Engineering and Applied Science Research 2022;49(6):731-743 Research Article

Engineering and Applied Science Research

https://www.tci-thaijo.org/index.php/easr/index

Published by the Faculty of Engineering, Khon Kaen University, Thailand

Strength and microstructural of geopolymer mortar from palm oil ash containing alumina powder with palm oil clinker aggregate

Abideng Hawa*

Infrastructure and Materials Innovation Research Unit, Department of Civil Engineering, Princess of Naradhiwas University, Narathiwat 96000, Thailand

Received 27 July 2022
Revised 15 September 2022
Accepted 10 October 2022

Abstract

This paper presents geopolymer mortars from palm oil ash (POA) containing alumina powder (AP) with palm oil clinker (POC) for fine aggregate. Different AP contents in the geopolymers were investigated along with binder (BD) to alkali activator (AK) ratio, BD to ordinary Portland cement (OPC) ratio, curing conditions and heat curing time. The geopolymer samples were studied for compressive strength, bulk density and microstructure. A combination of sodium hydroxide and sodium silicate was used as an activator. The microstructure was analyzed by scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray fluorescence (XRF) to detect changes in the geopolymerization process. Results showed that addition of AP increased the compressive strength of the geopolymer binders. At 28 days, compressive strength of up to 18.99 MPa was achieved in samples cured at 80 °C for 24 h, with BD to POC ratio of 1.4 and BD to AK ratio of 1.44 with 5% AP. SEM results showed that the 5% AP samples had a dense compact matrix with higher compressive strength. The POA based geopolymer containing AP with POC as fine aggregate had bulk density ranging from 1,004 to 1,911 kg/m^3 at 28 days.

Keywords: Geopolymer, Palm oil ash, Alumina powder, Palm oil clinker, Microstructure

1. Introduction

Geopolymers can be used to replace ordinary Portland cement (OPC) as construction materials in buildings and infrastructures. Using geopolymers allows elimination of OPC as a binder (BD), and replacement with products derived from natural resources, thereby reducing the carbon dioxide (CO_2) contribution to global warming by the cement industry. Geopolymers are made using alkali activators and pozzolanic materials in powder form that behave similarly to OPC. Previous investigations have shown that geopolymers exhibit similar or higher strength and durability compared to OPC [1]. Good geopolymer synthesis relies on the use of silicon (Si) and aluminum (Al) for the geopolymerization reaction. Copious studies have examined the properties of geopolymers prepared from fly ash (FA) [2, 3], metakaolin (MK) [4, 5] and granulated blast furnace slag [6, 7]. These raw materials contain silica (SiO_2) and alumina (Al_2O_3) as part of their main chemical compositions. Geopolymers have also been prepared from pumice powder [8] and volcanic ash [9] that contain SiO_2 and Al_2O_3, while industrial waste pozzolanic materials such as palm oil ash (POA), rice husk ash, bagasse ash and wood ash can also be used.

The potential of POA, especially treated, as a source material for geopolymer synthesis has not been adequately explored, along with FA, MK or calcined kaolin. Nowadays, increased interest has been shown in POA, especially by researchers from palm oil producing countries. To the best of our knowledge, geopolymerization using POA has not been previously reported. POA contains high amounts of silica but low quantities of alumina. Recent research has shown that inclusion of specific amounts of aluminum hydroxide ($Al(OH)_3$) [10] and calcium hydroxide ($Ca(OH)_2$) [11] enhanced the geopolymerization process. Parameters that affect POA geopolymer synthesis include alkali activator types (combination and concentration). Previous research concluded that combining Na_2SiO_3/NaOH at weight ratios of 1.0 and 2.5 produced high strength geopolymers [12, 13].

Geopolymer synthesis requires an alkali silicate material and alkali hydroxide plus water to mediate the reaction and increase workability. The availability of silica- and alumina-rich FA or MK clears the way for the use of these base materials as geopolymer binders. However, POA is mainly comprised of silica (SiO_2) with low alumina (Al_2O_3) content. Some research has been conducted on the use of POA and FA in geopolymer mixtures [14, 15], with results showing that partial replacement by POA increased compressive strength but mixing low alumina materials such as rice husk ash and POA in geopolymer systems has received scant attention. Research by [16] prepared geopolymer paste from POA with curing at 60 °C, 70 °C, 80 °C and ambient temperature. Results indicated that heat increased compressive strength in the early stages at a high rate. Increasing the curing temperature from 60 °C to 80 °C did not affect the compressive strength. POA activation resulted in compressive strength of 29.1 MPa at 28 days. Salih et al. [17] investigated geopolymer pastes prepared using solid to liquid and Na_2SiO_3 to NaOH ratios of 1.32 and 2.5, respectively. At 28 days, POA geopolymer paste had compressive strength of 32 MPa. Geopolymer mortars can be made using palm oil clinker (POC) by replacement of conventional sand as lower bulk density samples. Geopolymer mortars mixed with POC were compared to mixtures using conventional sand. Results gave bulk densities ranging from 1,710 to 1,754 kg/m^3 for different molarities of 8 to 16 [18].

---

*Corresponding author. Tel.: +88 7285 5328
Email address: abideng.hawa@gmail.com
doi: 10.14456/easr.2022.71

Palm oil ash (POA) and palm oil clinker (POC) are waste materials from the oil palm industry and accumulate as biomass at power plants in Southeast Asia. The southern region of Thailand is home to the highest level of palm oil manufacturing. Normally, POA and POC wastes are sent to landfills, resulting in an environmental problem, with more than 280,000 tons of waste produced since 2011 [19]. POA is classified as a pozzolanic material and can be used as a replacement for Portland cement in normal concrete for the development of compressive strength and durability [20]. Consequently, POA has been used as a partial replacement material in fly ash geopolymers [21, 22] and metakaolin geopolymers [23, 24] to develop compressive strength, while other by-products of oil palm production and biomass from power plants such as POC have been used as aggregates in geopolymer mixtures [18, 25].

In this study, POA was used as the main binder in geopolymer mortars and combined with alumina powder (AP) at 0%, 5%, 10% and 15% to develop the strength of geopolymer mortars. The samples used binder (BD) to alkali activator (AK) ratios and BD to POC ratios at 0.96, 104, and 1.44 and 0.2, 0.4, 0.6, 1.4 and 2.4, respectively. The geopolymers were cured at 80 °C for 24 h and 12 h and at ambient temperature for 24 h. Compressive strength of the samples was tested to determine the effect of AP and BD to POC ratio, BD to AK ratio, curing temperature and curing time. Analytical methods used to investigate the microstructure of POA-based geopolymers included scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray fluorescence (XRF). The geopolymers in this study were produced by mixing POA and AP with alkaline-activated aluminosilicate based on sodium silicate and sodium hydroxide, and POC was used for fine aggregate.

2. Materials and methods

2.1 Materials

2.1.1 Palm oil ash

Palm oil ash (POA) is a biomass by-product of power plants that burn palm kernel shells and palm mesocarp as fuel. The POA was collected from local power plants in Narathiwat Province, Southern Thailand. Incompletely combusted fibers and palm kernel shells were sieved (No. 8) after drying in an oven for 24 h at 105 °C. The POA was ground for 5 h using a Los Angeles abrasion machine in 5 kg batches. Particle size distribution of POA after grinding is shown in Figure 1, with particle morphology shown in Figure 2.

Chemical and mineral compositions of POA are shown in Table 1 and Figure 3, respectively. Chemical compositions of POA were analyzed using X-ray fluorescence (XRF). The major oxide was silica (SiO_2) with alumina (Al_2O_3) content 1.06%. Raw materials for geopolymer production must have high alumina content. The X-ray diffraction (XRD) pattern of POA presented in Figure 3 shows mineral phases including quartz.

2.1.2 Alumina powder

Alumina powder (AP), used in this research as an additive to enhance the strength and microstructure of POA-based geopolymer, was synthesized by the geopolymerization reaction. Wherewith, the geopolymerization process must be Al_2O_3 content but the analysis chemical composition of POA that low Al_2O_3 content. Particle size distribution of AP is presented graphically in Figure 4 and visually in Figure 5. A laser diffraction technique was used to analyze particle size distribution. The finely powdered AP had average particle size (d_{50}) of 8.35 μm and d_{90} of 15.2 μm. The XRD patterns presented in Figure 6 show the mineral phases in AP, including oxonium and corundum.

2.1.3 Alkali activator solution

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were used as alkaline activators in the geopolymerization reactions. Chemical compositions of sodium silicate were 14.85 wt% Na_2O, 29.45 wt% SiO_2 and 55.70 wt% H_2O, while sodium hydroxide flakes were 99% pure.

2.1.4 Palm oil clinker

In this study, palm oil clinker (POC) was used as fine aggregate. The POC was collected from local biomass power plants in Narathiwat Province, Southern Thailand at 5-50 mm size (Figure 7). The POC was then crushed and sieved in the laboratory for use as fine aggregate, with particle size distribution shown in Figure 8. Table 2 shows the physical properties of POC as porous and hence lightweight. POC density was in accordance with ASTM C331 [26], with maximum dry loose bulk density of fine aggregate as 1,120 kg/m^3.

