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Technical Paper

Optimization of factors affecting the silicate modulus of home–brewed sodium silicate solution from industrial & agro ashes

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Abstract: This study investigates the development of geopolymer mortars using a greener alternative to commercial silicate can be produced. Home-brewed sodium silicate (HBSS) solutions were synthesized via a hydrothermal method using agro-industrial byproducts like rice husk ash (RHA) and palm oil fuel ash (POFA) as silica sources. These materials offer high silica content (approximately 90% for RHA and 60% for POFA) compared to traditional activators, potentially reducing reliance on energy-intensive commercial processes. The research explores the influence of various parameters on the silicate modulus (SM) of the HBSS solution, including NaOH solution molarity, stirring time, and thermal treatment of the ash materials. Geopolymer mortars were then formulated using GGBS/FA blend as the main binder and the HBSS solutions as activators. The results demonstrate that the optimal combination of NaOH concentration and stirring time for achieving maximum compressive strength depends on the ash source. Ground RHA achieved a maximum strength of 44.7 MPa with a 5M NaOH solution and 1 hour of stirring, while treated ground POFA reached a higher strength of 51.6 MPa using a 3M NaOH solution and 3 hours of stirring. The higher silica content and amorphous silica fraction in RHA likely contribute to the observed differences in optimal SM values between the ash sources. This research paves the way for utilizing waste materials and a greener activator production method for the development of strong and sustainable geopolymer mortars.

1. Introduction

The construction industry is vital to global economies, with concrete being the most widely used building material, consumed at a rate of 1 m³ per person annually (Turner & Collins, 2013). However, concrete production is responsible for significant waste and greenhouse gas (GHG) emissions, with cement production contributing around 6% of global anthropogenic GHG emissions (Almutairi et al., 2021). The extraction of raw materials for cement also leads to environmental degradation. Geopolymers, introduced by Davidovits in 1978 (Davidovits, 1988), offer a promising alternative to traditional cement-based concrete. These materials are made by combining aluminosilicate sources with alkaline activators such as sodium hydroxide (NaOH). Geopolymer concrete (GPC) has advantages over conventional concrete, including reduced CO_2 emissions, enhanced mechanical strength, fire resistance, and long-term durability. GPC can cut CO_2 emissions by up to 80% compared to ordinary Portland cement (Singh Rajput et al., 2023). However, its adoption is hindered by high costs,

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limited awareness, and a lack of standardization (Li et al., 2019). A major challenge in geopolymer concrete (GPC) is the cost of alkaline activators, such as sodium silicate. Traditional sodium silicate is produced by heating silica and sodium carbonate at high temperatures, which is energy-intensive and contributes to CO_2 emissions. Additionally, excessive sand mining for silica harms ecosystems. Using industrial by-products like Rice Husk Ash (RHA) and Palm Oil Fuel Ash (POFA) as silica sources for sodium silicate production presents a more sustainable, cost-effective alternative, reducing both costs and environmental impact (Hadria Ghanim et al., 2025; Alnahhal et al., 2025).

The strength of sodium silicate is influenced by its silicate modulus (SM), a ratio of SiO_2 to Na_2O. Optimizing this ratio can improve the properties of the resulting geopolymer. This research aims to explore the synthesis of sodium silicate from RHA and POFA using a hydrothermal method and examine factors such as NaOH concentration, stirring time, and thermal treatment on the silicate modulus. The objectives of this experimental work were the following: a) To synthesize home-brewed sodium silicate (HBSS) using RHA and POFA as silica sources by the hydrothermal method. b) To optimize the silicate modulus (SM) values of different HBSS solutions and evaluate their impact on geopolymerization. c) To investigate the flow value and compressive strength of geopolymer mortar specimens prepared with HBSS and analyse their microstructural properties.

This research focused on the synthesis of sodium silicate using RHA and POFA as precursor materials through the hydrothermal method. The study has evaluated the effect of NaOH concentrations (5M and 10M), stirring times (1 hour and 3 hours), and thermal treatment (500°C) on the silicate modulus. The experiments have been conducted at a controlled temperature of 80°C with a stirring speed of 200 rpm to ensure consistent reaction conditions. The research has also explored the impact of these variables on the performance of geopolymer concrete, including compressive strength, flow value, and microstructural properties. The goal is to identify optimal conditions for sodium silicate production using waste-derived silica sources, reducing energy consumption, CO_2 emissions, and production costs.

2. Methodology

This research investigated methods to optimize factors affecting the silicate modulus (SM) of geopolymer mortars. Following a literature review, materials like rice husk ash (RHA), palm oil fuel ash (POFA), and activators were collected. Home-brewed sodium silicate (HBSS) solutions were synthesized using these ashes under hydrothermal conditions at various stirring times with 5M or 10M NaOH. The SM of the HBSS solutions was determined using X-ray fluorescence (XRF). Subsequently, geopolymer mortars were cast using the HBSS solutions and tested for fresh properties and compressive strength at different curing times. Based on the test results, the mortar mix design was optimized to achieve maximum compressive strength.

Based on Fig.1 above, the process began with a review of existing literature, providing the necessary background and context. After the literature review, materials are collected, including a silica source, binders (Fly Ash and GGBS), alkaline activators, and fine aggregates. The silica source is combined with Ground and treated Rice Husk Ash (RHA) or Palm Oil Fuel Ash (POFA). For the alkaline activators, both sodium hydroxide (NaOH) solutions of 5M and 10M concentration and commercial sodium silicate are prepared.

The first objective was to synthesize HBSS solution mixes using RHA and POFA as a silica source by the hydrothermal method with various parameters and silicate modulus (SM) values. the parameters set were NaOH to silica ratio of 2, a temperature of 80°C, and a stirring speed of 200 rpm. Factors influencing this process were the molarity of the NaOH solution, the use of Ground & treated RHA and POFA, and the stirring time (either 1 or 3 hours). The HBSS solution was then filtered using a centrifuge process. The silicate modulus of the HBSS solution was determined via X-ray fluorescence (XRF) analysis. After that, it was examined that the crucial factors and their optimization on the SM values of different HBSS solutions. The first and second objectives could be achieved.

