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Effect of quick lime addition for improving compressive strength of fly ash based (cement free) geopolymer concrete

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In this current investigation, a comprehensive exploration has been undertaken to advance the development of “Class F” fly ash-based Geopolymeric mortar and concrete, utilizing fly ash that has been sourced from the Satpura Thermal Power Station in Sarni, District Betul, Madhya Pradesh, India. The study has systematically investigated the influence of Quick Lime addition in the formulation of fly ash-based Geopolymeric mortar and concrete, with a particular focus on enhancing compressive strength within the constraints of ambient atmosphere conditions. The Geopolymeric binder has been meticulously crafted using sodium hydroxide and sodium silicate as alkaline activators. Systematic variations of Quick Lime dosages (ranging from 0% to 10% by weight of fly ash) have been introduced, and the ensuing specimens have undergone scrutiny for standard consistency and setting time under ambient temperature curing. The outcomes have underscored a discernible trend wherein the judicious addition of 7 to 9 wt.% calcium oxide (Quick Lime) to the fly ash matrix has precipitated a noteworthy reduction in setting time at room temperature, concurrently manifesting a substantial enhancement in compressive strength for Geopolymeric mortar and concrete formulations. The elucidation of the binder's microstructural phases and their chemical characteristics has been pursued through rigorous analytical methodologies, encompassing X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), and field emission scanning electron microscopy (FE-SEM). Cost analysis has also been conducted for 1m³ of concrete, comparing conventional concrete (M25) and fly ash-based Geopolymer concrete.

Keywords: Cement free concrete, Compressive strength, Cost analysis, Fly ash, Geopolymer, Industry by-product, Quick lime, Setting time

1 Introduction

Cement, a pivotal constituent in construction, operates as a binding element in concrete and cement mortar. However, the production of Ordinary Portland Cement (OPC) entails significant carbon dioxide (CO_2) emissions, thereby contributing to the global greenhouse gas inventory. The production process yields a one- to-one ratio of CO_2 emission per ton of cement produced, and the annual global output of OPC, reaching 1.6 billion tons, constitutes approximately 7% of the overall CO_2 emissions globally. Additionally, the production of one ton of OPC necessitates the consumption of 1.5 tons of limestone, thereby exacerbating global warming and engaging substantial natural resource and energy inputs. This predicament underscores an exigency for an expeditious alternative to traditional cementitious materials. Geopolymers, distinguished by their superior material attributes vis-à-vis conventional counterparts, are garnering attention across diverse applications. Characterized by rapid setting, elevated compressive strength, resistance to fire and corrosive agents, and minimal CO_2 emission, geopolymers emerge as a promising alternative. Fabricated through alkali activation of aluminosilicate raw materials, such as metakaolin, fly ash, and slag, and employing alkali solutions such as sodium hydroxide and potassium hydroxide, alongside silica sources like sodium and potassium silicates, geopolymers present environmental advantages and economic benefits derived from the valorization of by-