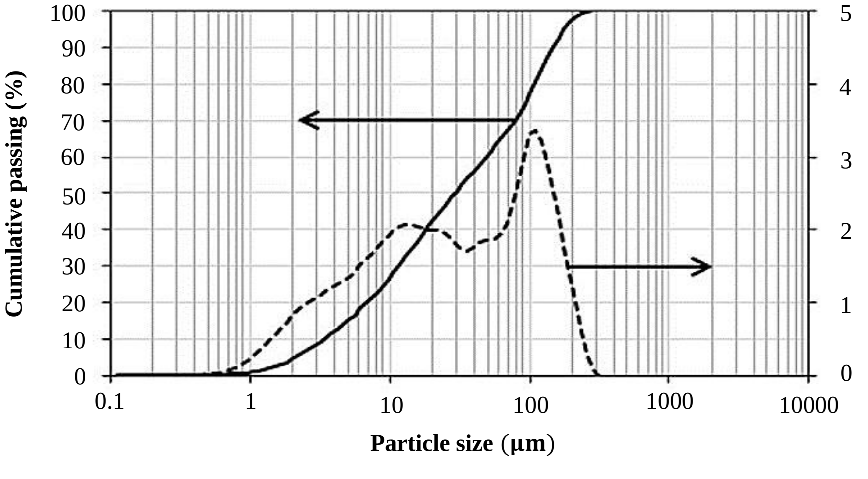
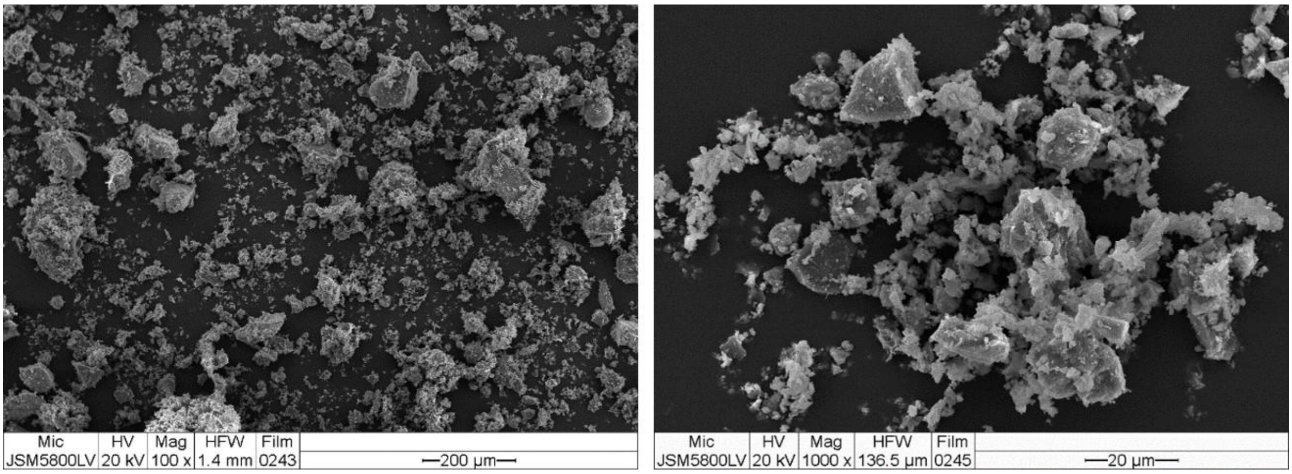


Figure 1 Particle size distribution of POA after grinding

Table 1 Chemical compositions of POA and AP

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	P_2O_5	Cl	MnO	TiO_2
POA	56.84	1.06	2.46	7.74	6.54	8.60	1.92	6.96	0.34	0.21	-
AP	0.11	99.66	-	-	-	-	-	-	-		0.1



(a) (b)

Figure 2 SEM Images of POA (a) 100x and (b) 1,000x

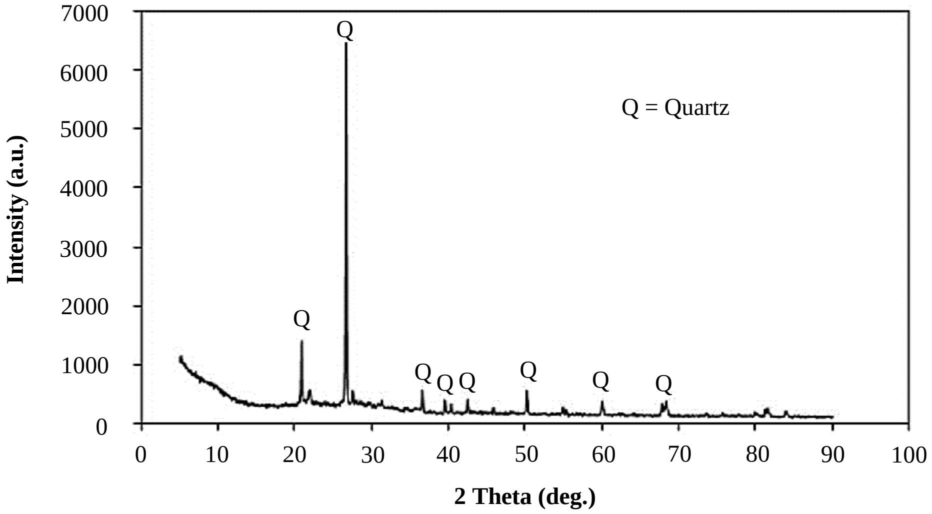


Figure 3 XRD Pattern of POA

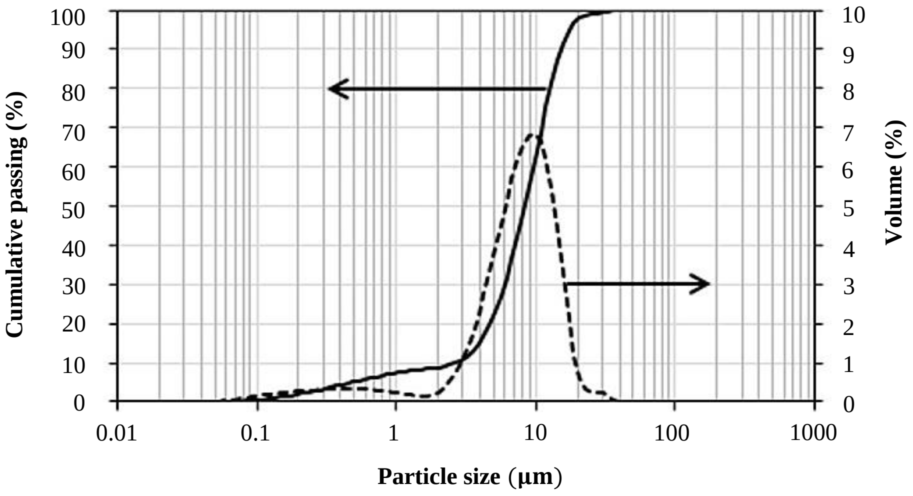
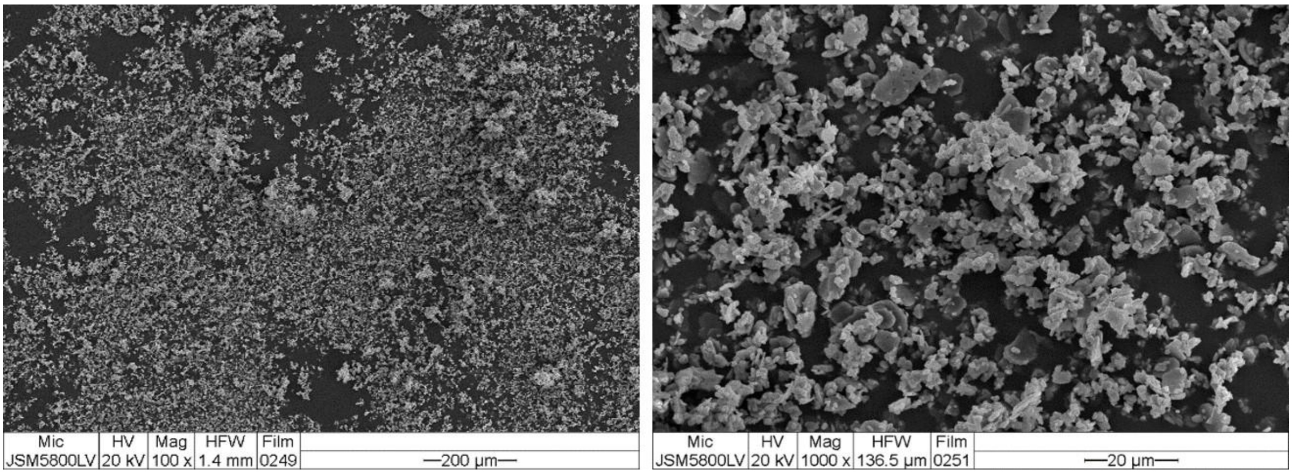


Figure 4 Particle size distribution of AP



(a) (b)