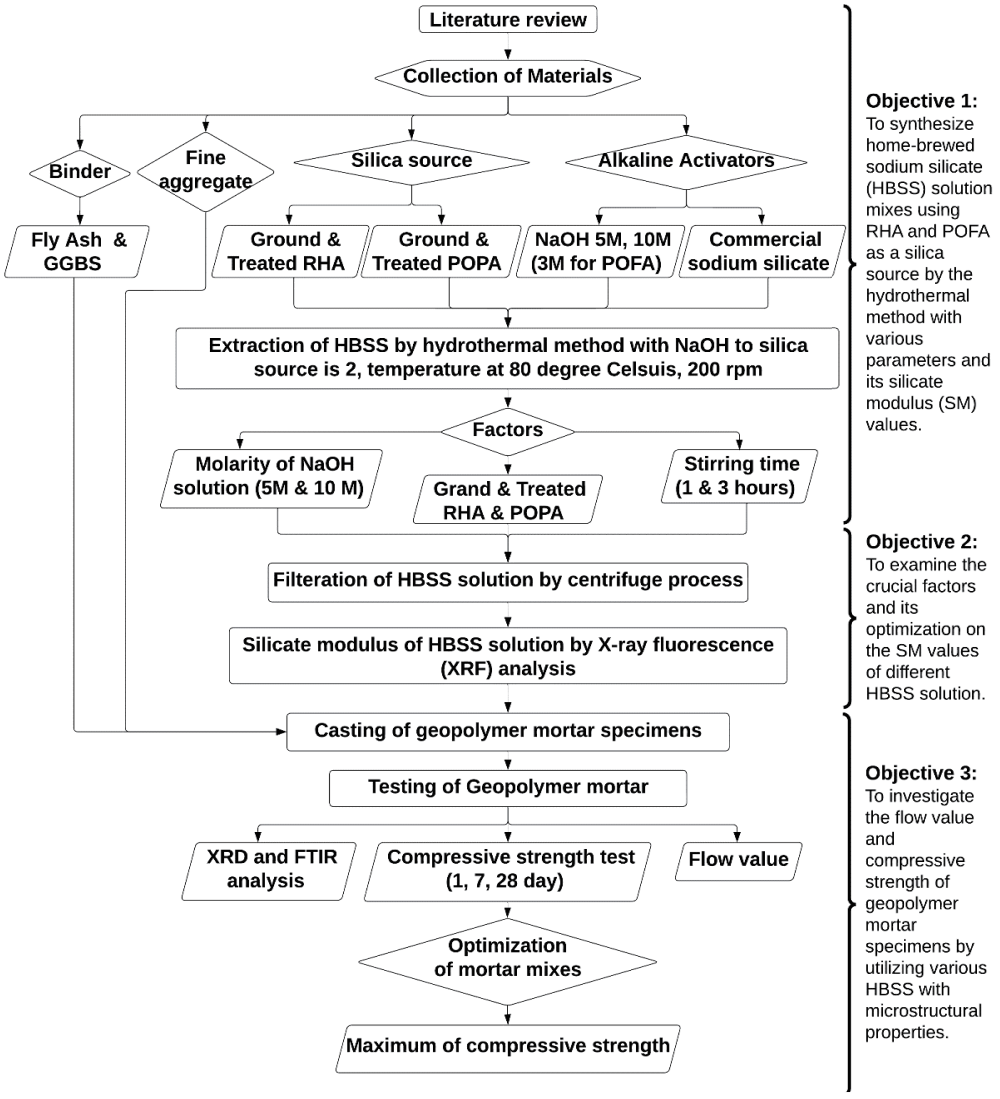


Fig.1. Flowchart of research work

The geopolymer mortar was then to be cast and underwent testing for fresh density and flow value. The mortar was subjected to a compressive strength test at intervals of 1, 7, and 28 days. Based on the test results, the mortar mixes were optimized. The final objective was to achieve the maximum compressive strength from the optimized mortar mixes.

2.1 Materials testing

Sieve analysis of fine aggregate was conducted by ASTM C136/C136M – 14 to determine the particle size distribution. A representative sample of the fine aggregate was first dried to a constant weight, and then sequentially passed through a series of standard sieves with varying mesh sizes. The material retained on each sieve was weighed, and the percentage passing each sieve was calculated. The results were used to assess the gradation of the fine aggregate, ensuring its suitability for the intended application in the geopolymer mix.

Specific gravity the specific gravity test using a Le Chatelier flask, as per ASTM standards (e.g., ASTM C128 for fine aggregates), involved measuring the volume of a sample by displacement of water. The sample was placed in the Le Chatelier flask, and water was added to fill the flask. The weight of the displaced

water was then measured, and the specific gravity was calculated by comparing the mass of the sample to the volume of water displaced. This method was commonly used for fine aggregates and provides a reliable measure of the material's density relative to water.

2.2 Preparation of 5M or 10 M of sodium hydroxide solution (1 L)

Grinding treatment: The silica source materials such as Rice husk ash (RHA) and Palm oil fuel ash (POFA) are ground to make finer particles using Los-Angeles abrasion machine with a capacity of 10 kg and it was designated as GRHA and GPOFA.

Thermal treatment: The thermal treatment process involves heating Ground Rice Husk Ash (GRHA) or Ground Palm Oil Fuel Ash (GPOFA) at 500°C for 1.5 hours to produce Treated Ground RHA (TGRHA) or Treated Ground POFA (TGPOFA). This treatment modifies the properties of the ashes, enhancing their suitability for applications like sodium silicate synthesis. The process includes weighing the materials, gradually heating them in a controlled furnace to 500°C, maintaining the temperature for 1.5 hours, and then cooling the treated ashes for storage until further use.