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*Corresponding author (E-mail: manish.ampri@csir.res.in) products like fly ash Benhelal[1]. The nuanced interplay of diverse factors, encompassing raw material attributes, calcination temperatures, particle dimensions, activating solutions, and treatment methodologies, exerts considerable influence on both the macrostructure and microstructure of geopolymers. Furthermore, the molarity concentration of the NaOH activator solution emerges as a pivotal determinant shaping the physical and chemical characteristics of the resulting geopolymer binders Turner[2]. Empirical findings corroborate that fly ash, when employed as a precursor and activated by an alkaline activator, surpasses ordinary cement in terms of compressive strength Turner[2], Albitar[3]. Additionally, diverse silica-rich precursors, such as rice husk ash, ceramic and glass waste, kaolin, and clay, have demonstrated the potential to yield geopolymer binders exhibiting compressive strengths exceeding 20 MPa Law[4], Matalkah[5]. Beyond compressive strength, the temporal dynamics encapsulated in the initial and ultimate setting times of geopolymer binders stand as critical parameters necessitating meticulous examination. The proportional adjustment of fly ash precursors and ground granulated blast furnace slag (GGBS) profoundly influences the setting times, with an optimum GGBS/fly ash ratio of 40% yielding specific setting time intervals Davidovits[6]. Process dynamics are further modulated by precursor mass and alkaline activator composition, with sodium silicate, as an alkaline activator, eliciting notable reductions in setting times. Augmenting the water- to-solid (W/S) ratio concomitantly amplifies compressive strength and bonding time. Furthermore, an elevated Silica/Alumina (Si/Al) ratio positively correlates with enhanced compressive strength while concurrently mitigating bonding time Yunsheng[7]. "Class C" fly ash sets faster than "Class F" fly ash. This work adopted a 14 M sodium hydroxide solution as an activator. He also explains how it is that $Ca(OH)_2$ is superior to CaO in terms of compressive strength. He also explains why a specimen cured at room temperature has a higher compressive strength than one treated in an oven at 70°C J. Temuujin[8]. The greater setting time was found in which contains less (7.07) CaO. Final setting time and Initial setting time were achieved from (21.08) CaO content, making it unsuitable for use in the concrete mixture, and it also received greater early age strength Abbas[9], Thaarrini[10]. The fineness of the geopolymer paste also contributes to its compressive strength. He also adds that the physical qualities of fly ash do not affect its strength or setting time, but the chemical composition does Adhitya Leonard Wijaya[11]. Increase in the binder ratio boosts compressive strength. Ground granulated blast furnace slag (GGBS), ordinary Portland cement (OPC), and calcium hydroxide ($Ca(OH)_2$) were commercially available additions. As alkaline solutions, sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) are utilized, with the concentration of sodium hydroxide in all mixes being 14 molars. As compared to OPC, fly ash geopolymers combined with GGBFS, OPC, and $Ca(OH)_2$ reduced the setting time. The use of additives in mortar reduces its workability slightly. It also demonstrates that the early age characteristics of geopolymer mortar improve noticeably as early as day one Pradip Nath[12]. The initial and final setting times of geopolymer paste at temperatures ranging from 65°C to 80°C were determined. The geopolymer paste was placed in the oven, and test readings were taken every 15 minutes. The initial and final settings took between 129 and 270 minutes. If the curing temperature is higher, less setting time is needed. Influence of sodium hydroxide solution, which results in a higher compressive strength of geopolymer mortar with a larger concentration of sodium hydroxide solution. 50 x 50 x 50 mm cube geopolymer mortar specimens were cast and oven cured for 24 hours at 65°C, 70°C, and 80°C for 28 days, with specimens treated at 65°C achieving higher compressive strengths in the range of 1.6 MPa– 20 MPa Djwantoro Hardjito[13]. Fly ash containing 9.42% CaO and 1.29% CaO was combined with Na_2SiO_3 and different hydroxide solutions [8M, 12 M, and 14M sodium hydroxide, potassium hydroxide, barium hydroxide, and lithium hydroxide]. Mixtures were created as samples [100 mm x 200 mm alkaline solutions/fly ash ratio: 0.31, Na_2SiO_3/ hydroxide solution ratio: 2.5] and cured under various conditions (dry oven curing at $55^{\circ C}$ and $70^{\circ C}$ and steam curing at 46°C for 24 hours) before being inspected on the seventh day. The results showed that the microstructure of fly ash particles and the calcium content impacted the setting time and compressive strength of geopolymer concrete. The concentration of NaOH solution, which has the greatest strength of all alkaline hydroxides (14M delivers better results than 8M), has been shown to alter the rate of polymerization. Because of the interaction of CaO with the water in the matrix, the C-S-H gel was formed in conjunction with higher CaO concentrations, resulting in larger strengths ranging from 36 N/mm^2 to 43 N/mm^2 Gaurav Nagalia[14]. The acid resistance of fly ash-based geopolymer mortar specimens with Na_2O

