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Journal of Advanced Concrete Technology, volume 15 (2017), pp. 684-699

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Journal of Advanced Concrete Technology, volume 12 (2014), pp. 127-137

Performance of Different Grades of Palm Oil Fuel Ash with Ground Steel Slag as Base Materials in the Synthesis of Alkaline Activated Mortar

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Journal of Advanced Concrete Technology, volume 12 (2014), pp. 378-387
POFA-Engineered Alkali-activated Cementitious Composite Performance in Acid Environment

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Abstract

The durability performance of palm oil fuel ash engineered alkali-activated cementitious composite (POFA-EACC) mortar exposed to different acid solutions is assessed in this study. 50 mm cubic specimens used for the study were prepared from 100% POFA, alkali-activator (Na₂SiO₃(aq)/NaOH(aq)) ratios of 2.5, different molarities (10, 12 and 14 M) of NaOH(aq) and 2% volume fraction of polyvinyl alcohol (PVA) fibres. Specimens were exposed to 10% H₂SO₄(aq), 10% HNO₃(aq) and 10% HCl(aq) at pH of 0.56, 0.52 and 0.42 respectively for 3, 6 and 9 months, with unexposed specimens as control. Small changes in compressive strength were identified with POFA mortar specimens during exposure to H₂SO₄(aq), while exposure to HNO₃(aq) and HCl(aq) greatly reduced the strength of the POFA mortar specimens. The results were supported through microstructural examinations using SEM, while the characterization was done using XRD and FTIR. The high resistance of POFA-EACC mortar to H₂SO₄(aq) is the contribution received through the formation of gypsum, which hinders the infiltration of more acids into the matrix microstructure.

1. Introduction

Developing a least permeable concrete to stand the test of time against deleterious agents such as acids have been an ongoing challenge in the research community. One of the many methods used to prevent the entrance of these harmful materials into the concrete was the optimization of the selected mixtures. Other strategies used against attack from acids were the use of durable coating materials on the surface of the developed concrete and the modification of the environment where the concrete “lives”. All of these solutions were all to either make the environment habitable for concrete or make the concrete durable or resilient in any environment regardless of the severity.

Despite all these, studies have not produced favorable results as the acid not only attack the hydrated and unhydrated cement, it decomposes any material that comes its way (Pacheco-Torgal et al. 2014). However, there are reports suggesting alkali-activated binder as a reliable alternative in withstanding the harshness of its external environment for service life improvement. Since its early development, alkali-activated binder have been widely reported as highly resistant to acid attack (Fernandez-Jimenez et al. 2006; Shi et al. 2006). However, few alkali-activated materials have been reported (Pacheco-Torgal et al. 2014) to undergo binder degradation due to acid attack, which in most cases was through ion-exchange reactions. The end products after the acid attack are calcium salts of the acid and/or decalcified residue of the hydrated cement products (Pavlík and Unčík, 1997). These reported negative effects of acids on alkali-activated binder awakens the need for more studies to ascertain the durability properties of alkali-activated binder especially with different precursor materials (Borges et al. 2016).

Bakharev (2005) started an early work to establish the actual durability performance of alkali-activated mortar in acid using fly ash as a base material. The resistance of the fly ash alkali-activated binder was studied using two acid solutions (5% CH₃COOH and 5% H₂SO₄) for 2 months. The performance of the alkali-activated concrete compared to the OPC concrete was found to be superior in resisting the deteriorating effect of the acids. Another study by Thokchom et al (2009) with fly ash as base material and a higher acid concentration (10% H₂SO₄(aq)) for 18 weeks revealed the high resistance of fly ash alkali-activated concrete to H₂SO₄(aq). After the exposure period, there was 52% loss in strength with alkali-activated mortar specimen with 5% Na₂O and 28% strength loss with 8% Na₂O. In addition to H₂SO₄(aq) used, HCl acid was used by Sreevidya et al (2012) to study the acid resistance of fly ash based alkali-activated mortar for 14 weeks. With weight loss, compressive strength and visual appearance as evalua-

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ion parameters, the fly ash-based alkali-activated specimen was reported to be highly resistant against $\text{H}_2\text{SO}_4$ and $\text{HCl}$ acids. In yet another study using lignite bottom ash, Sata et al (2012) studied the durability of this alkaline activated mortar in 3% sulfuric acid and 5% sodium silicate solution for 120 days. The results revealed that the lignite bottom ash was less susceptible to sulfuric acid attack compared to conventional OPC mortar.

