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Design of a Geopolymer Based on Fly Ash and Metakaolin with a Solid, Liquid, and Powdered Alkaline Activator at High Temperatures

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Abstract

In this work, the fresh and hardened properties of geopolymer concrete activated using solid, liquid, and powder alkaline activator systems, including fly ash and different amounts of metakaolin, are examined. Compressive and flexural strengths were assessed at 7 and 28 days in accordance with IS 516 (1959). Workability was evaluated using a slump test in accordance with IS 1199 (Part 2): 2018. At an ideal replacement level of 30%, the results indicate that strength improvement is directly proportional to the increase in metakaolin; above this level, limited workability and partial geopolymerization result in a decrease in strength. The strength performance of liquid activators is the highest among other activator systems, followed by powder activators; solid activators, on the other hand, create the least amount. However, the solid-activated system still led to strength development, enhancing handling ease and reducing safety risks relative to liquid chemicals, making it a far better choice for field applications where storing and transporting liquids pose problems. While SEM microstructural analysis showed lower porosity and a denser gel structure in liquid-activated systems compared to solid and powder systems, X-ray diffraction (XRD) data showed that these mixes exhibited greater geopolymerization and a larger amorphous phase. The best results were obtained with a liquid system containing 70% fly ash and 30% metakaolin. However, the good workability and durability of solid activators demonstrate that they can be used in less severe structural fill applications, thereby extending the use of geopolymer concrete.

Keywords: Geopolymerization, microstructural analysis, X-ray diffraction (XRD), geopolymer concrete, crystalline sodium aluminosilicate hydrate

INTRODUCTION

Significant environmental changes have led to global warming, including abrupt shifts in unseasonal rainfall or drought, rising ocean levels that threaten aquatic life, and ecological changes affecting both humans and animals – all of which are primarily due to global warming [1]. Various factors contribute to global carbon emissions, but one of the main sources of environmental damage from fly ash is carbon emissions [2]. This fly ash is generated by burning raw materials in the kiln, grinding minerals, and combusting fossil fuels. Additionally, the entire cement industry produces substantial CO_2 emissions [3]. Reducing this pollution remains a persistent challenge for researchers and conservationists. Dr Joseph Davidovits is one of the pioneers who first introduced this term to describe environmental waste. He conducted extensive research to develop Geo-polymer, a cement-like material made from fly ash [4, 5]. Inorganic binders called geopolymers can solidify at room temperature,

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low temperatures, or high temperatures. They are produced by mixing natural pozzolanic minerals and amorphous aluminosilicates, such as fly ash and metakaolin, with alkali activators, such as sodium silicate (Na2SiO3), sodium hydroxide (NaOH), or potassium hydroxide (KOH) [6, 7, 8]. The compressive strength, initial setting time, heat resistance, low water permeability, and acid resistance of Geo-polymer all surpass those of Portland cement [6, 9–14].

Over the past 10 years, two-part geopolymers have been a significant focus of research aimed at promoting the use of geopolymer concrete as a sustainable building material. High compressive strength [15–16]., strong bonding qualities with reinforcement [17]., and resilience to high temperatures [18]. have been reported for fly ash-based geopolymer concrete. They are suitable for small-scale applications because it is challenging to transport, store, and prefabricate large volumes of viscous, extremely corrosive alkaline solutions.

Thus, one factor enabling the widespread use of geopolymer in the construction industry is the development of a one-part, or "just adding water" geopolymer suitable for use as OPC. Water is added on-site after the solid alumino-silicate precursor and solid alkaline activator have been dry-mixed in advance for one-part geopolymer synthesis. However, when water is added to the mixture of the alumino-silicate precursor solid and the alkaline activator (solid NaOH, Na2-SiO3), the alkaline solid activator cannot be fully dissolved to react with the alumino-silicate in a short period of time. Consequently, the mechanical properties of one-part geopolymer were unable to match those of two-part geopolymer [19].

Using low-quality kaolin, Peng created single-part geopolymers by calcining it with NaOH at various high temperatures and then rehydrating it with water. After three days of curing at 80°C, the geopolymer's compressive strength reached 63 MPa [20]. This was significantly higher than the strength of the two-part geopolymer [21]. Nematollahi et al. produced alkali-activated mortars at room temperature by immediately mixing fly ash (FA) or ground granulated blast-furnace slag (GGBS) with a combination of sodium silicate and sodium hydroxide powders. Test results showed that while the FA mortars produced no appreciable compressive strength, the GGBS-based mortar achieved a 28-day compressive strength comparable to that of the control ordinary Portland cement (OPC) mortar [22]. Ma et al. examined the effects of three commercially available sodium metasilicate activators in fly ash and slag-based single-part geopolymer systems. The three activators were Na2SO3-anhydrous, Na2SO3-5H2O-pentahydrate, and Na2SO3-5H2O-nonahydrate. They concluded that the activated effects of various forms of sodium metasilicate powders in single-part geopolymer varied, and that anhydrous sodium metasilicate was the most effective among the three activators [23]. Luukkonen et al. investigated how different curing conditions affect the strength development of single-part alkali-activated binders. They found that curing conditions can significantly influence the development of strength and structural stability in alkali-activated slag/ceramic binders. Furthermore, it was demonstrated that hydrotalcite-like phases were present and that the strength properties of single-part alkali-activated slag increased at a lower silica modulus [24].

Based on the literature review, there has been limited research on the use of metakaolin and low-calcium F-class fly ash for one-part or in-situ alkali-activated concrete with enhanced curing procedures. To commercialise the use of one-part geopolymers, achieving the highest possible strength with minimal energy consumption during curing is essential [24].