percentages ranging from 5% to 8% was evaluated experimentally. For up to 24 weeks, the specimens were submerged in 10% Sulfuric acid and 10% Nitric acid solutions. At regular intervals, the acid resistance was assessed in terms of surface corrosion, residual alkalinity, weight variations, and compressive strength. Geopolymer mortar samples did not change colour and remained structurally sound, despite the exposed surface being somewhat softer. A corroded surface could be viewed using an optical microscope, which increased with the period of exposure. The alkalinity loss was determined by the alkali concentration of the geopolymer mortar. In both Sulfuric acid and Nitric acid solutions, mortar with less Na_2O lost alkalinity quicker than mortar with more Na_2O. Weight loss varied from 0.81% to 1.64% in Sulfuric acid and from 0.21% to 1.42% in Nitric acid after 12 weeks of exposure. After 24 weeks of exposure, the compressive strength of Sulfuric acid rose from 44% to 71%, while that of Nitric acid grew from 40% to 70%. This study revealed that geopolymers based on fly ash were extremely resistant to both sulfuric and nitric acid Partha Ghosh[15]. Geopolymers were used to explore the characteristics of concrete. The structure of the geopolymers generated from fly ash was largely AlQ4 (4 Si), SiQ4 (4 Al), and SiQ4 (2–3 Al). The fly ash-based geopolymer was a porous substance. The porosity of the geopolymers in the region of nanopores was fairly comparable, independent of the circumstances under which they were prepared. The strength of the geopolymers was significantly influenced by macro-pores (103 nm and larger) generated as a result of the air entrained into the geopolymers, which might be owing to the partial reaction of fly ash particles. Because of the presence of the geopolymer phase with the C-S-H phase, the addition of Ca- containing additives (slag, gypsum) significantly lowers porosity. The geopolymer concrete does not shrink due to hydration. The compressive strength-to-tensile strength ratio during bending varies between 10.0 and 5.5. In geopolymer concrete, there is no transition phase between the binder and the particles František Škvára[16].

In a discernible departure from antecedent inquiries into alkali-activated fly ash, this study scrutinizes the augmentation of mechanical properties through the introduction of calcium oxide within the framework of alkali-activated fly ash-based geopolymers. This scholarly pursuit seeks to unravel novel dimensions in the enhancement of the mechanical robustness of geopolymers.

2 Materials and Methods

The aluminosilicate precursor employed in the fabrication of fly ash-based geopolymers consisted of Class F fly ash sourced from the Satpura Thermal Power Station in Sarni, located 150 km from Bhopal city. The fly ash, exhibiting particle sizes within the range of 10 to 100 μm, underwent a preliminary drying process. Chemical composition analysis via X-ray fluorescence (XRF), detailed in Table 1, highlighted the preponderance of silica and alumina, collectively constituting 74.7% by weight of the fly ash. For the optimization of geopolymer binder characteristics, incremental additions of calcium oxide (CaO) were made at varying levels ranging from 0 to 10% by weight of fly ash. The synthesis of fly ash-based geopolymers employed a sodium hydroxide and sodium silicate flakes solution, procured from Central Drug House (Pvt.) Ltd. for Laboratory Supplies, boasting a purity of 96.0%. Augmenting both ambient and residual compressive strength, calcium oxide was introduced at 8% by weight of fly ash. Separate batches were cast for geopolymer mortar and geopolymer concrete. The calcium oxide (CaO) utilized, with a purity of 96%, was sourced from Central Drug House (Pvt.) Ltd. for Laboratory Supplies. Standard River sand, hailing from local resources in Bhopal City, India, served as the raw material for mortar specimens. Properties determined according to IS 383: 2016. yielded fineness modulus (FM), specific gravity, and absorption values of 2.658, 2.55, and 1.09%, respectively. Coarse aggregate from local resources in Bhopal City was employed for concrete specimen preparation.

Mortar mixtures, featuring a molar ratio of 12.5 for fly ash and alkaline additives, were prepared. With a 1:2 ratio of fly ash to river sand, the resulting mixtures were cast into 70.6 x 70.6 x 70.6 mm cubic molds, subjected to room temperature curing, and stored for compression tests as per IS 4031-6 (1988). A flexural strength test, conducted using a Jinan

Table 1 — Chemical compositions of the fly ash (wt.%) obtained via XRF analysis.

Silicon Dioxide (SiO_2)	Aluminium Oxide (Al_2O_3)	Ferric Oxide (Fe_2O_3)	Calcium Oxide (CaO)	Potassium Oxide (K_2O)	Sodium Oxide (Na_2O)	Sulphur Trioxide (SO_3)	Magnesium Oxide (MgO)	Titanium Dioxide (TiO_2)
47.1	27.6	19.2	1.57	1.27	0.118	0.459	0.698	1.35

testing equipment WDW-50 (UTM) universal testing machine at a loading rate of 50 N/s until failure, adhered to ASTM C348. Compressive strength assessments were conducted using a 3000 kN capacity compressive testing machine from Hydraulic and Engineering Instruments (HEICO), with specimens tested at 3 days, 7 days, 14 days, and 28 days.