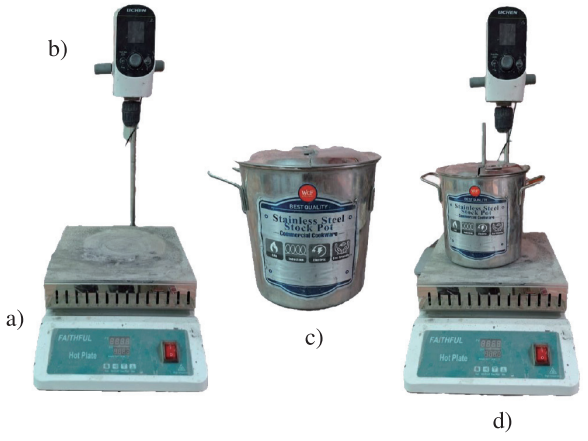


Fig. 2. Apparatus for extraction of sodium silicate solution, a): Hot plate, b) Stirrer, c) Reactant container, d) Assembly.

Extraction of Home-Brewed Sodium Silicate Solution: Fig.2 shows the apparatus of the silicate extraction. For the extraction of Home-Brewed Sodium Silicate Solution (HBSS), the hydrothermal method was employed. Table 1 Mix proportion for HBSS in this research. Ground Rice Husk Ash (GRHA), Ground Palm Oil Fuel Ash (POFA), TGRHA, and TGPOFA (Fig.3) are mixed with Sodium Hydroxide (NaOH) solutions at concentrations of 5M, 10M, and 3M for POFA testing. The mixture was heated to 80°C and stirred at 200 rpm in a stainless-steel pot to ensure uniformity in the reaction. The resulting HBSS is then stored for casting or further lab testing.

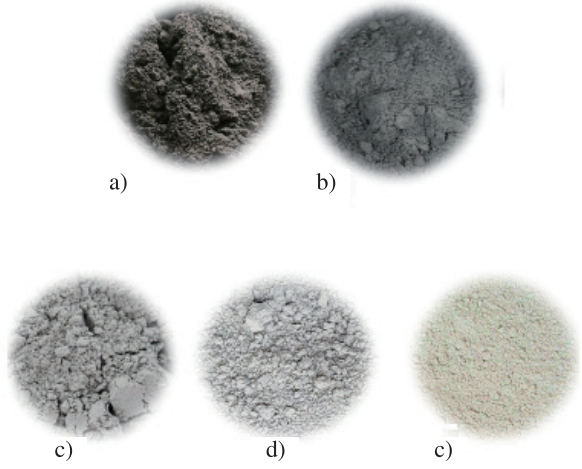


Fig. 3. Material for Extraction of Sodium Silicate Solution, a): Ground Rice Hush Ash (GRHA), b) Ground Palm Oil Fuel Ash (POFA), c&d) Treated Ground Rice Hush Ash (TGRHA), e) Treated Ground Palm Oil Fuel Ash (TPOFA),

Table 1 Mix proportion for HBSS

No	Binders		NaOH solution /Silica sources	Molarity of NaOH solution for HBSS preparation	Stirring temperature (°C)	Stirring time (hr)
1	Palm Oil Fuel Ash (POFA)	Treated Ground POFA (TGPOFA)	2	5M	80	1 hr
		Ground POFA (GPOFA)				
2	Rice Husk Ash (RHA)	Treated Ground RHA (TGRHA)		10M		3 hr
		Ground RHA (GRHA)				

2.3 Casting for Geopolymer Mortar Specimens

Table 2. Mix design for casting

No	Specific gravity		Ratios (to binder)		Weight (kg/ m^3)	
1	FA	2.55	Fly Ash (20%)	1	100	500
2	GGBS	2.88	GGBS (80%)		400	
3	SS	1.3	SS solution/binder	0.35	175	
4	Water	1	Water/ binder	0.35	175	
5	Mining sand	2.65	Sand/ binder	2.6	1300	
6	SH (10M)	1.2	SS/ SH ratio	1.0	175	

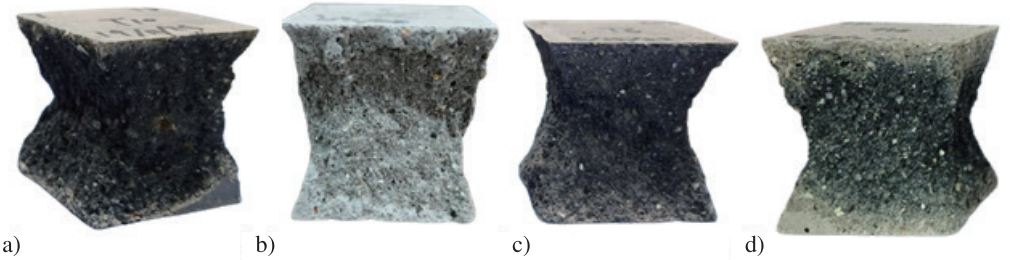
Table 3. Mix proportions of mining sand

Mix proportions of sand				
1.18 mm	0.6 mm	0.3 mm	0.15 mm	Total (g)
25%	35%	20%	20%	1682
420.5	588.7	336.4	336.4	

Mix Design: Due to the lack of a standard code for geopolymer concrete, assumptions are made based on conventional concrete practices. As shown in Tables 1 and 2, a binder content of 500 kg/m^3 is selected, with 20% fly ash and 80% Ground Granulated Blast Furnace Slag (GGBS) for optimal setting time and workability. The Home-Brewed Sodium Silicate Solution (HBSS) to binder ratio is set at 0.35, after trials showed that higher ratios (10-15%) resulted in lower strength. The water to binder ratio starts at 0.35 for better flowability but was later adjusted due to strength optimization with POFA-based HBSS mixes. The sodium silicate to sodium hydroxide ratio was set at 1 for environmental benefits. Different sand sizes were used to prevent high porosity and ensure optimal mix quality.

2.4 Compressive strength

A universal compression testing machine of a capacity of 2000kN was used for the compression test for cube specimens with a pacing speed of 0.5 kN/sec.



a) b) c) d)

Fig.4. Compressive test specimens, a) Ground Rice Hush Ash (GRHA) mortar, b) Treated Ground Rice Hush Ash (TGRHA) mortar c) Ground Palm Oil Fuel Ash (POFA) mortar, d) Treated Ground Palm Oil Fuel Ash (TPOFA) mortar,

3. Result

3.1 Material Test

3.1.1 Size distribution

Significance and Use of Fine Aggregate Grading: The particle size distribution of fine aggregates, analysed via sieve analysis, is crucial for obtaining a uniform concrete mix. Proper grading minimizes voids, improving concrete strength and durability. Well-graded aggregates reduce the amount of paste required, lowering binder usage and cost while enhancing workability and stability.

