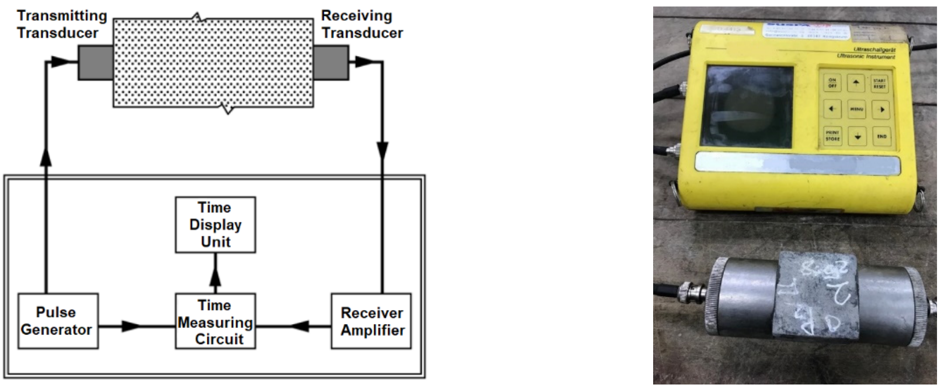
where V is pulse velocity (m/s), L is the distance between centres of transducer faces (m), and T is transit time (s).

3.3.4 Absorption and porosity

The absorption of water by immersion of a hardened mortar under vacuum is defined as the difference between the wet mass (saturated with water in a vacuum medium) and the dry mass of a given sample of hardened mortar, expressed in terms of the dry sample volume. ASTM C642-13 (2013) was used to determine both the water absorption and porosity of mortar samples. Vacuum saturation method was applied on 50 mm mortar cubes. The absorption and porosity were investigated at 7, 14, and 28 days of samples curing age in water. A total number of 81 cubes were cast and tested. For each mix, three cubes were prepared at every curing age. The average value of the three tests was recorded. The same specimens were used for calculating the porosity and absorption. Eq. (3) was used to determine the porosity (P), while Eq. (4) was used to calculate the absorption (A).

Table 7 Typical UPV values to assess the Portland cement mortars quality (Estévez *et al*. 2020)

UPV (m/s)	Mortar Quality
> 3800	Excellent quality
3500-3800	Good quality
3200-3500	Poor quality
< 3200	Very Poor quality



(a) (b)

Fig. 9 (a) Schematic of UPV Apparatus (ASTM C597-16 2016); (b) UPV for 50 mm mortar cube

$$P(\%) = \left(\frac{M_1 - M_2}{M_1 - M_3}\right) \times 100 \tag{3}$$

$$A(\%) = \left(\frac{M_1 - M_2}{M_2}\right) \times 100 \tag{4}$$

where M_1 refers to the mass in grams of samples during dry surface condition while the internal voids are water-saturated (saturated and surface dry), M_2 is the mass of the specimen in grams after drying in an oven at 100 ± 5°C for 24 hours (dry condition), and M_3 refers to the mass of specimen in grams during the immersion in water (apparent mass).

4. Results and discussion

4.1 Flowability

As has been known, the low flowability could cause the HPCM mix to become non uniform with regards to the distribution of its contents, which leads to a reduction in performance and mechanical properties such as compressive strength. The smooth texture of the HPCM mix allows it to flow effortlessly under gravity into various complex forms and moulds with little or no need for compaction. This high workability of HPCM can be significant and mandatory, particularly in

