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Experimental and Statistical Optimization of Low-carbon Binder Concrete under Varying Alkaline Activator Conditions

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Abstract

This study investigates the mechanical and microstructural performance of fly ash–GGBS-based geopolymer concrete activated using sodium carbonate and sodium silicate in varying ratios (1:1, 1.5:1, and 1:2), with a conventional sodium hydroxide–sodium silicate mix serving as the control. Workability was evaluated through slump tests, while compressive strength was measured at 7, 14, and 28 days. The control mix ($NaOH:Na_2SiO_3$ = 1:2.5) achieved a 28-day compressive strength of 48.6 MPa, while the carbonate-based mixes recorded strengths of 44.1 MPa (1:1), 36.3 MPa (1.5:1), and 41.2 MPa (1:2). SEM–EDS analyses confirmed that the 1:1 mix exhibited a dense matrix with a balanced distribution of Si, Al, Na, and Ca, indicative of the formation of both N–A–S–H and C–A–S–H gels. XRD patterns further corroborated the presence of these hydration products, with reduced unreacted phases in the optimized mix. Statistical evaluation using Response Surface Methodology (RSM) and ANOVA identified the alkaline activator ratio as a significant factor influencing compressive strength ($p < 0.05$), with the model achieving a high coefficient of determination (R^2 = 0.985). The predicted and experimental strengths showed close agreement, validating the model's predictive capability. Results demonstrate that a 1:1 Na_2CO_3:Na_2SiO_3 ratio provides an optimal balance between mechanical performance and sustainability, making it a promising alternative for high-strength, eco-friendly geopolymer concrete applications.

Keywords

geopolymer concrete, GGBS, fly ash, sodium carbonate, alkali activation

1 Introduction

The increasing demand for sustainable construction materials has prompted consideration of the reduction of dependence on Ordinary Portland Cement (OPC), whose production is responsible for approximately 7–8% of global CO_2 emissions [1]. Geopolymer Concrete (GPC), produced by activating industrial by-products like fly ash (FA) and Ground Granulated Blast-Furnace Slag (GGBS) with alkaline solutions, has emerged as a viable low-carbon alternative owing to its superior mechanical performance, durability, and diminished environmental impact [2]. Over 28 days, the compressive strengths of 45.8 MPa in FA-based mixtures that were ambient-cured by utilizing sodium hydroxide–sodium silicate ($NaOH–Na_2SiO_3$) were attained [3]. Although they are effective, conventional activators like $NaOH–Na_2SiO_3$ have several downsides, including high costs, a large carbon footprint, and potential risks when they are handled [4, 5].

As high-performance alternatives to OPC, FA–GGBS binary systems that are activated with conventional alkali ($NaOH–Na_2SiO_3$) have a long history of use. Under ambient curing conditions, compressive strengths exceeded 50 MPa after 28 days. They attributed this to the creation of synergistic gel networks [6, 7]. In the same way, it has been noted that replacing fifty percent FA with GGBS increased in early-age strength of more than forty percent, resulting in a dense C–A–S–H/N–A–S–H hybrid microstructure [6].

Traditional high-alkali activators, such as high-molarity NaOH (8–16 M), significantly improve strength. The compressive strength increases from lower values at an activator ratio of 1.5 to a maximum of 67.6 MPa at 2.5 when heat-cured, demonstrating the positive influence of higher silicate content on geopolymerization. However, these activators present issues in terms of cost, safety, and sustainability [8]. Alternatives that are low in carbon and alkali

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have been the focus of recent work. It has been shown in 2024 that low-carbon concrete mixtures can still fulfil structural performance requirements. For instance, a mix incorporating 30% slag and 100% pre-consumer coarse recycled aggregate achieved a 90-day compressive strength of 40.2 MPa. This was made possible by blending ultrafine slag with fly ash and using ambient curing conditions [9].

Sodium Carbonate (Na_2CO_3) provides cost and safety advantages as a result of its lower pH of 11.5. Nevertheless, it frequently fails to meet expectations when employed independently. For instance, slag-activated systems obtained only approximately 25 MPa after 28 days. To resolve this issue, composite systems have been evaluated [10].

Na_2CO_3 and Na_2SiO_3 are emerging as environmentally friendly alternatives, particularly under ambient curing circumstances, owing to their diminished ecological impact and safety benefits. Na_2CO_3 frequently results in a reduced rate of strength development unless accompanied by enough silicate concentration [11, 12]. Na_2CO_3, has shown 28-day strengths of 30–40 MPa when adjusted with silicate additions [13, 14]. On the other hand, Na_2CO_3 by itself frequently causes early-age impaired strength due to the development of phases such as gaylussite [15]. A solution that helps reduce these difficulties is the combination of Na_2CO_3 and Na_2SiO_3 due to the formation of a denser and more refined microstructure [4].