Figure 5 SEM Image of AP (a) 100x and (b) 1,000x

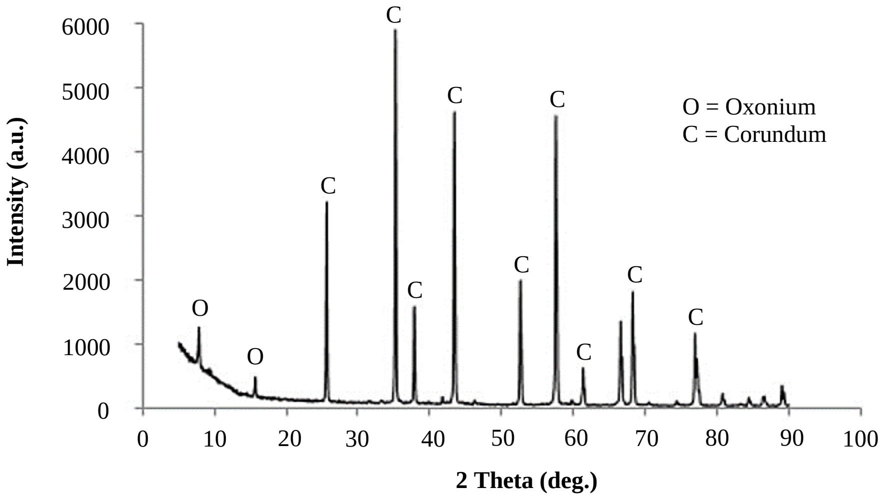


Figure 6 XRD Pattern of AP

Figure 7 Original POC

Table 2 Physical properties of POC

Property	Value
Loose bulk density (kg/m^3)	634
Compact bulk density (kg/m^3)	686
Specific gravity	1.60
Water absorption (%)	4.30

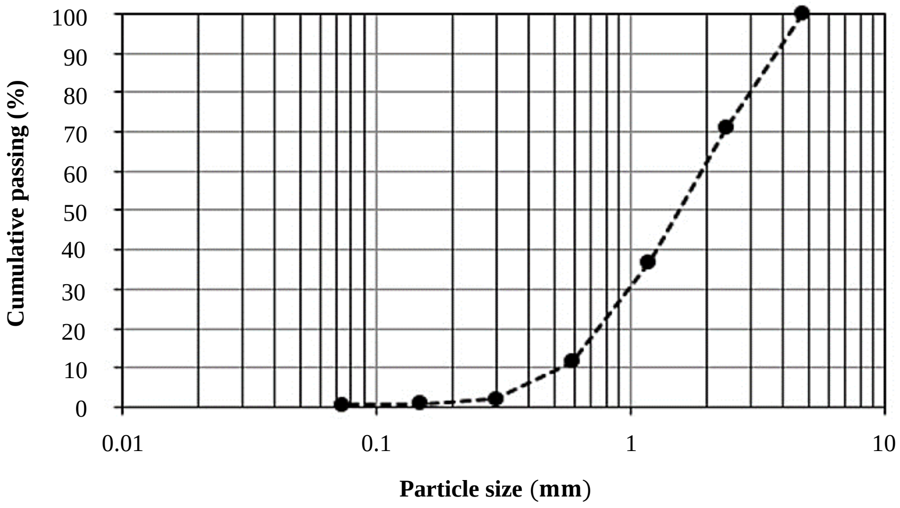


Figure 8 Particle size distribution of POC

2.2 Sample preparation

In this study, 17 mixtures were prepared, as shown in Table 3, containing alkaline activator solution, water, solid materials and aggregate. Five series of geopolymer samples were designed and investigated for ratios of POA to AP, BD to AK, BD to POC, temperature and curing time. Sodium hydroxide flakes were mixed with sodium silicate liquid by weight proportion of 1:2.5. The geopolymer mortar was mixed in three steps. First, BD (POA + AP) and POC were mixed by hand for about 3 minutes. Second, NaOH, Na_2SiO_3 and water (H_2O) were mixed into a homogeneous alkaline solution. Finally, the alkaline solution was added to BD and POC from the first step and mixed for about 5 minutes. The alkaline activator in the first step had an exothermic reaction conducive to fast setting [24, 27]. All samples were mixed manually. After mixing, the geopolymer slurries were poured into acrylic 50 mm cube molds to set and form samples for compressive strength testing and compacted in accordance with ASTM C109/C109M [28]. The geopolymer mortar samples in the acrylic molds were wrapped with polyvinyl sheets to prevent loss of moisture. The samples were cured in an electric oven at 80 °C and at room temperature after casting. After heat curing under different conditions, the samples were removed from the oven, demolded and stored at ambient temperature of 30±2 °C until testing. The geopolymer paste was investigated for microstructure, phases and chemical composition of the geopolymer matrix. Samples of geopolymer pastes were prepared with diameter 30 mm and thickness 10 mm and mixed and cured with the sample containing AP. After heat curing of different conditions, the geopolymer pastes were stored at ambient temperature for 28 days until testing.

Table 3 Mixture proportions of mortar samples (kg/m^3)

Symbol	POA (kg)	AP (kg)	AK (kg)	POC (kg)	BD/POC	BD/AK	Water (kg)	Temp. (◦C)	Curing (h)
CT	897	0	934	373.4	2.4	0.96	89.7	80	24
A1	852.2	44.9	934	373.4	2.4	0.96	89.7	80	24
A2	807.3	89.7	934	373.4	2.4	0.96	89.7	80	24
A3	762.5	134.6	934	373.4	2.4	0.96	89.7	80	24
B1	852.2	44.9	846	373.4	2.4	1.06	89.7	80	24
B2	852.2	44.9	640	373.4	2.4	1.44	89.7	80	24
C1	852.2	44.9	640	4400	0.2	1.44	89.7	80	24
C2	852.2	44.9	640	2400	0.4	1.44	89.7	80	24
C3	852.2	44.9	640	1500	0.6	1.44	89.7	80	24
C4	852.2	44.9	640	640	1.4	1.44	89.7	80	24
D1	852.2	44.9	640	2400	0.4	1.44	89.7	RT	24
D2	852.2	44.9	640	640	1.4	1.44	89.7	RT	24
D3	852.2	44.9	640	373.4	2.4	1.44	89.7	RT	24
E1	852.2	44.9	640	4400	0.2	1.44	89.7	80	12
E2	852.2	44.9	640	2400	0.4	1.44	89.7	80	12
E3	852.2	44.9	640	1500	0.6	1.44	89.7	80	12
E4	852.2	44.9	640	640	1.4	1.44	89.7	80	12
E5	852.2	44.9	640	373.4	2.4	1.44	89.7	80	12

CT = control sample; RT = room temperatures; BD = binder; AK = alkali activator

2.3 Characterization techniques

2.3.1 Compressive strength and density

Compressive strength testing was conducted on cast samples with dimensions of 50 cubic mm in accordance with ASTM C109/C109M [28] at 1, 7 and 28 days after demolding and curing at ambient temperature using a universal testing machine for mortar. The density of the geopolymer mortars was calculated by mass to volume at 28 days using the samples of compressive strength.

2.3.2 Microstructure

SEM Image testing was conducted to investigate the raw materials and geopolymerization products of the geopolymer pastes and identify pores in the matrices. To observe microstructure, only geopolymer pastes containing AP at 0-15% by weight and cured at 80 °C were tested as CT, A1, A2 and A3 (Table 3).

2.3.3 Mineral phases

XRD Tests were performed to analyze the crystalline phases of POA, AP and the geopolymer pastes using an X'Pert MPD X-ray Diffractometer at angles from 5° to 90° (2Θ) using the clay and rock 0.4 program. XRD testing identified the distinctive crystalline phases and detected the position and changes of the peaks.

2.3.4 Chemical composition

The XRF tests analyzed the different chemical compositions of POA, AP and geopolymer pastes after addition of AP in the binder.

3. Results and discussion

3.1 Compressive strength

Compressive strengths of geopolymer mortars with different percentages of AP are shown in Figure 9. POA based geopolymer mortars containing AP developed strength in all mixtures, especially the sample with partial replacement of 5% AP. Maximum compressive strength of 4.15 MPa was obtained at 28 days, providing evidence that AP contributed effectively to the high rate of reaction development. High compressive strength was attributed to the SiO_2/Al_2O_3 ratio in this geopolymer system. The geopolymerization system cannot use only the SiO_2 content with the geopolymerization process. POA has high SiO_2 content with low Al_2O_3 (Table 1). The activity of alkali-activated BD (POA+AP) reflected the ability of the powder to transition from the mortar matrix to a hard sample when mixed with the activator, compatible with other geopolymer binders using raw materials with metakaolin [29, 30] and fly ash [31-33]. Islam et al. [34] prepared a geopolymer mortar with 100% POA and achieved maximum compressive strength at 28 days of 18 MPa with curing at 65 °C for 24 h. Lower compressive strength was achieved due to the lower BD to AK ratio.