Significance of Fineness Modulus of Fine Aggregate: The fineness modulus indicates the average size of fine aggregates, influencing concrete mix design. A higher modulus corresponds to coarser aggregates, requiring less paste and resulting in stiffer mixes with lower slump values. An optimal fineness modulus range of 2.5–3.2 ensures adequate workability and strength, with a measured value of 3.1 in this study.

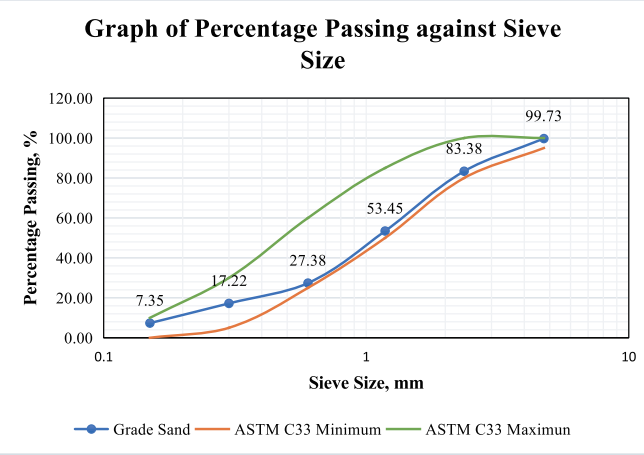


Fig. 5. Graph of Percentage Passing Against Sieve Size

Effects of Sand Grading on Mortar and Concrete Properties: Fig.5 shows that sand grading confirms the standard ASTM C 33; it is well established that sand grading impacts mortar and concrete properties significantly. Poorly graded sand with excess fines requires more water for workability, potentially leading to segregation. Finer sand reduces compressive strength, increases shrinkage, and weakens tensile bond strength in masonry. Proper grading minimizes voids, reduces cement needs, and improves strength.

Analysis of Specific Gravity of RHA: Rice Husk Ash (RHA) had a specific gravity of 2.14, indicating it was denser than water. This property improved the packing of concrete, reducing porosity and enhancing strength and durability. RHA's higher specific gravity helped fill voids, leading to a denser, more robust material. However, other factors such as particle size and chemical composition also influenced its performance in concrete mixes.

Viscosity Test: Dynamic viscosity, measured in MPa.s, assesses the fluid resistance of solutions. Higher viscosity indicates thicker solutions, which impacts mortar workability and performance. Optimal viscosity ensures smooth flow, facilitating proper filling of formwork and uniform reinforcement coverage. Table 4 shows the dynamic viscosity of different sodium silicate solutions.

Table 4. Dynamic Viscosity based on difference solution

Mix ID	Solution			Dynamic Viscosity (MPa.S)
	Binder	NaOH Molarity, M	Stirring time, hr	
T15	TGPOFA	10	3	4341.5
T16	TGPOFA	5	3	87.7
T14	TGPOFA	10	1	203
T11	TGPOFA	5	1	76.1
T13	TGRHA	10	3	2066
T12	TGRHA	5	3	52
T19	GPOFA	3	1	26.2
T20	GPOFA	3	3	12.1
T21	TGPOFA	3	1	14.4
C1	Commercial Sodium Silicate			12525
	NaOH	10		9.93
	NaOH	5		5.08

3.2 Observations and Discussion

3.2.1 Effect of Concentration

Higher concentrations of NaOH (higher M values) resulted in higher viscosities. For instance, T15 (TGPOFA with 10M NaOH) had a high viscosity of 4341.5 mPa.s, whereas T16 (TGPOFA with 5M NaOH) had a low viscosity of 87.7 mPa.s. This is due to more particles per unit volume or the presence of sediments, leading to greater resistance to flow. Higher NaOH concentrations promote the dissolution of silica and the formation of more silicate species, creating a thicker solution. According to Viscosity of NaOH: Understanding Its Influence on Industrial Processes - TechieScience (2022), higher NaOH concentrations generally increased viscosity because the greater number of NaOH molecules strengthens intermolecular forces, making the solution more resistant to flow.

3.2.2 Effect of Stirring Time

All the solution showed an increase in viscosity with longer stirring times due to the enhanced interaction between silica particles and the alkali solution. As stirring continued, silica particles disperse more uniformly throughout the solution, forming a stable colloidal system. This uniform dispersion increased the

viscosity because the solution became denser and more cohesive, thus resisting flow more effectively. However, GPOFA solutions showed a decrease in viscosity with longer stirring times (e.g., 3 hrs vs 1 hr). This could be due to breaking down larger particles or removing trapped air bubbles during stirring. For example, comparing T15 (TGPOFA 10M, 3 hrs) with T14 (TGPOFA 10M, 1 hr), the one with a longer reaction time (T15) had a higher viscosity.

The table allows for comparing the viscosity of TPOFA with GPOFA and TGRHA. This comparison can be useful in understanding how the material type and processing affect flow behavior. In short, NaOH concentration and reaction time might influence the viscosity of sodium silicate solutions prepared by the hydrothermal method. Result of the compressive strength of geopolymer mortar specimens

4. Discussion

4.1 Ground Rice Husk Ash (GRHA)

Workability (Fresh Properties)

Increasing the molar concentration of sodium hydroxide (NaOH) from 5M to 10M (T1 and T4) resulted in an increase in the flow value from 157.75 mm to 201.75 mm as shown in Table 6. This indicates that higher NaOH concentration improved the workability of the mix, allowing for better flow and ease of handling.

