

# Fresh and Hardened Properties of Alkali-Activated POFA-GGBFS Pastes Cured in Ambient Temperature – An Initial Mix Design

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#### **1. Introduction**

The presence of weak soils in construction sites is common with the advent of rapid development projects in emerging economic countries such as Malaysia. More specifically, the typical problematic soils encountered in Malaysian construction sites are organic soils, peat soil or soft clayey soil [1], which are characterised by their undesirable characteristics, i.e., low shear strength and high compressibility [2,3]. Soil stabilisation is the process of enhancing the strength and engineering properties of weak soils to ensure that the required geotechnical properties for the development project are met [4]. Typically, weak soil is stabilised using conventional stabiliser materials such as lime and cement, where both materials are associated with the escalation of global greenhouse gas emissions. In particular, Ordinary Portland Cement (OPC) production is responsible for around 7% of carbon dioxide emissions worldwide [5,6]. The ever-increasing use of OPC highlights the need for researchers to explore the use of agricultural waste or industry by-products as supplementary cementitious materials (SCM) for partial replacement of cement [7–9], to reduce the dependency of the construction industry on cement.

In recent years, the use of alkali-activated waste materials as a sustainable construction material has been the focus of researchers aiming to address the Sustainable Development Goals (SDGs), specifically Goal 11: Sustainable Cities and Communities [10,11]. The waste materials repurposed as geopolymer for soil stabilisation are derived from various sources, mainly from the agricultural sector, i.e., palm oil fuel ash [12–14], rice husk ash [15,16], and the industrial sector, i.e., coal fly ash [17–19], ground granulated blast furnace slag [20,21]. Currently, there is a limited number of published works which highlight the effectiveness of using waste-derived geopolymers as viable stabiliser materials for weak soils, and the outcome of this study aims to contribute to the existing body of knowledge.

Therefore, this study aims to ascertain the significant parameters capable of producing the highest compressive strength with adequate workability and setting time of POFA-slag-based geopolymer paste synthesised at ambient temperature, using the mix design outlined in this research project. Based on this approach, it is hoped to show that using waste or by-products from the industrial sector as sustainable soil stabiliser materials with a low carbon footprint is indeed possible. There are three parameters for the optimisation of the POFA-GGBFS geopolymer mix design, as recommended by Chen *et al.,* [21]: Alkali Equivalent (mass percentage of Sodium Oxide from Alkali Activators to the oxide content from the geopolymer source materials), Activator Modulus (molar ratio of Silica to Sodium Oxide in the Alkali Activators), and Slag Replacement (mass ratio of GGBFS to replace POFA). The produced specimens were then subjected to the Vicat test and flow table test to determine their initial and final setting times, workability and the compressive strength test at 28 days curing age to determine the material strength.

#### **2. Methodology**

*2.1 Geopolymer Mix Design and Specimen Preparation 2.1.1 Geopolymer source materials and alkali activators*

The geopolymer paste produced in this study uses two (2) precursor waste materials, namely Palm Oil Fuel Ash (POFA) sourced from Lumadan Mill, Beaufort, Sabah, and commercially available Ground Granulated Blast Furnace Slag (GGBFS). The POFA material was oven-dried, sieved with a 300µm sieve, and ground using a planetary milling machine at 300 rpm for three hours, as recommended by past research [22]. The authors have previously characterised the chemical composition of the POFA and GGBFS materials used in this study using X-Ray Fluorescence (XRF), as

shown in Table 1 below [23]. Table 1 shows that adding GGBFS as source material ensures that the geopolymer mix undergoes setting and hardening at ambient temperature within a reasonable duration due to the presence of Calcium Oxide (CaO). Without the content from GGBFS, Salih *et al.,* [24] reported that the final setting time for alkali-activated POFA synthesised in ambient temperature was recorded at 18 hours after the initial mix, and therefore this is not practical for *in-situ* works. Furthermore, the authors have also established the mean particle sizes ( $d_{50}$ ) for the POFA and GGBFS materials used in this study, which are 17.34 μm and 13.21 μm, respectively [23].

