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Pozzolanic activity of nanosized palm oil fuel ash: A comparative assessment with various fineness of palm oil fuel ash

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Abstract. As the nanosized POFA (nPOFA) is expected to be a better supplementary cementing material (SCM) compared to conventional POFA, the comparative assessment of pozzolanic activity with various fineness of POFA was investigated. The assessment of pozzolanic activity of as-received POFA (rPOFA), microsized POFA (mPOFA) and nPOFA were conducted via electrical conductivity, lime consumption (LC) and strength activity index (SAI) test. The particle size of nPOFA acquired from TEM analysis is in the range of 20 nm to 90 nm. The nPOFA-lime system demonstrates the highest relative loss of conductivity (RLC) and LC value compared to rPOFA-lime and mPOFA-lime system through the electrical conductivity and LC test, respectively. At 28 days curing age, the nPOFA mortar possesses a 96.9% of SAI as it shows the highest SAI value compared to mPOFA and rPOFA which has 93.8% and 73.6% of SAI values, respectively. The RLC and LC results indicated the high pozzolanic activity of nPOFA compared to mPOFA and rPOFA due to high fineness and specific surface area, resulting in enhancing the degree of pozzolanic reaction. The results confirmed that the high fineness of nPOFA accelerates the pozzolanic activity as it shows the suitability as better SCM compared to the conventional POFA.

1. Introduction

Palm oil fuel ash (POFA) is a by-product generated alongside the combustion of palm oil biomass at high temperature (800–1000ºC) in the boiler for steam production to generate electricity [1]. Currently, POFA is disposed directly onto landfills, creating an environmental burden and health hazard due to the presence of airborne silica and carbon [2–4]. At the same time, it reduces landfill space and causes high costs of waste management [2,3]. While there were researches conducted to address these concerns by utilizing POFA as supplementary cementing material (SCM) since its high silica content (44-66%) [2,4–6].

Nanoparticles are known as particles having sizes ranging from 1 to 100 nm [7]. Research on the implementation of nano-materials as additives and SCM in cement matrix has grown rapidly in recent years since these materials have high prospective as construction materials compared to the

conventional materials [8–10]. Literatures on utilizing nanosized-SCM such as silica fume [11,12], nano-silica [13,14], nano-clay [15,16], carbon nanotubes [17,18], nano-metakaolin [9,19], nano-titanium oxide [20,21] and nano-fly ash [22,23] in cementitious matrix revealed the ability to enhance fresh and hardened properties as it improved the durability of concrete and mortar in comparison with the conventional SCM.

Pozzolanic activity is defined as the proficiency and rate of reaction of pozzolanic materials to generate binding cementitious when it reacts with calcium hydroxide (CH) in the presence of water [24]. According to Donatello [25], pozzolanic activity of materials can be evaluated through direct and indirect methods. Direct methods such as Frattini test [26,27], lime consumption (LC) test [26,28], X-Ray diffraction (XRD) [29,30] and thermogravimetric analysis (TGA) [31,32] assessed the pozzolanic activity by monitoring CH consumption through pozzolanic reaction [25]. Meanwhile, indirect methods involve strength acitivity index (SAI) test [26,33], electrical conductivity (EC) test [34–36] and conduction calorimetry [37,38] in which most of them are designed to measure the progress of physical assets for the interpretation of the degree of pozzolanic activity [25].

According to Massazza [24], the capability of pozzolanic activity of materials strongly dependent on the chemical and mineralogical composition, active phase compounds, fineness of particles, ratio of lime to pozzolan, water content, curing time and temperature. The SCM with high silica content possesses a high pozzolanic reactivity since the silica is a main component in the pozzolanic reaction to form secondary calcium silicate hydrate (CSH) [39,40]. Meanwhile, the increase of fineness contributes to the increment of specific surface area, resulting in high pozzolanic reactivity of SCM due to the high dissolution of particle and rapid reaction with CH to form CSH [38,41].

