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Using Palm Oil Fuel Ash as a Source Material for Alumina Silicate

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ABSTRACT

This research investigated the utilization of palm oil waste as a source material for developing an alkali-activated binder with alumina-silicate properties. The geopolymer synthesis involved a combination of palm oil fuel ash (POFA) and fly ash (FA), as well as sodium silicate and sodium hydroxide as alkali activator solutions. The study assessed the physical, mechanical, water-transport and thermal performances of the binder, including the influence of oxide ratios on its strength-gain characteristic. The highest strength achieved was 54.7 MPa for a blend of POFA-FA in a ratio of 20:50 with a molarity of NaOH at 12M. The experimental results revealed good water-transport performance due to the dense nature of the binder that restricted water movement. However, the material's insulation performance did not produce significant results with the lowest thermal conductivity value of 0.59 W/mK. Overall, the developed binder has potential industrial applications, as it performed well in the technical aspects studied.

KEYWORDS: Palm oil fuel ash, Alkali-activated binder, Waste, Sustainability, Water-transport performance.

INTRODUCTION

The concept of sustainable development aims to meet the present societal needs while ensuring that future generations have access to Adequate resources. In the construction industry, sustainability has become a crucial factor, with stakeholders increasingly aware of the need to develop and use materials in sustainable ways. Achieving sustainability in construction materials involves reducing the use of non-renewable resources and adopting strategies that emphasize the use of agricultural and industrial by-products. While many researchers, like Yang et al. (2019), have explored the potential of using municipal, agricultural (Raut et al., 2016) and industrial waste (Naganathan et al., 2013; Kinuthia et al., 2011) to develop sustainable alternatives to traditional construction materials, it is important to provide a comprehensive solution that meets all industry standards (Raut et al., 2023). There is a risk that these alternatives may not perform as well as traditional materials and practitioners may not adopt them unless they meet the required quality standards.

According to recent statistics, palm oil remains one of the most widely consumed vegetable oils globally, with an estimated production of over 77 million tons in 2021. As of 2020, palm oil accounted for nearly 30% of the world's vegetable-oil production and consumption (Bolina et al., 2021). In Malaysia, the area of land used for palm oil cultivation has grown to 5.8 million hectares in 2021. Despite its popularity, the production of palm oil generates various by-products, including palm oil, palm kernels, palm fibers, palm shells and empty fruit bunches, which require proper disposal. Researchers have investigated various applications for palm oil by-products, including using palm oil ash as a supplementary cementitious material for construction (Raut et al., 2017a). Other agricultural waste by-

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products, such as rice-husk ash and sugarcane-bagasse ash, have also been explored for their potential use as supplementary cementitious materials.

Supplementary cementitious materials are currently being utilized to create cement-less binders through alkali-activation. These materials contain silica and alumina, making them ideal for the development of alkali-activated binder material (Singh et al., 2023). The reaction between the alkali and alumina-silicate monomers creates a polymeric chain, which provides the material with strength and bonding properties like those of CSH gel found in traditional cementitious materials. The resulting cement-less concrete has physical characteristics equivalent to conventional concrete, but with superior performance in terms of strength, setting time, reduced shrinkage, resistance to freezing and thawing and increased durability (Sumathi et al., 2008; Foo et al., 2009). Additionally, studies have shown that alkali-activated binders have an 80% lower carbon footprint than traditional concrete (Singh et al., 2021). Researchers are investigating various alumino-silicate precursors, such as fly ash (Singh et al., 2021), blast furnace slag (Kumar et al., 2010), silica fume (Khater et al., 2013), rice-husk ash (He et al., 2013), metakaolin (Khan et al., 2021), coal and biomass ash (Samadhi et al., 2017), copper slag (Ahmari et al., 2015), red mud from aluminum extraction (Zhang et al., 2010), volcanic ash (Tchakoute Kouamo et al., 2012), sugarcane-bagasse ash (Cataldelli et al., 2013) and glass powder (Rivera et al., 2018) for the development of alkali-activated binders.