Table 5 Standard Deviation error for compressive strength

Mix ID	Binders	Average compressive strength and standard deviation (Day)					
		1		7		28	
T1	GRHA	26.2	±1.53	36.0	±1.25	44.7	±1.44
T5	GRHA	20.6	±1.76	36.5	±2.52	43.8	±2.08
T4	GRHA	18.1	±1.44	28.8	±1.53	41.1	±1.25
T7	GRHA	16.8	±1.15	30.9	±1.73	35.5	±1.15
T2	GPOFA	6.4	±0.76	10.9	±0.88	15.3	±0.58
T6	GPOFA	8.6	±0.88	16.2	±1.15	19.2	±0.76
T3	GPOFA	9.4	±0.76	13.4	±0.58	18.1	±0.44
T8	GPOFA	9.5	±0.58	13.2	±0.76	19.5	±1.15
T10	TGRHA	17.4	±1.15	32.0	±1.00	38.2	±1.25
T12	TGRHA	20.8	±1.25	32.6	±1.53	42.3	±1.15
T9	TGRHA	19.7	±1.44	33.5	±1.25	36.8	±0.88
T13	TGRHA	18.2	±1.15	31.9	±1.15	36.9	±1.53
T11	TGPOFA	7.7	±0.76	14.2	±0.76	15.4	±0.58
T16	TGPOFA	9.2	±0.58	11.5	±0.44	14.0	±0.58
T14	TGPOFA	5.3	±0.58	8.5	±0.76	10.1	±0.76
T15	TGPOFA	11.2	±1.15	17.3	±1.73	24.2	±1.25

Table 6. Overall result for 16 basic mixes

Mix ID	Variables for sodium silicate			Average compressive strength (MPa)			Fresh density	Avg flow value (mm)	SM
	Binders	Molarity of NaOH solution for HBSS preparation (M)	Stirring time (hr)	Day 1	Day 7	Day 28			
T1	GRHA	5	1	26.2	36.0	44.7	2.7	157.75	2.3
T5	GRHA	5	3	20.6	36.5	43.8	2.19	196.25	2.1
T4	GRHA	10	1	18.1	28.8	41.1	2.14	201.75	1.98
T7	GRHA	10	3	16.8	30.9	35.5	2.11	> 250	1.6
T2	GPOFA	5	1	6.4	10.9	15.3	2.6	> 250	1.04
T6	GPOFA	5	3	8.6	16.2	19.2	2.11	> 250	1.5
T3	GPOFA	10	1	9.4	13.4	18.1	2.12	> 250	1.17
T8	GPOFA	10	3	9.5	13.2	19.5	2.20	212.25	1.1
T10	TGRHA	5	1	17.4	32.0	38.2	2.17	175.5	1.8
T12	TGRHA	5	3	20.8	32.6	42.3	2.16	141.25	2.15
T9	TGRHA	10	1	19.7	33.5	36.8	2.18	182.5	2.05
T13	TGRHA	10	3	18.2	31.9	36.9	2.18	200.29	1.97
T11	TGPOFA	5	1	7.7	14.2	15.4	2.21	> 250	0.96
T16	TGPOFA	5	3	9.2	11.5	14.0	2.23	> 250	1.07
T14	TGPOFA	10	1	5.3	8.5	10.1	2.23	> 250	1
T15	TGPOFA	10	3	11.2	17.3	24.2	2.25	230	1.3

Note: NaOH solution/Silica sources is 2

4.2 Compressive Strength

Increasing the NaOH concentration from 5M to 10M lead to a decrease in compressive strength. For GRHA, there was no significant difference in compressive strength between 5M and 10M NaOH with 1 hour of stirring time in Figure 4.2 below. Thus, 5M NaOH with 1 hour stirring (Mix T1) is sufficient to achieve the maximum compressive strength of 44.7 MPa. According to the research (Mejía et al., 2013), Rice Husk Ash (RHA) can replace commercial sodium silicate in geopolymer binders, achieving 7-day strengths of around 42 MPa, with both amorphous

and some crystalline silica in RHA contributing to the activation process. An increase in flow value resulted in decreased compressive strength can be shown in the Table 4-1 above.

4.3 Treated Ground Rice Husk Ash (TGRHA)

The optimal mix for TGRHA was achieved with 5M NaOH and a stirring time of 3 hours in Mix T12, which resulted in a maximum compressive strength of 42.3 MPa from the Figure 4-3 below.

4.4 Ground Palm Oil Fuel Ash (GPOFA)

The compressive strength tests showed that the lowest strength of 15.3 MPa was observed in Mix T2, which used 5M NaOH with a stirring time of 1 hour. In contrast, the highest compressive strength of 19.5 MPa was achieved in Mix T8, which utilized 10M NaOH and a stirring time of 3 hours. These results indicate that higher NaOH molarity and longer stirring times may enhance the compressive strength of the sodium silicate solution mixes. (Salih et al., 2013)

4.5 Treated Ground Palm Oil Fuel Ash (TGPOFA)

According to Fig. 6, the lowest compressive strength observed was 10 MPa in Mix T14 with 10M NaOH and 1 hour of stirring time, while the maximum compressive strength is 24.2 MPa in Mix T15 with 10M NaOH and 3 hours of stirring time.

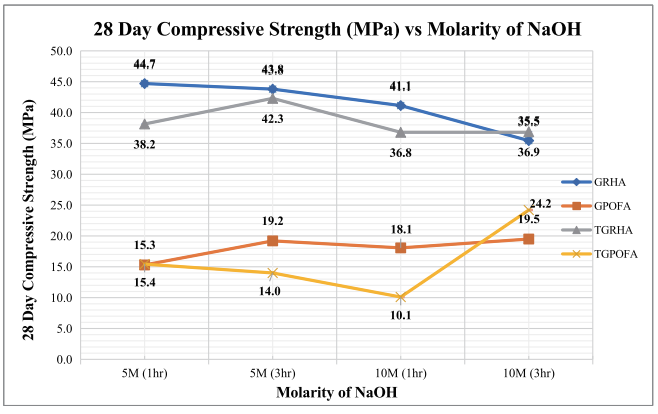


Fig.6. 28-Day Compressive Strength (MPa) vs Molarity of NaOH

4.6 Effect of NaOH molarity on compressive strength for silicate preparation and geopolymer mortar casting

10M to 5M NaOH: When reducing the NaOH molarity from 10M to 5M for both silicate preparation and geopolymer mortar casting, the maximum compressive strength achieved was only of 20 MPa (Mix T6a) in Table 7. Even with this reduction, the strength was not significantly different, indicating that 10M NaOH was not necessary for achieving the desired strength.