Barbhuiya, S., & Pang, E. (2022). Portland cement production is considered a major contributor to greenhouse gas emissions, accounting for about 6–7% of global CO2 emissions, according to the International Energy Agency. Consequently, over the past few decades, efforts have increased to reduce or eliminate Portland cement in concrete. Among the alternatives, geopolymer has attracted significant attention for its early strength development, low permeability, high chemical resistance, and excellent fire resistance. This research investigates the strength and microstructure of geopolymers made from fly ash and a metakaolin-fly ash blend. Compressive strengths were evaluated at 7, 14, and 28 days, while microstructural analysis was performed using SEM and XRD [25].

Due to the growing threat of greenhouse gas emissions from the global cement industry, various sustainable solutions have emerged over recent decades. The development of geopolymer concrete (GPC) is a key contribution toward eco-friendly construction. This research optimises GPC by adding a fixed amount of fly ash (FA) and alkali activator to an acceptable aggregate ratio of 0.5, with molarity varied from 12 M to 16 M and Na_2SiO_3/NaOH ratios from 1.5 to 2.5. The properties and effects of heat and ambient curing were tested at various ages. The optimised mixture was then blended with locally sourced Metakaolin (MK) at 10%, 20%, 30%, and 40% dosages. The study investigates the combined effect of FA and MK on GPC performance. Results indicated that a 30% FA-MK GPC showed improvements of 21%, 19%, and 26% in compressive, split tensile, and flexural strengths, respectively, over FA-based GPC under heat curing. This is primarily due to MK's binary action, which enhances compaction, and its pozzolanic activity, which accelerates the development of strength. FTIR and the Modified Chapelle test confirmed these findings, and SEM images examined GPC morphology. This work aims to use fly ash from coal power plants to support sustainable cities and development goals in communities [26].

MATERIAL & METHODS

Material

Metakaolin from Kaolin Minerals in Vadodara, Gujarat, and low-calcium fly ash (F-class) that complies with IS 3812 PART 1 [27]. specifications have been used as the binder and activator, respectively, with granular sodium metasilicate as the alkaline activator. The physical and chemical makeup of fly ash, metakaolin, and the ignition loss values obtained using the X-ray fluorescence (XRF) test method are listed in Tables 1 and 2. While the metakaolin loss on ignition value is 13.38%, the fly ash loss on ignition value of 1.07 is extremely low and well below the IS 3812 (Part 1) norms. Natural sand, which has a specific gravity of 2.68 and a fineness modulus of 2.96, is the fine aggregate used. Three different coarse aggregate sizes were used in this study: 20 mm (specific gravity: 2.66), 14 mm (specific gravity: 2.65), and 10 mm (specific gravity: 2.65). The weight proportions of 20 mm (25%), 14 mm (20%), and 10 mm (35%) coarse aggregates were employed to create a well-graded mix. The remaining 20% of the aggregate content consisted of fine aggregate. In accordance with IS: 2386 (Part 1): 1963, the procedure for assessing particle size distribution, a sieve analysis was conducted on the fine aggregate [28]. Figure 1 displays the gradation aggregate curve. Table 1 represents the physical and chemical composition of Metakaolin. Table 1 presents the physical and chemical properties of fly ash. Table 2 presents the physical and chemical properties of Metakaolin

Table 1. Physical and chemical compositions of fly ash.

Test	**Unit**	**IS 3812 PART -1 Specification**	**Test Result**
Fineness – Specific Surface by Blaine's Permeability Method (Min)	m^3/kg	320	412
Lime Reactivity (Minimum)	mmN/mm^3	4.5	5.18
Moisture Content (Max.)	%	2	0.22
Autoclave Expansion (Max.)	%	0.8	0.023
Chemical Analysis			
Loss on Ignition (Max.)	%	5	1.03
$SiO_2+Al_2O_3+Fe2O_3$	%	70 min by mass	92.11
SiO_2	%	35 min by mass	56.87
MgO	%	5 min by mass	1.29
SO_3	%	3 min by mass	0.45
Na_2O	%	1.5 min by mass	0.42

Total Chlorides	%	0.05 min by mass	0.024

Table 2. Physical and chemical compositions of Metakaolin

Physical Parameters	**Standard Limits**	**Results**	**Method**
Residue 300# (45 Micron) Wet Method	<3.0%	1.80%	Wet Sieving Method
Appearance	Creamish White	Creamish white	
Brightness	77±1	76.00	ISO 2470
pH (00%) Solid	8.5 to 9.5	9.3	IS 4031 (Part 11) Determine pH of metakaolin slurry
Bulk Density (gm/Ltr) (Untapped)	0.4 to 0.5	0.45	IS 12474
Specific gravity	2.5 – 2.6	2.56	IS 2720 (Part 3) Use the pycnometer method with kerosene.
Moisture content	3.0% Max	2.88%	IS 2720 (Part 2) Oven dry at 105 ± 5°C for 1–2 hours.
Absorption powder (gm/100gm)	30-40	32.14	ASTM D281
Chemical Analysis			XRF
SiO2	46%±2	47.88%	
Al2O3	32%±2	32.41%	
L.O.I	14%±1	13.38%	
Moisture content	3.0% Max	2.88%	