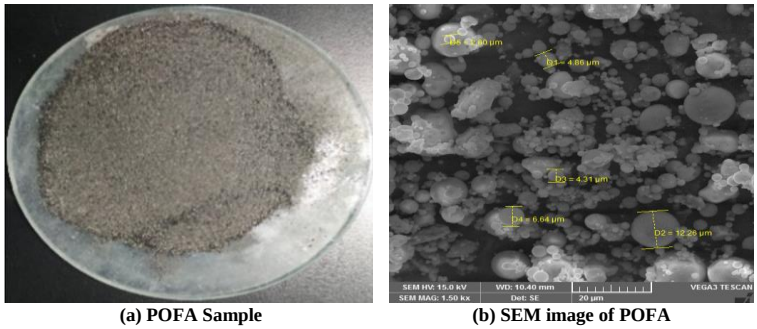
The utilization of palm oil fuel ash as a prospective component in the development of alkali- activated binder is becoming increasingly popular, especially in Southeast Asian countries, where it is widely available. It is crucial to assess the performance of the binder in terms of physical, mechanical, thermal and water-transport characteristics. A comprehensive analysis of these properties is necessary to ensure that palm oil fuel ash can be used as a reliable and effective ingredient in the production of alkali-activated binder (Raut et al., 2023). Such an evaluation will be important to gain the consensus of researchers and practitioners in the industry, encouraging them to incorporate palm oil as a potential ingredient in the production of alkali-activated binder material.

POFA (Palm Oil Fuel Ash) is a by-product generated through engineering processes from the waste of the palm oil industry (Amran et al., 2021). Within the steam boiler, combustion of palm kernel and husk shell occurs, resulting in the production of POFA. This by-product contains a substantial quantity of silica-oxide content, meeting the criteria for pozzolanic properties. Consequently, it holds potential as a suitable replacement for cement in various applications (Rasid et al., 2023). However, several researchers have asserted the utilization of POFA in regular concrete applications (Hamada et al., 2018), as well as in high-strength concrete (Sata et al., 2004), in addition to light-weight and foamed concrete (Al-Shwaiter et al., 2022).

Raw Material

Palm Oil Fuel Ash

The primary focus of this study was to evaluate the potential of palm oil fuel ash (POFA) as an alumino-silicate precursor for developing alkali-activated binder. The physical form of POFA is shown in Figure 1(a). To assess the characteristics of POFA, a scanned microscopy image (SEM) was obtained and is presented in Figure 1(b). The reactivity of the raw material is strongly influenced by its fineness; so, particle-size analysis was conducted for POFA and the results are presented in Figure 2.



(a) POFA Sample **(b) SEM image of POFA**

Figure (1): Palm oil fuel ash

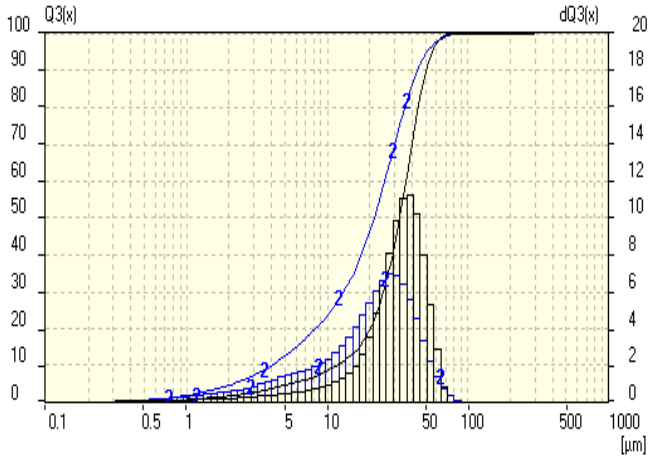


Figure (2): Particle-size analysis of POFA

(a) Fly ash **(b) SEM image of fly ash**

Figure (3): Fly ash: Physical form and SEM

Fly Ash

The Class-F Fly Ash (FA) utilized in this research was sourced from Narla Tatarao Thermal Power Station (NTTPS), located near Vijayawada City, Andhra Pradesh. The physical form of FA is shown in Figure 3(a). SEM Analysis is shown in Figure 3(b). To remove excess moisture, the fly ash underwent a heating process at 110°C. Following this, it was sieved to ensure that particle-sizes remained below 1.18 mm, as illustrated in Figure 4, displaying the particle-size distribution. The reactivity of the raw material is significantly impacted by its fineness. Furthermore, the presence of oxides in the raw SS material can significantly impact its performance; hence, X-ray fluorescence (XRF) analysis was performed and the results are shown in Table 1.