Presently, no study has been reported on the pozzolanic activity assessment of POFA in various fineness. Hence, the comparative pozzolanic activity assessment would be valuable to comprehend the effect of fineness towards the pozzolanic reactivity of POFA as SCM. The present study examined the pozzolanic activity of as-received POFA (rPOFA), microsized POFA (mPOFA) and nanosized POFA (nPOFA) with the intention to explore the significant of POFA in nanosized particles to be used as SCM.

2. Experimental

2.1. Raw material

The rPOFA specimens were sieved through a No. 100 (150 μm) sieve to remove large contaminants and were dried overnight in an oven at 105 ± 5 °C. The rPOFA was subjected to the modified Los Angeles Abrasion test machine to obtain a mPOFA particles as it is compiled in the ASTM C618 – 15 [42]. Then, the mPOFA specimen was subjected to a ceramic ball milling for 30 h to acquire nPOFA [43,44]. The chemical composition of POFA and Ordinary Portland Cement (OPC) type I was shown in Table 1. Meanwhile, the physical properties of mPOFA and nPOFA were shown in Table 2. Particles size of nPOFA was analyzed via Transmission electron microscopy.

Table 1. Chemical composition of POFA and OPC [43].

Component	SiO_2	CaO	Al_2O_3	MgO	Fe_2O_3	SO_3	LOI
POFA	54.8	14.0	7.24	4.14	4.47	0.71	8.5
OPC	21.45	60.98	3.62	1.22	4.89	2.30	-

Table 2. Physical properties of mPOFA and nPOFA.

Physical properties	mPOFA	nPOFA
Passing 45 μm sieve (%)	96.0	100
S_{BET} (m^2/g)	16.63	145.35
True density (g/mL)	1.78	1.71

2.2. Pozzolanic activity assessment

2.2.1. Electrical conductivity.
Assessments of the pozzolanic activity of POFA specimens were executed using an EC test by determining the RLC value of a pozzolan-lime water suspension [35,36]. A saturated lime solution was prepared in 250 mL deionised water. The solution was continuously stirred at 40 ± 1ºC. The POFA-lime system solution was formulated by adding 5.0 g POFA into the lime solution. Then, the conductivity value reading was monitored for 6 h. The procedure was repeated for the POFA-water system. The experiment was performed in inert environment to evade carbonation reaction to occur. The relative loss of conductivity (RLC) of POFA was computed using Eq. 1.

$$(\%RLC)_t = [(C_0 - (C_{POFA})_t)/C_0] \times 100 \tag{1}$$

Where $(\%RLC)_t$ is the relative loss of conductivity at given time, C_0 is the conductivity value of saturated lime solution without POFA addition while $(C_{POFA})_t$ is the difference between conductivity value of POFA-lime-water system $(C_{POFA-L})_t$ and conductivity value of POFA-water system $(C_{POFA-W})_t$ at a given time.

2.2.2. Lime consumption.
The POFA-saturated lime suspension was prepared with 2.5 g POFA and 25 mL saturated lime solution. The suspensions were kept at 40 ± 1ºC for 1, 3, 7 and 28 days of curing age. At the specified curing ages, the suspension was filtered off and ready to be used for acid-base titration by using 0.02 N hydrochloric acid solution to determine the concentration of hydroxide ion. The LC in percentage was calculated using Eq.2.

$$LC = (([OH\text{-}]_0 - [OH\text{-}]) / [OH\text{-}]_0) \times 100\% \tag{2}$$

Where $[OH\text{-}]$ is a concentration obtained from the POFA- saturated lime suspension and $[OH\text{-}]_0$ is an initial hydroxide ion concentration in a saturated lime solution before it comes into contact with POFA.

2.2.3. Strength activity index.
Mortar cubes were prepared in compliance with ASTM C311-11b [45] to determine the SAI value. The mixing procedure was performed in accordance to the ASTM C305-12 [46]. Binder mixtures using rPOFA, mPOFA and nPOFA were prepared with 20% weight by weight (w/w) of POFA for cement replacement. The binder: sand ratio was kept constant as 1:2.75 by weight [45]. A flow table test was conducted to determine the required amount of water for binder mixture, which gives a consistent flow of mortar in the range of 110 ± 5 cm [47]. Table 3 shows the mix design to cast nPOFA, mPOFA and rPOFA mortars. After 24 h of casting, all cubes were cured the saturated lime solution until the testing day. A compressive strength test of mortars from each POFA specimens was conducted triplicate at 7 and 28 days curing age in accordance to ASTM C109 [48]. The percentage of SAI of mortars was calculated accordance to the ASTM C311-11b [45].