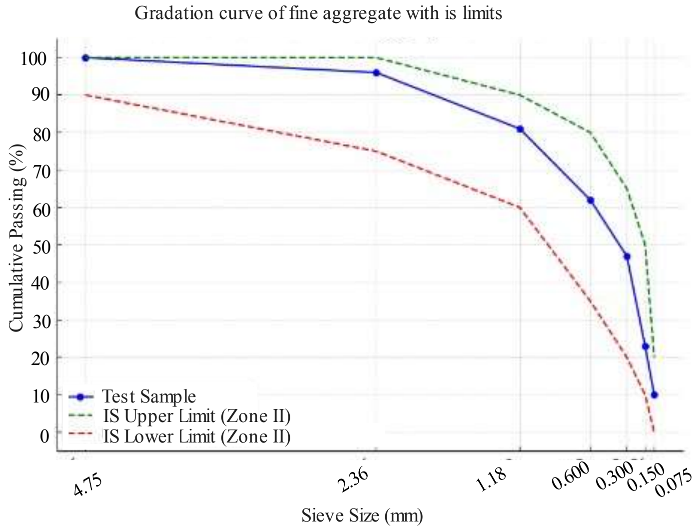

Figure 1. Fine aggregate gradation curve

Alkaline Activator

According to the study by Bong et al [29]., the most effective alkali activators are sodium hydroxide, sodium silicate, and sodium metasilicate. However, handling and storing these highly caustic alkaline activators pose challenges. Additionally, some research indicates that the anhydrous and pentahydrate forms of sodium metasilicate show better geopolymerization properties. A study reports that powdered alkali activators are easy to apply and enhance geopolymerization [30]. This research examines the performance of dry alkali activators. Anhydrous sodium metasilicate pentahydrate (Anhy.Na2SiO3•5H2O), with the following composition, was used in five mixed formulations: SiO2 at 27–29 weight per cent, water at 43 weight per cent, and Na2O at 28–30.5 weight per cent, sourced from Tina Labs. Additionally, five more mixes were prepared using liquid-based alkaline activators containing sodium hydroxide and sodium silicate. Furthermore, careful attention is required when mixing sodium hydroxide (NaOH) solution with sodium silicate to produce a liquid alkaline solution. Based on the amount of sodium hydroxide solution and sodium silicate relative to NaOH, a (Na2SiO3) gel is used to create an alkaline solution. The solution should be prepared the day before. To make a 14 M NaOH solution, dissolve 560 g of NaOH pellets (molecular weight 40) in distilled water until the total volume reaches 1 L. The remaining five binders were prepared by combining predetermined amounts of Class F fly ash, metakaolin, and powdered activators. The activator, a powder containing sodium hydroxide and sodium silicate, was used to produce these binders. The activator-to-source material ratio for both binders was 0.3 [31–33].

Sample Preparation

The blending of Fly Ash and Metakaolin was conducted at partial fly ash replacement levels ranging from 0 to 40% to identify the optimal mix proportions with various types of alkaline activators. These mixes were tested for compressive, flexural, and split tensile strengths after 7 and 28 days.

In this research, notation is used for solid anhydrous sodium metasilicate pentahydrate, i.e., 100F.A.S, 90F.A. 10MK.S, 80F.A. 20MK.S, 70F.A. 30MK.S, 60F.A. 40MK.S. Similarly, notation for liquid-based activators is 100F.A.-L, 90F.A. 10MK.-L, 80F.A. 20MK.-L, 70F.A. 30MK.-L, 60F.A. 40MK.-L.

Lastly, the notation for a powdered mixture of sodium silicate and sodium hydroxide is as follows: 100F.A. P, 90F.A. 10MK.-P, 80F.A. 20MK.-P, 70F.A. 30MK.-P, 60F.A. 40MK.-P. Table 3 provides the Mix Proportion of (Anhy.Na2SiO3•5H2O) for the one-part geopolymer mixes. In all batches, we standardised the amount of sodium silicate activator to 12 wt.% of the binder, as established from our own trial tests and prior work in this area (32). The liquid-based geopolymer mixes are prepared with an SS/SH ratio of 2.5 and a w/b ratio of 0.5, which were determined after various trial tests and prior work in this area (33). Lastly, the solid mixture of both sodium silicate and sodium hydroxide was prepared from the liquid-based geopolymer after removing the water content. During mixing, the one-part, solid-based geopolymer is prepared using an inclined mixer, where fly ash and metakaolin are added simultaneously for approximately 10 to 15 minutes. Then, the prescribed amount of water is added until a homogeneous mixture is achieved.

Table 3. Mix proportion of various samples

Notation of the sample	**Fly Ash (kg/m^3)**	**Metakaolin (kg/m^3)**	**NaOH (kg/m^3)**	**Na_2SiO_3 (kg/m^3)**	**Fine Aggregate (kg/m^3)**	**Coarse Aggregate (kg/m^3)**	**Water (Kg/m^3)**
100F.A. S	405	-	-	48.6	669.10	1242.62	81
90F.A.10MK.S	364.5	40.5					
80F.A.20MK.S	324	81					
70F.A.30MK.S	283.5	121.5					
60F.A.40MK.S	243	162					
100F.A.-L	405	-	57.86	144.64	669.10	1242.62	121.8
90F.A.10MK.-L	364.5	40.5					
80F.A.20MK.-L	324	81					
70F.A.30MK.-L	283.5	121.5					
60F.A.40MK.-L	243	162					
100F.A. P	405	-	34.71	86.79	669.10	1242.62	174.93
90F.A.10MK.P	364.5	40.5					
80F.A.20MK.P	324	81					
70F.A.30MK.P	283.5	121.5					

60F.A.40MK.P	243	162					

When preparing a liquid-based alkaline geopolymer solution, it is ready one day before activation. This solution is carefully mixed with fly ash and metakaolin to ensure proper geopolymerization. After preparation, all samples are placed in a hot-air oven for elevated curing at about 80°C for 24 hours. Following proper curing, these samples are tested at 7 and 28 days, respectively.