Response Surface Methodology (RSM) is widely adopted in geopolymer concrete research for optimizing mix design variables with minimal experimental effort. RSM has been used to optimize sand-to-fly ash and water-to-binder ratios, achieving less than 5% error between predicted and experimental compressive strength under ambient curing [16]. It has also improved the mechanical properties of fly ash–slag-based geopolymer systems [17] and demonstrated prediction errors below 2% in GGBS- metakaolin based mixes validated through ANOVA [18]. Comparative analysis with machine learning techniques such as regression trees and random forests showed that RSM achieved a Prediction Correlation Coefficient (PCC) of 0.994 and a Mean Absolute Percentage Error (MAPE) of 0.708 [19].

This study uniquely investigates the influence of varying Na_2CO_3:Na_2SiO_3 ratios (1:1, 1.5:1, and 1:2) at a fixed 3 M Na_2CO_3 concentration under ambient curing conditions a combination that has not been systematically explored in previous research. While several studies have highlighted the potential of sodium carbonate-based binders, a detailed comparison of these specific ratios under practical, low-energy curing conditions remains limited. To address this gap, RSM using a Box-Behnken Design was employed to statistically evaluate and optimize the effects of activator ratios on the fresh and hardened properties. Key performance metrics such as slump and compressive strength (at 7, 14, and 28 days) were assessed, alongside microstructural characterization using SEM, EDS, and XRD. The findings aim to support the development of a low-carbon, cost-effective, and field-applicable alternative binder system for sustainable construction.

2 Materials and methods

2.1 Aluminosilicate source materials

The primary binder materials used were GGBS and Class F fly ash (FA). GGBS, a calcium-rich by-product of the steel industry, was blended with siliceous fly ash to form a reactive aluminosilicate base. This binary binder system combines the rapid setting characteristics of GGBS with the long-term pozzolanic activity of fly ash, ensuring both early strength gain and long-term durability [20–22].

2.2 Alkaline activators

A combination of Na_2CO_3 and Na_2SiO_3 was employed as the alkaline activator. Na_2CO_3 was selected due to its lower alkalinity and reduced environmental and handling hazards compared to conventional NaOH, thereby contributing to lower CO_2 emissions [23]. Na_2SiO_3 solution was added to improve the dissolution of the aluminosilicate source materials and to facilitate the formation of a stable geopolymer matrix [24]. The alkaline solution was prepared 24 h before mixing to allow complete dissociation of sodium carbonate and to increase the reactivity of the system.

2.3 Aggregates

The fine aggregate used was Manufactured Sand (M-sand) conforming to Zone II grading as per IS 383:2016 standard [25]. M-sand were chosen for its consistent particle size distribution, clean surface texture, and angular shape, which collectively improve the workability and packing density of the mix. The coarse aggregate was natural crushed stone with a nominal maximum size of 12.5 mm, also conforming to IS 383:2016 standard [25] specifications. Its uniform grading and mechanical stability made it suitable for use in geopolymer concrete.

A summary of the materials used in this study is illustrated in Fig. 1, including the source and role of each component in the geopolymer concrete mix.

Fig. 1 Materials for GPC preparation

2.4 Alkaline activator preparation

3 M sodium carbonate (Na_2CO_3) solution was selected as the primary alkaline activator, balancing effectiveness in promoting geopolymerization with improved handling safety and environmental performance. To prepare 1 liter of this solution, 318 g of anhydrous sodium carbonate (molar mass = 106 g/mol) were gradually dissolved in potable water. The water was preheated to approximately 40–50 °C to aid in dissolution.

The sodium carbonate was added slowly while continuously stirring the solution using a magnetic or mechanical stirrer. This method ensured uniform dispersion, prevented clumping, and promoted complete solubilization. Stirring was continued for 10 to 15 min, or until the solution appeared completely homogeneous with no undissolved particles.

After preparation, the solution was allowed to cool and stabilize at ambient temperature (25 ± 2 °C) for at least one hour to ensure chemical equilibrium. Following this resting period, the required quantity of sodium silicate solution was incorporated to formulate the complete alkaline activator system. The combined activator solution was stored in a sealed, air-tight container and left to condition for 24 h at room temperature to allow for adequate chemical interaction and stabilization before being used in the geopolymer concrete mixture. The overall process of activator preparation is illustrated in Fig. 2.

2.5 Characterization of aggregates and binder system

To confirm the suitability of raw materials for geopolymer concrete production, the physical and mechanical properties of the aggregates and binder components were thoroughly evaluated. The test results are summarized in Tables 1 and 2.