5M to 3M NaOH: By further reducing the NaOH molarity from 5M to 3M for silicate preparation, while keeping 5M constant for casting, the maximum compressive strength reached was 51.6 MPa with a 3-hour stirring time (Mix T18) according to Fig.7. The drop in NaOH molarity for silicate preparation, combined with the constant 5M for casting, had resulted in a significant increase in compressive strength.

Table 7. Overall result for POFA in reducing the molarity of NaOH

Mix ID	Variables for sodium silicate			Average compressive strength (MPa)			Fresh density	Avg flow value (mm)
	Binders	Molarity of NaOH solution for HBSS preparation (M)	Stirring time (hr)	Day				
				1	7	28		
T8	GPOFA	10	3	9.5	13.2	19.5	2.20	212.25
T6	GPOFA	5	3	8.6	16.2	19.2	2.11	> 250
T6a	GPOFA	5	3	9.5	18.8	20.7	2.19	> 250
T19	GPOFA	3	1	7.2	14.3	19.1	2.23	227.25
T20	GPOFA	3	3	20.8	36.6	43.4	2.18	210.5
T21	TGPOFA	3	1	11.0	18.9	25.4	2.22	175.5
T18	TGPOFA	3	3	21.1	41.5	51.6	2.20	219.5

Note: NaOH solution/Silica sources is

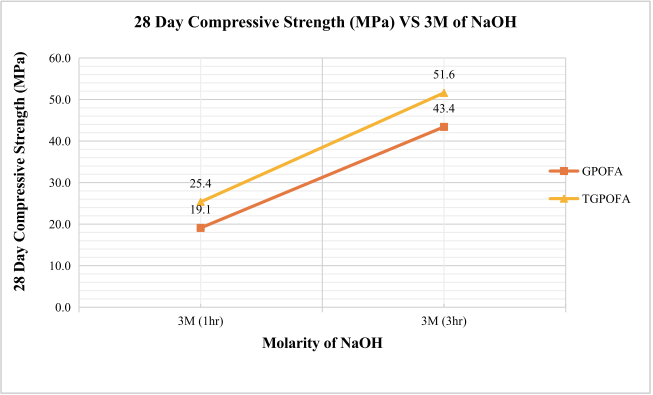


Fig. 7. 28-Day Compressive Strength (MPa) VS 3M of NaOH

4.7 Microstructural property

X-ray fluorescence (XRF) analysis was used to determine the elemental composition of a material. It works by bombarding a sample with high-energy X-rays, which excite the atoms in the sample. These excited atoms then emit fluorescent X-rays of specific energies, characteristic of the elements present. By measuring the energy and intensity of these fluorescent X-rays, an XRF spectrometer can identify and quantify the elements in the sample. Silicate Modulus (SM) value can be determined using this analysis by obtaining SiO_2 and Na_2O contents from the liquid powder of the HBSS. It was expected that the solution with the highest SiO_2 content would provide the highest binder activation and compressive strength (Alnahhal et al., 2023).

POFA and RHA are preferred for HBSS solution preparation due to their high Silicon Dioxide (SiO_2) content. The SiO_2 content in POFA varies between 50% and 80% depending on burning processes and palm fruit components (Joshi et al., 2021) As shown in Table 8 and Fig. 8, RHA has a higher SiO_2 content of 90.1%. This acidic SiO_2 likely reacts with other HBSS components, adjusting the solution's pH to the optimal range for cell cultures. Fly Ash and GGBS, byproducts of coal combustion and steel production respectively, are not typically used in HBSS due to their focus on concrete applications through pozzolanic properties (Alnahhal et al., 2024). It is important to note that the ideal SiO_2 content for HBSS might depend on the desired final pH and specific recipe, and other factors like impurities in the ash can also influence its suitability.

Table 8. Percentage components of the Ashes

Components	RHA	Treated RHA (500c)	Treated POFA-500c	POFA	FA	GGBS
SiO_2	90.1	89.2	60.3	54.3	45.5	29.3
Al_2O_3	0.481	0.432	1.33	1.83	20.9	14.2
CaO	1.75	2.12	8.9	11.9	8.69	43.3
Fe_2O_3	0.952	1.29	5.06	10.7	16.9	0.5
K2O	3.33	3.25	14.5	12.5	1.67	0.484
SO3	0.372	0.756	1.15	0.943	1.19	2.09
MgO	0.316	0.945	2.75	1.71	1.33	7.82