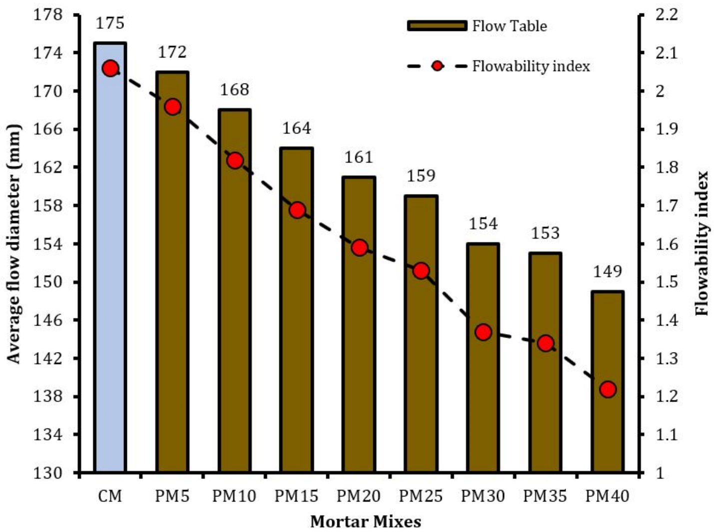


Fig. 10 Flow characteristics of fresh HPCM

concrete structural members that have heavily congested reinforcements. Good workability of a fresh HPCM mixture can be achieved if sufficient segregation resistance and flowability in the fresh state have been reached. Based on the above requirements, the amount of high range water reducer was controlled to obtain the required flow characteristics (Zeyad 2013).

Fig. 10 shows the average flow diameter as well as the flowability index of the fresh mortar mixes. It is clear that as the POFA content increased, the flow reduced, and the effect was more noticeable at the higher POFA content, whereby as the replacement ratio of POFA relative to OPC increased, the flowability of HPCM reduced significantly. This can be mainly attributed to the different in the physical properties between the OPC and POFA. Since the POFA has much smaller particle size and greater surface area than OPC, its incorporation as partial replacement of OPC significantly increased the water demand. As a result, increasing the level of POFA from 0 to 40% greatly reduced the flow diameter from 175 to 149 mm. Nevertheless, all the HPCM mixes containing POFA including the PM40 were able to be placed and compacted fully without any problem. These observations agreed with the ones reported by previous researchers (Huseien *et al*. 2018, Salami *et al*. 2018).

4.2 Compressive strength

The results obtained from the compressive strength tests for all specimens at 7, 14, and 28 days are shown in Fig. 11. It is clear that the control HPCM mix (CM) and four of the HPCM mixes containing POFA (PM5, PM10, PM15 and PM20) achieved the target 28-day strength of at least 90 MPa. The HPCM mixes containing 25%, 30%, 35% and 40% POFA, namely PM25, PM30, PM35 and PM40 mixes, respectively registered slightly lower 28-day strength of 89.2, 88.8, 87.8 and 87.4 MPa. Based on the strength achieved, the HPCM mixes (PM25, PM30, PM35 and PM40 mixes) would be suitable for many applications requiring high strength mortar or concrete, despite not achieving the target 28-day strength.

The bar chart in Fig. 11 shows good development of compressive strength for the OPC mortar

(CM). The strength of the CM mortar specimens at 7, 14, and 28 days was 76.6, 84.8, and 93.1 MPa, respectively. It is worth noting that the mortars containing POFA had lower compressive strengths than the CM mortar at the same curing age, and the strength reduction was greater for mortars with higher content of POFA. At 40% replacement level, the compressive strength reduced by 21.9% and 6.1% with compressive strength values of 59.8 and 87.4 MPa at 7 and 28 days, respectively when compared with the control mortar. Hence, the reduction in strength was much greater at the early age of 7 days than at 28 days. The significant reduction in strength at the early age of 7 days could be mainly explained by the dilution effect as a result of the reduction in cement content in particular in the HPCM containing 40% POFA (PM40) as well as the slower rate and delayed onset of pozzolanic reaction of the POFA. Nonetheless, the reduction in strength of the HPCM containing POFA (PM40) lessened significantly at longer curing period of 28 days as a result of the pozzolanic reaction of the POFA with $Ca(OH)_2$ to produce C-S-H binding gel that improved the compressive strength. Comparing the development in strength from 7 to 28 days for the CM and the PM40 mixes, the CM recorded 17.7% increase in strength, whereas the PM40 registered a significantly greater increase of 31.6%. Again, the greater development in strength from 7 to 28 days for the PM40 could be linked to the pozzolanic reaction of POFA. Based on the observed trend on strength development, it is envisaged that the sustained pozzolanic reaction could contribute towards comparable if not higher strength of the HPCM mixes containing POFA than the CM mortar at much longer curing period. This behaviour has been previously observed in high strength concrete by Zeyad *et al*. (2016).