Alkali-activated binder presents a better mechanical and durability performances as shown by the studies above; however, its production is presently expensive with materials like OPC, fly ash, etc. As a result, alkali-activated binder development needs materials like Palm oil fuel ash (POFA) with no economic value posing environmental and social risks to people. POFA have been used wholly (Salami et al, 2016, 2017) and partially (Megat Johari et al, 2012; Yusuf et al, 2014a) in the development of alkali-activated mortar and concrete. The reality of the durability performance of alkali-activated binder is dependent on the source material used and the concentration of exposure acids. A number of researchers (Altwait et al, 2012; Johari et al, 2013) have studied fresh and hardened alkali-activated properties of POFA and its blends with other materials, but limited studies are presently available on its durability performance. One of the very few studies was where Ariffin et al (2013) utilized POFA blended with pulverized fuel ash (PFA) in the development of alkali-activated concrete and its durability performance when exposed to 2% sulfuric acid was studied. The study revealed a better durability performance in comparison to OPC concrete. Another work into the acid resistance of POFA-based alkali-activated mortar was undertaken by Monita et al (Olivia et al, 2016) where acid resistance comparison was drawn between fly ash (FA)-based and POFA-based alkali-activated mortar for 180 days. The exposure environment for the mortar specimens was an organic acid water in swampy peat soil, which consists of humic acid that is potentially corrosive to concrete and metal structures. The results conclude that the FA-based geopolymer mortar performed relatively better than the POFA-based alkali-activated mortar because of the stable aluminosilicate-bonding present in the FA-based alkali-activated mortar. It is worthy of note that in the study, the POFA-based alkali-activated mortar performed better than the OPC concrete used as control specimen. To the best of the knowledge of the authors, the work of Monica et al was the only study on the durability of POFA-alkali activated binder with 100% POFA and that cannot be representative as only one mixture was used to study the POFA binder durability. Therefore, there is need for more studies into different factors affecting the mechanical properties and durability performance of POFA alkali-activated binder.

This research work studies the effect of different acidic environments (10% $\text{H}_2\text{SO}_4$, 10% $\text{HNO}_3$ and 10% $\text{HCl}$) on POFA-based engineered alkali-activated cementitious composite (POFA-EACC) mortar during a 9-month exposure. The parameters used in the durability study are residual compressive strength, weight loss and visual appearance of the mortar specimens after exposure to the different acids. The results obtained were compared with the 28-day compressive strength unexposed to the acidic environment. It is the hope of the authors that this work will be synergic to all other works to stimulate the increase and beneficial use of POFA.

### 2. Experimental program

#### 2.1 Materials

##### 2.1.1 Palm oil fuel ash

POFA was sourced from United Oil Palm Industries Sdn. Bhd. in Nibong Tebal, Penang, Malaysia. It was generated as a by-product from the combustion of palm kernel shells, fibres and empty fruit bunches to heat the boiler for electrical generation in the palm oil mill. The sourced raw POFA was dried in an oven at $100 \pm 5^\circ\text{C}$ to remove moisture and sieved using a set of sieves (600 µm and 300 µm) to remove coarser and unwanted materials. The after-sieve ash was then ground by a mechanical ball mill containing 150 steel balls of sizes from 6 mm to 32 mm and rotating at speed of 180 rpm. This was done to reduce the POFA particle size and to increase its surface area, which ultimately will aid in its reactivity. This grinding is followed by calcination at $550^\circ\text{C}$ in a gas-powered furnace for 90 min to remove unburnt carbon, prevent glassy phase crystallization and agglomeration of particles. To further improve the surface area, the calcined POFA underwent another round of grinding in the ball mill. It should be noted that apart from increased fineness after grinding, the density also increased (Jaturapitakkul and Cheerasot 2003). This is because porous samples characterised by low density have improved porosity hence increased density (Payà et al, 1996). The loss on ignition (LOI) values after heat treatment is 2.3% an indication of low unburnt residue in the POFA (Chandara et al, 2010). The reduction in the LOI value is compensated for by the increase in mass percentages of other oxide components. With total oxides of Silicon, Aluminium and Iron of 78.07%, it complies with the specification of ASTM C618 class F. Tables 1 and 2 respectively show the oxide compositions and physical properties of POFA, which were determined using X-ray fluorescence (XRF) technique.

| Materials/Oxides | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | CaO | MgO | Na$_2$O | K$_2$O | P$_2$O$_5$ | SO$_3$ | LOI | Total |
|------------------|--------|-------------|-------------|-----|-----|--------|--------|---------|-------|-----|-------|
| POFA             | 66.91  | 6.44        | 5.72        | 5.56| 3.13| 0.19   | 5.20   | 4.13    | 0.33  | 2.3 | 99.88 |

**Table 1 Chemical composition of POFA.**
2.1.2 Aggregates
The fine aggregate used in the study is dune sand with fineness modulus of 1.85 and density in the saturated and surface dry (SSD) condition of 2.62.

2.1.3 Synthesis of alkaline activators
Laboratory-grade Na$_2$SiO$_3$(aq) with an initial silica modulus (Ms = SiO$_2$/Na$_2$O) of 3.3 and NaOH (aq) were used as alkaline activators (AA). The percentage compositions of the Na$_2$SiO$_3$(aq) are as follows: Na$_2$O (8.76%), SiO$_2$ (29.13%) and H$_2$O (62.11%) All the available water was from added water (10 wt. % of POFA) and water contained in the AA. The distilled water and NaOH pellets of 99% analytical-grade were used in the preparation of the xM NaOH(aq) (where x = 10, 12 or 14). The mass of solids (pellets) used is 399.98g, 479.97g and 559.97g in 1 litre of solution for 10, 12 and 14M NaOH(aq) respectively.