To achieve uniformity and consistency across all batches, a strict mixing protocol was followed. All dry components – fly ash, metakaolin, and both fine and coarse aggregates – were initially dry-mixed for 3 minutes in a pan mixer to ensure even distribution. For liquid-activated mixes, the NaOH–sodium silicate solution was prepared 24 hours prior to use and cooled to room temperature. During mixing, the prepared alkaline solution was added slowly in two stages while the mixer operated, ensuring gradual wetting and preventing localised stiffening. For solid and powder activators, the dry activator was pre-blended with the fly ash–metakaolin mixture for 2 minutes to avoid clumping, followed by the addition of the required water in a thin stream over 60–90 seconds. Mixing continued for an additional 3–4 minutes until a homogeneous, lump-free paste was obtained. Each batch was immediately tested with a slump test (IS 1199 Part 2: 2018). If the slump varied by more than ±5 mm from the target, water adjustments of up to 1–2% were made to ensure consistent flow. All samples were mixed under the same conditions – time, moisture, and a 5-minute rest before casting – to minimize variability. Table 3 presents the mix proportion of various samples.

Testing

The engineering properties of concrete were assessed in this study in both its hardened and plastic (fresh) states. Slump measurement was used to determine the workability of fresh concrete in accordance with IS 1199 (Part 2):2018. The properties of hardened concrete were examined in terms of its compressive and flexural strengths. According to IS 516 (1959), the compressive strength of concrete composed of three different alkaline materials was measured at 7 and 28 days to investigate the evolution of strength at both early and later ages. Similarly, flexural strength tests of concrete were conducted at 28 days in accordance with IS 516. Scanning electron microscopy (SEM) and X-ray diffraction have been employed to investigate the effect of varying the fly ash-to-metakaolin ratio and the alkaline activator on the microstructure of geopolymer concrete. To enhance the statistical reliability of the mechanical strength data, each compressive and flexural strength value is the average of three specimens tested under identical conditions. The standard deviation (SD) for each data point was determined from the three readings, and these SD values are included in the result tables. The graphs have also been updated to display error bars based on SD, offering a clearer visual of data variability. This method ensures that the reported mechanical performance accurately represents a statistically significant trend.

RESULT AND DISCUSSION

Workability

The workability of geopolymer concrete mixes was assessed using the slump test, as specified in IS 1199 (Part 2): 2018. The results are presented in Table 4.

It was observed that the slump values strongly depended on the type of activator and the percentage of fly ash replaced with metakaolin. For solid activators, slump values increased from 150 mm in the control (100F.A.S) to 160 mm at 40% metakaolin replacement (60F.A.40MK.S); these improvements are due to metakaolin's higher reactivity and finer particle size compared to fly ash. The increased reactivity of metakaolin accelerates the dissolution of aluminosilicate phases, releasing reactive species that improve paste cohesiveness without significantly increasing mixing water consumption. These factors collectively enhance the workability of solid-activated geopolymer mixes. Unlike solid activator mixes, liquid activator mixes showed lower slump values, ranging from 140 mm to 150 mm. This reduced flowability compared to solid activators may result from the rapid release of gases in the liquid phase, which speeds up geopolymerization and gel formation during mixing. Early gelation traps free water, reducing its lubricating effect on particles and thus lowering the slump.

Table 4. Slump test of geopolymer concrete

Notation of Sample	**Slump Value (mm)**
100F.A. S	150
90F.A.10MK.S	152
80F.A.20MK.S	154
70F.A.30MK.S	155
60F.A.40MK.S	160
100F.A.-L	140
90F.A.10MK.-L	140
80F.A.20MK.-L	143
70F.A.30MK.-L	145
60F.A.40MK.-L	150
100F.A. P	130
90F.A.10MK.P	134
80F.A.20MK.P	135
70F.A.30MK.P	135
60F.A.40MK.P	135

Interestingly, as metakaolin content increased, the liquid-activator series showed only moderate changes, suggesting that metakaolin's impact on workability is less significant in liquid-activated systems. The consistent results across different replacement levels suggest that liquid activators have a significant influence on fresh properties, often overshadowing the effect of precursor composition.

The powder activator series produced the lowest slump values, ranging from 130 mm to 135 mm. This limited workability is likely due to the slower dissolution rate of powder activators, which delays the availability of alkaline ions during the early mixing stage. Consequently, the dispersion of fly ash and metakaolin particles is restricted, resulting in a stiffer mix with reduced mobility. Moreover, the slower reaction rate prevents the formation of sufficient gel phases during the initial minutes of mixing, leading to particle clumping and reduced flow. The slight variation in slump with different metakaolin contents indicates that, in powder-activated systems, the behaviour of the activator during dissolution has a greater impact on fresh-state properties than the precursor composition. Overall, these results clearly demonstrate that the type of activator has a significantly greater influence on the workability of geopolymer concretes than the proportions of fly ash and metakaolin. Solid activators yielded the highest slump values, indicating good dispersion and delayed reactivity, providing sufficient time for mixing and placement. Liquid activators yielded moderate slump values with stable trends across various replacement levels, rendering them suitable for mixes that require balanced reactivity and workability. However, powder activators produced stiff, poorly flowing mixes, limiting their practical application unless measures such as superplasticisers, higher liquid-to-binder ratios, or longer mixing times are employed. These findings are important for field applications where ease of placement and compaction are critical. Therefore, selecting the appropriate activator system is essential for developing workable geopolymer concretes suitable for structural or precast uses.