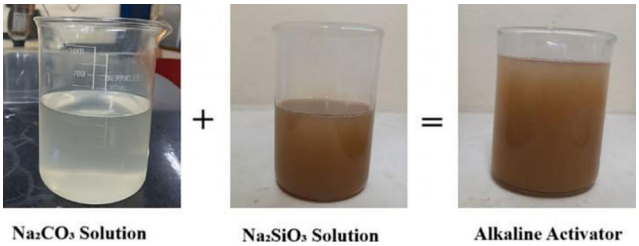


Fig. 2 Alkaline activator preparation

Table 1 Basic properties of natural aggregate and M-sand

Parameter	Natural aggregate	M-sand
Specific gravity	2.67	2.64
Water absorption (%)	1.125%	1.24%
Impact value (%)	22.5	–
Crushing value (%)	24.3	–
Abrasion value (%)	28.6	–

Table 2 Basic properties of binder materials

Parameter	GGBS	FA
Specific gravity	2.9	2.3
Specific surface area (m^2/kg)	400	320
Finer than 90 µm (%)	97.4%	86.0%

2.5.1 Aggregate suitability assessment

Coarse aggregates and manufactured sand (M-sand) were tested as per IS 2386 (Parts I–IV):1963 [26]. The coarse aggregates were subjected to impact, crushing, and abrasion tests to determine their mechanical strength, all of which met the specified standards. The M-sand used for fine aggregate conformed to Zone II grading, exhibiting good gradation, angularity, and cleanliness, making it ideal for geopolymer applications in terms of both strength and workability.

2.5.2 Binder material properties

The physical characteristics of GGBS and Class F fly ash, including specific gravity, fineness, and surface area, were evaluated to assess their reactivity potential. The high fineness and increased surface area of these binders are critical in achieving effective geopolymerization, especially when activated with the sodium carbonate-sodium silicate system.

3 Experimental methodology

The experimental methodology was designed to evaluate the influence of sodium carbonate-based alkaline activators on the fresh and hardened properties of geopolymer concrete cured at ambient conditions.

The key objective was to investigate the effect of varying the Na_2CO_3:Na_2SiO_3 ratio on workability, compressive strength, and microstructural development, while keeping the sodium carbonate concentration constant at 3 M. Three ratios (1:1, 1.5:1, and 1:2 by weight) were explored by adjusting the quantity of sodium carbonate relative to

sodium silicate. All mixes maintained a constant activator-to-binder (A/B) ratio of 0.50, and the binder phase consisted of 50% GGBS and 50% low-calcium fly ash. The fine aggregate was M-sand, and the coarse aggregate was 12.5 mm crushed stone, both sourced in accordance with IS 383:2016 standard [25]. No chemical admixtures or heat curing were applied to isolate the performance effects of the alkaline activator system.

For effective comparison, a conventional geopolymer concrete mixture was developed utilizing NaOH (commonly at 10 M) alongside Na_2SiO_3 solution, maintaining a $NaOH:Na_2SiO_3$ mass ratio of roughly 2.0, which is extensively recognized in previous studies as optimal for ambient-cured fly ash-based geopolymers [27, 28]. This conventional mixture functioned as a reference benchmark to evaluate the comparative performance of sodium carbonate-based systems. All specimens were cured at ambient temperature (25 ± 2 °C) to simulate real-world settings and evaluate the viability of employing sodium carbonate as a substitute for traditional alkali activators, eliminating the necessity for thermal curing. To ensure statistical reliability, all tests were conducted on triplicate specimens for each mix combination, as shown in Fig. 3.

The dry constituents, including fly ash, GGBS, and M-sand, were first blended in a pan mixer for about 2 min to achieve uniform distribution. The alkaline activator solution was then added gradually while mixing continued for an additional 4 min until a homogeneous mixture was obtained. The prepared mix was immediately placed into cube molds in two layers, each compacted using a table vibrator for approximately 30 s to remove entrapped air and ensure uniform density. This standardized mixing and compaction procedure was followed for all batches to maintain consistency in fresh and hardened properties.

Workability was evaluated using the slump flow test as per IS 1199:2018 standard [29]. Fig. 4 represents the slump flow test of the specimen. The flow characteristics were anticipated to vary with the ratio of sodium silicate to sodium carbonate, as sodium silicate contributes significant viscosity to the activator mix.

The viscosity of the activator solution was found to increase with higher sodium silicate content because of the polymeric nature of soluble silica species in water glass. These polymer chains enhance interparticle attraction and restrict the free movement of solid particles within the paste, thereby reducing flowability. This elevated viscosity influences the mixture of homogeneity and indirectly affects the formation of pores during casting, which in turn governs the strength development of the hardened matrix. In addition to viscosity effects, the degree of precursor dissolution also plays a key role in workability; greater dissolution of aluminosilicate particles enhances the mix fluidity, whereas incomplete dissolution increases stiffness and heterogeneity [30].

Compressive strength tests were conducted on cube specimens measuring 100 mm × 100 mm × 100 mm, which were cast and tested at 7, 14, and 28 days following IS 516 (Part 1/Sec 1):2018 standard [31]. These results provided insights into early-age strength gain and long-term performance, helping to understand the impact of sodium carbonate on geopolymerization kinetics. Fig. 5 shows the compressive strength test of the casted specimen.