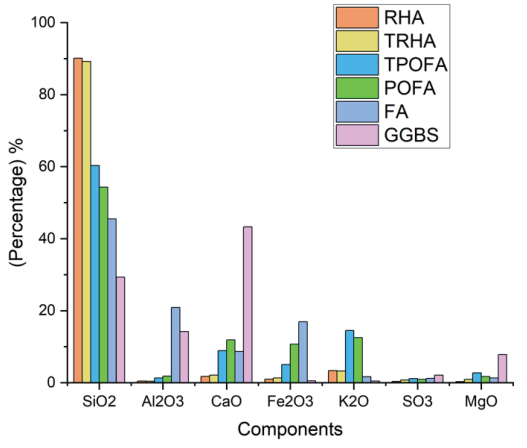


Fig. 8. Percentage components of the Ashes

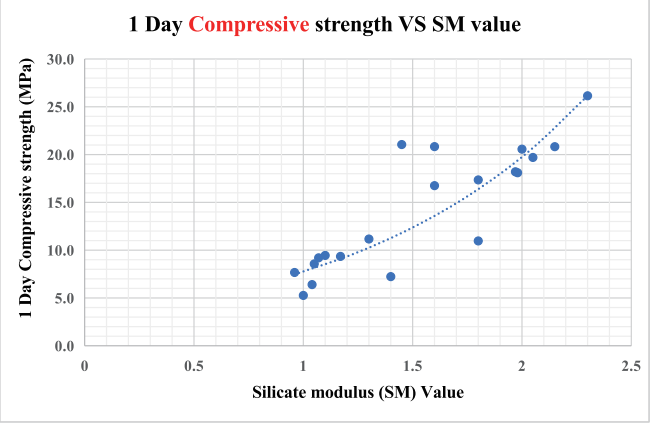


Fig.9. 1-Day Compressive strength Vs SM value

4.8 SM values of HBSS solution

Fig. 9 shows that increasing the NaOH concentration from 5 to 10 M, and reducing the SM values from 2.3 to 1.98, led to higher viscosity and lower flowability in geopolymer mortar. For RHA-derived HBSS solution, specimen T1 achieved a 28-day compressive strength of 44.7 MPa with an SM value of 2.3 and an average flow of 157.75 mm. Thermal treatment of ground RHA had little effect on mortar properties, with specimen T12 reaching a compressive strength of 42.3 MPa, an SM value of 2.15, and a flow of 141.23 mm. In contrast, palm oil fuel ash (POFA)-derived HBSS solution showed lower compressive strength, with specimen T8 reaching only 19.5 MPa due to a lower SM value (1.1) and SiO_2 content (32%). Thermal treatment of POFA (specimen T15) improved compressive strength to 24.2 MPa with an SM value of 1.3, and the optimal NaOH concentration for TGPOFA was 3 M, achieving a maximum compressive strength of 51.6 MPa.

X-ray diffraction (XRD) analysis is used to determine the crystalline structure of materials, providing complementary information to X-ray fluorescence (XRF), which focuses on elemental composition. In this study, the powder from the geopolymer mortars prepared using the HBSS and the Commercial sodium silicate was analyzed by using PANalytical XRD equipment with a Cu-kα 1.54 Å radiation (45 kV, 30 mA) at 2θ range of 5 – 90°at a step size of 0.026° and 150 sec/step. (Alnahhal et al., 2023)

Fig. 10 shows an X-ray diffraction (XRD) pattern, with intensity (counts) on the y-axis and 2θ (degrees) on the x-axis. The pattern reveals several peaks corresponding to different crystallographic planes in the sample, identified using Highscore Plus software. The peak at 26.6° (2θ) for Quartz (SiO2-crystalline) showed a noticeable increase in intensity for T1 with 5 M NaOH compared to the commercial sodium silicate mix (C1). The calcite (CC) peak at 29.6° was more intense in HBSS-activated geopolymer mixes (T1, T15, T18), due to the presence of calcium from GGBS and Fly ash reacting with CO2. The sodium aluminosilicate (NAS) peak at 20.6° was stronger in HBSS mixes than in the C1 mix, influenced by additional ions in the HBSS solution. The mortar specimen T1 possesses the lowest crystalline compounds (calcite and quartz) among the mixes T15 and T18 because T1 comprises of RHA which contain more amorphous content than POFA (T18) that resulted in a higher intensity of sodium aluminosilicate (NAS) crystal. On the other hand, the 28-day compressive strength of T18 was maximum (51.6 MPa) compared to T1 (44.6 MPa). The intensity of calcite was higher in T18 which could act as a filler and responsible for the maximum compressive strength.

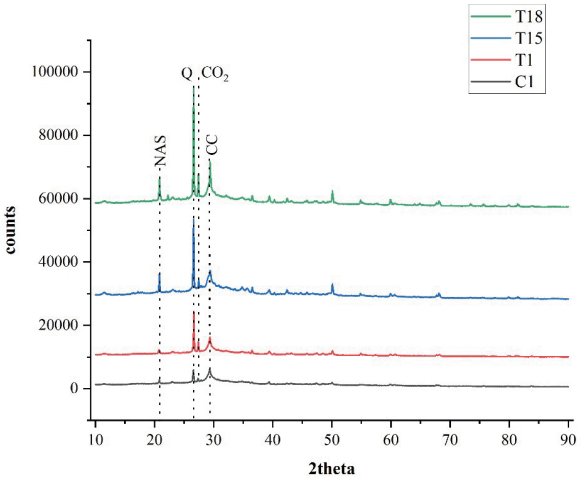


Fig.10. XRD plot of 28-day compressive strength tested geopolymer mortar specimens C1, T1, T15 and T18

Note: NAS - Sodium aluminosilicate, Q - Quartz (SiO_2), CC - Calcite ($CaCO_3$), CO_2 – Carbon dioxide

X-ray fluorescence (XRF) analysis is a non-destructive technique used to determine the elemental composition of materials. When a sample is exposed to high-energy X-rays, it emits secondary (fluorescent) X-rays characteristic of the elements present. The intensity and energy of these emitted X-rays are measured to identify and quantify the elements in the sample. XRF is widely used in fields such as materials science, geology, environmental monitoring, and forensic analysis due to its speed, sensitivity, and ability to analyse a wide range of materials without sample preparation.

The results from XRF analysis in Table 9 show that thermal treatment has different impacts on the silica content of POFA and RHA. For POFA, there is a noticeable increase in silica after treatment, rising from 56.03% in the untreated sample to 61.02% in the treated sample. This suggests that the thermal process

boosts the silica concentration in POFA. However, for RHA, the silica content remains almost unchanged, increasing only slightly from 90.09% to 90.96% after treatment. This indicates that thermal treatment does not have a significant effect on the silica content of RHA, highlighting the contrasting responses of POFA and RHA to thermal treatment.