Compressive Strength

The specimens are designated according to the percentage replacement of fly ash with metakaolin (0–40%) and the curing/exposure condition (S, L, or P). The data provide a comparative assessment of early-age and later-age strength development, highlighting the influence of metakaolin content and curing condition on the compressive performance of the mixes. Table 5 presents the compressive strength results of fly ash–metakaolin (FA–MK) blended samples at curing ages of 7 and 28 days.

The assessment of compressive strength in various geopolymer systems – solid, liquid, and powder activators – showed distinct differences in early-age and long-term characteristics based on the type of activator and the percentage of metakaolin (MK) mixed with fly ash (F.A.). For solid activators, the strength values were relatively low.

Table 5. Compressive strength of geopolymer concrete for 7 and 28 days

Notation of the sample	Compressive strength (MPa) of 7days	Compressive strength (MPa) of 28days
100F.A. S	15.50	16.45
90F.A.10MK.S	17.45	18.70
80F.A.20MK.S	18.65	19.45
70F.A.30MK.S	18.70	19.80
60F.A.40MK.S	17.25	17.65
100F.A.-L	29.10	30.95
90F.A.10MK.-L	30.35	31.05
80F.A.20MK.-L	31.45	32.24
70F.A.30MK.-L	31.95	33.10
60F.A.40MK.-L	29.25	31.80
100F.A. P	20.30	21.55
90F.A.10MK.P	21.85	23.34
80F.A.20MK.P	22.60	24.24
70F.A.30MK.P	23.55	24.86
60F.A.40MK.P	21.40	22.45

The control mix with solely fly ash (100F.A.S) reached 15.50 MPa at 7 days and 16.45 MPa at 28 days. Integrating MK gradually increased strength up to 30% substitution, with the mix (70F.A.30MK.S) achieving a peak of 19.80 MPa at 28 days. Additionally, increasing the MK content (40%) resulted in a decrease, suggesting that excess MK could hinder effective geopolymerization due to increased water requirements and reduced workability. Different behaviour was observed in the liquid activator system, which consistently provided the highest compressive strengths. The sample containing only fly ash (100F.A.-L) showed remarkable results, reaching 29.10 MPa at 7 days and 30.95 MPa at 28 days. The incorporation of MK significantly boosted the strength, with the ideal blend (70F.A.30MK.-L) achieving 33.10 MPa at 28 days. Nevertheless, as observed with the solid activator system, an increased MK content (40%) led to a minor decrease in strength, indicating that the fly ash-to-MK ratio is essential for optimising geopolymerization effectiveness.

The powder activator system produced results between the solid and liquid activator mixtures. The base sample (100F.A.P) reached 20.30 MPa after 7 days and 21.55 MPa after 28 days. With MK incorporation, compressive strength increased consistently, reaching a maximum of 70F.A.30MK.P (24.86 MPa) at 28 days. An additional rise to 40% MK once more resulted in a minor decrease, confirming the pattern that excessive MK substitution does not always result in increased strength. Generally, comparative analysis indicates that the nature of the activator is a critical factor in determining the compressive strength of geopolymer concrete. Liquid activators were found to be most effective, resulting in relatively higher early-age and long-term strengths. Powder activators yielded moderate enhancement, whereas solid activators yielded the lowest values throughout. In all three activator systems, the addition of MK increased strength to a maximum of 30% replacement, above which the gains decreased. This indicates that MK plays a helpful role by accelerating the geopolymerization reaction and optimising the microstructure, but high levels may upset the system's equilibrium. These results highlight that both the selection of the activator and the MK ratio are key factors in tailoring the mechanical properties of geopolymer binders. Figure 2 represent the compressive strength graph of geopolymer concrete or 7 and 28 days.

Flexural Strength

The specimens are identified by the percentage replacement of fly ash with metakaolin (0–40%) and the respective curing/exposure conditions, denoted as S, L, and P. The results illustrate the effect of metakaolin incorporation on the flexural performance of the mixes, enabling comparison of strength enhancement trends across different replacement levels and curing conditions at a later age. Table 6 summarizes the flexural strength of fly ash–metakaolin (FA–MK) blended samples measured at 28 days of curing.

The flexural strength results at 28 days reveal clear performance patterns among geopolymer samples activated by solid, liquid, and powder activators. In the solid activator series (F.A.–S), the strength ranged from 7.10 to 9.75 MPa. A gradual increase was observed with the partial replacement of fly ash by metakaolin up to 30% (70F.A.30MK.S), which achieved the highest flexural strength of 9.75 MPa, indicating improved matrix densification and crack bridging at this replacement level. However, higher substitution (40% MK) caused a slight decrease (8.10 MPa), suggesting that excessive metakaolin could increase brittleness and decrease flexural strength. Conversely, the liquid activator series (F.A.–L) consistently outperformed the solid system, reaching flexural strengths of 10.10-12.30 MPa. The highest strength was observed for 70F.A.30MK.-L (12.30 MPa), demonstrating the excellent dissolution and polymerisation capabilities of liquid activators, which promote better bonding within the geopolymer matrix. Even at higher replacement levels, the liquid-activated samples showed significantly higher strengths than their solid counterparts, highlighting their effectiveness in activating both fly ash and metakaolin. In the powder activator series (F.A.–P), the strength measurements were moderate, ranging from 8.30 to 9.10 MPa.