Meanwhile, two types of Alkali Activators were used in this study, namely Sodium Hydroxide (NaOH, or NH) and Sodium Silicate (Na2SiO3, or NS) provided by a local supplier. Based on the data specification sheet, the NH pellets obtained for this study are classified as Analytical Reagent (AR), with a purity level of 99%. Subsequently, the NS solution used in this study has a bulk density of 1410 kg/m<sup>3</sup>, with the following oxide content: Na<sub>2</sub>O = 17%, SiO<sub>2</sub> = 35.4% and 47.6% H<sub>2</sub>O, with a modulus  $(SiO<sub>2</sub>/Na<sub>2</sub>O ratio)$  of 2.1.



**Table 1**

#### *2.1.2 Geopolymer specimen preparation*

As mentioned previously, three parameters were selected to determine the most significant factors influencing the setting time, workability and strength characteristics of the POFA-GGBFS geopolymer paste material. More specifically, the mix design parameters selected comprised of three factors: alkali equivalent ranging from  $8 - 11.5$ %, activator modulus ranging from  $0 - 0.7$ , and slag replacement of POFA fixed at 0.3 (30%). The Liquid/Solid ratio applied for this study was 0.38, as recommended by other researchers [12,16]. These factors and their respective ranges were chosen based on pilot studies conducted prior to the start of this Trial Mix Design testing program. The mixture proportions were determined using the mix design method proposed by Ghosh and Ghosh [25], and a total of eight mixes were designed for this study, as shown in Table 2.

The freshly prepared geopolymer paste was subsequently cast in 50 mm cube iron moulds in two layers, where the specimens were subjected to 30 seconds vibration for each layer, using a vibration table to ensure proper compaction was achieved. The iron moulds were kept at an ambient temperature of 25–30°C for 24 hours before the specimens were de-moulded. Specimens were subjected to air curing, where the specimens were kept in zip lock bags to prevent moisture loss and stored in the laboratory storage area at ambient temperature with a Relative Humidity range of 60– 75 % until the date of testing. Four specimens were prepared for each of the eight (8) mixes, listed in Table 2. Figure 1 shows the POFA-GGBFS geopolymer specimens prepared for TM-Mix 7 and TM-Mix 8, respectively. As seen in the figure, the author notes that specimens containing NS had a shinier appearance due to the silicate content within the geopolymer mix.

#### **Table 2**





\*NOTE: Repl. = Replacement; NH = Sodium Hydroxide; NS = Sodium Silicate; POFA = Palm Oil Fuel Ash; GGBFS: Ground Granulated Blast Furnace Slag



**Fig. 1.** POFA-GGBFS geopolymer specimens for TM Mix-7 and Mix-8

# *2.2 Experimental Methodology*

#### *2.2.1 Vicat test*

The method to assess the setting time of cementitious materials is to determine the initial and final setting time, as detailed in ASTM C191-08 [26] using the Vicat needle. The initial set is defined as the time when the material has achieved partial plasticity, while the final set is the time when the material has reached a state of adequate hardening. To carry out the Vicat needle test, the geopolymer specimen was placed inside a ring mould of 75 mm diameter and 50 mm height. The 1.13 mm diameter needle was used to measure the penetration depth to determine its initial setting time, achieved when the penetration reached a depth of 25 mm from the mould base. Penetration readings for geopolymer were taken at 5-minute intervals. This is due to the nature of the geopolymer, which undergoes rapid setting compared to cementitious materials [27]. Meanwhile, the final setting time is defined as the time taken for the penetration measurement that does not leave a circular impression on the specimen. The elapsed time for both initial and final setting times was measured from the first contact between the geopolymer source materials and the alkali activators.

# *2.2.2 Geopolymer source materials and alkali activators*

The method to evaluate the workability of the POFA-GGBFS geopolymer material is the Flow Table test, as outlined in ASTM C 1437-07 [28], by measuring the spread diameter of the geopolymer sample on the table surface. The flow table used in this study adhered to the standard specified in ASTM C230C-03 [29], with a 255 mm diameter rigid table top. After mixing, the geopolymer paste was poured into the bronze conical mould in two (2) layers, with each layer subjected to tamping for 20 times to ensure proper compaction was achieved. A palette knife was used to remove excess paste

from the top of the conical mould. After one (1) minute, the mould was lifted from the table before the table was dropped 25 times within 15 seconds. The specimen diameter was then measured with a calliper along the four lines etched on the tabletop, and the result of this flow test was expressed as the average of the four readings in millimetres. Figure 2 illustrates the geopolymer specimen after being subjected to the vertical drop of 25 times.