2.1 Characterization of fly ash

Figure 1 presents scanning electron microscopy (SEM) micrographs of the fly ash material, revealing that the particles exhibit a predominantly spherical morphology characterized by a smooth surface texture.

Styszko-Grochowiak[17] indicate that fly ash is primarily composed of fine spherical grains, which consist of a complex mixture of aluminosilicates and calcium compounds. SEM microscope made with a scanning electron microscopy (Jeol Jem -35 -CF).

The X-ray diffraction (XRD) analysis of fly ash, as illustrated in Fig. 3, reveals distinct patterns indicative of its mineralogical composition. The XRD pattern prominently features pronounced peaks corresponding to quartz (SiO_2) and mullite ($Al_2O_3 \cdot nSiO_2$) minerals. A broad hump observed in the 25° to 30° (2θ) range is attributed to a significant presence of the amorphous aluminosilicate phase within the fly ash matrix. According to Bhagwanjee Jha[18] this amorphous phase predominantly resides in the outer layers of the spherical fly ash particles, whereas the crystalline quartz and mullite are located in the intermediate and inner core regions, respectively. The stronger and more intense peaks associated with quartz and mullite suggest a lower reactivity of these crystalline phases compared to the amorphous constituents.

Field emission scanning electron microscopy (FE-SEM) analyses of paste specimens were executed, with a 20-nm thick carbon coating applied using an SC7640 auto/manual high- resolution sputter coater. The mineralogical examination of specimens was carried out through X-ray diffraction (XRD) using an Ultima IV X-ray diffractometer machine, operating at 40 kV and 40 mA with Cu X-ray radiation.

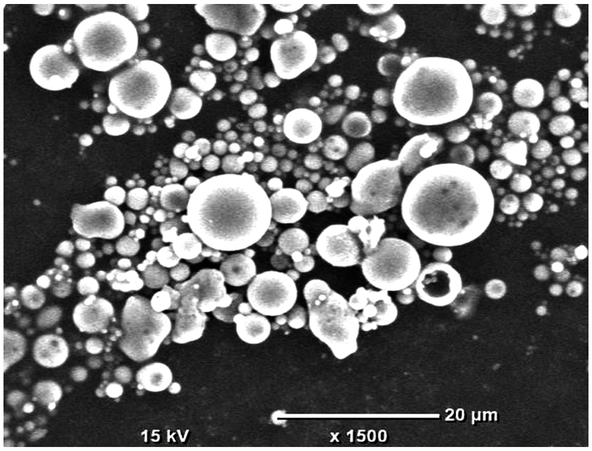

Fig. 1 — SEM image of fly ash

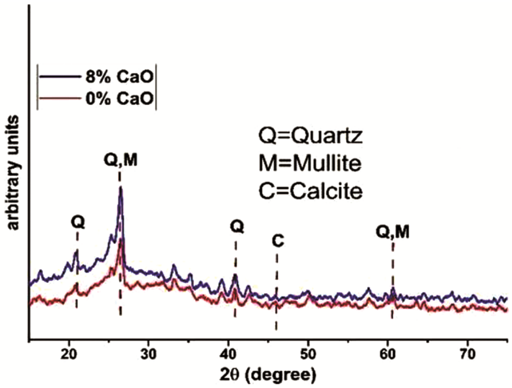

Fig. 2 — X-Ray Diffraction Analysis for the fly ash-based geopolymer binder without added calcium oxide and after adding 8% calcium oxide fly ash-based geopolymer binder.

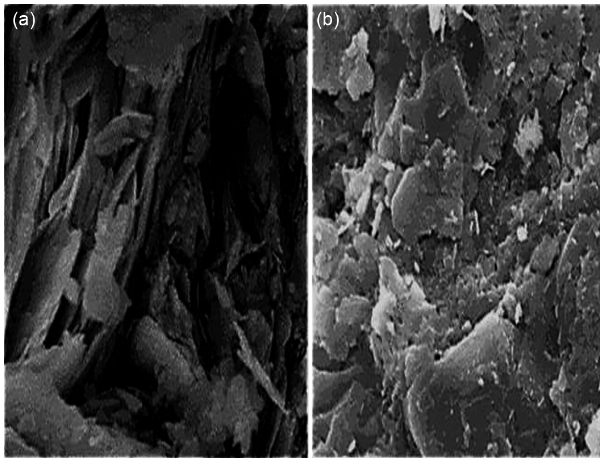

Fig. 3 — FE-SEM images for the (a) fly ash-based geopolymer binder without added calcium oxide and (b) adding 8% calcium oxide into the geopolymer binder.