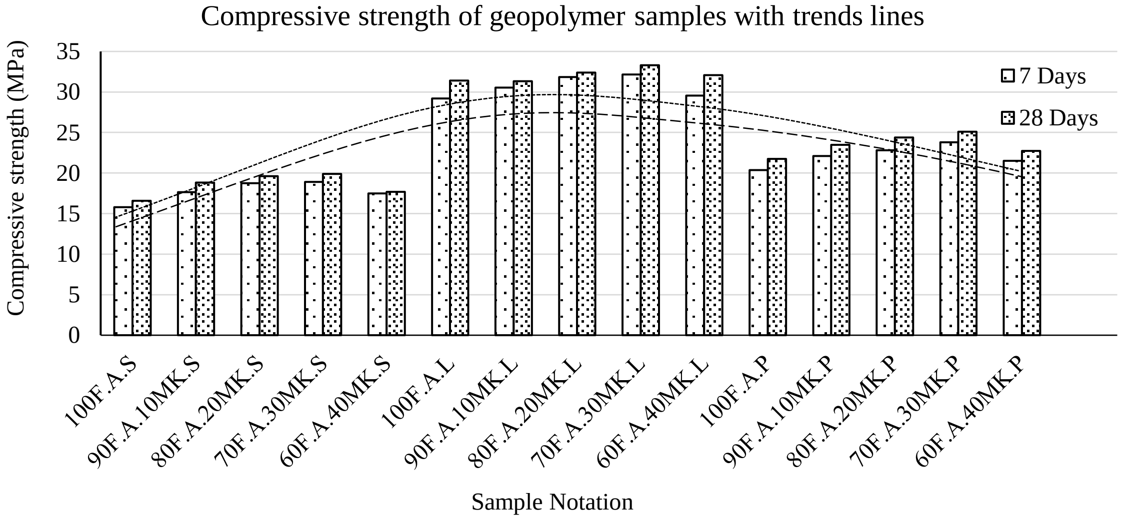

Figure 2. Compressive strength of geopolymer concrete for 7 and 28 days

Table 6. Flexural strength of geopolymer concrete after 28 Days

Notation of the sample	Flexural strength (map) of 28days
100F.A. S	7.45
90F.A.10MK.S	7.55
80F.A.20MK.S	8.25
70F.A.30MK.S	9.75
60F.A.40MK.S	8.10
100F.A.-L	10.25
90F.A.10MK.-L	11.45
80F.A.20MK.-L	11.55
70F.A.30MK.-L	12.30
60F.A.40MK.-L	10.10
100F.A. P	8.75
90F.A.10MK.P	8.90

80F.A.20MK.P	8.95
70F.A.30MK.P	9.10
60F.A.40MK.P	8.30

The improvement with higher metakaolin content was less pronounced than in the liquid system but showed a slight increase up to 30% substitution (9.10 MPa for 70F.A.30MK.P). Beyond this level, strength decreased, exhibiting a performance pattern similar to that of solid activators, but with somewhat better results. Overall, the findings indicate that the liquid activator system produces the highest flexural strength, followed by the powder and solid activator systems. The optimal replacement level of metakaolin appears to be around 30%, regardless of the activator type, after which flexural strength generally declines. This highlights the importance of balancing the proportions of fly ash and metakaolin in geopolymer formulations to optimise flexural performance.

Figure 3 represent the flexural strength graph of geopolymer concrete for 28 days

SEM Images

SEM images of the geopolymer concrete, composed of 70% fly ash and 30% metakaolin, at ×500 magnification show a mixed matrix. Both unreacted and partially reacted particles can be seen alongside the binding gel, illustrating the complex geopolymerization process. In Figure 4, the round fly ash particles and angular metakaolin fragments are clearly visible, all of which are encapsulated by a continuous gel phase, indicating some degree of dissolution and reaction. The surface texture appears uneven, with micro-voids and delicate pores scattered throughout the matrix. This unevenness suggests that incomplete reactions and potential air entrapment during casting may have contributed to localised porosity. Furthermore, the presence of thin, needle-like crystals within the matrix indicates the formation of secondary aluminosilicate products, which help densify the matrix and improve mechanical interlocking between particles.

In Figure 5, a denser, more compact area within the geopolymer matrix is observed. Here, the elongated needle-shaped crystals cluster together, radiating from the gel surface. This close arrangement is characteristic of crystalline sodium aluminosilicate hydrate (N-A-S-H) phases, which act as the primary binding agents in alkali-activated materials. Their uniform growth and strong connection to the surrounding gel suggest robust chemical bonds, likely enhancing both mechanical strength and long-term durability. The well-developed crystalline features also indicate that the alkali activation process was particularly effective in specific regions, resulting in lower porosity and a more stable microstructure.