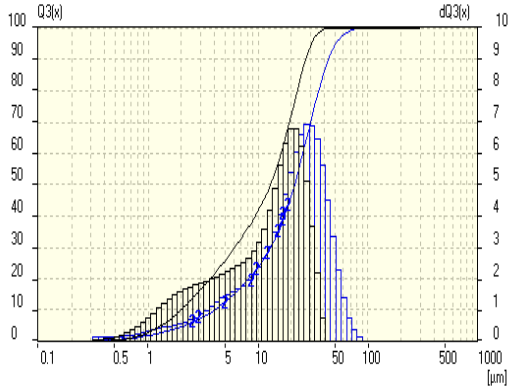


Figure (4): Particle-size analysis of fly ash

Table 1. XRF analysis of POFA and FA

Sample	POFA (%)	Fly Ash (%)
SiO_2	61.66	50.47
TiO_2	-	9.6
Al_2O_3	5.13	28.76
Fe_2O_3	5.29	4.3
CaO	9.86	0.81
MgO	4.17	0.39
Na_2O	0.49	0.09
K_2O	8.42	0.77
P_2O_5	3.71	0.31
SO_3	2.98	4.3

As shown in Table 1, the predominant chemical component of the ground palm oil fuel ash (POFA) was SiO_2, constituting 61.66% of the composition. There is a possibility that, in the presence of moisture and a suitable pH environment, SiO_2 from the ash could dissociate to release Si^{4+} ions, a phenomenon observed in previous studies. Additionally, under similar conditions, Al_2O_3 (6.27%) might also dissociate, producing Al^{3+} ions. Notably, these dissociated Al^{3+} ions from the ground POFA could potentially undergo isomorphous substitution by replacing the Si^{4+} ions within the silica tetrahedral sheets without altering the overall silica structure. These substitution reactions could result in a deficit of positive charge, leading to the development of a negative charge on the kaolinite mineral, as described in previous research (Nadziri et al., 2018).

To quantify the amorphous and crystalline phases of the POFA material, X-ray-diffraction analysis was conducted and the results are presented in Figure 5. It is evident that the material has no detectable crystalline phase and is amorphous in nature within the range of 20°-40°. The Rietveld refinement detected quartz, which is rich in silica within the amorphous phase, as well as cristobalite. Cristobalite is a polymorph of silicon dioxide (SiO_2), indicating that it shares the same chemical composition as quartz, but possesses a distinct crystal structure. Quartz is another prevalent variation of SiO_2 with a different arrangement of its atoms and gismondine (with the chemical formula $CaAl_2Si_2O_8{\cdot}4(H_2O)$). By conducting these tests and analyzing the results, researchers can better understand the physical and chemical properties of POFA and its potential as a raw material for the development of alkali-activated binder.

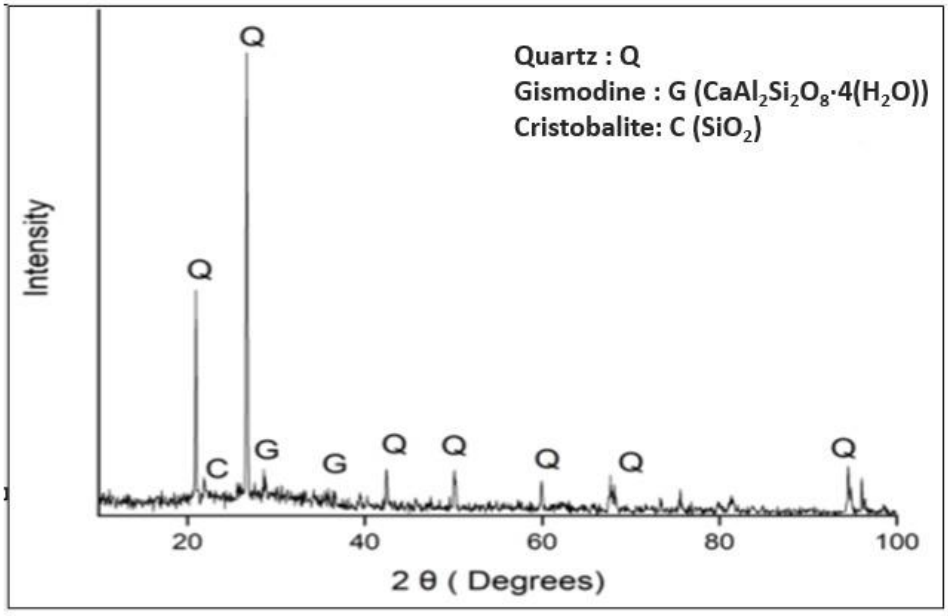


Figure (5): XRD outline of POFA

Material Development

The raw materials required to prepare alkali-activated binder are divided into two categories: solid and liquid. The solid materials include fly ash, palm oil fuel ash and crusher dust. The inclusion of crusher dust in the mix was restricted to 30% due to its role as a filler

material and its minimal variance in properties compared to the geopolymer matrix. Crusher dust was chosen for its ready availability as a local waste material. This decision was made in place of river sand, making crusher dust a suitable choice for geopolymer applications. The liquid materials consist of sodium silicate, sodium hydroxide and water. Table 2 shows the mix design used for preparation. To understand the effect of molarity on the performance of the final product, sodium hydroxide (NaOH) was used at both 10M and 12M levels, with the ratio of sodium silicate to sodium hydroxide kept at 2.5. The liquid solution used as the alkali activator was mixed one day prior to the preparation of the alkali-activated binder.