3 Results and Discussion

3.1 Geopolymer paste

Figure 3 presents a comparative morphological assessment via FE-SEM for two distinct states of a fly ash-based geopolymer binder. Specifically, Fig. 3a depicts the geopolymer binder in its unaltered form without the introduction of calcium oxide, while Fig. 2b showcases the binder subsequent to the inclusion of an 8% calcium oxide admixture. In the absence of added calcium oxide, the as-received fly ash- based geopolymer manifests a plate-like morphology, characterized by particles predominantly smaller than 10 µm. The inherent structure exhibits a pseudo- hexagonal configuration with plate entities that have the potential to attenuate the reactivity of the geopolymer binder. Following the introduction of 8% calcium oxide, Fig-3b delineates a discernible metamorphosis where the sheets of the fly ash- based geopolymer binder exhibit heightened deformation and local condensation into bundled formations, displaying reduced crystalline regularity.

3.2 Mechanical properties

3.2.1 Effect of CaO on the standard consistency and initial and final setting on fly ash based- geopolymer

The preeminent outcome in the evaluation of geopolymer paste's initial and final setting times was achieved at an 8% calcium oxide concentration, corresponding to a standard consistency of 26% by weight of fly ash within the designated 25-30% range for all 11 conducted tests. It is imperative to note that these experiments were conducted under controlled ambient conditions, specifically at room temperature. Figure 4 is a graphical representation of the influence of calcium oxide on the initial and final setting times of fly ash- based geopolymers. Throughout all tests, standard consistency was rigorously maintained within the prescribed 25-30% range based on the weight of fly ash. In the absence of supplemental calcium oxide, the initial setting time was notably protracted, registering at 1365 minutes, while the final setting time exhibited an even greater duration, lasting 7200 minutes. Conversely, the introduction of calcium oxide, particularly Sat an 8% concentration, yielded the most favorable outcome. This composition demonstrated a significantly abbreviated initial setting time of 120 minutes and a final setting time of 330 minutes, indicating a substantial enhancement in both initial and final setting characteristics attributed to the incorporation of calcium oxide.

3.2.2 Effect of CaO on the mechanical properties of the fly ash based –Geopolymer mortar

In Fig. 5, the impact of calcium oxide (CaO) supplementation on the compressive strength of fly ash-based geopolymer mortar mixes is elucidated. The addition of 8 wt.% CaO resulted in a discernible augmentation in compressive strength, attaining 49.91 MPa, in stark contrast to control specimens devoid of CaO, which exhibited a compressive strength of approximately 45.75 MPa. The assessment of geopolymer mortar flexural strength was undertaken on