The lower strength at early ages for mortar and concrete containing POFA and other pozzolanic materials namely fly ash and ground granulated blast-furnace slag have previously been observed by other researchers. It has been reported that the inclusion of pozzolanic materials in mortars or concretes matrix as partial OPC replacement leads to an increase in the SiO_2 and Al_2O_3 compositions while at the same time reducing the CaO content from the lesser cement content, thereby affecting the hydration process at early age and reducing the compressive strength (Wi *et al*. 2018a). With lower cement content as it was replaced with POFA, the composition of tricalcium silicate (C_3S) which is responsible for early strength development of cement will be

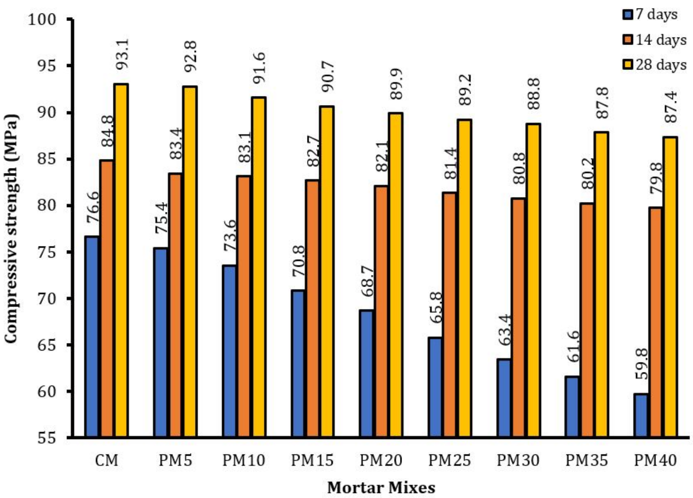


Fig. 11 Compressive strength for various HPCM mixes at the ages of 7, 14, and 28 days

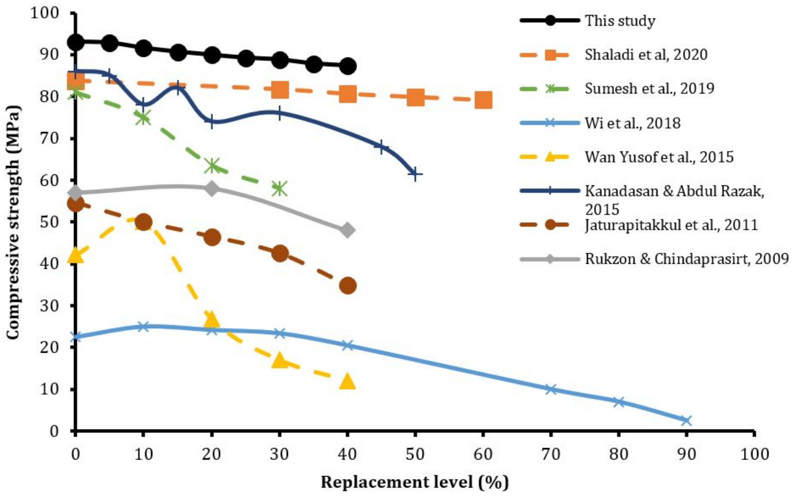


Fig. 12 Comparison between compressive strength at 28 days with previous studies

reduced, hence reducing the early age strength of the HPCM mixes containing POFA.