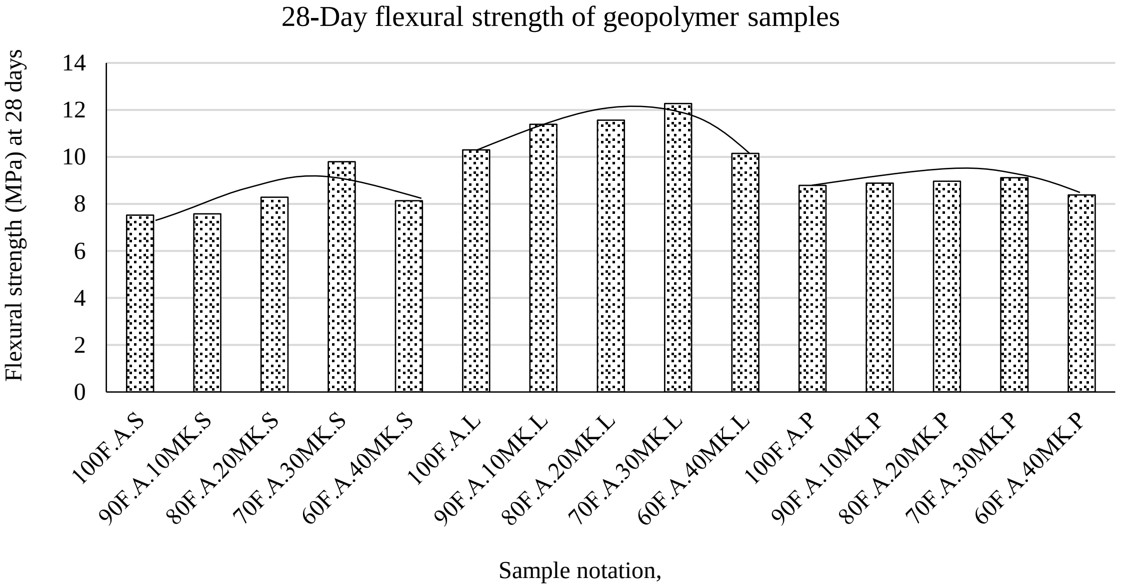

Figure 3. Flexural strength of geopolymer concrete for 28 days.

In Figure 6, we observe that the microstructure has a rough, uneven surface, filled with numerous pores and microcracks within the hardened matrix. These gaps could serve as channels for aggressive agents and act as weak points in the structure. Conversely, some areas in the same micrograph display a dense, compact gel. This indicates that the geopolymerization reaction caused localised densification, although it was not uniform throughout the entire matrix. The coexistence of porous and dense regions highlights the importance of carefully adjusting curing conditions and activator composition to achieve a more uniform reaction throughout the material.

Overall, in Figure 7 the SEM observations confirm that the microstructure of the geopolymer concrete, composed of 70% fly ash and 30% metakaolin, includes unreacted fly ash spheres, partially reacted metakaolin particles, needle-like crystalline products, and a gel matrix with varying density. While the development of well-formed crystalline compounds contributes to strength and bonding, the presence of microvoids and cracks indicates that we need to refine processing parameters to minimise porosity and enhance durability.

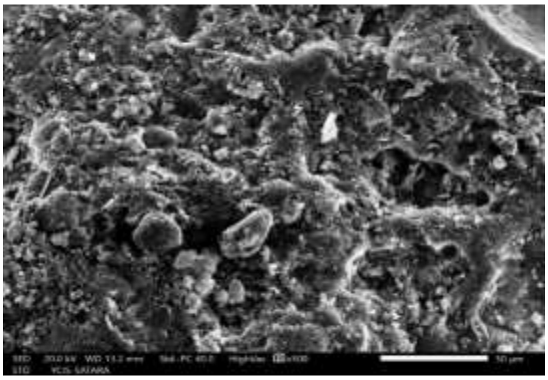

Figure 4. Microstructure of 70FA 30MK Solid by using SEM under different magnifications

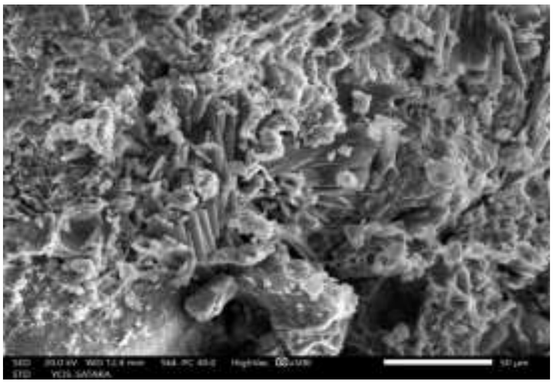

Figure 5. Microstructure of 70FA 30MK Liquid by using SEM under different magnifications

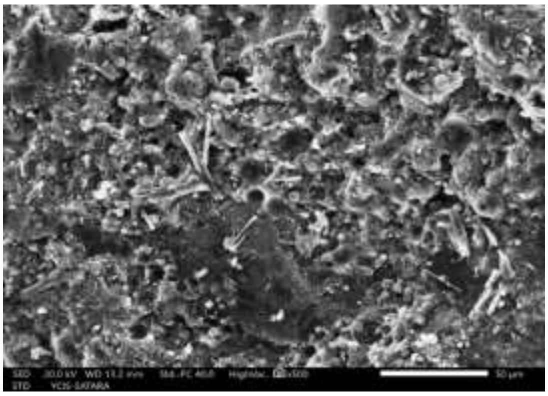

Figure 6. Microstructure of 70FA 30MK powder by using SEM under different magnifications

XRD Images

X-ray diffraction (XRD) analysis of the Solid 30 MK, Liquid 30 MK, and Powder 30 MK samples reveals distinct differences in their phase composition and degree of geopolymerization. All samples exhibit a broad, diffuse hump between 15° and 35° (2θ), characteristic of amorphous aluminosilicate gel, confirming the formation of geopolymeric binding phases. Superimposed on this amorphous background, several crystalline reflections vary in intensity and sharpness across samples. The Powder 30 MK shows sharper, more intense peaks at 20–24°, 26–29°, and 51–52° (2θ), indicating a higher presence of unreacted crystalline phases, such as quartz and mullite, from the raw metakaolin. Conversely, the Liquid 30 MK exhibits the lowest crystalline peak intensities and a more prominent amorphous hump, suggesting greater geopolymerization and less residual crystalline material. The solid 30 MK presents an intermediate pattern with visible crystalline peaks, indicating some crystalline phases remain while strong gel formation occurs. The Powder 30 MK's crystalline peaks are more pronounced, with minor reflections at approximately 26.3° and 29.2° (2θ) corresponding to quartz (SiO_2) and calcite ($CaCO_3$), likely due to surface carbonation during curing or storage. Additional features between 20° and 24° and 27° and 29° (2θ) may indicate secondary crystalline phases, such as zeolitic aluminosilicates. These findings suggest that the precursor form influences the extent of the reaction: Liquid 30 MK produces the most amorphous gel, followed by Solid 30 MK, with Powder 30 MK showing significant crystalline residues. Figure 7 represents the Comparison of XRD test of various proportions of geopolymer concrete.