# *2.2.3 Compressive strength test*

For the assessment of the hardened properties of POFA-GGBFS geopolymer paste material, the Compressive Strength test was carried out on 50 mm cube specimens, as detailed in ASTM C109-02 [30]. As stated previously, four samples for all eight (8) mixes were prepared and air-cured until the date of testing. The compressive strength tests were conducted at the curing age of 28 days at an independent testing laboratory (Geospec Sabah Pvt. Ltd.). The testing was performed by crushing the 50 mm cube samples using a Universal Testing Machine (UTM) with a loading rate of 1.8 kN/second, according to ASTM C109-02 [30]. Maximum load (in Newton) at failure was recorded for every specimen, and this value was divided with the cube cross-sectional area (in  $mm<sup>2</sup>$ ) to obtain the sample's compressive strength. However, out of the four available readings, only triplicate readings were considered for each mix to acquire the best average compressive strength.



**Fig. 2.** Spread diameter of POFA-GGBFS geopolymer

# **3. Results and Discussion**

#### *3.1 Fresh Properties*

In this section, the fresh and hardened properties of the POFA-GGBFS geopolymer pastes synthesised based on the proportions shown in Table 2 shall be presented and discussed in detail. The material's fresh properties (setting time and workability) are important to be studied to ensure that the geopolymer can be handled and cast *in-situ* with no issues, as applying the wrong alkali activator dosage may pose problems. More specifically, when the activator content is inadequate, this delays the setting time and does not harden in a timely manner. In contrast, excessive activator content causes flash setting of the synthesised geopolymer. On the other hand, the hardened properties of the geopolymer material were also inspected to ensure that the compressive strength at 28 days curing age is comparable to concrete of C20 (20 MPa) characteristic strength as stated in JKR Standard Specifications for Building Works 2020 [31].

# *3.1.1 Initial and final setting time*

Table 3 shows the results obtained for the Vicat test to determine the initial and final setting time and the Flow Table test results to establish the average flow diameter of the POFA-GGBFS geopolymer paste material. Meanwhile, Figure 3 shows the initial setting time observed for the geopolymer material, with increasing NH Molarity values ranging from 7M to 10M. It can be observed that for the mixes without NS content, the initial setting time typically ranges from 55 to 60 minutes, while the initial setting time was reduced to half (25 to 35 minutes) with the presence of NS.





**Fig. 3.** Initial setting time of POFA-GGBFS geopolymer (sorted by NH molarity)

The same trend of longer setting times was observed for the final setting time of POFA-GGBFS geopolymer, as seen in Figure 4, where the NH mixes had achieved a similar final setting time of 85 minutes. Conversely, for mixes consisting of both NH and NS, the final setting times were reduced to a range of 45 – 60 minutes. The faster setting time phenomenon for specimens containing NS (TM-Mix 2,4,6 and 8) is attributed to the higher silica content that is responsible for the accelerated rate of setting, resulting in shorter setting times and denser gel matrix [14,32,33]. As discussed previously in Section 2.1.1 regarding the Calcium Oxide content in GGBFS, Salih *et al.,* [24] reported a shorter





**Fig. 4.** Final setting time of POFA-GGBFS geopolymer (sorted by NH molarity)

#### *3.1.2 Flow diameter*

Subsequently, the flow table test results for TM-Mix 1 to Mix 8 are shown in Figure 5, with the inclusion of error bars representing the standard errors. In a previous study, Salih *et al.,* [35] reported that for geopolymer mixes made with pure POFA material, the flow of POFA geopolymers experiences a linear reduction in the diameter value as the NH molarity increases. Salih *et al.,* [35] state that the flow diameter reduction at higher NH molarity is attributed to the dissolution of aluminosilicate ions from POFA, which contributes to the higher solid content (in the form of SiO2) and the increase of Na<sub>2</sub>O compounds originating from the NH solution. As such, this consequently increases the solid content, which causes a reduction in the flow diameter.

In the context of this study, a blend of POFA and GGBFS activated with NS and NH produced a different correlation between the alkali concentration and the flow diameter. As seen in Figure 5, the main difference is that the presence of GGBFS changes the flowability characteristics of the geopolymer mix once the alkali activator content is gradually increased. According to Ghosh and Ghosh [25], the inclusion of slag in the geopolymer mix increases its reactivity due to the high surface area of the slag material, and this causes an increase in its flow diameter compared to POFA-based geopolymers.