To prepare the binder, all the solid ingredients were first batched and mixed until a homogeneous mixture was obtained. Then, the alkali activator solution that was prepared one day prior was added to the mix. Wet mixing was conducted for 2-5 minutes in a rotary mixer until the mixture became homogeneous. Once the mixture was homogeneous, it was transferred to molds of dimensions of 100x100x100mm³. The demolding process was done once the binder had attained a certain degree of hardness. The binder was then temperature-cured at 60°C to activate the reactivity between alkalis and alumino-silicate precursors.

The physical, mechanical and thermal properties of the developed alkali-activated binder samples were evaluated to investigate their performance. The physical characteristics, including bulk density, porosity and water absorption, were determined in accordance with the ASTM C373-18(2023) standard. The compressive strength and flexural strength were evaluated using ASTM C109/C109M-21(2021) and ASTM C348-21(2021), respectively. The thermal conductivity and heat capacity of the material were measured using Hot Disk TPS 2500S using ASTM C177-19e1 (2023). Moreover, the water-transport properties, such as percentage water absorption (PWA), velocity of water absorption (VMA) and coefficient of water absorption (CWA), were also assessed. The calculation methodology for performing these tests was based on a study conducted by Raut et al. (2016).

Table 2. Mix design

Mix No.	POFA (%)	Fly Ash (%)	Crusher Dust (%)	Na_2SiO_3 (g)	Molarity of NaOH	NaOH Sol. (g)	Water (ml)
C1	40	30	30	1375	10	157.1	392.9
C2	30	40	30	1375	10	157.1	392.9
C3	20	50	30	1375	10	157.1	392.9
C4	40	30	30	1375	12	178.2	371.8
C5	30	40	30	1375	12	178.2	371.8
C6	20	50	30	1375	12	178.2	371.8

RESULTS AND DISCUSSION

Physical Properties

The findings regarding the physical performance of the alkali-activated binder are shown in Figure 6. It is evident from the results that the density of the binder increased as the molarity of the alkali activation solution increased. This can be attributed to the formation of additional binding compounds resulting from increased reactivity, thereby occupying more volume in the matrix. This increase in molarity also caused a reduction in porosity due to the formation of alumino-silicate polymeric chains occupying the spaces. The porosity of the binder ranged from 9.4% to 11.1%, while the bulk density varied from 1.7 kg/m^3 to 1.92 kg/m^3. Furthermore, the addition of POFA led to a decrease in density and an increase in porosity, possibly due to the lower specific gravity of POFA compared to fly ash.

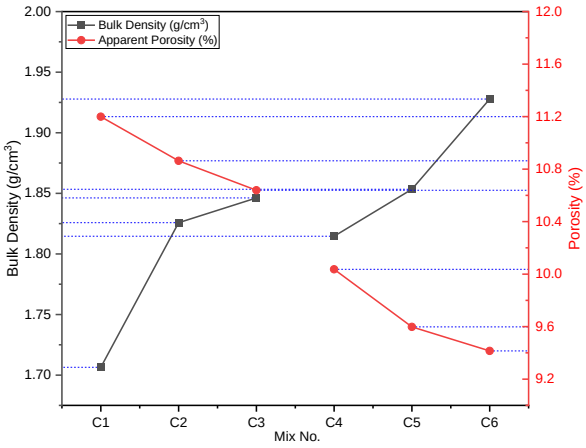


Figure (6): Relationship between density and porosity of developed alkali-activated binder