2.1.4 Polyvinyl alcohol fibre
Polyvinyl alcohol (PVA) fibres “REC15”, manufactured by Kuraray Co - Japan, with data provided by the manufacturer as shown in Table 3, was used in this study. The fibers are coated with a proprietary hydrophobic oiling agent of 1.2% by weight to control the interfacial bonding properties between the fiber and matrix for composite performance (Li et al. 2002).

2.2 Experimental procedure
2.2.1 Mixture proportion and mixing for POFA-based EACC mortar
POFA-EACC mortar was prepared with 100% POFA as the binder with a Sand/POFA ratio of 1.8. The initial silica modulus (Ms = SiO$_2$/Na$_2$O) was obtained from activator’s relative proportion = (Na$_2$SiO$_3$ (aq)/XM NaOH(aq)) such that the ratio is 2.5:1. The water was added such that water to POFA ratio was maintained at 10 wt. %. To study the durability performance of POFA-EACC mortar to acid attack, three different acidic solutions (10% H$_2$SO$_4$(aq), 10% HNO$_3$(aq) and 10% HCl(aq)) were prepared for the mortar specimen exposure. Table 4 shows the proportions of the constituent materials in the POFA geopolymer mixtures.

The constituent materials were mixed in the Hobart planetary floor mixer model HSM 20 at room temperature. The mixing of POFA geopolymer mortar was in two stages. First, The POFA and sand were mixed for 4 minutes until a homogeneous mixture was achieved in dry stage. Secondly, the alkaline solution (NaOH (aq) + Na$_2$SiO$_3$(aq)) was added to the homogenous mixture of POFA and sand for the wet mixing stage. The mixture was then continued for another 5 minutes to achieve fresh geopolymer mortar. The mixing continued until a uniform and homogeneous fresh mixture was achieved. After achieving homogeneity and making sure there are no solid materials left sticking to the base of the bowl, the PVA fibres were gradually spread into the fresh mortar matrix by hands as the mixture is mixed at slow speed until all fibres were evenly distributed. The fibres were added slowly to ensure proper dispersion with no agglomeration. This mixing procedure was considered by many authors (Cheng and Chiu 2003; Kong and Sanjayan 2010; Swanepoel and Strydom 2002) as required to attain good compressive strength. The slump of the mortar mixture was measured before casting into the molds. The cube molds were smeared with oil and specimens were cast in three layers of standard rod compaction and was then vibrated on the vibrating table for 20 sec. On the second day of casting, the cubes were demolded and subjected to temperature curing at 65 °C for 24 hrs. After that, the specimens were cured under a condition of 20 ± 5 °C and 95% relative humidity until the age of exposure. The curing age is 28 days before subjecting them to sulfate tests. The 28-day compressive strength of the specimens was used as the control for the study.

2.2.2 Test procedure
To study the durability performance of POFA-EACC mortar specimens in acidic environment, specimens were immersed in three different acid solutions (10%
H₂SO₄(aq), 10% HNO₃(aq) and 10% HCl(aq) at 28-day curing for 9 months. The initial pH of the solutions are 0.56, 0.52 and 0.42 for sulfuric acid, nitric acid and hydrochloric respectively. The 50 mm x 50 mm x 50mm POFA-EACC mortar specimens were used for the study. After the 28-day curing, the specimens were immersed in the acid solutions for the required exposure periods at room temperature. Solution were made 24 hrs prior to the specimen immersion, thus replaced every 4 weeks in order to maintain as much as possible the exposure environment. The weight loss and residual compressive strength after 3-month, 6-month and 9-month period of exposure were recorded.

2.3 Analytical methods
Scanning electron microscopy (SEM), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) were used to study the deterioration after exposure to the different acids. Samples were taken from at approximately 2mm from the surface of the samples exposed to the solutions for the analysis. XRD was conducted on POFA alkali activated powder using Rigaku miniflex II powder automated diffractometer with the following parameters (Cu-Kα radiation (λ 1.54178 Å) operated at 30kV and 15 mA). The XRD patterns were obtained by scanning at angle ranging from 10-80°, with sampling width and the scan speed of 0.02° and 2°/min, respectively.

The specimens for SEM analysis were prepared by cutting into a distance of 2-3mm of 0.5mm thick slices from the POFA alkali-activated mortar specimens to expose fresh surfaces. The cut specimens were then coated with gold for the avoidance of distortion in image and also to give allowance for discharge of electron on the specimen surface.

For XRD specimen preparation and analysis, POFA alkali-activated specimens were pulverised until smooth powder using an electronic grinding machine. 10-12 g of the powder was taken with spatula, and gradually dropped to fill the specimen holder to avoid agglomeration. Flushing of the specimen in the holder was achieved using a piece of flat glass with smooth surface. After careful removal of the flushing glass, the specimen holder was placed in the diffractometer. The different phases present in the POFA alkali-activated specimens were identified and analysed using X’Pert Highscore Plus software in order to compare the identified spectra with the global database.