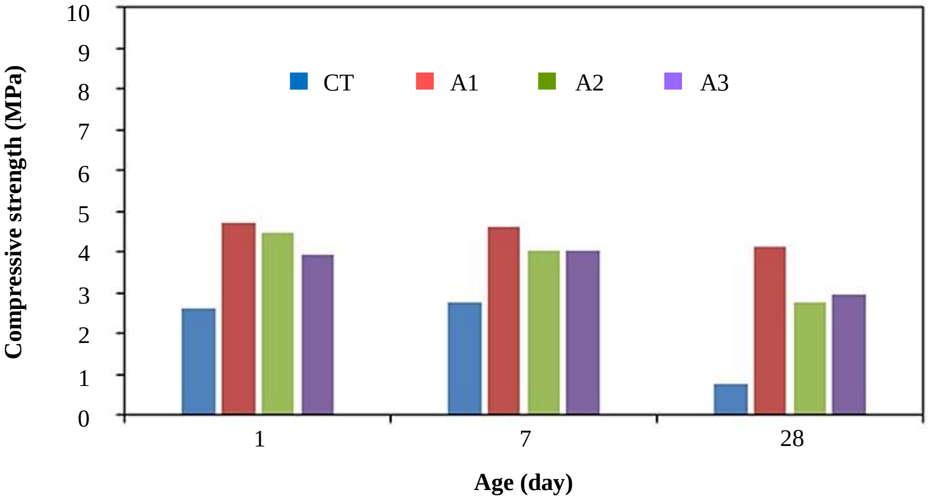


Figure 9 Compressive strength of samples with different AP percentages

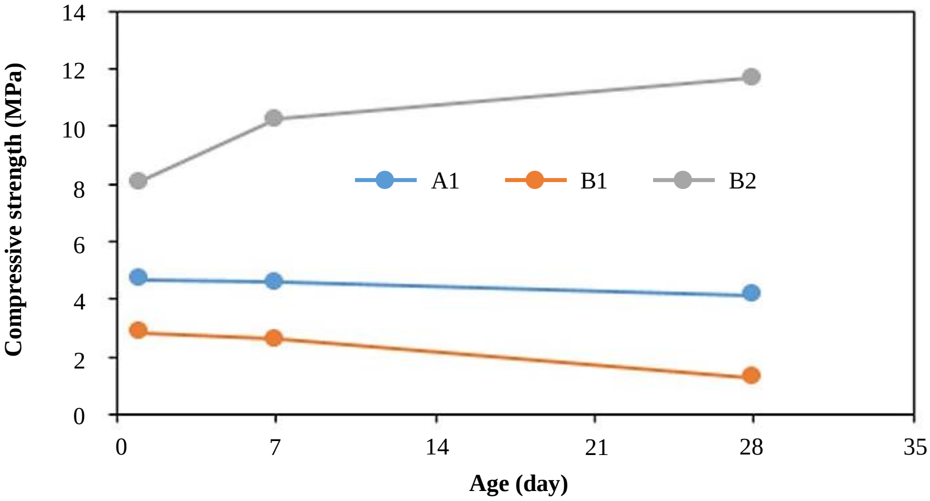


Figure 10 Compressive strength of samples with different BD to AK ratios

Moreover, in this study, the geopolymers were prepared using POC with lower bulk density as low strength. Figure 9 shows the strength of geopolymer samples as lower after 7 days, and lowest strength gain during this period was significant. Compressive strength of the CT sample reduced after curing at ambient temperature for 7 days and a viscous liquid appeared on the surface. Incomplete reaction in the system adversely impacted the strength of the geopolymer mortars.

Figure 10 presents the compressive strength of geopolymer mortars with different BD to AK ratios of 0.96, 1.06 and 1.44 for A1, B1 and B2, respectively. The B2 result shows that compressive strength increased with aging time. After 7 days, compressive strength increased up to the 28th day. For A1 and B1 samples, compressive strength decreased after 7 days. Strength gains up to 7 days were significant due to the mixtures using high alkali activators and low reactivity of geopolymerization.

The POA based geopolymer with a solid to liquid ratio of 1.44 showed the highest compressive strength, while the sample with a solid to liquid ratio of 0.96 presented the lowest compressive strength. Salih et al. [16] used a palm oil fuel ash (POFA) based geopolymer paste with solid to liquid ratio of 1.32, giving compressive strength at 28 days of 29.1 MPa for samples cured at 80 °C for 2 h, while Khal and Chaudhary [35] reported that high solid to liquid ratio of fresh geopolymer was less workable. A solid to liquid ratio of 1.44 was chosen for the next experiment to investigate how curing time impacted POC properties. Fresh geopolymer mixture had low workability, resulting in difficulty in compaction during the molding process, this study has the objective for the application of geopolymer block.

Figure 11 illustrates the compressive strength of geopolymer mortars with different BD to POC ratios at 1, 7 and 28 days. The geopolymer mortars were prepared with BD to POC ratios of 0.2, 0.4, 0.6, 1.4 and 2.4 by weight. The C1 (BD to POC of 0.2) sample had the lowest compressive strength results of 0.23, 0.59 and 0.59 MPa at 1, 7 and 28 days, respectively while the C4 sample had the highest strength results of 20.10, 18.33 and 18.99 MPa at 1, 7 and 28 days, respectively. Increasing increments of BD to POC ratio (low aggregate) reduced compressive strength at curing times. These results concurred with previous literature that identified compressive strength of POFA based geopolymer mortars with manufactured sand for fine aggregate at 18 MPa after 28 days [34]. Saeli et al. [36] reported that compressive strength increased at higher volume because high volume POC with low binder content in the matrix resulted in high porosity and large pores (Figure 12(a)).

Figure 12(a) shows that specimens with low BD to POC ratios had large pores and high porosity, while geopolymer mortars with high BD to POC ratios (Figure 12(b)) had dent-compact and higher homogeneity, with the lowest number and lower size of pores compared to the other samples. This sample also had highest compressive strength of 18.99 MPa after 28 days, suggesting that BD and POC interacted appropriately and inserted in pore structures among the fine aggregate.

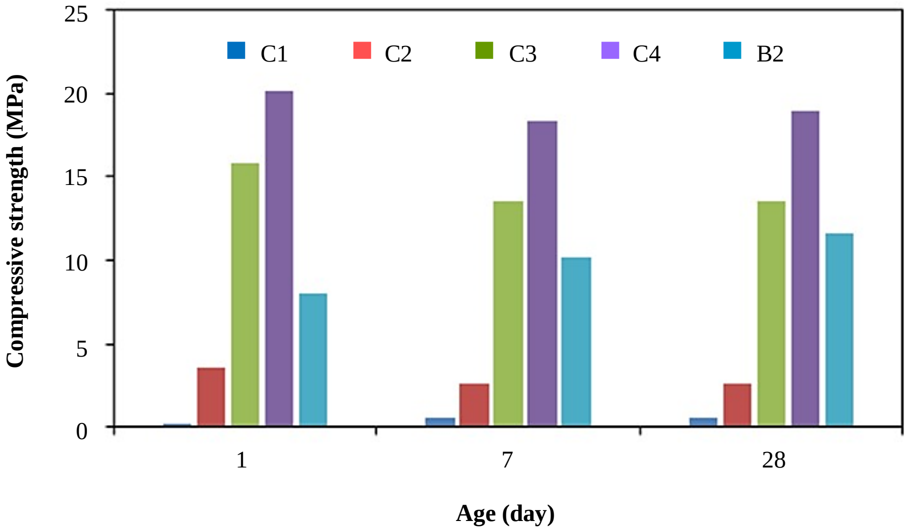
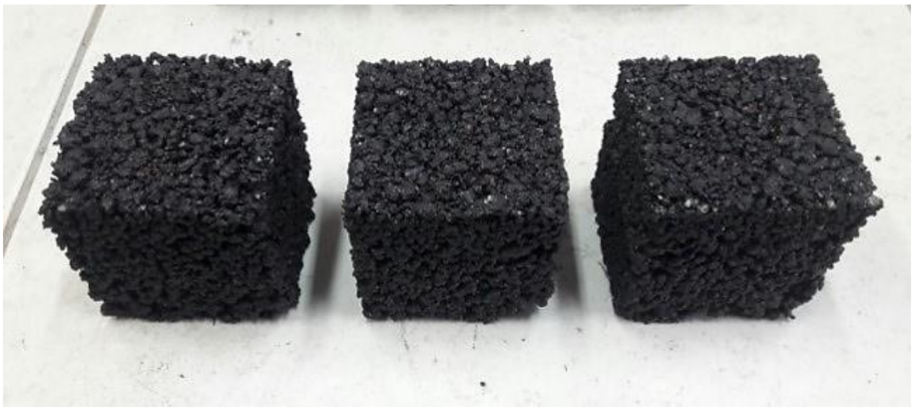
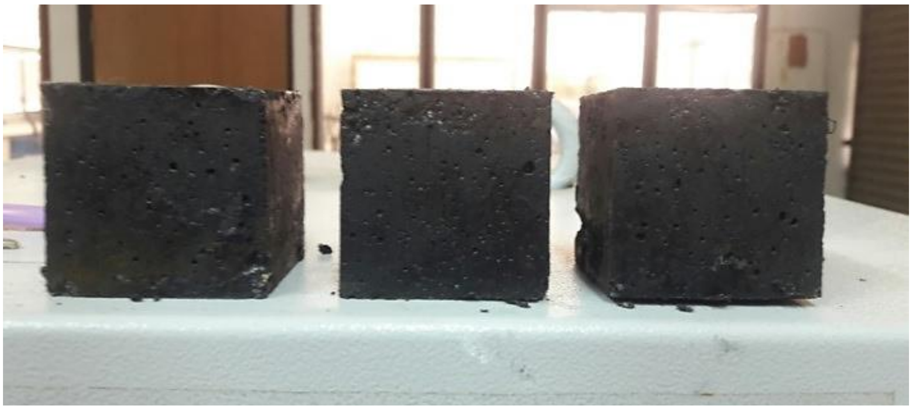