Table 3. Mix design for rPOFA, mPOFA and nPOFA.

Specimen	Cement (g)	POFA (g)	Sand (g)	Water Cement + POFA	Flow (cm)
OPC	500	-	1375	0.48	110
rPOFA	400	100	1375	0.67	108
mPOFA	400	100	1375	0.67	110
nPOFA	400	100	1375	0.66	110.5

3. Results and discussions

3.1. Particle size of nPOFA

Figure. 1 shows the TEM images of nPOFA particle dispersion. It was to be found that the average particles size of nPOFA in the range of 20 to 90 nm [43]. The grinding technique using ball milling is success in producing POFA in nanosized particles. It is expected the POFA in nanosized particles will give a different dimension in pozzolanic activity of POFA since it provides a high surface area in cementitious matrix [38,41].
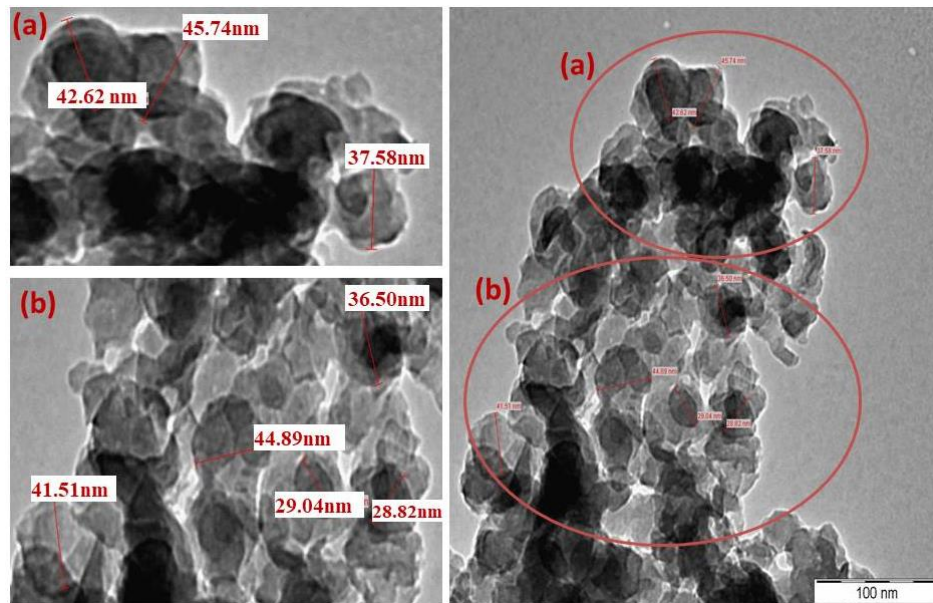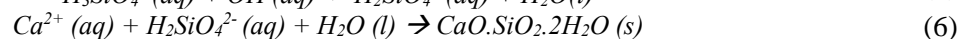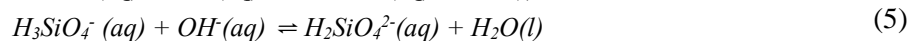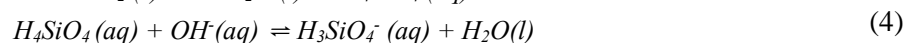

Figure 1. Particles size of nPOFA [43]

3.2.   Pozzolanic activity assessment

The pozzolanic activity assessments of rPOFA, mPOFA and nPOFA were carried out via the EC, LC, and SAI test. The pozzolanic reactivity is discussed based on the effect of fineness on the rate of pozzolanic activity.

3.2.1.Electrical conductivity.