For FTIR specimen preparation and analysis specimen preparation, approximately 100 mg of kBr powder was mixed with 1mg of the POFA alkali-activated powder specimen. Both kBr and POFA powder were mixed carefully and thoroughly for homogeneity and best results. The homogeneous mixtures were then placed in a die machine tool where pressure was applied under vacuum for approximately 4 minutes. Flat pelleted samples ready for FTIR scanning, was placed in the specimen holder and then scanned. The spectra results were analysed using OMNIC software were functional groups were identified.

3. Results and discussion
3.1 Compressive strength
3.1.1 Compressive strength of unexposed POFA-EACC mortar
The mechanical strength of alkali-activated POFA-EACC mortar specimens (NH10, NH12 and NH14) after 28 days of curing is shown in Fig. 1. These unexposed POFA-EACC mortar specimens to acid solutions were used as control specimens. The mortar specimens showed good compressive strength with strengths above 24MPa. In the POFA alkali-activated binder system, there exists a low availability of Al₂O₃ in the developed cementitious matrix. As a result of this, there exists a strong dominance of C-S-H gel with traces of Al₂O₃ in its microstructure, which slowed down its setting time and eventually its low strength. (Salih et al. 2014). The binding capability of the C-S-H gel could have been improved provided there was enough Al₂O₃ to be incorporated in the bridging sites of the SiO₂ chains (Andersen et al. 2006; Harvey and Dent Glasser 1989). The figure also shows clearly the dependence of the compressive strength among other factors on the molarities of NaOH(aq). This aided the dissolution of the POFA base material, which increases with increased concentration of NaOH(aq), aiding the precipitation of the Al₂O₃-SiO₂ phases early in the reaction. As a result of this, the polycondensation reaction was hastened in forming the polymeric chain, which improved the strength development of the POFA alkali-activated mortar specimens. The low Al₂O₃ content mentioned above in the chemical composition of POFA reduces the availability of Al(OH)₄⁻ for condensation expected to occur between alumina and silica species. This failure to create more alumina-silica species due to low presence of Al₂O₃ created the environment for more condensation between the silicate and silicate species. And as a result, relatively low matrix compressive strength was recorded with the POFA-based alkali-activated binder. The strength of alkali-activated binder is expected to increase with an increase in the SiO₂/Al₂O₃ ratio because.

Fig. 1 Compressive strength of unexposed specimens at 28 days.
Si–O–Si bonds, which are stronger than Si–O–Al and Al–O–Al bonds, will be proliferated with the increase in SiO$_2$/Al$_2$O$_3$ ratio (Yusuf et al. 2014b). However, there is strong correlation between SiO$_2$/Al$_2$O$_3$ ratio and compressive strength of alkali-activated mortar, strength increases with increase in SiO$_2$/Al$_2$O$_3$ within the required range (Yusuf et al. 2014b), outside which the strength starts to drop. This type of binder fits into the family of alkali-activated binders with high SiO$_2$/Al$_2$O$_3$ ratio.

3.1.2 Effect of acids exposure on compressive strength of POFA-EACC mortar

Plotted and shown in Figs. 2-4 are the results of the exposed POFA-EACC mortar specimens to H$_2$SO$_4$, HNO$_3$, and HCl solutions after 28-day curing respectively. Subsequently, the exposed specimens were tested for residual compressive strength after 3, 6 and 9 months of exposure periods. The unexposed POFA-EACC mortar specimens had a gentle failure with some delay in its crack propagation. However, after exposure especially with the POFA-EACC mortar specimens exposed to HCl and HNO$_3$, due to the brittle nature after exposure, the crack propagation was relatively faster in comparison with the unexposed mortar specimens. The H$_2$SO$_4$ exposed specimen had a soft failure with a much more delayed crack propagation. It is also worthy of mention that the applied loads were largely sustained in all the POFA-EACC, however more with the H$_2$SO$_4$ exposed specimen.

As shown in Figs. 2-4, during the early months of exposure, all the POFA-EACC mortar specimens lost strength. However, the extent of strength loss is dependent on the type of acid, the period of exposure to the acid solution and the molarity of NaOH$_{(aq)}$ used as alkaline activator. A deeper look at Figs. 5-7 revealed sharply the effect of NaOH$_{(aq)}$ molarities on the residual strength of POFA-EACC mortar specimens after acid exposure. Depending on the exposure acids as shown in the plots (see Figs. 5-7), two behavioural trends were observed with the POFA-EACC mortar specimens. For the specimens exposed to H$_2$SO$_4$ solution, the percent strength loss in compressive strength decreases with increase in not only the molarity of NaOH$_{(aq)}$ but also with exposure period.

The POFA-EACC mortar specimens exposed to H$_2$SO$_4$ lost the least strength in all periods of exposure as shown in Figs. 2 and Figs. 5-7. The first 6 months of H$_2$SO$_4$ exposure witnessed losses in strength, which was due to insufficient gypsum shield formed from the interaction between the H$_2$SO$_4$ solution and Ca-compounds in the alkali activated products (Allahverdi and Skvra 2005). This was not enough to prevent the acid from attacking the specimens due to less Ca$^{2+}$ ions in the solution to form the needed gypsum. After 6 months of exposure, there was some strength improvement, reaching a maximum percentage strength gain of approximately 4% with the 10M POFA-EACC mortar specimen. The formation of gypsum (CaSO$_4$) deposited in the eroded layer (Ruiz-Santaquiteria et al. 2012) forming a protective shield against further deterioration and also provided support to the matrix. The insolubility and stability of gypsum compared to silica gel from the alkaline activation of POFA helps at later periods of exposure in sustaining the matrix strength. The percentage strength gain of 8.1% and 18.7% was recorded with 12M and 14M mortar specimens after 9 months of exposure to H$_2$SO$_4$ solution.