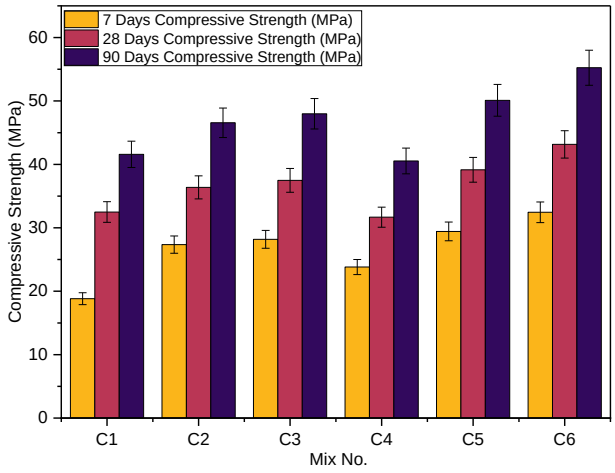
To prevent water percolation and damage to building elements, it is important to understand the water-transport properties of materials. Table 3 displays data on the water-transport performance of the alkali-activated binder at various ages. As the material ages, the percentage of water absorption decreases due to the formation of alumina-silicate polymeric chains and reduction in pore size. Porosity plays a key role in water-transport properties, as a decrease in porosity results in lower water-absorption velocity and coefficient. The C6 mix exhibited the lowest PWA (6.14%) after 90 days, while the velocity of water absorption ranged from 10.1×10^{-6} to 13.59×10^{-6} m/s$^{1/2}$. A higher molarity of NaOH resulted in greater deposition of the geopolymer matrix, leading to lower water-transport properties. Lower velocity of water absorption is critical to reduce the percolation of water through capillary pores. The coefficient of water absorption, which correlates with the permeability of materials, ranged from 2.13×10^{-10} to 3.2×10^{-10} m/s and was found to decrease with higher molarity of the sample.

Table 3. Water-transport performance of developed alkali-activated binder

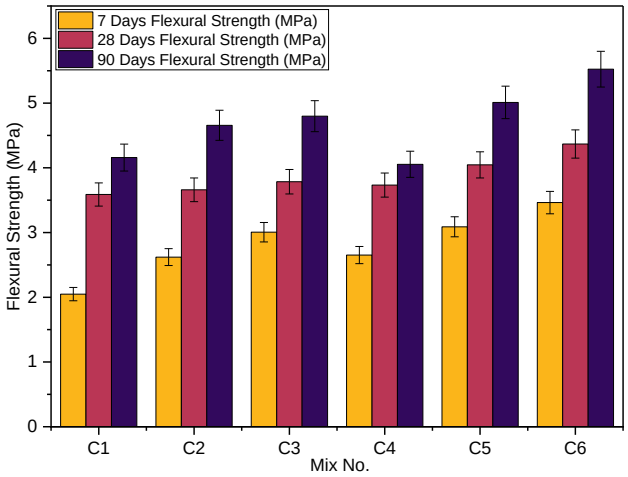
Mix No.	**PWA (%)**	**PWA (%)**	**PWA (%)**	**VMA**	VMA	**VMA**	**CWA**	**CWA**	**CWA**
	7 days	**28 days**	**90 days**	**7 days**	**28 days**	**90 days**	**7 days**	**28 days**	**90 days**
C1	9.0678	7.3848	7.0278	13.5966	12.1278	11.1282	3.2742	2.9478	2.601
C2	8.8434	7.1808	6.8646	13.4334	11.9238	10.9242	2.9682	2.7234	2.3766
C3	8.6394	7.0584	6.7932	12.7908	11.3424	10.3326	2.9274	2.5806	2.2848
C4	8.6088	7.0074	6.3342	12.9234	11.5056	10.6488	3.0702	2.8152	2.5398
C5	8.3946	6.5382	6.2934	12.699	11.2506	10.4448	2.9784	2.7132	2.3562
C6	8.1906	6.4566	6.1404	12.3012	11.0058	10.1082	2.6316	2.3562	2.1318

Mechanical Properties

The compressive strength of the alkali-activated material developed from palm oil fuel ash (POFA) is shown in Figure 7(a). The material exhibited a gain in strength with age, like that of any cementitious material. The C6 sample achieved a maximum strength of 54.7 MPa. The gain in strength was due to the reaction between alumina and silica monomers present in the blend of fly ash and POFA with the alkali activators, which formed polymeric chains, such as polysilicates (Al-O-Si), polysialatesiloxo (Al-O-Si-Si) and polysialatedisiloxo (Al-O-Si-Si-Si) (Ranjbar et al., 2014). The morphology of both FA and POFA also played an important role in the reaction, as the spherical texture of POFA increased its reactivity and formed a denser matrix. The percentage of fly ash was found to be beneficial for strength gain. The molarity of NaOH was also observed to have a crucial role in enhancing the compressive strength of the alkali-activated binder. An increase in molarity of NaOH increased the concentration of alkali solution, affecting the dissolution of silica and alumina monomers, which caused the formation of additional alkali-activated binder. However, further addition of Na+ ions due to increased molarity had a detrimental effect on the formation of the binder and caused cracking (Ranjbar et al., 2014).