SEM analysis was conducted on 28-day-old fractured surfaces to examine the microstructure of the binder matrix. The test focused on identifying the gel morphology, compactness, and any remaining unreacted particles, offering evidence of the degree of geopolymerization. The High-Resolution Scanning Electron Micro-

Fig. 3 Casted specimens

Fig. 4 Slump flow test

Fig. 5 Compressive strength test

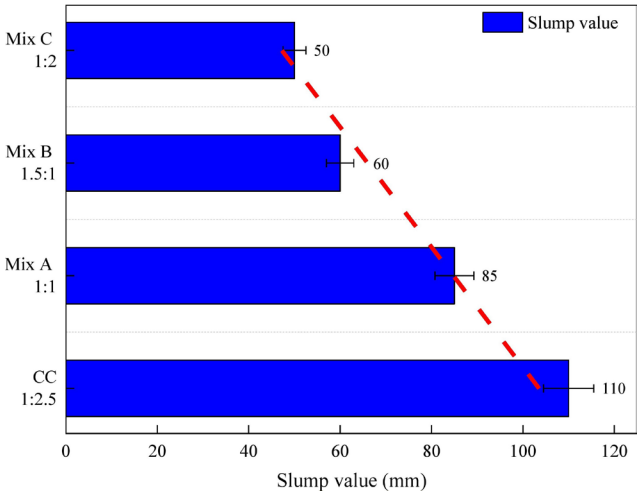


Fig. 6 Slump values for different activator ratios

scope (HRSEM) Thermo Scientific Apreo S model, was utilized to carry out the process of microstructural characterization of geopolymer concrete.

The elemental composition was analyzed using the Energy Dispersive X-ray Spectroscopy (EDS) system integrated with the SEM. The distribution of key elements Si, Al, Na, and Ca provided insight into the formation of binding phases such as N–A–S–H and C–A–S–H gels, which are essential indicators of successful geopolymerization X-ray Diffraction (XRD) analysis was performed using a Bruker USA D8 Advance, Davinci to examine the phase composition of geopolymer concrete specimens. The analysis aimed to identify crystalline and amorphous phases formed during geopolymerization. The presence of C–S–H and N–A–S–H gels, along with reduced crystalline peaks at higher sodium silicate content, confirmed enhanced gel formation and successful alkali activation.

4 Results and discussion

4.1 Slump flow test results

The workability of geopolymer concrete mixtures with varying sodium carbonate to sodium silicate ratios and also for Conventional Concrete (CC) was assessed using the standard slump cone method according to IS 1199:2018 standard [29]. The slump values documented for the all the mixtures are presented in Table 3, with the respective graphs shown in Fig. 6.

Table 3 Slump flow test results

Mix ID	Alkaline activator ratio	Alkaline activator type	Slump (mm)
CC	1:2.5	$NaOH:Na_2SiO_3$	110
Mix A	1:1	$Na_2CO_3:Na_2SiO_3$	85
Mix B	1.5:1	$Na_2CO_3:Na_2SiO_3$	60
Mix C	1:2	$Na_2CO_3:Na_2SiO_3$	50

It is evident from the results that the 1:1 ratio mix exhibited the maximum slump value of 85 mm, which suggests that it has superior flow ability and workability in comparison to the other mix. As the proportion of sodium carbonate increased (in Mix B with 1.5:1), the slump value decreased substantially, indicating a decrease in workability as a result of the higher viscosity and lower silicate content. Conversely, the workability was diminished when the sodium silicate content was increased (in Mix C with 1:2), which is likely a result of the high viscosity of the sodium silicate solution, which renders the mix less fluid and more cohesive. The 1:1 ratio mix's superior workability can be ascribed to a more balanced activator composition, which optimizes the silica contribution from sodium silicate and the alkalinity from sodium carbonate. This equilibrium allows for the proper dissolution of aluminosilicate precursors during the mixing process, which leads to a homogeneous and flowable mixture that is free of excessive stiffening or segregation.

In comparison, the conventional mix, which used sodium hydroxide and sodium silicate as alkaline activators, exhibited the highest slump value of 110 mm, highlighting its superior workability. This can be attributed to the lower viscosity of sodium hydroxide solution, which enhances mix fluidity and dispersion, allowing better flow without compromising cohesion.

These results are in accordance with previous research that has emphasized the significance of preserving an optimal balance between the carbonate and silicate components in the activator system to accomplish satisfactory rheological behavior in geopolymer concrete [30]. Therefore, the fresh concrete workability under ambient curing conditions was determined to be the most suitable at a 1:1 ratio of sodium carbonate to sodium silicate.

4.2 Compressive strength results

The compressive strength development of geopolymer concrete was assessed to determine its compliance with the target strength of M40 grade concrete under ambient curing circumstances. Standard cube specimens of 100 mm × 100 mm × 100 mm were utilized, in accordance with IS 516 (Part 1/Sec 1):2018 standard [31]. Nine specimens were cast for each mix ratio, and three were tested at 7, 14, and 28 days; the average value of each set was documented.