Figure. 2 illustrates the EC of rPOFA, mPOFA and nPOFA in lime suspension. As can be seen, the rPOFA-lime suspension shows the highest EC compared to both mPOFA-lime and nPOFA-lime suspensions. On the other hand, the drop in EC of nPOFA-lime suspension is greater than in both rPOFA-lime and mPOFA-lime suspensions. The EC increased during the first minute and decreased from the first minute onwards. The increase in EC at an early period of the test is accredited to the dissolution of CH as well as the distribution of soluble POFA particles in the suspension [35]. Meanwhile, the drop in EC is suggested due to the occurrence of the pozzolanic reaction between silica and CH to form CSH gel, as the observation is similar with the previous studies [35]. The pozzolanic reaction between silica and CH can be summarized in Eq. 3, Eq. 4, Eq. 5 and Eq. 6.
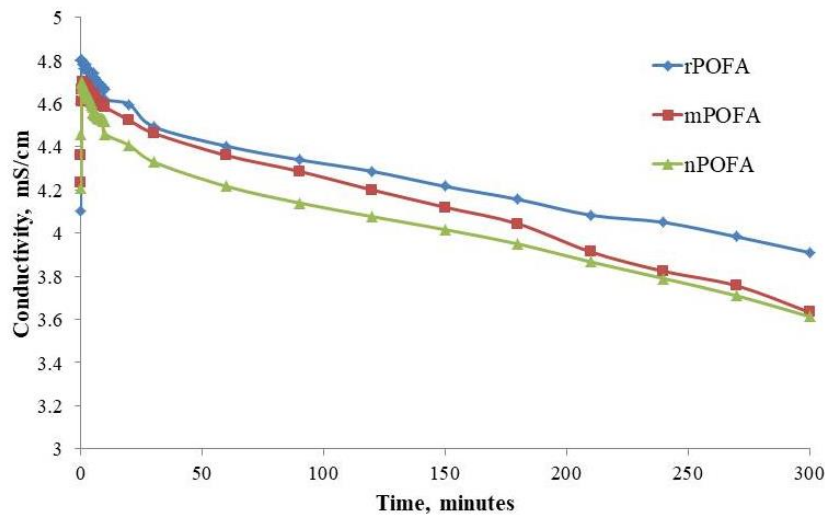
$$SiO_2\ (s)\ +\ \ 2H_2O\ (l)\ \rightleftharpoons H_4SiO_4\ (aq) \tag{3}$$

$$H_4SiO_4\ (aq) + OH^-(aq) \rightleftharpoons H_3SiO_4^-\ (aq) + H_2O(l) \tag{4}$$

$$H_3SiO_4^-\ (aq) + OH^-(aq) \rightleftharpoons H_2SiO_4^{2-}(aq) + H_2O(l) \tag{5}$$

$$Ca^{2+}\ (aq) + H_2SiO_4^{2-}\ (aq) + H_2O\ (l) \rightarrow CaO.SiO_2.2H_2O\ (s) \tag{6}$$

Figure 2. Conductivity of POFA-lime suspension for rPOFA, mPOFA and nPOFA.

As shown in Table 2, the high specific surface area and small diameter of nPOFA gives a better dissolution of silica, followed by rapid pozzolanic reactions with CH to form CSH gels. Subsequently, the EC drops faster compared to mPOFA and rPOFA due to a decrease in the concentration of free ions in suspension. However, these findings do not directly describe the pozzolanic behavior of the POFA since the conductivity value was also contributed by other soluble ions from POFA, which are dissolved in the suspension [35].