Referring to Figure 5, we can establish the fact that NH mixes produce lower flow diameters, while NH and NS activated geopolymer mixes produce higher flow diameter readings. According to Xia *et al.,* [17], larger flow diameter readings observed in mixes with both NS and NH activators are attributed to the increased amounts of dissolved aluminosilicate ions, hence causing higher interparticle electrostatic repulsion and eventually increasing the flow diameter.



**Fig. 5.** Flow diameter of POFA-GGBFS geopolymer (sorted by NH molarity)

Meanwhile, the reduction seen in TM-Mix 5 and TM-Mix 6 (from TM-Mix 3 and TM-Mix 4, respectively) despite the high NH concentration of 9M is because the NH was able to fully dissolve the silicate compounds contained in the POFA and GGBFS materials. As stated by Ghosh and Ghosh [25] and Salih *et al.,* [35], elevated amounts of silica within the geopolymer mix decrease the flow diameter since silica is a solid compound that causes the paste to turn stiff. Lastly, an increase in the flow diameter is seen in both TM-Mix 7 and TM-Mix 8 (from TM-Mix 5 and TM-Mix 6, respectively), indicating an excess in the alkali activator compound and high liquid content within the geopolymer mix, a similar finding which was also reported by Kwek *et al.,* [14] at the Liquid/Solid ratio of 0.85.

# *3.2 Hardened Properties*

# *3.2.1 Compressive strength and failure modes*

In this section, the results of the Compressive Strength tests carried out for TM-Mix 1 to Mix 8 are discussed in detail. Table 4 lists the average values of the compressive test results (for triplicate specimens) of the POFA-GGBFS geopolymer material studied in this research project and the failure mode observed for each synthesised mixture. The failure mode observation was based on the guidelines stipulated in MS EN 12390-3: 2009 [36], which defined the types of satisfactory failure in the cube specimens, as shown in Figure 6.



Based on the data presented in Table 4, the plotted graphs for Compressive Strength with correlation to the alkali activator content and dosage are illustrated in Figure 7, with the standard error shown in the form of error bars. As seen in Figure 7, it is apparent that the mixes comprised of only NH yielded low compressive strength values, ranging from 13.87 MPa (TM-Mix 5) to 22.80 MPa (TM-Mix 1) at 28 days curing age. Only two mixes activated with NH had achieved the target strength of 20 MPa (C20) set for this study, namely TM-Mix 1 with 22.80 MPa and TM-Mix 7 with 21.20 MPa. Unfortunately, the compressive strength values at 28 days curing age recorded for TM-Mix 3 (19.17 MPa) and TM-Mix 5 (13.87 MPa) fell short of the target strength value. Furthermore, it was observed that the failure mode for NS-activated geopolymer materials had a satisfactory failure of the nonexplosive and semi-explosive failure modes.



**Fig. 6.** Satisfactory failure of cubes: (a) non-explosive (b) semiexplosive and (c) explosive failure modes [36]



**Fig. 7.** Compressive strength of POFA-GGBFS geopolymer 28 days curing age (sorted by NH molarity)

Based on Figure 7 above, the strength values recorded for TM-Mix 1,3,5 and 7 indicate that the chosen NH Molarity range of 7 to 10M used in this study is considered inadequate to properly dissolve the  $Al^{3+}$  and  $Si^{4+}$  ions from the geopolymer source materials. When the NH molarity is weak, this adversely affects the formation of geopolymer gels that contribute to the material's overall strength [17]. To overcome this problem, the NH concentration can be increased from 10M to 18M, as demonstrated by Hanjitsuwan *et al.,* [37], that managed to record the compressive strength value of 56 MPa for single source alkali (18M NH) activated geopolymer.

However, the use of both NH and NS as alkali activators in geopolymer materials is a better solution since Kwek *et al.,* [14] have reported that excessive NH content in single-source alkaliactivated geopolymers causes delays in setting time and adversely affects the geopolymerisation process. At the same time, Ghosh and Ghosh [25] stated that surplus NH solutions lead to flash setting and hinder the proper formation of C-S-H gels, resulting in lower compressive strength values for the compound. The advantages of dual-source alkali activators are clearly evident, as seen in the compressive strength values reported for the POFA-GGBFS geopolymer pastes activated with both NH and NS, which will be discussed in the subsequent paragraphs.