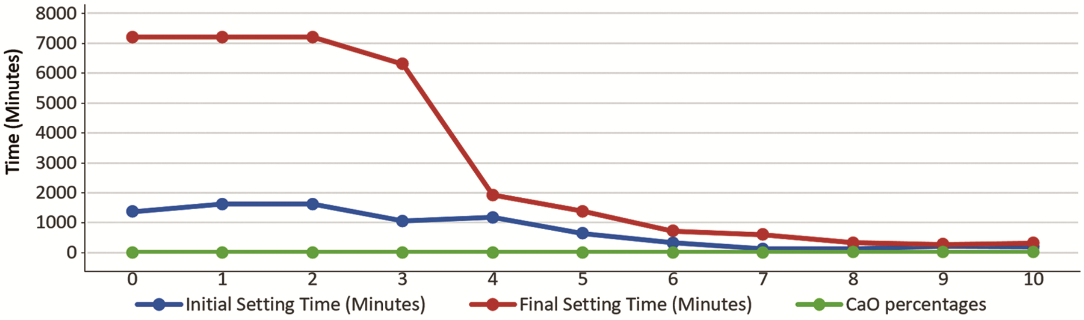

Fig. 4 — Initial and final setting time with CaO percentage

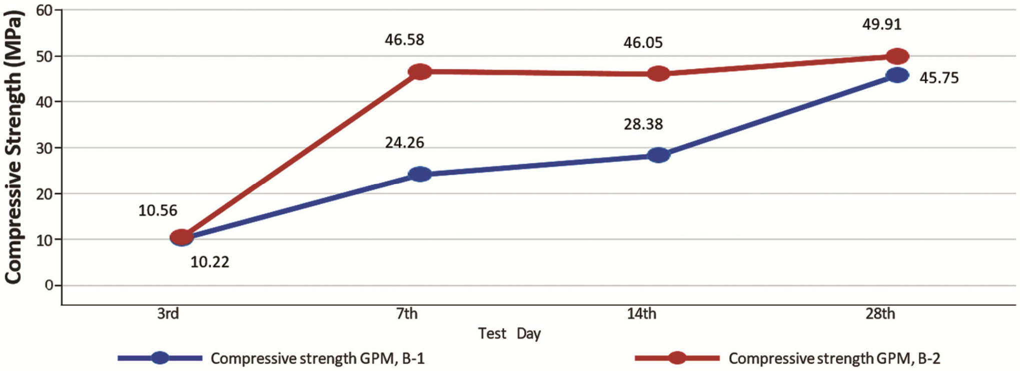

Fig. 5 — Effect of CaO on the mechanical properties of fly ash based-Geopolymer mortar.

the 28th day. Geopolymer mortar lacking added CaO (GPM, B1) manifested a maximum flexural strength of 4.21 MPa on the 28th day, whereas the incorporation of CaO in geopolymer mortar (GPM, B-2) yielded a heightened flexural strength of 5.56 MPa on the same day. These results underscore the salient influence of CaO addition under ambient room temperature curing conditions on the compressive strength of fly ash-based geopolymers mortar. The supplemented CaO is postulated to engage in reactions with silica components within sodium silicate and/or fly ash, culminating in the formation of C-A-S-H hydrates. This mechanistic insight elucidates the observed enhancement in compressive strength. Effect of CaO on the mechanical properties of fly ash based-geopolymer concrete. In Fig. 6, the nuanced impact of calcium oxide (CaO) augmentation on the compressive strength of fly ash-based geopolymer concrete mixes is graphically presented. Specifically, the introduction of 8 wt.% CaO resulted in a marked augmentation of compressive strength, attaining 31.36 MPa, in stark contrast to control specimens devoid of CaO, exhibiting a compressive strength of approximately 14.65 MPa. These results underscore the substantive influence of CaO addition under controlled room temperature curing conditions on the compressive strength of fly ash-based geopolymers concrete. The observed enhancement in compressive strength is attributed to plausible chemical interactions wherein added CaO reacts with silica constituents within sodium silicate and/or fly ash, precipitating the formation of C-A-S-H hydrates. This elucidation provides a detailed mechanistic insight into the consequential role played by CaO in fortifying the compressive strength attributes of fly ash-based geopolymers concrete.

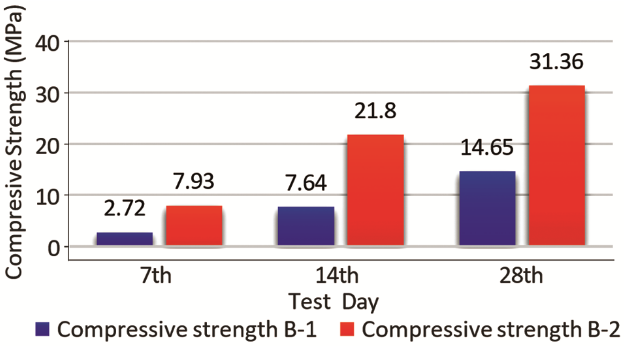

Fig. 6 — Effect of CaO on the mechanical properties of fly ash-based-geopolymer concrete. Effect of CaO on the mechanical properties of fly ash based-geopolymer mortar

3.3 Cost comparison of geopolymer concrete and M25 grade conventional Cement Concrete

This section undertakes a comparative cost analysis of geopolymer concrete in relation to M25 grade conventional cement concrete. In the construction industry, the critical dimensions of time, cost, and quality significantly influence overall project efficacy and industry standards. The field of civil engineering is particularly attentive to advancements that offer improvements in these dimensions, as innovations that enhance these factors can substantially influence industry practices and outcomes. The compressive strength test is a fundamental and straightforward procedure frequently employed to assess concrete. This test, typically conducted at 28 days after casting, serves as a critical indicator of concrete quality, commonly referred to as the concrete grade. Concrete undergoes a continuous process of strength development through hydration over time. Initially, this development occurs at an accelerated rate; however, the rate of strength gain progressively diminishes as the material ages. The 28-day compressive strength is widely accepted as an approximation of the concrete's ultimate strength, reflecting its long-term performance characteristics.