The concrete mixtures were formulated with three distinct sodium carbonates to sodium silicate ratios (1:1, 1.5:1, and 1:2), while maintaining a consistent sodium carbonate content of 3 M. The objective was to ascertain the optimal activator ratio for attaining M40 strength. Table 4 delineates the strength performance of various mixes, incorporating a Conventional Concrete (CC) mix for comparative analysis. Tested samples after 28 days are depicted in Fig. 7.

Among the geopolymer mixes, Mix A (Na_2CO_3:Na_2SiO_3 = 1:1) had the maximum compressive strength at all curing durations, attaining 44.1 MPa at 28 days, which is comparable to the strength of conventional concrete, reported at 48.6 MPa. Mix A demonstrated a consistent increase in strength, achieving 31.2 MPa at 7 days and 37.4 MPa at 14 days, signifying efficient geopolymerization and advancing gel formation. The balanced activator ratio of 1:1 facilitated optimal dissolving of aluminosilicate precursors and enhanced the formation of strength-contributing gels such as N–A–S–H and C–A–S–H.

Mix B (1.5:1) exhibited the lowest strength development, reaching only 36.3 MPa at 28 days. The higher sodium carbonate content may have hindered adequate silicate activation, slowing down the geopolymer reaction and reducing gel formation. On the other hand, Mix C (1:2), with increased sodium silicate, improved the performance relative to Mix B, attaining 41.2 MPa at 28 days. Although the silicate-rich activator exhibited higher viscosity, which marginally reduced workability, all specimens were compacted under identical vibration conditions to maintain uniform density and eliminate compaction-related variability. Therefore, the lower strength in this mix is attributed to limited precursor dissolution and slower gel formation rather than inadequate compaction. This confirms that the observed mechanical variations are governed by chemical rather than physical effects.

Table 4 Compressive strength test results

Mix ID	Alkaline activator ratio	Alkaline activator type	7-day (MPa)	14-day (MPa)	28-day (MPa)
CC	1:2.5	$NaOH$:Na_2SiO_3	35.3	41.6	48.6
Mix A	1:1	Na_2CO_3:Na_2SiO_3	31.2	37.4	44.1
Mix B	1.5:1	Na_2CO_3:Na_2SiO_3	24.6	30.1	36.3
Mix C	1:2	Na_2CO_3:Na_2SiO_3	28.1	34.5	41.2

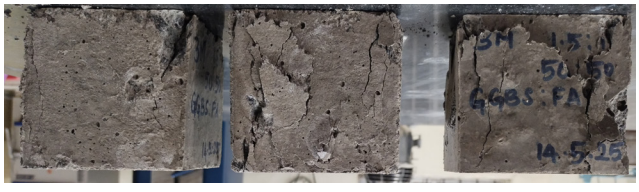


Fig. 7 Tested samples after 28 days

Conventional concrete, activated with $NaOH$:Na_2SiO_3 in a 1:2.5 ratio, attained the highest overall strength of 48.6 MPa after 28 days, with early-age values of 35.3 MPa and 41.6 MPa at 7 and 14 days, respectively. The strength results of the conventional OPC based mix were consistent with the expected M40 performance, confirming the reliability of the experimental procedure and providing a benchmark for evaluating geopolymer concrete. Furthermore, the reduced flowability of silicate-rich mixes may have increased internal porosity by restricting the release of entrapped air and limiting particle rearrangement during compaction. The presence of microvoids and weak interfacial zones consequently contributed to lower strength values. In contrast, mixes with balanced activator ratios exhibited smoother flow and denser particle packing, resulting in reduced pore volume and higher compressive strength.

In conclusion, Mix A exhibited performance equivalent to CC and was identified as the superior sodium carbonate-based geopolymer formulation. This finding demonstrates the viability of employing sodium carbonate-activated geopolymer concrete as a sustainable substitute for traditional concrete, especially under ambient curing circumstances. Fig. 8 represents the compressive strength results of the samples.

4.3 Microstructural analysis

The microstructural characterization was performed on 28-day-cured samples using SEM, EDS, and XRD to qualitatively support the mechanical performance trends. The analyses focused on gel morphology, matrix compactness, and reaction products, acknowledging that these techniques offer qualitative insights into phase evolution rather than absolute quantification. SEM observations helped identify overall matrix density and gel formation, while EDS spectra confirmed the presence of key elements (Si, Al, Na, and Ca) associated with N–A–S–H and C–A–S–H gels. XRD patterns were correlated with the precursor composition, revealing quartz and mullite