Figure. 3 shows the EC of mPOFA-water system. While the increase in the value of EC was observed for all POFA-water systems, the rPOFA-water system shows a greater EC compared to mPOFA-water and nPOFA-water systems. It is suggested that the increase in EC POFA-water system is attributed to the dissolution of silica (see Eq. 3, Eq. 4 and Eq. 5) and other soluble components in POFA [35]. The high EC of rPOFA could be attributed to the presence of soluble impurities which were not isolated from rPOFA [35]. Meanwhile, the differences between EC of mPOFA and nPOFA are due to the high rate of dissolution of silica and other soluble ions of nanosized particles in water. This trend was also in general agreement with other researchers [28,36].
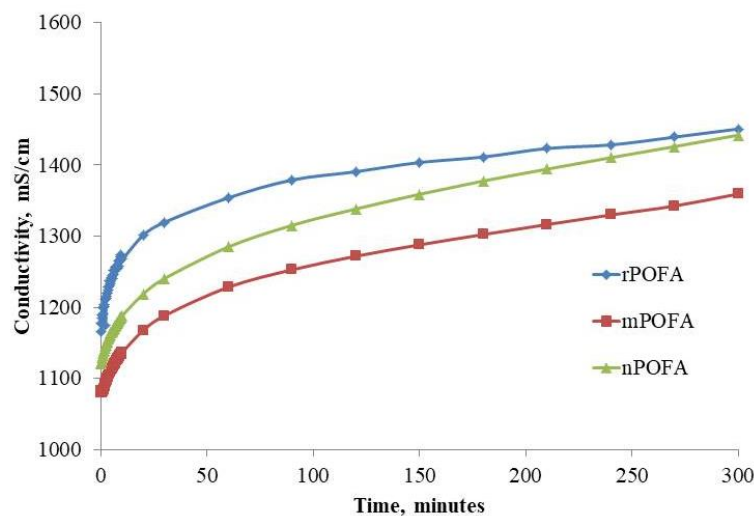

Figure 3. Conductivity of POFA-water suspension for rPOFA, mPOFA and nPOFA.

The RLC result for EC value was obtained using Eq. 1 with an aim to remove the effect of other soluble compounds in POFA which do not contributes to the pozzolanic reaction. The RLC of POFA-lime suspensions, as shown in Figure. 4 signifies the pozzolanic performance of POFA specimens. The nPOFA-lime system possesses the highest RLC value compared to mPOFA and rPOFA-lime systems. The RLC values of POFA specimens increased as a consequence of the pozzolanic reaction which occurred in the suspension. The high RLC value nPOFA is attributed to the high dissolution of silica, followed by rapid pozzolanic reaction. These findings suggest that nPOFA displays a better pozzolanic activity compared to mPOFA and rPOFA specimens.
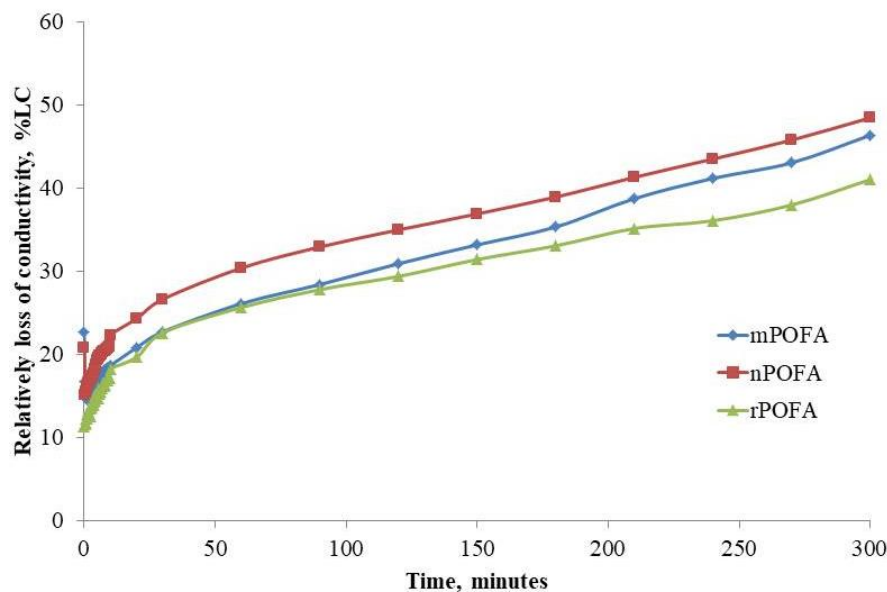

Figure 4. Relative loss of conductivity for rPOFA, mPOFA and nPOFA.