The M25 grade conventional concrete is characterized by a 28-day compressive strength of 32.15 MPa. In contrast, geopolymer concrete formulated with 100% fly ash (FA100%) demonstrates a compressive strength of 31.36 MPa to achieve a comparable structural performance. This chapter presents a detailed cost analysis, comparing the expense per cubic meter of geopolymer concrete at this specific mix proportion with that of M25 grade conventional concrete.

Table 2 — Geopolymer concrete versus. conventional cement concrete cost analysis

Material	Unit	Rate (Rs)	Control Cement Concrete (M25)		GPC (FA100%)	
			Quantity	Amount (Rs)	Quantity	Amount (Rs)
Cement	bags	340/-	540 kg	3400/-	0	0.00
Fly ash	Kg	2/	0	0	540	1080/-
CA	Tones	1320/-	1200 kg	1584/-	1200 Kg	1584/-
Sand	Tones	1400/-	650 kg	910/-	650 kg	910/-
Alkaline Activator	Kg	32/-	0/-	0/-	95 kg	3040/-
Water	Liters	0.1/-	290 L	29/-	110 Ltr	11/-
Total (Rs)				5887/-		6625/-

According to Table 2, the initial material cost of Geopolymer Concrete (FA100%) was approximately 11% greater than that of Conventional Cement Concrete (M25). This disparity in material costs indicates that geopolymer concrete incurs a significantly higher expense relative to Conventional concrete when both are used to achieve comparable structural strength. The elevated production costs associated with geopolymer concrete in the current study should not be regarded as a barrier to continued research. This is because the materials utilized in the study were of higher quality than those typically required for construction applications, and their prices were based on prevailing market rates rather than the intrinsic production costs.

4 Conclusion

The empirical investigation delineates the nuanced impact of calcium oxide (CaO) and ambient curing on the engineering properties, particularly compressive and flexural strengths, of geopolymeric mortar and concrete. The findings are systematically expounded as follows:

Standard consistency testing: Adherence to IS 4031 (Part 4) (1988) protocols facilitated the determination of

a. standard consistency for geopolymeric paste in 11 distinct samples. The outcomes, ranging between 25 and 30% by weight of fly ash, conform to the established standards for cement (25-35% standard consistency).
b. Initial and final setting Time: An intricate analysis of the initial and final setting times of geopolymer paste across 11 samples was conducted. The CaO-free paste exhibited an initial setting time of 1365 minutes and a final setting time of 7200 minutes. Introduction of 7 to 9 wt.% CaO significantly truncated these durations to 120 and 330 minutes for initial and final setting, respectively.
c. Flow table tests for workability: Quantitative assessment of flowability through flow table tests delineated the workability of geopolymer mortar. Batch-1 (without added CaO) demonstrated superior flowability (45%) in comparison to Batch-2 (with added CaO) registering at 35%. The slump test for geopolymer concrete further underscored this trend, with Batch-1 exhibiting a 140 mm slump and Batch-2 displaying an 80 mm slump.
d. Compressive strength testing: Rigorous examination of compressive strength at the 28th day revealed 45.75 MPa for GPM, B-1, and an elevated 49.91 MPa for GPM, B-2 in the geopolymer mortar domain. Geopolymer concrete at the same age exhibited 14.65 MPa for GPC, B-1, and a substantially augmented 31.36 MPa for GPC, B-2—reflecting a twofold increase.
e. Flexural strength testing: In-depth scrutiny of flexural strength for geopolymer mortar elucidated a peak strength of 4.21 MPa for GPM, B-1, and 5.56 MPa for GPM, B-2 on the 28th day.
f. This comprehensive analysis underscores the intricate interplay of CaO and ambient curing conditions, providing a scholarly insight into the discernible alterations in the engineering properties of geopolymeric mortar and concrete.
g. Cost analysis: Cost of $1m^3$ geopolymer concrete is around 11% higher than conventional cement concrete (M25), because of the alkaline activator solution used in it. The chemical which we used to make AAS, it increases the cost of geopolymer concrete.

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