Figures 3 and 4 reveal the changes in compressive strength of POFA-EACC mortar specimens after exposure to HCl and HNO$_3$ respectively. In all periods of exposure, the figures reveal losses in compressive strength of POFA-EACC mortar specimens with severity more in the HNO$_3$ exposed specimens. As seen in Figs. 5-7, the loss in strength also increased with increase in the molarity of NaOH$_{(aq)}$. In the first 3 months, the loss in strength was more and subsequently contin-
ued however, at a reduced rate as the ages of the mortar specimens increased up until the 9th month. This could be as a result of the formation of soluble nitrates after interaction with the activation products (C(N)-A-S-H), which leads to the weakening of the POFA-EACC specimens.

For the specimens exposed to HCl, the residual compressive strength was more in comparison to those exposed to HNO3 solution as shown in Fig. 4-7. However, the HCl solution also has the capacity to dissolve the alkali-activated binder and leach out some materials from within the POFA-EACC mortar specimens. The EDS microanalysis results (see Fig. 13) revealed the absence of the major elements (Ca, Na and Al) of the alkali-activated binder framework. This is due to the leaching action of HCl solution on these materials. HCl have been reported (Klinowski 1984) to be used in the treatment of Zeolites rich in silicate to remove aluminium from its framework. This could be an evidence in the ability of HCl solution to remove alumina from the POFA alkali-activated microstructure, a major part of alkali activation product responsible for improving the mechanical properties. This is possible due to the corrosive reaction that occurred during exposure where some ions (Ca, Na, Al, K) within the POFA-EACC mortar framework were exchanged with the hydrogen ions in the acid solution. This allows for a direct attack on the Si-O-Al bond, which ultimately impedes the creation of much of Si-OH and Al-OH in the HCl solution reducing the mass and weakening the strength of the POFA-EACC mortar specimen.

### 3.2 Variation in pH of acid solution after exposure

The immersion of POFA-EACC mortar specimens into the different acid solutions (H2SO4, HNO3 and HCl) as expected caused an imbalance in the pH of the acid solutions. The difference in the level of pH in all the acids at different exposure periods are plotted in Fig. 8. Before exposing the POFA-EACC mortar specimens to the acid solutions, the initial pH of the solutions were 0.42, 0.52 and 0.56 for 10% HCl, 10% HNO3 and 10% H2SO4 respectively. To study the early period leaching effect of the acids, the mortar specimens were exposed for 6 weeks and the pH obtained were between 1.8 and 2.88. The trend recorded in the pH plots is similar irrespective of the acid the specimens are exposed to, which increased with increase in the days of exposure. The changes in the pH of the acid solution from being acidic (pH~0.5) to being relatively less acidic (pH~2.5) is as a result of movement of alkali products of alkali-activated POFA-EACC mortar into the exposed solution. The H2SO4 solution amongst the three exposure solutions records the least rate of pH increase in all molarities of NaOH(aq) and at any time in the exposure period of the POFA-EACC mortar specimens. This is an indication that the most alkali products as a result of exposure to the H2SO4 were not leached out of the mortar specimens due to low solubility of these compounds. The pH values of HCl and HNO3 are close in their rates of increase. Most of nitrate and chloride compounds formed are soluble, hence found their way into the solutions and increased their pH.

### 3.3 Percentage loss in weight of exposed specimens to acid solutions

The percentage loss in weight of POFA-EACC mortar specimens after exposure to the three acid solutions; 10% HCl, 10% HNO3 and 10% H2SO4 is shown in Fig. 9. In all the NaOH(aq) molarities, the mortar specimens exposed to H2SO4 solution showed the least level of weight loss while those exposed to HCl and HNO3 solution showed more weight loss as revealed in Fig. 9. Largely, there are consistent changes in weight in all the exposed mortar specimens except some fluctuations in the 9-month result experienced with the 10M NaOH(aq) exposed to HNO3, which obviously might be due to
measurement error. It is important to note that while the percentage loss in weight for mortar specimens exposed to H$_2$SO$_4$ solution decreased with increase in NaOH$_{aq}$ molarity, with HNO$_3$ and HCl solutions, the weight loss increased with increase in NaOH$_{aq}$ molarity. The increase in weight of specimens exposed to H$_2$SO$_4$ solution is due to the filling of the pore spaces by CaSO$_4$ formed from the reaction between the alkali activation products (Ca$^{2+}$ from C(N)-A-S-H) and the H$_2$SO$_4$ solution. This offers additional weight to the mortar specimens, which normally contain empty pores because of evaporation of water from the reaction product. However, the percentage loss in weight of the POFA-EACC specimens exposed to HCl and HNO$_3$ solutions is due to the movement of corrosion products formed from acid exposure into the solution leaving behind internal cavities. The solubility of the products from exposure plays a major role in reducing the weight of the exposed POFA-EACC mortar specimens.