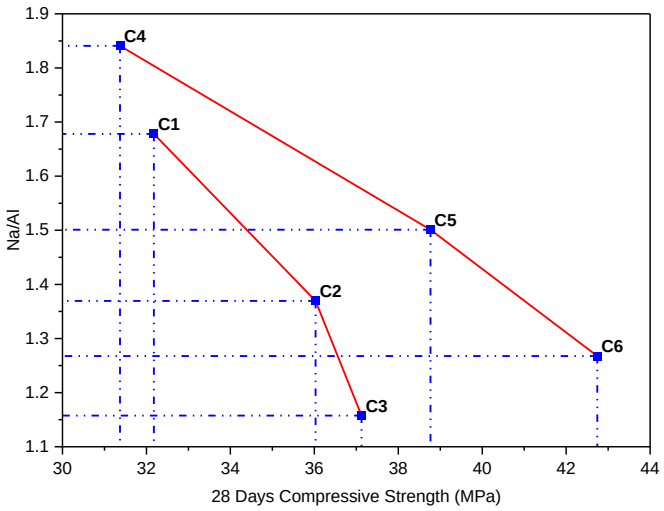
(a): Compressive strength



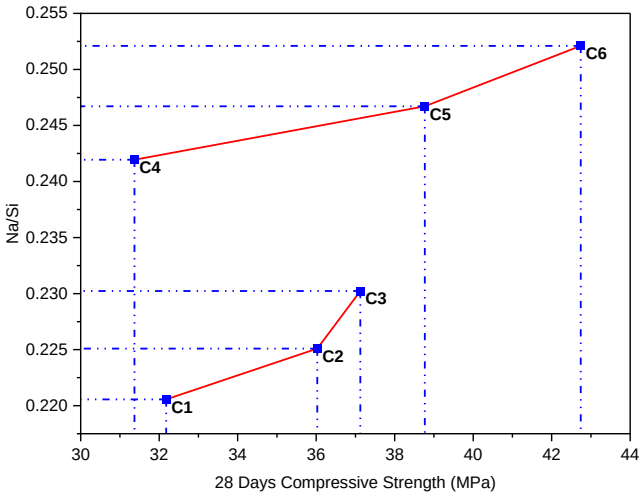
(b): Flexural strength

Figure 7: (a) Compressive strength and (b) Flexural strength of developed alkali-activated binder

Figure 7 (b) illustrates the flexural behaviour of the alkali-activated binder, which follows a trend similar to that of the compressive strength. The flexural strength is attributed to the reactivity of the alumina-silicate precursor material with the alkali activators, but the binder is brittle in nature and exhibits features similar to those of cementitious binders. The C6 mix achieved the highest flexural strength of 5.52 MPa. The dense micro-structure of the geopolymer leads to higher flexural strength due to the inter-molecular bonding caused by continuous polymerization. The chemical composition of the ingredients, specifically the Na/Al, Na/Si and Si/Al oxide ratios, plays a crucial role in the reactivity and strength development of the material. Higher Na/Al ratios can cause stiffening of the matrix and lead to micro-cracks, ultimately affecting the overall strength. As shown in Figure 8(a), the C1 and C4 mixes had higher Na/Al ratios due to the low percentage of alumina in the POFA, resulting in lower strength compared to other mix proportions. The C6 mix achieved a strength of 53.11 MPa with the lowest Na/Al ratio of 1.15 among the other mixes.



(a) Na/Al oxide ratio



(b) Na/Si oxide ratio

Figure (8): (a) Na/Al oxide ratio and (b) Na/ Si oxide ratio effect on compressive strength

Figure 8 (b) demonstrates the impact of the ratio of Na/Si on the strength of the alkali-activated binder. The molarity of NaOH had a significant influence on the Na/Si ratio in the mixes. For instance, the Na/Si ratio for

mixes C1-C3 ranged from 0.22-0.23 for 10M NaOH. Figure 8(b) clearly illustrates the difference between the molarity and its impact on the Na/Si ratio. As the Na/Si ratio increased, more Na+ ions were available for dissolution, resulting in increased reactivity and formation of polymer chains. The addition of fly ash to the mix also decreased the silica content, leading to an increase in Na/Si, which negatively affected the strength of the binder. According to Davidovits et al. (2019), the ratio of Na/Si should be within 0.2-0.28 to develop strength in alkali-activated binders.

Figure 9 revealed that the Si/Al ratio ranged from 5-7.6, which affected the compressive strength of the binder. A higher Si/Al ratio resulted in lower strength, since the excess Na+ ions remained unreacted, affecting the strength development of the binder. Davidovits et al. (2019) suggested a proposed Si/Al ratio of 3.5-4.5 for the development of binders. However, all the developed mixes had a higher silica and alumina ratio. The strength gain was highest for the mixes with ratios closer to 5. The C6 mix had the lowest Si/Al ratio of 5.02 among all mixes, proving to have the highest strength characteristic. While the C3 and C6 mixes had similar Si/Al ratios, other factors such as Na+ ions also played a role in enhancing the strength of the C6 binder.