Figure 11 Compressive strength of samples with different BD to POC ratios



(a)

Figure 12 Geopolymer mortar samples after compression testing (a) BD to POC = 0.2 and (b) BD to POC = 1.4



(b)

Figure 12 (Continued) Geopolymer mortar samples after compression testing (a) BD to POC = 0.2 and (b) BD to POC = 1.4

Influences of curing condition and duration on the compressive strength of POA based geopolymer mortars at 1, 7 and 28 days using POC fine aggregate are shown in Figure 13. The samples were heated at 80 °C for 24 h and 12 h, and cured at ambient temperatures for 24 h. Compressive strengths of the geopolymer mortars increased with longer curing time from 12 to 24 h because heat curing formed higher amounts of reaction products. Hawa et al. [24] reported that longer heat curing expedited the degree of geopolymerization reaction because of the formation of mineral phases, while Noushini and Castel [37] found that compressive strength increased at higher curing temperatures and longer curing time from 8 h to 24 h. Geopolymer samples cured at 90 °C showed increased compressive strength from 8 h to 24 h, while samples cured at 60 °C and 75 °C showed increased strength over time. POA geopolymers with POC of fine aggregate cured for 24 h at 80 °C showed highest compressive strengths at 1, 7 and 28 days of 20.10, 18.33 and 18.99 MPa, respectively. Sample curing at ambient temperature (26-33 °C) did not set the geopolymer matrix, even at 28 days because using POA with the binder could not achieve the geopolymerization process.

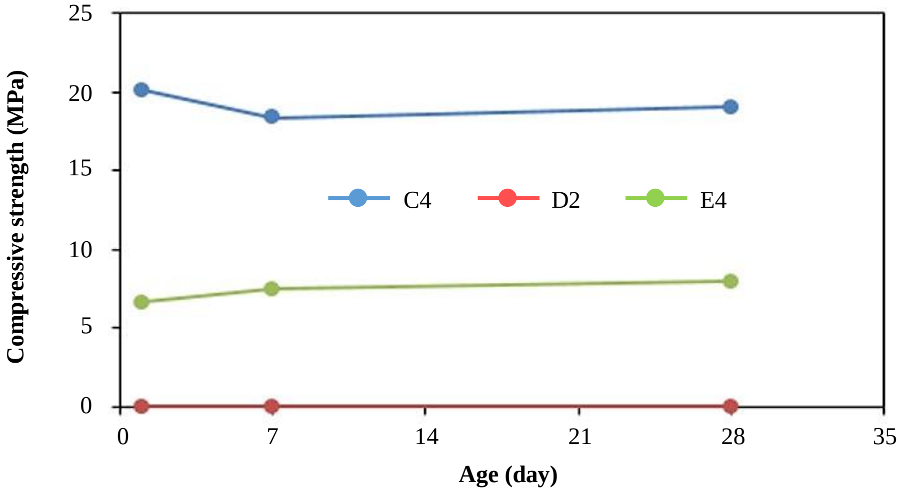


Figure 13 Compressive strength of samples under different curing conditions

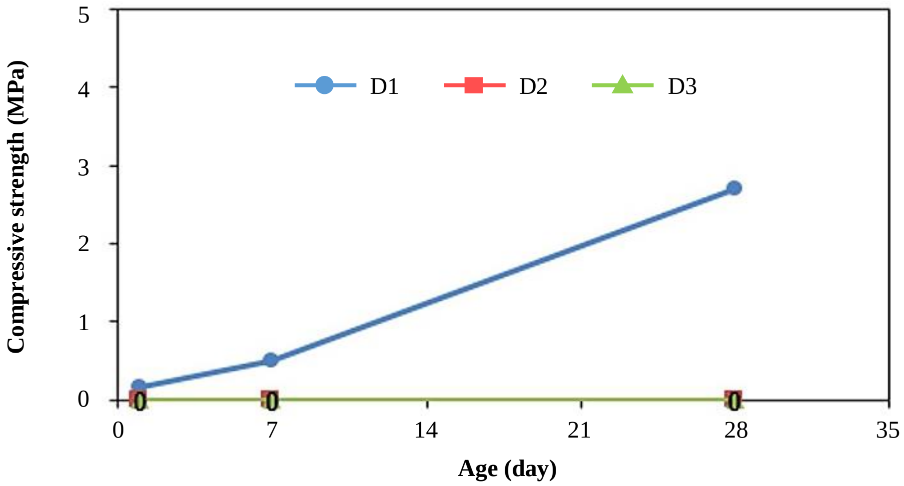


Figure 14 Compressive strength of samples cured at ambient temperature

Compressive strength testing results of geopolymer mortars cured at ambient temperature with different BD to POC ratios of 0.4, 1.4 and 2.4 are shown in Figure 14. Lowest BD to POC ratio (0.4) resulted in the development of strength after long curing of up to 28 days, while samples prepared with higher BD to POC ratios did not experience geopolymerization setting at ambient temperature, despite curing the specimens for 28 days. Strength development in the geopolymer matrix with low BD to POC ratio (low binder) was impacted by the thinness of the geopolymer binder matrix. At low curing temperature, the POC was coated by sparse geopolymer binder. Figure 15 shows POA geopolymer mortars cured at 80 °C for 12 h with different BD to POC ratios. The E3 sample had the highest compressive strength, comparable with the C3 sample cured at 80 °C for 24 h, while the E1 and E2 samples had similar compressive strengths to the C1 and C2 samples. Low powder content decreased compressive strength of samples cured at different temperatures.

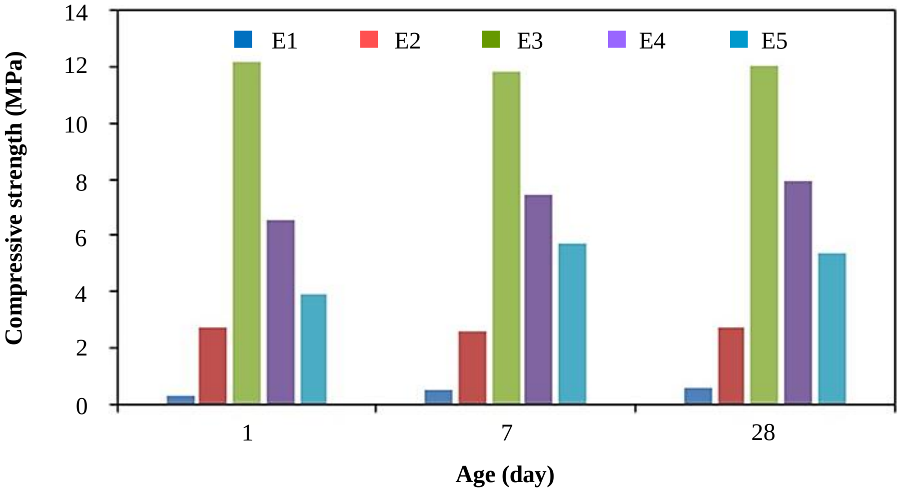


Figure 15 Compressive strength of samples with different BD to POC ratios cured at 80 °C for 12 h

3.2 Bulk density

Bulk densities of POA geopolymer mortars at BD to POC ratios of 0.2, 0.4, 0.6, 1.4 and 2.4 with partial replacement of 5% POA are shown in Figure 16. The BD to POC ratio influenced changes in sample density. Results showed that higher density mortar samples were obtained with increased BD to POC ratio. POC because POC has lower density and specific gravity compared to POA, which has specific gravity of 1.89-2.39 [38, 39]. Curing times of 24 h and 12 h resulted in different bulk densities of the geopolymer samples at 28 days, related to the evaporation of moisture in the geopolymer matrix after long curing at ambient temperature (28 days).

In this study, the POA geopolymer mortars had bulk densities up to 1,864-1,880 kg/m^3 (1.4) at 28 days, higher than geopolymer mortars with POA and fly ash [15] using normal sand. Here, the geopolymer mortars were prepared using fine aggregate OPC which has lower density and specific gravity. Findings concurred with the literature that identified bulk density of geopolymer mortars with palm oil clinker ranging 1,754-1,842 kg/m^3 (different GGBS) [18]. However, in this study, the testing of bulk density not heat for repel moisture in the samples at the oven.