The high RLC values of mPOFA and nPOFA are due to its highly active silica content. Yet, the nPOFA particles increase the rate of dissolution of silica and rapid pozzolanic reaction. This is in agreement with the similar finding reported by other researchers [16,23]. Consequently, the RLC value of nPOFA is higher than mPOFA. Study on the electrical conductivity test suggests that the nPOFA exhibits a better pozzolanic activity compared to mPOFA and rPOFA.

3.2.2.Lime consumption.
The LC value of rPOFA, mPOFA and nPOFA at different curing ages was shown in Figure. 5. As can be seen, the nPOFA displays high LC values compared to both rPOFA and mPOFA specimens. The nPOFA and mPOFA consumed 66.1% and 64.6% of CH at 1 day of curing age, respectively, while the rPOFA displays only 39% of lime consumption. At 3 days of curing age, the nPOFA consumed up to 82.9% of CH whereas mPOFA consumed 79.3% of CH. At this curing age, the lime consumption of rPOFA remains indicates a lower CH consumption, which is 46%. As the CH consumption continuously increased at 7 days curing age, the nPOFA and mPOFA react with more than 80% of CH in the lime suspension. The nPOFA demonstrates a better pozzolanic ability at 28 days curing age, as only 4.8% of CH remains in the lime suspension. Meanwhile, the mPOFA achieved 89.5% of CH consumption while the rPOFA retained in lower CH consumption, which is 65%.
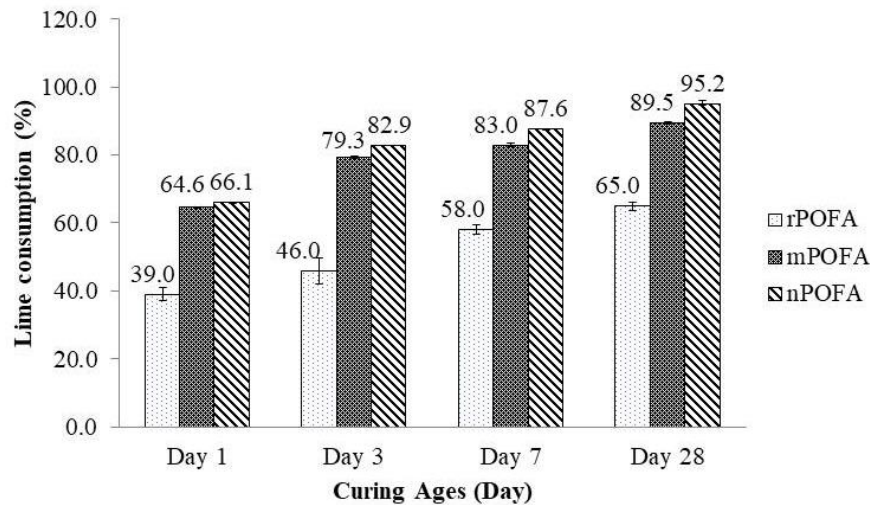
Figure 5. Lime consumption by rPOFA, mPOFA and nPOFA specimen.

The LC results indicate the occurrence of pozzolanic reaction between silica and CH (see Eq. 6). The increases LC in Figure. 5 is attributed to the high pozzolanic reaction rate at later curing ages. This is in agreement with the similar finding reported by other researchers [25,28]. It can be suggested from LC results that the nPOFA demonstrates a better pozzolanic activity compared to mPOFA, while rPOFA shows a poor pozzolanic activity. In addition, the LC results confirmed the electrical conductivity result which proposed the high specific surface area and small diameter nPOFA particles increase the dissolution of silica, resulting in high rate of pozzolanic reaction. Further explanation of the effects of fineness on the LC value between rPOFA, mPOFA and nPOFA are similar with findings in the electrical conductivity value, as discussed in previous section.