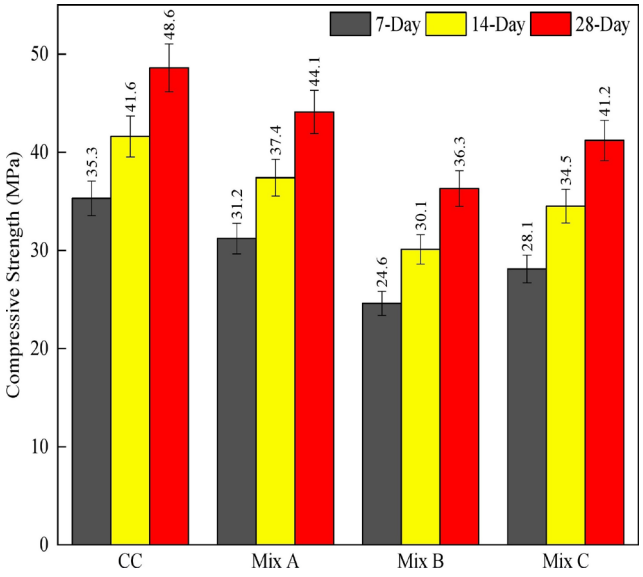


Fig. 8 Compressive strength results

from unreacted minerals and a broad amorphous hump representing geopolymeric gel formation. XRF data of fly ash and GGBS further substantiated the chemical basis for geopolymerization. Overall, the microstructural findings serve as qualitative evidence complementing the mechanical and chemical results rather than as quantitative proof.

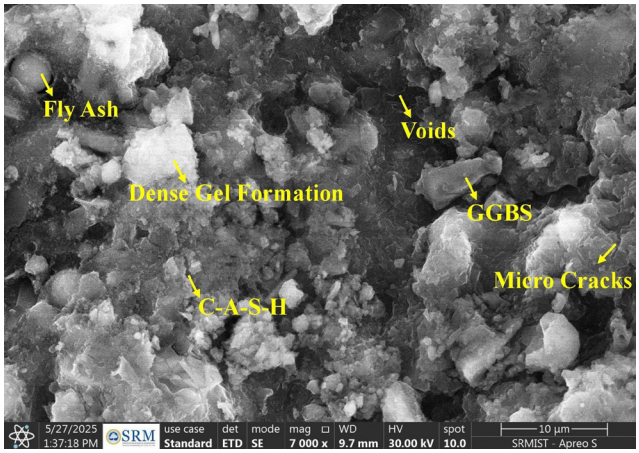
4.3.1 SEM analysis

Significant variances in microstructural morphology were evident in the SEM images of the geopolymer matrix at a magnification of ×7000 and 30000 kv were used. Small fragments (5–10 mm) were extracted from the interior of the geopolymer specimens to prevent surface contamination. The samples were subjected to oven drying at 60 °C for 24 h to eliminate residual moisture while maintaining the inherent microstructure. Loose debris was eliminated using mild air propulsion. The specimens were subsequently sputter-coated with a thin layer of gold (~10 nm) to improve surface conductivity prior to imaging [32].

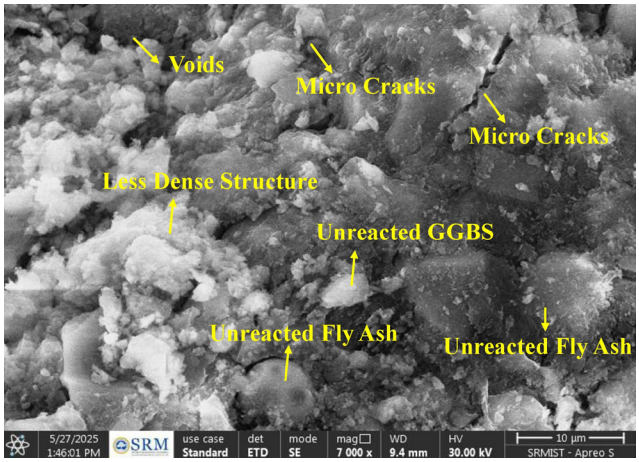
Mix A (1:1 ratio) displayed a solid, compact, and uniform gel matrix with little microcracks and fissures. The binder phase exhibited well-formed geopolymer gel networks, indicating effective polymerization of the aluminosilicate precursors. The compactness of the matrix is correlated with the increased compressive strength seen in mechanical tests.

Mix B (1.5:1 ratio) exhibited a less dense and more porous structure, characterized by discernible microvoids and unreacted particles, signifying incomplete geopolymerization. The surplus sodium carbonate probably hindered precursor dissolution, diminishing gel formation and compromising the matrix.