As seen in Table 4, a marked improvement in the compressive strength values was observed for specimens activated with both NH and NS, except for TM-Mix 8, which recorded a relatively low average strength value of 22.30 MPa. In particular, TM-Mix 2 produced specimens with the highest average compressive strength value of 67.27 MPa, followed by TM-Mix 4 with an average value of 60.57 MPa, and TM-Mix 6 with an average value of 53.57 MPa. This strength enhancement can be attributed to the higher content of soluble silicate originating from the dissolution of POFA and GGBFS materials by the NH solution and the silicate compounds contributed by the addition of NS in the geopolymer mix. More specifically, Duxson *et al.,* [27] explained that the elevated levels of soluble silicate play a role in the formation of oligomers and promote the polycondensation process, which subsequently improves the hardened properties of the synthesised geopolymer paste. Another consequence of the improved hardened properties of POFA-GGBFS geopolymer is seen in its failure mode: the specimens activated by NH and NS mostly failed in the explosive mode, an indication that the material has higher stiffness compared to the specimens produced with NH activator only [33].

Conversely, for TM-Mix 8 (at 10M NH and 0.5 NS/NH ratio), the compressive strength of 22.3 MPa was the lowest recorded strength value amongst all specimens activated with both NH and NS solutions. According to Kwek *et al.,* [14], the subpar compressive strength is attributed to the high liquid content within the geopolymer mix, as the excess liquid from both NH and NS solutions increases the void volume within the specimen. In addition, Xia *et al.,* [17] and Chen *et al.,* [21] reported the adverse effects of excessive NS content within the geopolymer mix: they stated that the increased amounts of silica cause the rapid formation of geopolymeric gels, which quickly adheres to the aluminosilicate materials and stops the dissolution of the remaining geopolymer material, eventually disrupting the formation of geopolymer gels and producing specimen with lowered viscosity and lower compressive strength.

From the detailed discussions on the alkali activator dosage and types, it can be concluded that TM-Mix 2 is the optimum alkali activator content, producing the highest average compressive strength value of 67.27 MPa with adequate workability and setting times (226 mm flow diameter, 30 minutes initial setting time and 45 final setting time) for the synthesis of geopolymer in ambient temperature conditions. As seen in Figure 7, it is also evident that an increase in the NH Molarity with the presence of NS solution in the geopolymer mix adversely affects the compressive strength, which may be attributed to the high amount of alkaline activator solutions that deter the geopolymerisation process [38].

# **4. Conclusions**

In this paper, the study on POFA-GGBFS binary waste blends activated with two types of alkali activators, Sodium Hydroxide (NH) and Sodium Silicate (NS), to produce POFA-GGBFS geopolymer pastes synthesised at ambient temperatures was examined to assess its suitability as a weak soil stabiliser material. The following conclusions were made based on the findings of this study:

- i. Two parameters, Alkali Equivalent and Activator Modulus, were studied by examining their fresh properties (setting times and workability) and hardened properties (compressive strength) to determine the most suitable mix design with adequate strength, satisfactory setting times and acceptable workability. The third parameter, slag replacement, was fixed at 30%.
- ii. Single-source alkali activators (NH only) used on POFA-GGBFS produced specimens with smaller flow diameters, longer initial and final setting times, and lower compressive strength at 28 day curing age compared to specimens activated with both NH and NS.
- iii. Geopolymer specimens synthesised with NH and NS produce stiffer compounds, characterised by higher compressive strength values and explosive-type failure modes, due to the higher silica content within the geopolymer material.
- iv. The highest compressive strength POFA-GGBFS geopolymer paste was achieved for TM-Mix 2 (7% alkali equivalent and 0.7 activator modulus) with 67.3 MPa after 28 days curing period, with 227 mm flow diameter, initial setting time of 25 minutes and final setting time of 45 minutes.
- v. The results of this study show that the preliminary mix design of POFA-GGBFS geopolymer paste was capable of producing material with acceptable strength, suitable setting times and sufficient workability. In the near future, further experiments will be conducted to determine the optimised mix design of this geopolymer material for the purpose of weak soil stabilisation.

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