The mechanical performance in this study aligns with previous research, which indicates that metakaolin enhances the geopolymerization process due to its high content of amorphous aluminosilicates. Likewise, other studies have shown better strength development in one-part geopolymer systems when using fine reactive materials like metakaolin. Ma et al. (2018) also found that metasilicate-based activators promote gel formation, aligning with the superior performance of liquid activators observed in this study. These comparisons confirm the behaviour seen in our results and support the use of a 70% FA–30% MK blend as an effective geopolymer mixture.

CONCLUSION

The experimental study revealed that the mechanical performance of geopolymer concrete is significantly influenced by the type of activator and the degree of metakaolin replacement. Strength steadily increased with up to 30% metakaolin substitution, but then slightly declined due to higher water demand and decreased flowability.

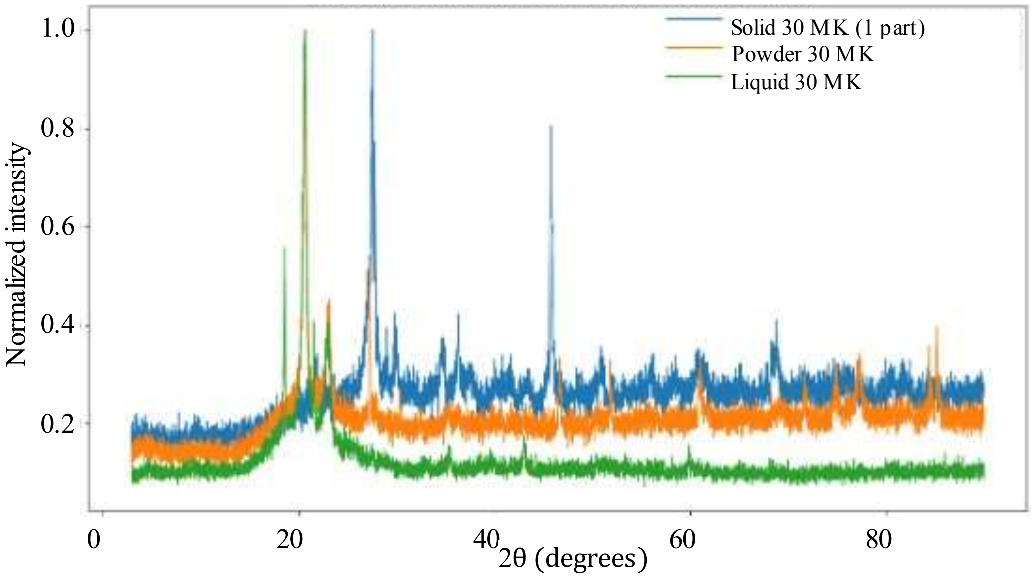

Figure 7. Comparison of XRD test of various proportions of geopolymer concrete.

Liquid alkaline activators yielded the highest compressive and flexural strengths, followed by powder activators. Solid activators resulted in lower strengths due to slower dissolution and limited geopolymerization. SEM analysis revealed that liquid-activated systems yielded the densest N–A–S–H gel with fewer microvoids. At the same time, XRD results indicated a greater amorphous phase, which aligns with the enhanced strength.

In terms of durability, the denser gel matrix, reduced porosity, and well-formed aluminosilicate networks observed in liquid-activated and powder-activated systems indicate strong resistance to chemical ingress and potential long-term stability. The presence of fewer microcracks and lower amounts of unreacted crystalline residue suggests that optimised fly ash–metakaolin blends (especially the 70% FA – 30% MK combination) are likely to exhibit improved sulfate resistance, reduced permeability, and better performance under thermal cycling – characteristics desirable for durable infrastructure materials. Although the current study did not directly quantify durability parameters, the microstructural evidence strongly supports their potential for high durability compared to traditional OPC systems.

From an industrial-scale application perspective, the findings highlight significant practical opportunities. The liquid activator system offers high strength and superior microstructure, making it suitable for precast products, structural elements, and high-performance applications. Powder activators, despite slightly lower strength, present a safer and more manageable alternative for large-scale onsite use due to easier storage, transport, and reduced chemical handling risks. Solid one-part geopolymer systems offer the most extraordinary operational simplicity. They are promising for mass production of non-structural components, low-load pavements, bricks, blocks, and backfill materials where moderate strength is sufficient. The reliance on fly ash and metakaolin – both widely available industrial by-products – also enhances scalability and economic feasibility.

Overall, this study demonstrates that a 70% fly ash and 30% metakaolin blend activated with a liquid system provides the best balance of strength, microstructural stability, and potential durability. With further optimisation of curing regimes and durability-specific testing, these geopolymer systems present a strong pathway for industrial adoption as low-carbon, sustainable construction materials.

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