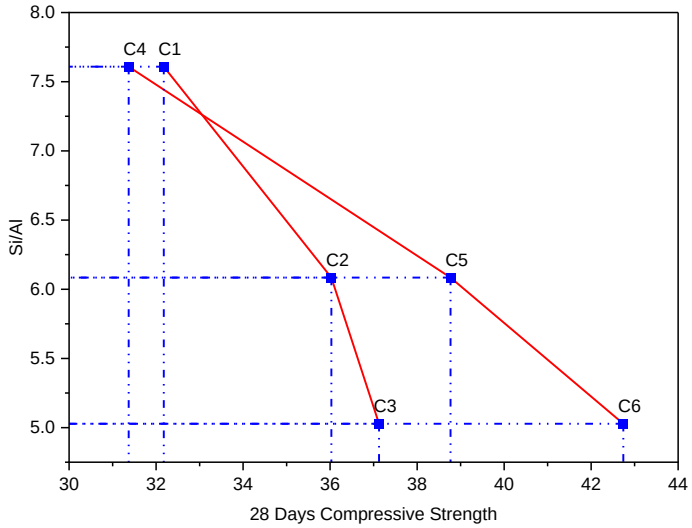


Figure (9): The effect of Si/Al oxide ratio on compressive strength

Thermal Properties

The ability of building materials to insulate against heat transfer is a critical feature of building envelopes. To achieve effective insulation, the material's thermal conductivity should be as low as possible. However, the strength of the material is directly correlated with its thermal conductivity. As shown in Table 4, the sample with the highest compressive strength (C6) has the highest thermal conductivity (0.79 W/mK). On the other hand, the sample with the lowest thermal conductivity (0.59 W/mK) is C6, which has the lowest density (1.7 kg/m^3). The variation in thermal-conductivity values can be attributed to the porous nature of the geopolymer samples. A higher porosity leads to a lower transmission of heat through the material, as air-filled voids interrupt particle-to-particle heat transfer.

Aside from thermal conductivity, other material properties, such as heat capacity, thermal diffusivity and thermal effusivity, should be considered when analyzing steady-state heat transfer. Heat capacity and storage are critical properties for determining a material's thermal mass. The diffusivity of a material refers to the speed of heat propagation when the temperature changes, which is important as building envelopes are exposed to outdoor temperatures that vary over time. Thus, the diffusivity of a material should be as low as possible to minimize heat transfer during non-steady-state heat-transfer processes. The thermal-diffusivity values in Table 4 range from (1.99 to 2.16 x 10^{-7} (m^2/s)), with the highest value belonging to the C6 sample.

The thermal effusively values, which range from 1326 to 1688 (Ws$^{1/2}$/m^{2}K), suggest that the rate of heat absorption by the material will be higher when the absorption rate affects the heat propagation of the material. For optimal insulation performance, the thermal diffusivity and effusively values should be as

low as possible. However, materials with high density and strength may not support effective insulation due to their closely packed arrangement within the matrix of the developed geopolymer.

Table 4. Thermal performance of developed alkali-activated binder

Mix No.	Density (kg/m^3)	Thermal Conductivity (W/mK)	Heat Capacity (J/kg^oc)	Thermal Diffusivity (m^2/s)	Thermal Effusivity ($WK^{-1}m^{-2}\ s^{1/2}$)
C1	1.70	0.59	1759.1	1.99E-07	1326.38
C2	1.82	0.64	1805.16	1.96E-07	1446.07
C3	1.84	0.71	1877.68	2.05E-07	1563.15
C4	1.81	0.66	1729.7	2.12E-07	1431.99
C5	1.75	0.62	1697.36	2.10E-07	1351.99
C6	1.92	0.79	1899.24	2.16E-07	1688.90