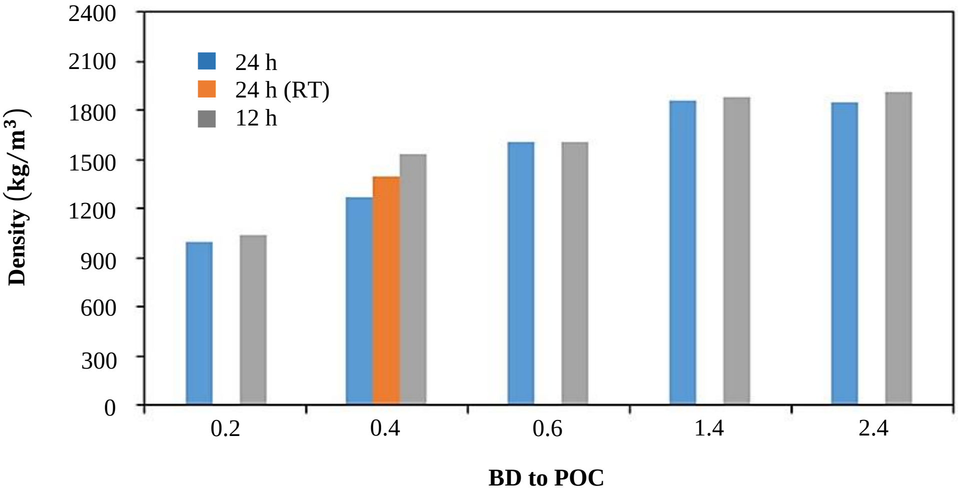
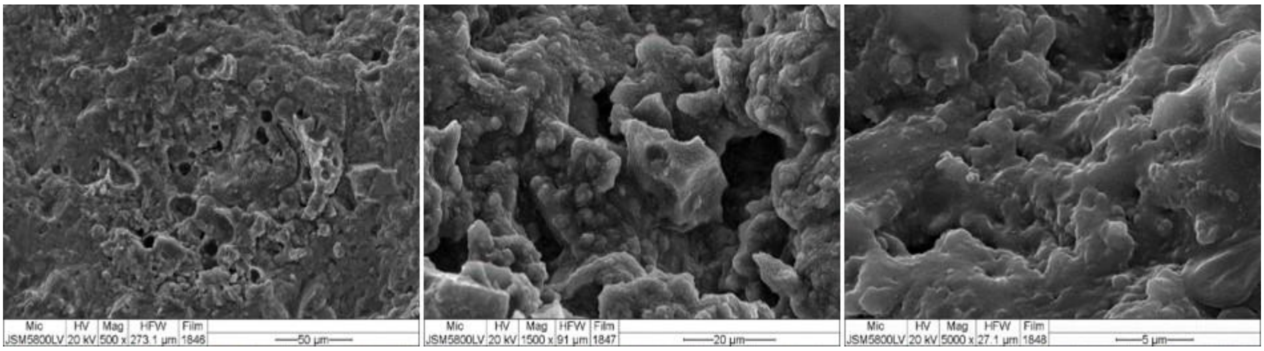


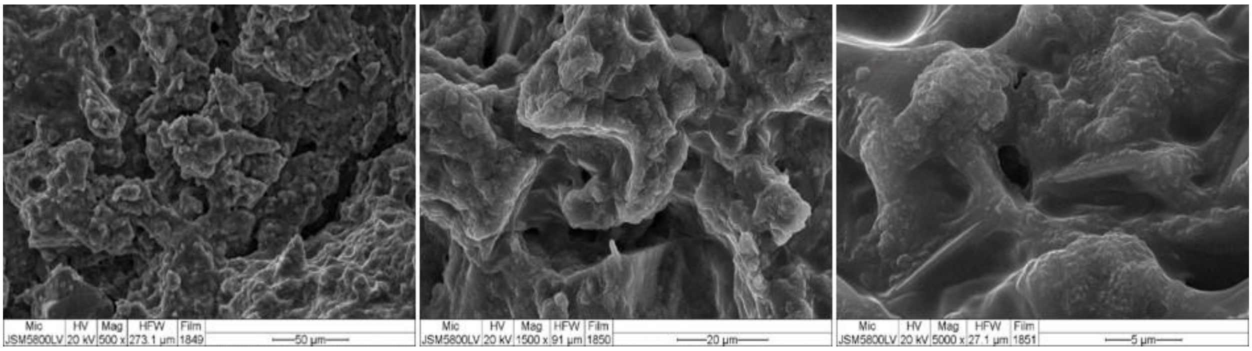
Figure 16 Density of samples with different BD to POC ratios

3.3 Scanning electron microscopy

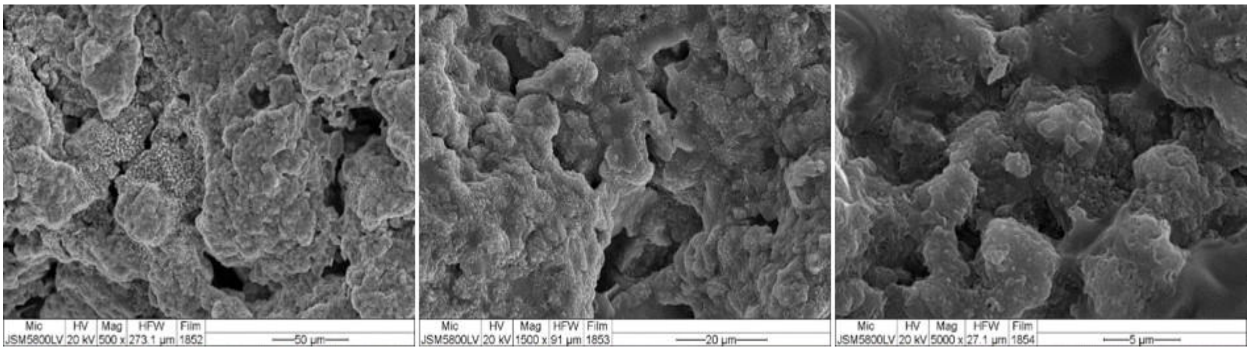
Figure 17(a)-(d) shows SEM analyses of the microstructure of geopolymer pastes at different AP contents at 28 days. The samples were taken from a small crushed section of the pastes. Figure 17(a) shows varying numbers of small pores on the surface of the control (CT) sample that underwent hot curing without AP. Spherical holes are visible in Figure 17(a). The CT, A2 and A3 matrices were not homogeneous and showed small pores, especially in the CT sample. The CT sample had many pores, whereas in the A1 matrix, a lower proportion of unreacted binder materials was detected (Figure 17(b)). The A1 sample had higher homogeneity, lowest number and lower size of pores with the least unreacted POA and AP from the alkaline activator compared to the other samples. The pore network in the matrix structure did not show a complete reaction. This geopolymer sample also produced the highest compressive strength of the series at 4.15 MPa after 28 days. Highest compressive strength was achieved with dissolution of aluminosilicate in the geopolymerization process in the A1 sample. The increased compressive strength was ascribable to the structure of the geopolymer samples with a dense compact matrix that contained less unreacted raw materials. The SEM image of 5,000x (Figure 17(b)) shows the dense compact matrix with fewer pores and uniform structure. Increased superficial continuity improved mechanical consistency.



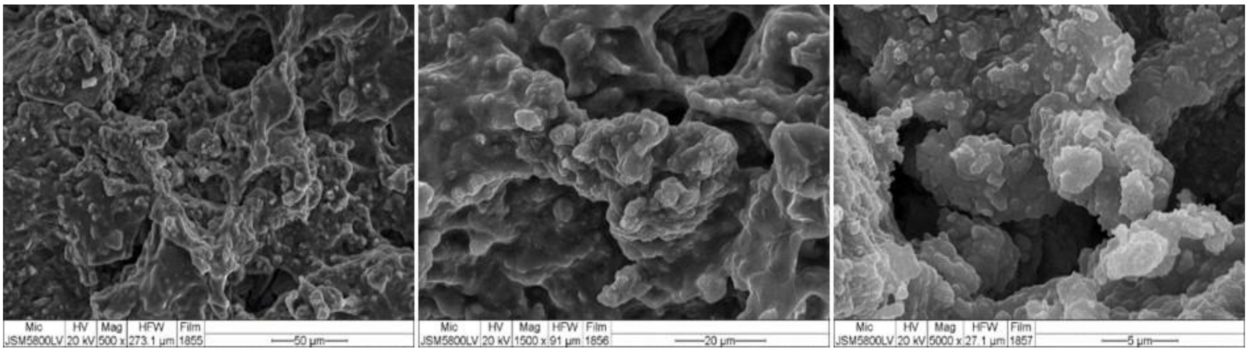
(a) CT Sample



(b) A1 Sample



(c) A2 Sample



(d) A3 Sample

Figure 17 SEM Images of geopolymer pastes

3.4 XRD Analysis

The XRD patterns of POA geopolymers containing 5, 10 and 15% AP cured at 80 °C for 24 h and kept at ambient temperature until testing on day 28 are shown in Figure 18. All mixtures presented a characteristic high background between 23° and 38° 2θ with increase in the crystalline peaks associated with base materials. The characteristic high background of the geopolymer pastes moved from 18° and 32° 2θ with initial materials to 23° and 38° 2θ after the geopolymerization process, indicating formation of an amorphous sodium aluminosilicate hydrate (N-A-S-H) gel and three dimensional network [40]. Geopolymers are also known as alkali activated aluminosilicate materials. The gel is formed either by geopolymerization of individual Si and Al atoms as building blocks in the system and the main reaction product of alkali activated aluminosilicate materials derived from low calcium content [41]. Peaks of quartz (silicon oxide; SiO_2) from the POA and corundum (aluminum oxide; Al_2O_3) from the AP were observed, confirming the geopolymerization reaction. Peaks of corundum of the geopolymer pastes were also detected, while the peak of quartz content was noted at around 27° 2θ due to the remaining quartz in POA. XRD patterns for the A1, A2 and A3 samples showed that corundum content was detected most conspicuously at 26°, 35°, 43°, 58° and 68° 2θ compared to the CT sample. Salih et al. [17] reported that the geopolymerization process of the POFA geopolymer impacted the XRD patterns. The geopolymer changed the hump shape by changing the widespread halo peak in the center toward the long wide base angles, representing formation of new amorphous compounds [42, 43].