Rukzon and Chindaprasirt (2009a) reported that the strengths of mortar slightly decreased with the increases in the dosage of the ash included. Zeyad *et al*. (2021a, b, c) also observed reduction in early age strength of high strength concrete containing high volume of ultrafine POFA, but this was compensated via the application of steam curing which accelerates both the cement hydration as well as the pozzolanic reaction of POFA. Huseien *et al*. (2018) experimented with specimens having POFA dosages ranging from 0 to 50% and confirmed that the effect of early-age strength reduction is more prevalent with higher POFA contents. Fig. 12 shows the influence of POFA content on the 28-day compressive strength of HPCM based on the results of this study and several relevant previous studies. Shaladi *et al*. (2019) observed a reduction in strength from 81.70 to 79.20 MPa at 28 days, with the increase in the u-TPOFA replacement level from 30 to 60 wt% in binary blended cement mortar.

4.3 Ultrasonic pulse velocity

The UPV could provide assessment of the uniformity and quality of paste, mortar and concrete. It has been reported in a previous study that cement mortars including those containing POFA typically have a uniform interfacial transition zone (ITZ) and properly distributed voids within the mortar (Kanadasan and Abdul Razak 2015). Fig. 13 shows the UPV of all the HPCM mixes at 7, 14, and 28 days of curing ages. It is evident that the UPV values for all the mixes range from the lowest UPV value of 4494 m/s, recorded by the PM40 mix at 7 days to the highest UPV value of 5213 m/s registered by the CM mix at 28 days. Hence, in term of quality, all mixes could be classified as having excellent quality at the three testing ages since the recorded UPV values are greater than 3800 m/s (Estévez *et al*. 2020).

In addition, it is also clear that the CM and the PM40 mixes exhibited the highest and the lowest UPV values, respectively at the three testing ages. Hence, the effect of incorporating POFA is to reduce the UPV, with greater reduction at the higher POFA content and at the early age. As in the case of strength, this trend could be explained by the reduced cement content of the HPCM

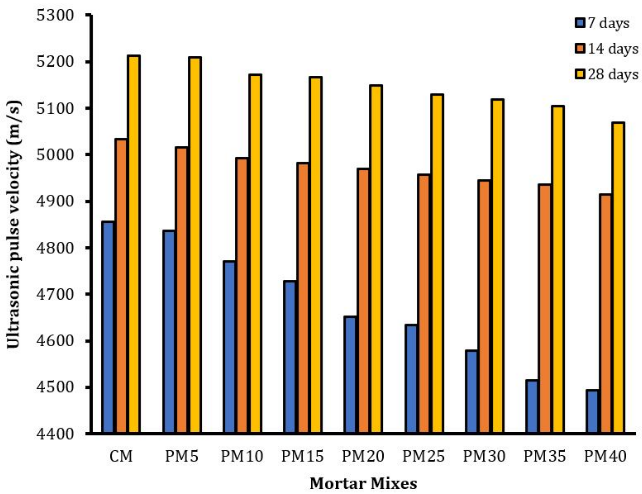


Fig. 13 UPV for various HPCM mixes at the ages of 7, 14, and 28 days

mixtures containing POFA coupled with slower rate and delayed onset of pozzolanic reaction of POFA. Nonetheless, the positive contribution of POFA could be seen by comparing the change in UPV values from early age of 7 days to 28 days between the CM and HPCM containing POFA. The PM mix recorded the increase in UPV of 6.8%, while the mixes containing POFA registered higher increase in UPV that ranges from 7.14 to 11.6%, and this greater increase in UPV could be attributed to the delayed pozzolanic reaction of POFA.