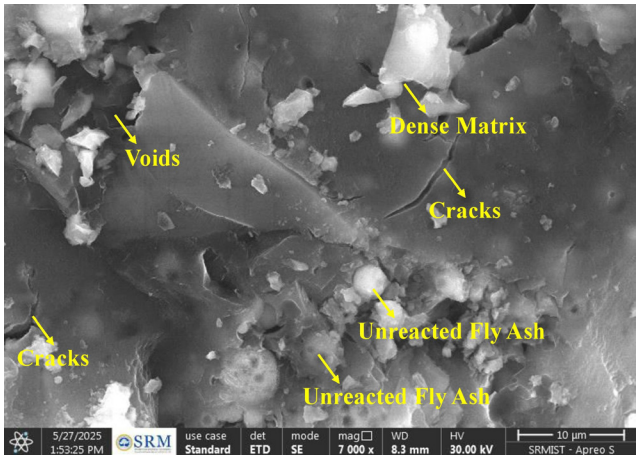
Mix C (1:2 ratio) exhibited a moderately dense matrix, characterized by heightened porosity and considerable aggregation of unreacted fly ash particles in comparison to Mix A. An elevated sodium silicate concentration augmented solution viscosity, hindering the formation of a homogeneous gel. Fig. 9 presents a SEM observations for various mixes.



(a)



(b)



(c)

Fig. 9 SEM analysis results: (a) Mix A; (b) Mix B; (c) Mix C

Overall, the SEM observations demonstrate that mixtures with denser matrices and fewer pores correspond to higher compressive strength, confirming the influence of microstructural compactness on mechanical performance.

4.3.2 EDS analysis

The microstructural and elemental analysis of the geopolymer concrete samples was performed utilizing Energy Dispersive X-ray Spectroscopy (EDS) in conjunction with Scanning Electron Microscopy (SEM). The SEM-EDS investigation was carried out at a magnification of 5000×, facilitating precise examination of the gel phases, unreacted particles, and pore structures. The results are tabulated in Table 5.

Fig. 10 (a) illustrates that Mix A had a consistent distribution of Si, Al, Na, and Ca, signifying the creation of a homogenous gel. The Ca:Si ratio was roughly 1.12, promoting the simultaneous presence of both C–A–S–H and N–A–S–H gels, which collaboratively enhance a denser and more cohesive microstructure. The increased calcium from GGBS and the balanced sodium and silica levels from the activator indicate a chemically stable geopolymer matrix. This balance corresponds with the maximum compressive strength recorded among the mixtures, validating a well reacted and structurally robust geopolymer network.

In Fig. 10 (b), Mix B demonstrated a comparatively reduced silicon concentration and an irregular distribution of calcium and sodium. The Ca:Si ratio was determined to be 1.45, signifying calcium-rich areas and a potential deficit in silica necessary for complete geopolymerization. The imbalanced ratio may have led to the development of a poorly linked gel network, evidenced by the reduced compressive strength and observable microstructural voids. The surplus sodium carbonate may have resulted in inadequate breakdown of aluminosilicate precursors and impeded effective gelation.

Fig. 10 (c) illustrates that Mix C exhibited elevated silicon content and comparatively diminished calcium concentration, resulting in a Ca:Si ratio of approximately 0.82. The microstructure exhibited evidence of heterogeneous gel formation, characterized by discrete areas of elemental clustering, particularly in the Si and Na regions. Although increased silicate concentration typically enhances polymerization, the diminished calcium amount may have resulted in a predominance of N–A–S–H gel, leading to inferior mechanical integrity relative to Mix A. Furthermore, the absence of homogeneity in elemental distribution indicates the development of less cohesive gel network.

4.3.3 XRD analysis

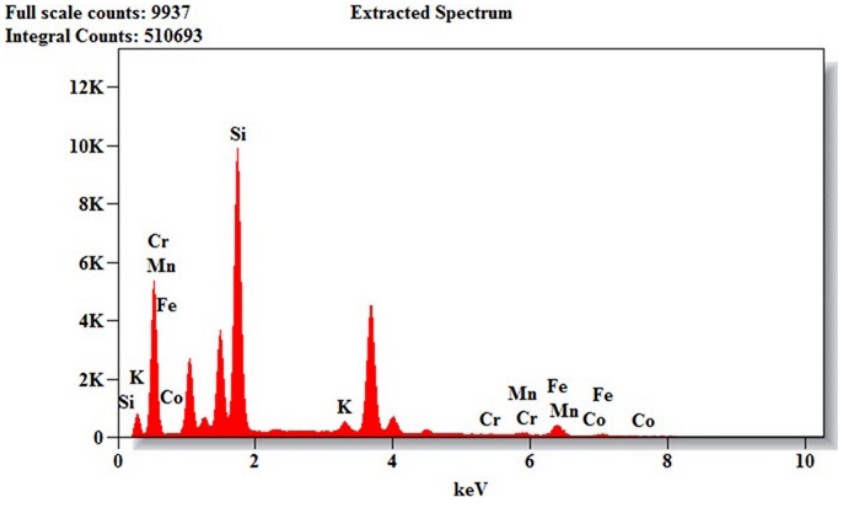
The XRD patterns of the geopolymer concrete mixtures – Mix A (1:1), Mix B (1.5:1), and Mix C (1:2) sodium carbonate to sodium silicate ratios are illustrated in Fig. 11. Each mix exhibited distinct diffraction peaks indicative of crystalline phases, including quartz (SiO_2), hematite (Fe_2O_3), mullite ($3Al_2O_3{\cdot}2SiO_2$), calcite ($CaCO_3$), gaylussite [$Na_2Ca(CO_3)2{\cdot}H_2O$], and hydrotalcite [$Mg_3Al_2CO_3(OH)_8{\cdot}4H_2O$]. The peaks have been attributed to unreacted precursors (fly ash and GGBS) and products of secondary carbonation or alkali–carbonate reactions.