3.2.3.Strength activity index.
Figure. 6 represents the SAI of rPOFA, mPOFA and nPOFA while OPC mortar functioned as the control specimens. The control specimen, OPC mortar is expected to achieve 100% of SAI value at 7 and 28 days curing age. At 7 days curing age, the SAI of mPOFA and nPOFA mortars achieved a 75% of the target strength as specified by ASTM C618-15 [42], which is 76.6% and 76.7% respectively. However, the mortars strength at this curing age is lower than SAI of control. Meanwhile, the SAI of rPOFA mortar is lower than SAI value (minimum 75%) as required in ASTM C618-15. At 28 days curing age, the nPOFA mortars possess a 96.9% of SAI as it shows the highest SAI value compared to mPOFA and rPOFA which has 93.8% and 73.6% of SAI values. The SAI of mPOFA and nPOFA mortars confirmed to the requirement as specified in ASTM C618-15 [42]. The rPOFA achieved the lowest SAI as it does not meet the specification in ASTM C618-15. Furthermore, the result indicates the increase of SAI values for all mortar with curing time up to 28 days. In spite of this, the SAI of all mortars did not reach the SAI of control specimens.
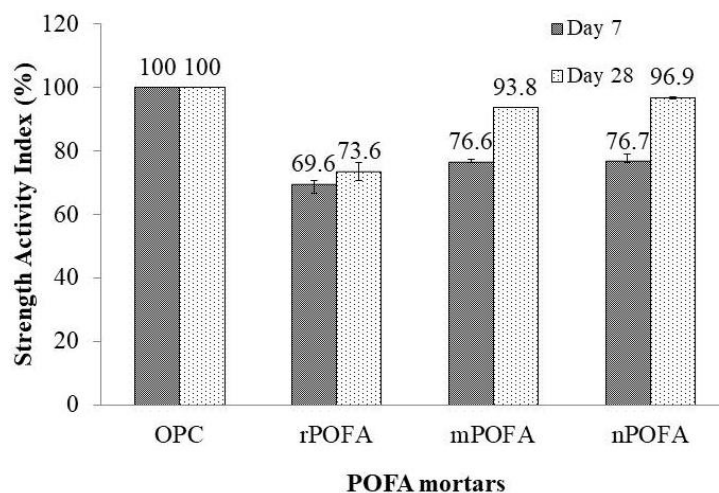
Figure 6. SAI value of rPOFA, mPOFA and nPOFA mortars

It is suggested that the differences in the SAI values of rPOFA, mPOFA and nPOFA are mainly attributed to the fineness of POFA particles. Similar findings from the previous literature also show the fly ash with various fineness contributes to different SAI value of mortars [23,49]. The high SAI value of nPOFA mortars could be attributed to the high specific surface area and small diameter of nPOFA particles. These factors enhance the distribution of nPOFA in the hardened cement paste (filler effect) and the potential of nPOFA as nucleation sites in a hardened mortar. It also could escalate the dissolution of amorphous and crystalline silica in the pore solution of the hardened cementitious matrix as well as increase the pozzolanic reaction rate to produce secondary CSH. Consequently, it refines the microstructure of hcp and enhances the strength of nPOFA. Further explanations on the effect of nPOFA toward its filler effect and pozzolanic reaction are similar with the findings on previous studies [19,50]. The crystalline silica could give a low dissolution in water due the high stability in Si-O framework [51]. However, the dissolution of C_3S and C_2S (OPC) in hcp increase the pH in pores solution [52]. This suggested that the high pH of pore solution in the hardened cementitious matrix, as well as the small diameter of nPOFA particles, could be attributed to the dissolution of crystalline silica. Meanwhile, the low SAI value of rPOFA mortars in Figure. 6 could be related to the low active silica content besides the low fineness as well.

As can be seen in Figure. 6, the low SAI values of rPOFA, mPOFA and nPOFA mortars at 7 days curing age, which is 69.6%, 76.6% and 76.7%, respectively, compared to the SAI values at 28 days curing age could be attributed to the retardation and dilution effect. Meanwhile, the increases of SAI value in rPOFA, mPOFA and nPOFA mortars at 28 days curing age, which is 73.6%, 93.8% and 96.9%, respectively; could be explained by the pozzolanic reaction in producing secondary CSH which contributes to the strength of mortars.