The trend of the findings is generally in agreement with that observed by of several other previous researchers (Kanadasan and Abdul Razak 2015, Usman *et al*. 2017). Fig. 14 portrays that there is a good relation between 28-day compressive strength at the y-axis and UPV at the x-axis. That relationship is represented by a linear equation (Eq.5) with a coefficient of determination R^2 = 0.972.

$$F_c' = 0.0421UPV - 126.37 \quad (5)$$

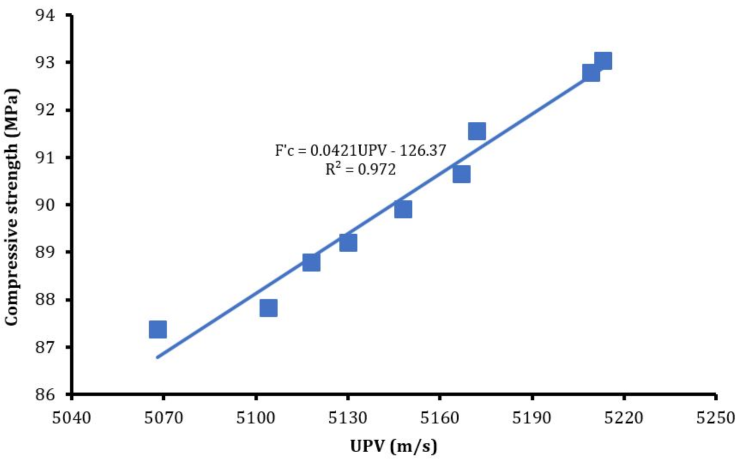


Fig. 14 Relationship between compressive strength and UPV at 28 days

4.4 Absorption

The effect of replacing the OPC with POFA on the water absorption of the hardened mortar mixes is shown in Fig. 15. In general, the variation in the POFA replacement levels did not produce a great difference in the water absorption when compared to the control specimens. It can be seen that the control HPCM (CM) exhibited slightly lower absorption than the HPCM mixes containing POFA at the three testing ages of 7, 14 and 28 days. However, at 28 days the PM5 HPCM mix recorded similar absorption with the CM. In addition, comparing the reduction in absorption from 7 to 28 days, all the HPCM mixes containing POFA generally demonstrated greater reduction in absorption with longer curing age. The CM recorded a reduction of 19.2%, whereas the HPCM mixes displayed greater reduction which ranges from 19.9% to 29.4%, as a result of the contribution of the delayed pozzolanic reaction of POFA. The trend of the results of water absorption is generally in agreement with the study of Kanadasan and Abdul Razak (2015).

4.5 Porosity

Fig. 16 shows the effect of the variation in POFA dosage on the level of porosity of the HPCM. It could be observed that the relationship between POFA content and the porosity was directly proportional, whereby the porosity increased with the incorporation of POFA with greater increase in porosity at the higher POFA content. When compared with the reference mix, as the POFA content increased from 0% (CM) to 40% (PM40), the porosity values increased from 9.8% to 11.5%, respectively at the age of 28 days, which is an increase of 17% from the porosity of the CM. The lower porosity of the CM from the mixes containing POFA, in particular at the early age of 7 days could be attributed to the greater rate of hydration of cement from the higher cement content of the CM which led to faster formation of cement hydration products that occupied the pores, reducing the porosity. In the case of mortars containing POFA, the reduction in cement content as it was replaced with POFA lessened the rate of hydration and coupled with slower rate as well as delayed onset of pozzolanic reaction of POFA slowed down and delayed the rate of