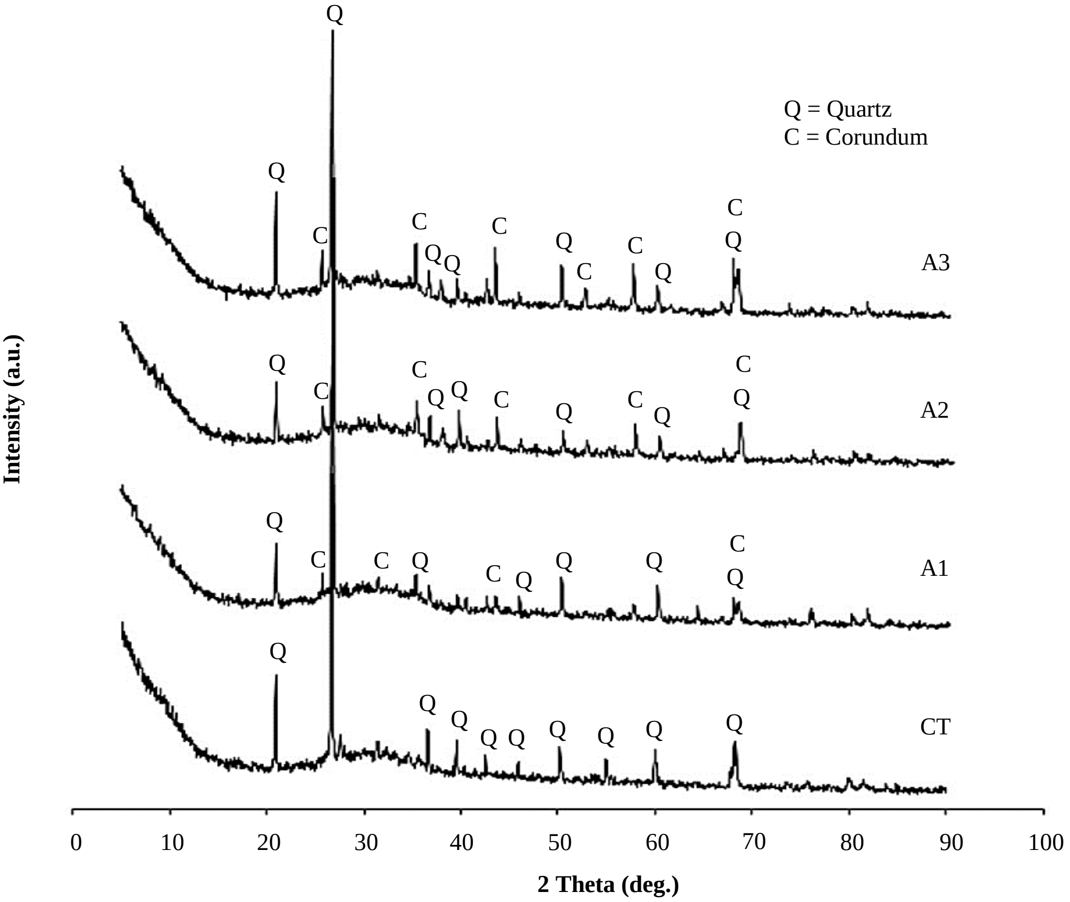


Figure 18 XRD Patterns of geopolymer pastes

3.5 Analysis of chemical composition

The rate of the geopolymerization process is influenced by parameters such as time of heat curing, curing temperature, sodium silicate to sodium hydroxide ratio, sodium hydroxide concentration and powder to alkaline ratio. The oxide composition of a geopolymer matrix impacts the strength of geopolymer paste, mortar and concrete. Table 4 compares the major oxide compositions of both materials i.e. POA and AP and alkaline activator. AP with increased Al_2O_3 oxide was added to the geopolymer pastes. Chemical compositions of paste were investigated after adding AP to the matrix. For AP with high Al_2O_3 oxide, the A1 sample achieved highest compressive strength compared to the control sample (CT) and samples containing 10% AP (A2) and 15% AP (A3). The CT sample (without AP) resulted in the lowest compressive strength compared to the other mixtures. Table 4 shows that samples containing AP had low SiO_2, while ratio of SiO_2/Al_2O_3 (Table 5) differed with Al_2O_3 oxide from AP. The rate of geopolymerization is influenced by parameters such as curing temperature, alkali concentration, initial solid content, silicate and aluminate ratio. Table 5 compares the major oxide compositions of the four geopolymer samples. The A1 mix achieved highest strength and CT achieved lowest strength. CT had the highest ratio of SiO_2/Al_2O_3 (57.57). The lowest compressive strength of CT containing 100% POA was attributed to the highest SiO_2/Al_2O_3 ratio of 57.57, while mixes A1, A2 and A3 had lower SiO_2/Al_2O_3 ratios of 19.8, 9.90 and 7.20, respectively. Islam et al. [34] prepared geopolymer mortar with POFA by adding raw materials. The resulting mortar sample had SiO_2/Al_2O_3 ratio of 11.43. By contrast, lowest compressive strength of the CT sample containing 100% POA was attributed to the highest SiO_2/Al_2O_3 ratio of 57.57 Investigation of the geopolymer pastes detected sodium content from the alkaline activator.

Table 4 Chemical compositions of geopolymer pastes

Oxide	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**K_2O**	**SO_3**	**P_2O_5**	**Cl**	**MnO**	**Na_2O**
CT	50.09	0.87	3.02	4.48	2.74	5.74	1.10	3.57	0.14	0.14	19.34
A1	45.55	2.30	2.76	4.16	1.87	5.48	0.77	3.24	0.14	0.13	23.49
A2	45.64	4.61	2.47	3.76	1.83	4.85	0.89	2.95	0.15	0.12	22.80
A3	45.50	6.32	2.54	3.57	2.19	4.76	0.86	2.87	0.12	0.12	20.72

Table 5 SiO_2/Al_2O_3 Mixture ratios

Mixture	**CT**	**A1**	**A2**	**A3**
SiO_2/Al_2O_3	57.57	19.80	9.90	7.20

4. Conclusions

In this study, 18 geopolymer mixtures were prepared by blending local BD with POA and AP. The tests included physical properties, compressive strength and microstructural analysis by SEM, XRD and XRF to investigate the feasibility of POA based geopolymer mortars using POC as fine aggregate. Based on the results, the following conclusions were drawn:

- The mixture with 5% AP had the highest compressive strength because of the reaction of aluminium oxide. Geopolymerization was dominated by the reaction of silicate, leading to increased compressive strength. SEM Image results showed that the sample had a dense compact matrix which contributed to its superior compressive strength.

The compressive strength of POA geopolymer mortars with binder to alkali activator ratio of 1.44 was higher than samples with binder to alkali activator ratios of 0.96 and 1.06.

- Heat curing increased the compressive strength in the early stages at a higher rate. However, long curing at ambient temperature from day 1 to day 28 did not notably impact strength development.
- Lower BD to POC ratio decreased the bulk density of the geopolymer samples.
- SEM Images of alkali activated POA with 5% AP displayed a fully reacted matrix with a homogeneous surface structure and low porosity.
- XRD Patterns of geopolymer POA suggested major changes compared to the other raw materials. The hump shape changed with widespread halo peaks at larger angles. Mixtures with higher AP showed higher intensity peaks of corundum (aluminum oxide; Al_2O_3), while Al_2O_3 intensity increased in alkali activated POA, resulting in the appearance of higher peaks.
- Higher AP content in the geopolymer matrix showed higher Al_2O_3.

5. Acknowledgements

The authors gratefully acknowledge the support of the Infrastructure and Materials Innovation Research Unit and Department of Civil Engineering, Princess of Naradhiwas University, Amphur Muang, Narathiwat.

6. References

[1] Kamseu E, Alzari V, Nuvoli D, Sanna D, Lancellotti I, Mariani A, et al. Dependence of the geopolymerization process and end-products to the nature of solid precursors: challenge of the sustainability. J Clean Prod. 2021;278:123587.