3.4 Visual appearance of the exposed specimens

Figure 10 shows the appearance of POFA-EACC mortar specimens after 9-month exposure to the different acid solutions. From visual observation, the appearance

Fig. 8 Changes in pH values for HCl, HNO$_3$ and H$_2$SO$_4$ solution after specimens’ exposure.

Fig. 9 Percentage change in weight of POFA-EACC mortar specimens exposed to acid solutions.

Fig. 10 Surface change of POFA-EACC mortar specimens to acid solutions exposure.
of the mortar specimens revealed that of partial surficial degradation. Inasmuch as all the mortar specimens undergo some surface deterioration the level of degradation is dependent on the different acid solutions the mortar specimens are exposed to. The very slight semblance to pumice surfaces of all the POFA-EACC mortar specimens reveal some surface leaching, which might be due to dissolution of some of the products of alkali activated binder. As seen from Fig. 10, the H$_2$SO$_4$ exposed specimen were the least affected and the effects on the HCl and HNO$_3$ exposed specimens were more. In addition, the colors of the specimens after exposure were slightly affected as those exposed to H$_2$SO$_4$ solution looks abit darker in grey than those exposed to the HCl and HNO$_3$ solutions with lighter grey.

3.5 SEM/EDS Analysis
3.5.1 Microstructure of the unexposed (control) POFA-EACC mortar specimens
Samples for microstructural and elemental analysis of the POFA-EACC mortar were taken from some mm into the surface of the exposed and unexposed specimens. Scanning electron microscopy (SEM) monographs and Energy Dispersive Spectroscopy (EDS) spectra are shown in Fig. 10 for the control mortar specimens (unexposed to acid solutions) with respect to their NaOH$_{(aq)}$ molarities. The good bonding between the PVA fibres and POFA binder matrix was seen through the revelation of a well formed homogenous and uniform microstructure as shown in the monographs. This is a result of a strong interaction of the PVA fibres with polar solvents (water in this case) through the existence of hydrogen and hydroxyl bonding from water and alkali-dominating internal structure of POFA binder (Salami et al. 2016). Relatively low compressive strength was recorded, which increases with increase in NaOH$_{(aq)}$ molarity. This is buttressed further with the SEM monographs where the denseness increases with increase in NaOH$_{(aq)}$ molarity as revealed in Fig. 11. As shown from the EDS also, the Si/Al ratio is high which have

![Fig. 11 SEM/EDS of POFA-EACC mortar specimens with different NaOH$_{(aq)}$ molarities unexposed to acid solutions.](image-url)
been reported (Duxson et al., 2005, 2007; Silva et al., 2007) to be responsible for low compressive strength recorded with POFA binder. The SiO$_3$:Al$_2$O$_3$ ratio plays crucial role in the outcome of the compressive strength after alkaline activation of POFA. It is largely responsible for the mechanical properties, which is used to determine the durability characteristics of the POFA-EACC mortar specimens.

3.5.2 Microstructural effect of H$_2$SO$_4$ on the exposed POFA-EACC mortar specimens

Figure 12 shows the SEM micrographs and EDS spectra respectively for POFA-EACC mortar specimens exposed to H$_2$SO$_4$ solution. The SEM micrograph revealed a better internal structure compared to the controlled specimen with the presence of some crystalline particles, which increased with the NaOH molarity. The crystalline particles are actually gypsum in the POFA-EACC mortar specimens as revealed in the EDS spectra with the presence of calcium, sulfur and oxygen. The presence of gypsum explains further the reason the H$_2$SO$_4$ exposed mortar specimens recorded the highest residual strength in all exposures.

3.5.3 Microstructural effect of HCl on the exposed POFA-EACC mortar specimens

After 9-month of POFA-EACC mortar specimen exposure to HCl solution, observable differences were found in the microstructure (Fig. 13). It revealed through the SEM, a more porous microstructure and in the EDS, there were absence of the major element; Al, Ca and Na. There is high possibility of the reaction between the hydrochloric acid and the calcium compounds as shown in (Eq. 1) to form a highly soluble calcium chloride, which explains its depletion from the specimens as revealed by EDS.

\[
\text{Ca}^{2+} (s) + \text{HCl} (aq) \rightarrow \text{CaCl}_2 (aq) + \text{H}^+ (l)
\] (1)

This shows the high capacity of the acid to dissolve the microstructural component of POFA-EACC mortar
specimen responsible for its strength build-up. This also explains why low residual strength was recorded after exposure to hydrochloric acid solution.