X-Ray Diffraction Analysis

To conduct XRD analysis and study the compound and mineralogical composition developed in alkali-activated solutions incorporating fly ash and palm oil fuel ash, the samples were first cured in an oven at 60°C for one day and then kept at ambient temperature for 28 days. XRD was conducted at KL University, by using bruker apparatus. The mixture was heated at 400°C in a controlled atmosphere for 6 hours. The characteristic diffraction peaks at specific 2θ values were determined using Scherrer's formula ($D = K\lambda/ \beta\cos\theta$). Afterwards, the specimens were crushed to a powder form, refined by a 20-micron sieve and immersed in an isopropyl solution to stop hydration. XRD analysis was carried out on specimens C1 to C6 to investigate the main phases formed in POFA-FA-based geopolymer. The results showed that the major compounds formed with POFA-FA-based mortar were quartz, albite, sodalite and mullite. The diffractogram of original POFA and FA changed noticeably with the addition of alkaline solutions, activating the precursor ions present in the ash and leading to a change in the formation of alumino-silicate hydrate gel, which is the primary product of the formation of geopolymer mortar. As seen from Figure 10, an increase in the percentage of POFA in FA-based geopolymer led to the formation of an albite N-A-S-H phase ($NaAlSi_3O_8$), resulting in an increase in the compressive strength of C3 and C6 due to the highest amount of amorphicity. To discern the peaks in the XRD data, the X-pert High Score Plus software was employed. A thorough analysis was conducted using the star and indexed ICDD PDF-2 file in combination with the open crystallography database.

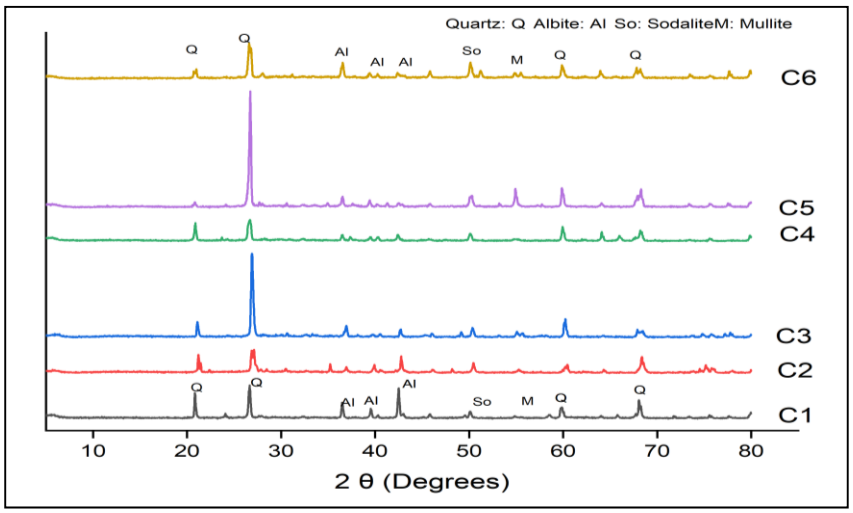


Figure (10): XRD pattern of specimen FA and POFA-based geopolymer mortar

CONCLUSIONS

The results obtained in the present study clearly indicate that the presence of alumina and silicate monomers within the palm oil fuel ash in combination with fly ash can be used for the preparation of alkali-activated binders. Based on the experimentation on the developed binder, some conclusions were drawn, which are listed as follows:

1. The blend of POFA-FA provides strength to the geopolymer, where the highest strength observed was 54.7 MPa for C6 sample. The gain of strength is due to the formation of polymerics; i.e., polysilate (Al-O-Si), polysialate-siloxo (Al-O-Si-Si) and polysialate-disiloxo (Al-O-Si-Si-Si) chains, which bond to form a dense matrix. Also, the increase in molarity of NaOH in the solution enhanced the dissolution of silica and alumina monomer, thus enhancing the strength performance of the binder.
2. It is observed that the formation of polymeric chain proves dense micro-structure, which in turn affects the water-transport property. The pores within the matrix occupy void spaces due to the alkali-activation reaction, thus restricting the movement of water through capillary pores giving rise to improved water-transport performance. The lower susceptibility to water percolation enhances the life of the structural element.
3. Also, from the experimental analysis, it was found that the combination of molar ratios in an appropriate amount can provide a binder with higher-strength characteristics. It was observed that oxide ratios were Na/Al (1.15), Si/Al (5.02) and Na/Si (0.25), providing highest strength for C6 mix amongst other mixes. The oxides interact with each other and provide the alkali-activated product; however, variation in this oxide can affect reactivity greatly.
4. The thermal performance of the alkali-activated binder does not show promising results, as it has higher thermal values that affect the heat-transfer rate. Higher density and lower porosity have an adverse effect on insulation performance of the binder. It is suggested that to enhance the insulation characteristics of the binder developed, it needs to have a porous nature without affecting the mechanical performance.

Thus, based on the conclusions, it can be interpreted that palm oil fuel ash, which is a waste product, can be easily utilized for a meaningful development of the sustainable material.

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Conflict of Interests

On behalf of all authors, the corresponding author states that there is no conflict of interests.

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