[2] Wongkvanklom A, Sata W, Sanjayan JG, Chindaprasirt P. Setting time, compressive strength and sulfuric acid resistance of a high calcium fly ash geopolymer containing borax. Eng Appl Sci Res. 2018;45(2):89-94.

[3] Hosseini S, Brake NA, Nikookar M, Gűnaydin-Sen Ö, Snyder HA. Mechanochemically activated bottom ash-fly ash geopolymer. Cem Concr Compos. 2021;118:103976.

[4] Kumar ML, Revathi V. Microstructural properties of alkali-activated metakaolin and bottom ash geopolymer. Arab J Sci Eng. 2020;45(5):4235-46.

[5] Selamni S, Sdiri A, Bouaziz S, Joussein E, Rossignol S. Effects of metakaolin addition on geopolymer prepared from natural kaolinitic clay. Appl Clay Sci. 2017;146:457-67.

[6] Topala Ö, Karakoç MB, Özcan A. Effects of elevated temperatures on the properties of ground granulated blast furnace slag (GGBFS) based geopolymer concretes containing recycled concrete aggregate. Eur J Environ Civ Eng. 2022;26(10):4847-62.

[7] Hasnaoui A, Ghorbel E, Wardeh G. Optimization approach of granulated blast furnace slag and metakaolin based geopolymer mortars. Constr Build Mater. 2019;198:10-26.

[8] Safari Z, Kurda R, Al-Hadad B, Mahmod F, Tapan M. Mechanical characteristics of pumice-based geopolymer paste. Resour Conserv Recycl. 2020;162:105055.

[9] Tchadjié LN, Ekolu SO, Quainoo H, Tematio P. Incorporation of activated bauxite to enhance engineering properties and microstructure of volcanic ash geopolymer mortar composites. J Build Eng. 2021;41:102384.

[10] Rattanasak U, Chindaprasirt P, Suwanvitaya P. Development of high volume rice husk ash alumino silicate composites. Int J Miner Metall Mater. 2010;17(5):654-9.

[11] Rashad AM. Alkali-activated metakaolin: a short guide for civil engineer-an overview. Constr Build Mater. 2013;41:751-65.

[12] Rattanasak U, Chindaprasirt P. Influence of NaOH solution on the synthesis of fly ash geopolymer. Miner Eng. 2009;22(12):1073-8.

[13] Liew YM, Kamarudin H, Bakri AMMA, Binhussain M, Luqman M, Nizar IK, et al. Influence of solids-to-liquid and activator ratios on calcined kaolin cement powder. Phys Procedia. 2011;22:312-7.

[14] Khankhaje E, Hussin MW, Mirza J, Rafeizonooz M, Salim MR, Siong HC, et al. On blended cement and geopolymer concretes containing palm oil fuel ash. Mater Des. 2016;89:385-98.

[15] Ranjbar N, Mehrali M, Alengaram UJ, Metselaar HSC, Jumaat MZ. Compressive strength and microstructural analysis of fly ash/palm oil fuel ash based geopolymer mortar under elevated temperatures. Constr Build Mater. 2014;65:114-21.

[16] Salih MA, Farzadnia N, Ali AAA, Demirboga R. Effect of different curing temperatures on alkali activated palm oil fuel ash paste. Constr Build Mater. 2015;94:116-25.
[17] Salih MA, Ali AAA, Farzadnia N. Characterization of mechanical and microstructural properties of palm oil fuel ash geopolymer cement paste. Constr Build Mater. 2014;65:592-603.
[18] Darvish P, Alengaram UJ, Poh YS, Ibrahim S. Yusuff S. Performance evaluation of palm oil clinker sand as replacement for conventional sand in geopolymer mortar. Constr Build Mater. 2020;258:120352.
[19] Hawa A, Tonnayopas D, Prachasaree W, Taneerananon P. Investigating the effects of oil palm ash in metakaolin based geopolymer. Ceram-Silik. 2013;57(4):319-27.
[20] Chalee W, Cheewaket T, Jaturapitakkul C. Enhanced durability of concrete with palm oil fuel ash in a marine environment. J Mater Res Technol. 2021;13:128-37.
[21] Bashar II, Alengaram UJ, Jumaat MZ, Islam A. The effect of variation of molarity of alkali activator and fine aggregate content on the compressive strength of the fly ash: palm oil fuel ash based geopolymer mortar. Adv Mater Sci Eng. 2014;2014:245473.
[22] Liu MYJ, Alengaram UJ, Santhanam M, Jumaat MZ, Mo KH. Microstructural investigations of palm oil fuel ash and fly ash based binders in lightweight aggregate foamed geopolymer concrete. Constr Build Mater. 2016;120:112-22.
[23] Hawa A, Tonnayopas D, Prachasaree W. Performance evaluation of metakaolin based geopolymer containing parawood ash and oil palm ash blends. Mater Sci. 2014;20(3):339-44.
[24] Hawa A, Tonnayopas D, Prachasaree W. Performance evaluation and microstructure characterization of metakaolin-based geopolymer containing oil palm ash. Sci World J. 2013;2013:857586.
[25] Darvish P, Alengaram UJ, Alnahhal AM, Poh YS, Ibrahim S. Enunciation of size effect of sustainable palm oil clinker sand on the characteristics of cement and geopolymer mortars. J Build Eng. 2021;44:103335.
[26] ASTM. ASTM C331-03: Standard specification for lightweight aggregates for concrete masonry units. West Conshohocken: ASTM International; 2003.
[27] Hawa A, Tonnayopas D, Prachasaree W, Taneerananon P. Development and performance evaluation of very high early strength geopolymer for rapid road repair. Adv Mater Sci Eng. 2013;2013:764180.
[28] ASTM. ASTM C109/C109M-07: Standard test method for compressive strength of hydraulic cement mortars (using 2-in. or [50-mm] cube specimens). West Conshohocken: ASTM International; 2007.
[29] Hawa A, Prachasaree W, Tonnayopas D. Effect of water-to-powder ratios on the compressive strength and microstructure of metakaolin based geopolymers. Indian J Eng Mater Sci. 2017;24:499-506.
[30] Upshaw M, Cai CS. Feasibility study of MK-based geopolymer binder for RAC applications: effects of silica fume and added CaO on compressive strength of mortar samples. Case Stud Constr Mater. 2021;14:e00500.
[31] Hawa A, Salaemae P, Prachasaree W, Tonnayopas D. Compressive strength and microstructural characteristics of fly ash based geopolymer with high volume field Para rubber latex. Rev Romana Mater/ Rom J Mater. 2017;47(4):462-9.
[32] Hawa A, Prachasaree W. The development of compressive strength, drying shrinkage and microstructure of fly ash geopolymer with field Para rubber latex. Rev Romana Mater/ Rom J Mater. 2020;50(1):59-68.
[33] Prachasaree W, Limkatanyu S, Hawa A, Sukontasukkul P, Chindaprasirt P. Development of strength prediction models for fly ash based geopolymer concrete. J Build Eng. 2020;32:101704.
[34] Islam A, Alengaram UJ, Jumaat MZ, Bashar II. The development of compressive strength of ground granulated blast furnace slag-palm oil fuel ash-fly ash based geopolymer mortar. Mater Des. 2014;56:833-41.
[35] Khale D, Chaudhary R. Mechanism of geopolymerization and factors influencing its development: a review. J Mater Sci. 2007;42(3):729-46.
[36] Saeli M, Senff L, Tobaldi DM, Seabra MP, Labrincha JA. Novel biomass fly ash-based geopolymeric mortars using lime slaker grits as aggregate for applications in construction: influence of granulometry and binder/aggregate ratio. Constr Build Mater. 2019;227:116643.
[37] Noushini A, Castel A. The effect of heat-curing on transport properties of low-calcium fly ash-based geopolymer concrete. Constr Build Mater. 2016;112:464-77.
[38] Yusuf MO, Johari MAM, Ahmad ZA, Maslehuddin M. Effects of H_2O/Na_2O molar ratio on the strength of alkaline activated ground blast furnace slag-ultrafine palm oil fuel ash based concrete. Mater Des. 2014;56:158-64.
[39] Sathonsaowaphak A, Chindaprasirt P, Pimraksa K. Workability and strength of lignite bottom ash geopolymer mortar. J Hazard Mater. 2009;168(1):44-50.
[40] Criado M, Fernández-Jiménez A, de la Torre AG, Aranda MAG, Palomo A. An XRD study of the effect of the SiO_2/Na_2O ratio on the alkali activation of fly ash. Cem Concr Res. 2007;37(5):671-9.
[41] Temuujin J, van Riessen A, MacKenzie KJD. Preparation and characterisation of fly ash based geopolymer mortars. Constr Build Mater. 2010;24(10):1906-10.
[42] Somna K, Jaturapitakkul C, Kajitvichyanukul P, Chindaprasirt P. NaOH-Activated ground fly ash geopolymer cured at ambient temperature. Fuel. 2011;90(6):2118-24.
[43] Ahmari S, Ren X, Toufigh V, Zhang L. Production of geopolymeric binder fromblended waste concrete powder and fly ash. Constr Build Mater. 2012;35:718-29.