3.5.4 Microstructural effect of HNO₃ on the exposed POFA-EACC mortar specimens
The SEM/EDS micrograph of POFA-EACC mortar specimens exposed to nitric acid is as shown in Fig. 14. The SEM reveal some microstructural shortfalls as a result of the nitric acid attack on the products responsible for its mechanical strength. The mortar matrix as revealed looks similar with internal porosity of the microstructure as observed in the HCl exposed mortar specimens. The microanalysis also revealed the absence of Na and Ca, which implies that some reaction had taken place between the alkali activated products (C(N)-A-S-H) and the $\text{NO}_3^-$ ion from the exposure acid. This reaction led to the soluble nitrates that are dissolved into the exposure acid leaving behind empty pore spaces in the matrix of the POFA-EACC mortar specimens.

3.6 X-Ray diffraction (XRD) analysis
Figure 15 reveals the XRD diffractograms of POFA-EACC mortar specimens prepared with different NaOH molarities unexposed to the different acid solutions. The diffractogram shows the different phases formed as a result of alkali activation synthesis of the POFA binder, which is characterized by low intensity diffusion halo of amorphous material between 25 and 20 2-theta. The major identified crystalline phases are quartz, cristobalite, NASH, jadeite, stishovite and amorphous silica. The dominant peaks are found at approximately 22, 26, 51 and 68 two-theta (°) and others weak peaks are found at 29, 37, 39, 55 and 61 two-theta (°). The presence of these dominant phases have been reported (Kumar et al. 2010) to fortify the microstructure of alkali activated binder.

The XRD diffractogram of POFA-EACC mortar specimens exposed to different acid solutions for 10, 12 and 14M NaOH is shown in Figs. 16-18 respectively. In comparison with the unexposed specimens, newly
formed phases were observed most especially with H$_2$SO$_4$ exposed specimen, which is as a result of the interaction of the mortar specimens and the acid solutions. The case of H$_2$SO$_4$ was different because of the formation of CaSO$_4$.2H$_2$O (gypsum) whose relative insolvibility allow it be deposited inside the mortar specimens. In addition is the formation of basanite (Ca$_3$(SO$_4$)(H$_2$O)$_{1.8}$, a stable crystalline precursor phase

Fig. 14 SEM/EDS of POFA-EACC mortar specimens with different NaOH(aq) molarities exposed to HNO$_3$ solutions.

Fig. 15 XRD diffractograms of specimens unexposed to sulfate solutions (Control).
during calcium sulfate formation (Van-Driessche et al., 2012). The dual effect of gypsum and basanite on the microstructure strengthens it through pore filling ability because their relative insolubility prevent their leach-out from the mortar specimen. Some phases like cristobalite, stishovite, quartz and amorphous silica remained unchanged after exposure which offers the residual strength after exposure, however new phases like coesite, gypsum and basanite were formed after the acid exposure added to the mortar strength.

Fig. 16 XRD diffractograms of 10M NaOH specimens exposed to acid solutions (H$_2$SO$_4$(aq), HNO$_3$(aq) and HCl(aq)).

Fig. 17 XRD diffractograms of 12M NaOH specimens exposed to acid solutions (H$_2$SO$_4$(aq), HNO$_3$(aq) and HCl(aq)).

Fig. 18 XRD diffractograms of 14M NaOH specimens exposed to acid solutions (H$_2$SO$_4$(aq), HNO$_3$(aq) and HCl(aq)).
In Figs. 15-18, comparing the XRD diffractogram of HCl exposed POFA-EACC mortar specimens to the unexposed diffractogram for all the molarities of NaOH solution, it revealed that most of the activation products were lost to the acid exposure. In addition, no new phases are formed due to the detrimental effect of leaching the acid solutions had on the products of alkalai-activation of POFA material. Some of the earlier formed products like stishovite, jadeite and NASH were lost to the acid exposure leaving behind in the POFA-EACC specimens materials like quartz resistant to the acid solution (Allahverdi and Skvara 2001a, 2001b) and cristobalite. Some of the products of activation also were left behind especially in the core.

The POFA-EACC mortar specimens exposed to HNO₃ also suffered some deterioration, as revealed in the XRD diffractogram in Figs. 15-18. Similar situation recorded with HCl specimens was observed in the POFA-EACC mortar specimens exposed to HNO₃ solution. The products of activation were also absent due to the dissolution of these alkali-activated materials into the exposure solution leaving behind resistant phases like quartz.

3.7 Fourier Transmission Infrared (FTIR) analysis

Figures 19-21 reveal the FTIR transmittance spectra (400-4000 cm⁻¹) of unexposed (control) and exposed POFA-EACC specimens to acid (HCl, HNO₃ and H₂SO₄) solutions. There is a distinct difference between the FTIR spectra of the unexposed and exposed specimens to the acids. For the unexposed specimens, in all molarities of NaOH, the transmittance signals were approximately same. Seven major adsorption bands can be identified in the FTIR transmittance spectra of the unexposed POFA-EACC specimens to different acids namely; 3460 cm⁻¹, 1650 cm⁻¹, 1450 cm⁻¹, 1025 cm⁻¹, 800 cm⁻¹, 465 cm⁻¹ and 435 cm⁻¹. The broad absorption intensity distributed between 2500 cm⁻¹-3700 cm⁻¹ corresponds to stretching vibration of hydroxyl (OH⁻) and bending vibration of H-O-H at approximately 1650 cm⁻¹. Asymmetric stretching vibration assigned to bands between 1430-1450 cm⁻¹ indicates some carbonation of the POFA-EACC specimens due to the presence of environmental CO₂. The vibration bands at approximately 875 cm⁻¹ and 1025 cm⁻¹ is the feature of asymmetric stretching vibration of Si-O in the silicate tetrahedrons. A sharp signal appearing at 472 cm⁻¹ is assigned to symmetric stretching vibration of Si-O-Si while between 690-800 cm⁻¹ bands correspond to asymmetric...
stretching vibration of Al-O bonds of AlO₄. Another very sharp signal appears at 460-480 cm⁻¹, which corresponds to the stretching vibrations of the Si-O-Si.