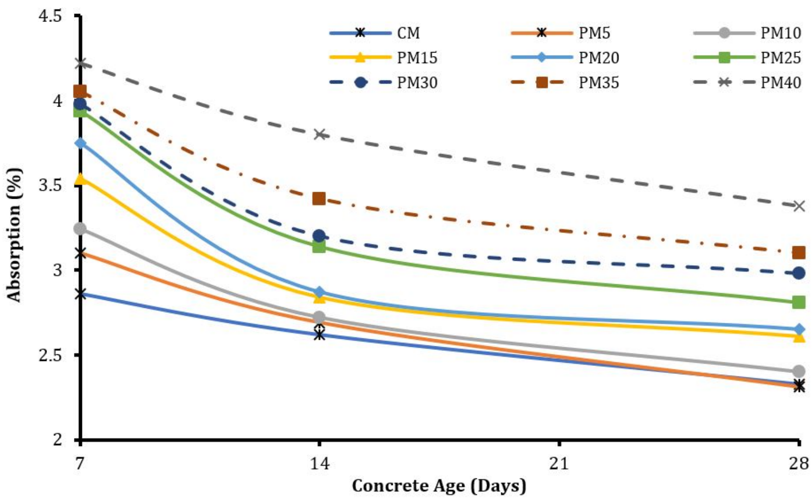


Fig. 15 Absorption for various mixes at 7, 14, and 28 days of HPCM age

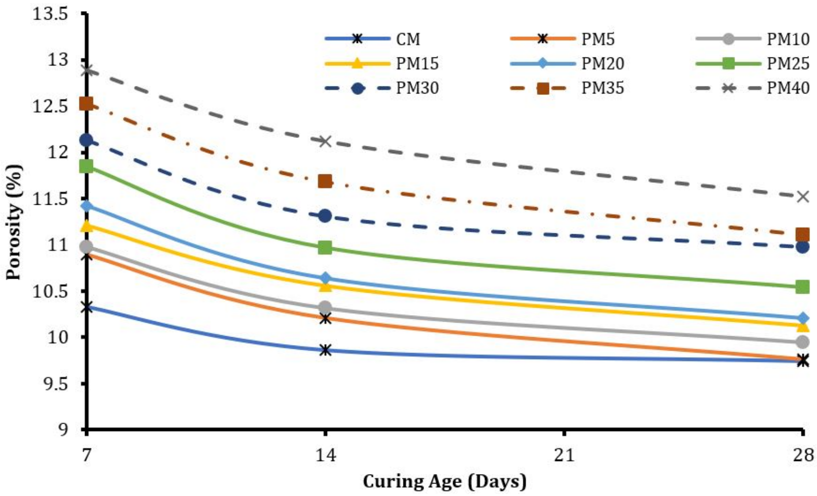


Fig. 16 Porosity for various mixes at 7, 14, and 28 days of HPCM age

C-S-H formation, leading towards higher porosity. The mixes containing POFA seemed to portray greater reduction in porosity from 7 to 28 days than the CM, as a result of the sustained pozzolanic reaction of the POFA. The same observation on the proportionality between porosity and POFA content was indicated by Huseien *et al*. (2018). Also, Kroehong *et al*. (2011) confirmed that adding POFA particles having the same size as the cement results in a paste with higher total porosity than that containing OPC.

5. Conclusions

- The workability of HPCM experienced a reduction when replacing cement with POFA, and the effect magnified as the POFA content increased. At 40% replacement level, the PM40 HPCM mix achieved the lowest flowability index of 1.22, yet full compaction of the mixes containing POFA was able to be achieved.
- The early age compressive strength of the HPCM was significantly affected by the incorporation of POFA. The PM40 HPCM mix recorded 22% lower compressive strength than the control at the age of 7 days. Nonetheless, at the age of 28 days, the PM40 HPCM mix recorded a compressive strength of 87.37 MPa which is only 6.1% lower than that of the control HPCM, indicating greater development in compressive strength at later age as a result of pozzolanic reaction of POFA.
- All HPCM mixes recorded UPV values in excess of 3800 m/s, indicating excellent quality. The UPV decreased as the content of POFA increased, indicating a reduction in mortar quality. The UPV went down from 4856 m/s to 4494 m/s and 5213 m/s to 5068 m/s at 7 and 28 days, respectively as 40% POFA was incorporated in the HPCM mix. This trend exhibits that the negative effect of POFA on UPV values lessened tremendously at longer curing period. Moreover, there is a good correlation between compressive strength of the HPCM and UPV with the correlation coefficients (R^2) equal to 97.2%.
- The absorption and porosity increased with the incorporation of POFA with greater

absorption and porosity at higher POFA content. The HPCM mixes containing POFA exhibited greater reduction in absorption and porosity from 7 to 28 days curing periods than the PM mix, due to the sustained pozzolanic reaction of POFA at longer curing period.

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