Table 9 X-ray fluorescence (XRF) on untreated and treated materials

Components	POFA	Treated POFA	RHA	Treated RHA
SiO_2	56.03	61.02	90.09	90.96
Al_2O_3	1.68	1.63	0.21	0.38
CaO	10.26	8.86	1.86	1.51
Fe_2O_3	9.27	6.19	0.89	0.88
K_2O	13.88	12.83	3.41	2.86
SO_3	0.89	0.92	0.43	0.43
MgO	1.72	1.86	0	0
P_2O_5	5.15	5.13	2.66	2.42
Na_2O	0	0	0	0

Fourier-Transform Infrared Spectroscopy (FTIR) is a powerful technique used to measure the absorption or emission spectra of materials, providing detailed insights into their chemical properties. By exposing a sample to infrared light, FTIR generates a spectrum based on the absorption of specific wavelengths of light, which are absorbed by the sample's molecules and converted into vibrational or rotational energy. This spectrum acts as a molecular fingerprint, allowing for the identification of various functional groups and compounds within the material. FTIR is commonly used across solids, liquids, and gases, and typically produces a spectrum ranging from 4000 cm^{-1} to 400 cm^{-1}.

Functional Groups: Fig.11 shows the identified bands through FTIR that include Si-O-Si around 450 cm^{-1}, which indicates silicon dioxide structure in geopolymer networks; Si-O-T between 940-1100 cm^{-1}, reflecting Si-O bonds with aluminum or other tetrahedral elements; CO_3^{2-}around 1300 cm^{-1}, linked to calcite; H-O-H around 1650 cm^{-1}, representing water molecule bending; C-O around 2350 cm^{-1}, from carbonyl or residual carbonates; and O-H stretching between 2950-3700 cm^{-1}, related to hydroxyl groups from water or silanol. FTIR analysis of the RHA-based HBSS solution activated geopolymer mortar (T1) shows similarities to the commercial sodium silicate mix (C1), with Si-O-T bonds detected at 940 cm^{-1} and calcite at 1400 cm^{-1}. The 3000-3600 cm^{-1} range indicates water molecules from unreacted NaOH, with C1 showing lower unreacted NaOH, suggesting better dissolution. Lowering NaOH concentration reduces carbonate and OH intensities, implying higher NaOH concentrations promote better geopolymerization. FTIR thus provides valuable chemical information but requires material-specific knowledge for accurate interpretation concentration from 10M (T15) to 3M (T18) reduced carbonate and OH group intensity, suggesting that higher NaOH promotes more complete geopolymerization. FTIR analysis helps identify chemical composition but requires knowledge of the material.

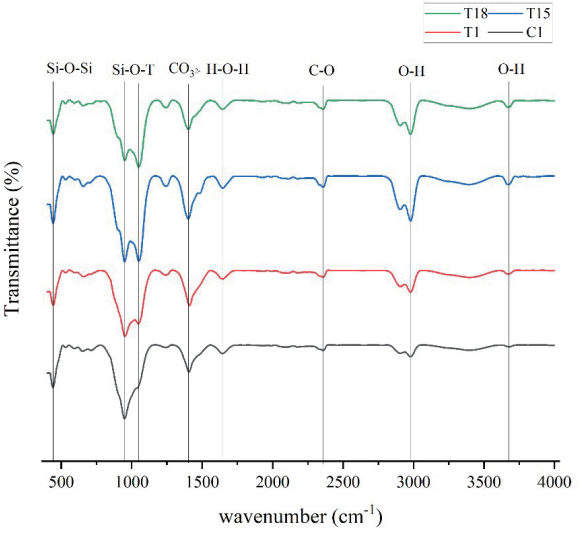


Fig.11. FTIR plot of 28-day compressive strength tested geopolymer mortar specimens C1, T1, T15 and T18

5. Conclusions

Home-brewed sodium silicate (HBSS) solutions were synthesized using the hydrothermal method with RHA and POFA as silica sources. The study identified the crucial factors influencing the silicate modulus (SM) of HBSS solutions. These factors are arranged in ascending order as follows: molarity of NaOH solution, amorphous content of SiO_2 content in POFA and RHA,

and stirring time.

The study effectively examined the influence of sodium hydroxide (NaOH) concentration and stirring time on the workability and compressive strength of alkali-activated binders made from ground rice husk ash (GRHA) and treated ground palm oil fuel ash (TGPOFA).

For GRHA, a 5M NaOH solution with 1 hour of stirring time (Mix T1) yielded the maximum compressive strength of 44.7 MPa and the flow value was around 157.75 mm. Treated GRHA achieves an optimal compressive strength of 42.3 MPa with 5M NaOH and 3 hours of stirring (Mix T12) together with its flow value of 141.25 mm.

For GPOFA, the highest compressive strength of 19.5 MPa was observed with 10M NaOH and 3 hours of stirring (Mix T8) followed by the 212.25 mm of its flow value while TGPOFA shows a maximum compressive strength of 24.2 MPa with 10M NaOH and 3 hours of stirring (Mix T15) come with the flow value 230 mm. However, when the molarity decreases to 3M in the TGPOFA, the maximum strength increases to 51.6 MPa with 3M NaOH and 3 hours of stirring (Mix T18). Totally 8M is adequate for POFA based mortar mix to achieve 50 MPa strength, hence it can be concluded that the mix T18 produced the optimum performance in 28-day compressive strength.

Increasing NaOH concentration from 5M to 10M significantly improved the workability of the mixes, enhancing flow values. All RHA samples had higher compressive strength when compared to POFA due to a higher amount of silica content.

The solution derived from Rice Husk Ash (RHA) had a high compressive strength comparable to Geopolymer (GP) mortar mixed with a 5 M sodium hydroxide (NaOH) solution. This implies that RHA-derived solutions could be used as a binding agent with similar strength properties to GP mortar mixed with a strong alkaline solution. Higher SM values were obtained in RHA-derived HBSS solution due to a high silica content in RHA and high amorphous percentage, suggesting that RHA-derived solutions have a higher silica content compared to other materials.

For POFA, the strength development was not significantly influenced by variations in the molarity of the NaOH solution. For instance, in T2 with 5M NaOH and T3 with 10M NaOH for both at 1 hour, the strength did not show major changes; rather, it only changed by around 2-3 MPa. Thus, it may be concluded that doubling or altering the molar concentration of POFA is not necessarily due to low SiO_2 content.

Overall, GRHA can effectively replace commercial sodium silicate, achieving high compressive strengths, with RHA contributing significantly to the activation process. The study highlights the potential of using treated agro-industrial by-products in geopolymer binders, promoting sustainability and cost-efficiency in construction materials.

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