As can be seen from the transmittance spectra, there were some differences in the transitions, which obviously is an indication of change in the absorption rate of the infrared of the POFA-EACC due to exposure to the different acids. For the specimens exposed to HCl acid solutions, the two peaks for water component and chemically bonded water with 3460 cm⁻¹, shifted to between 3440-3455 cm⁻¹ and 1650 cm⁻¹ to 1638 cm⁻¹ respectively. This is an indication of the presence of chemically bonded water and free water in the microstructure. Another major change experienced was the absence of the CO₂ band present in the unexposed specimens at 875 cm⁻¹ and 1430-1450 cm⁻¹. This is expected, as there was no reaction between HCl acid solution and CO₂ being an acidic oxide. After exposure to HCl acid, the main vibration fingerprints with peaks at 980-1040 cm⁻¹, 680-800 cm⁻¹ and 450-470 cm⁻¹ attributed to the alkali-activated materials according to previous studies were not affected.

The same bands observed in HCl exposed specimens were also observed in the H2SO₄ and HNO₃ exposed specimens except for the sharp intensity band at 1384 cm⁻¹ that showed the presence of O-C-O stretching in HNO₃ specimens for all molarities of NaOH. Generally, there is a shift between 800-1000 cm⁻¹, which despite the shift lies within the asymmetric stretching vibration of Si-O or Al-O. No shift was observed between the bands 470-1000 cm⁻¹ largely maintaining the alkali-activated binder attributes. There is also appearance of a band 435 cm⁻¹ after exposure to all acids.

4. Conclusions

The paper reports the durability performance of the developed POFA-EACC mortar subjected to different acid solutions namely, 10% H₂SO₄, 10% HNO₃ and 10% HCl solutions. Compressive strength, weight loss and visual appearance before and after exposure to the acid solutions were the metrics used on the study. The following conclusions can be drawn from the data garnered from the research work:

The compressive strength of unexposed POFA-EACC mortar specimens are all above 25MPa despite low alumina content. The strength increased with NaOH molarity increase with NH₁₂ increased by 8% more than NH₁₀ and NH₁₄ by 4.1% more than NH₁₂. After exposure to acid solutions, two trends were recorded depending on the type of acid solution and NaOH molarity. For H₂SO₄, the percentage loss in compressive strength decreases with increase in NaOH molarity. The strength of H₂SO₄ specimens improved after 6-month exposure reaching a maximum percentage loss of 19.5% with 10M POFA-EACC mortar specimens. The second trend is, for HCl and HNO₃, the percentage loss in compressive strength increases with increase in NaOH molarity. The strength of HCl and HNO₃ specimens did not improve after 6-month of exposure until a maximum percentage loss of 63.5% and 70% were reached respectively for HCl and HNO₃ mortar specimens.

The different acids affected the alkalities of the POFA-EACC mortar specimens differently. There was a reduction in the mortar specimens’ alkalinity as shown or correlated with the pH increase of the different acid solutions. The H₂SO₄ solution recorded the least rate of pH increase in all NaOH molarities amongst the three acid solutions, which is an indication of slow release of alkali-activated products from within the POFA-EACC mortar specimens. The slow release of the alkali-activated products was aided by the formation of CaSO₄, an insoluble product that acted as barrier or shield in the way of the acid solution. This prevented the movement of the H₂SO₄ solution into the POFA-EACC mortar specimens thus leaching from inside of the mortar was reduced. Comparable pH increase in HCl and HNO₃ solutions was recorder, however were higher than the pH of H₂SO₄ solution. This perhaps was due to the formation of more soluble products from the acid reaction leaching out of the mortar specimens.

The POFA-EACC mortar specimen weight loss is de-
pended on both molarity of NaOH and the exposure acid solutions. For HCl and HNO₃, the weight loss increases with increase in the NaOH molarity due to more alkali activated products dissolved into the acid solution since the products increase with increase in NaOH molarity. However, for H₂SO₄, the weight loss decreases as the NaOH molarity increase. This is probably due to more products due to acid exposure, which is directly proportional to the products of the alkali-activated POFA products.

Acknowledgement
The authors gratefully acknowledge the Universiti Sains Malaysia for providing the needed support including financial support through the Research University (1001/PAWAM/814191) Grant Scheme for undertaking of the research work. The supports from Center of Engineering Research (CER) of Research Institute, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia are specially acknowledged. Thanks are also due to United Palm Oil Industries for providing the palm oil fuel ash.

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