The comparison between the SAI of mortars and LC value in lime-POFA suspension at 28 days curing age are shown in Figure. 7. As can be seen in Figure. 7, as the fineness of POFA particles increases, the LC value and SAI value of mortars increase as well. It can be seen in Figure. 7, the nPOFA achieved the highest CH consumption and SAI value of mortar, which is 95.2% and 96.9%, respectively. Meanwhile, the rPOFA shows the lowest CH consumption and SAI value of rPOFA mortar, which is 65% and 73.6%, respectively. The CH consumption in mPOFA-lime suspension and the SAI value of mPOFA mortar are 89.5% and 93.8%, respectively.
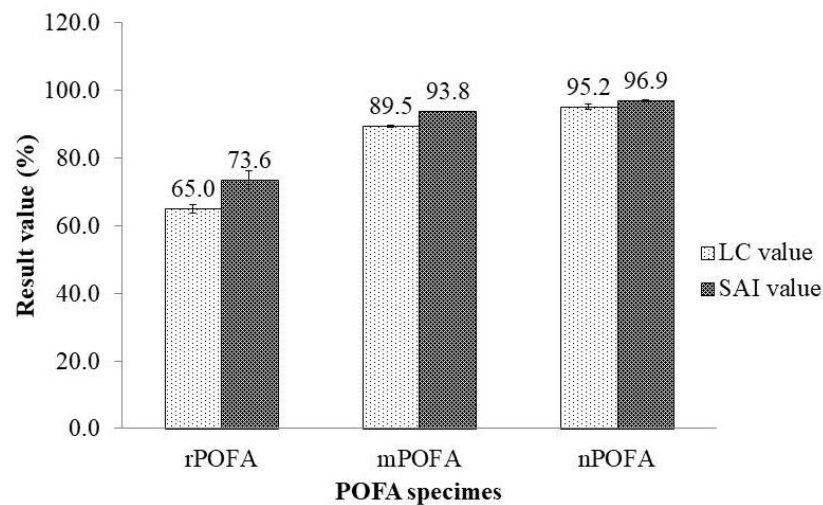
Figure 7. The SAI values of mortars and LC value of POFA-lime suspension at 28 days curing age.

From Figure. 7, it is evident that the differences of CH consumption and SAI value of mortars are likely to be due to the fineness of POFA particles. Altogether, the high fineness of nPOFA could attribute to the high dissolution of silica in the lime-POFA suspension (LC test) and pore solution (in the hardened mortars) which increase the rate of pozzolanic reaction. This result shows the relationship between CH consumption and SAI value of mortars. It is suggested that high LC value indicates high pozzolanic reaction which could increase the formation of secondary CSH and supply the additional strength to mortars. Overall, the obtained data for the influence of fineness POFA particles on the strength of mortars suggests that the high specific surface area and small diameter of nPOFA particles increase its pozzolanic activity, while the high rate of pozzolanic reaction in nPOFA mortars enhance the formation of the secondary CSH, resulting in a high SAI value of nPOFA mortar.

Conclusions

The outcome of this study can be summarized as follows:

• A better reactivity of nPOFA compared to mPOFA and rPOFA with CH can be observed based on the results of EC and LC test. It is suggested that the small diameter of nanosized particles and high specific surface area of nPOFA increase the dissolution of silica which led to the rapid pozzolanic reaction as the formation rate of CSH precipitate is increasing.

• The results of EC and LC test confirmed the SAI of mortars. The high SAI of nPOFA mortars could be attributed to the high fineness of nPOFA particle, which enhances the filler effect, increases the potential to act as nucleation sites, and escalates the rate of pozzolanic reaction in hardened mortars. As results, the high formation of secondary CSH in hardened cementitious matrix increases the strength of mortars.

• Overall, the assessments of the pozzolanic activity of POFA with various fineness (rPOFA, mPOFA and nPOFA) suggested that the pozzolanic activity of SCM is strongly affected by the fineness of the particle. Thus, it can be considered that nPOFA has the highest pozzolanic activity compared to mPOFA and rPOFA.

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