Among the three, Mix A had a more extensive and pronounced amorphous hump in the 20°–35° 2θ range, signifying the presence of significant quantities of geopolymeric gel phases such as C–A–S–H and N–A–S–H. This indicates an enhanced interaction between the aluminosilicate sources and alkaline activators in Mix A, supported by a more equitable availability of silicate and carbonate. In contrast, Mix B and Mix C exhibited distinctly sharper crystalline peaks, especially those of quartz and mullite, indicating a higher presence of unreacted raw materials and a significantly lesser extent of geopolymerization.

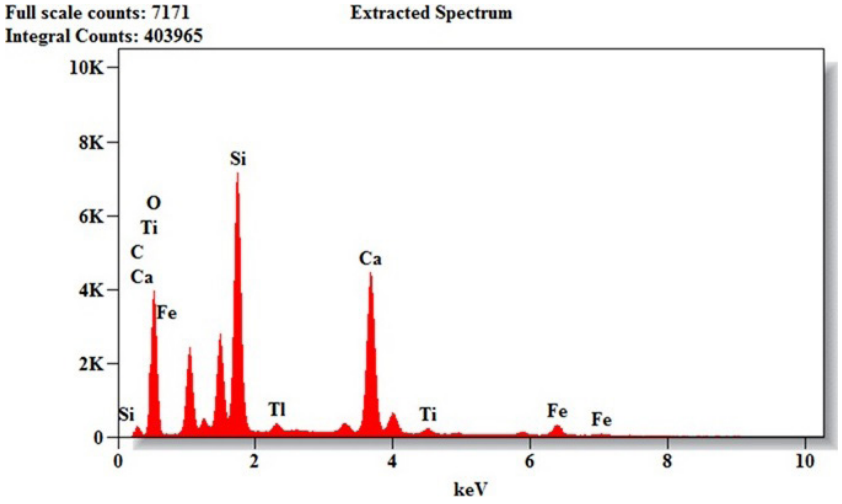
Conversely, Mix B and Mix C exhibited more pronounced crystalline peaks associated with unreacted quartz, mullite, and other residual phases, signifying an incomplete reaction attributable to an imbalance in the composition of the alkaline activator.

Table 5 Comparative observation of EDX for various mixes

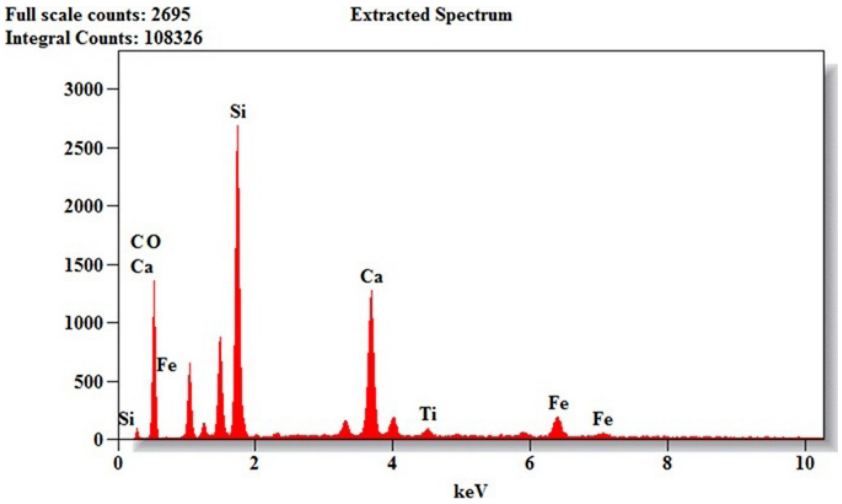
Feature	Mix A	Mix B	Mix C
Si peak intensity	Very high (dominant peak)	High	Noticeably lower
Ca peak intensity	High	Moderate to high	Lower than Mix A and Mix B
Fe peak intensity	Moderate (multiple peaks)	Moderate	Low
Oxygen and carbon peaks	Present (O strong; C visible)	Present	Clearly visible (C and O closely spaced)
Ti peak intensity	Present, moderate	Present	Present (lower)
Total counts	510,693 (highest among the three)	403,965	108,326 (lowest, indicating less dense matrix)
Overall elemental distribution	Rich in reactive components; likely better geopolymerization	Slightly lower reactivity	Possibly lower degree of reaction or dilution effect



(a)



(b)



(c)

Fig. 10 Comparison of EDX results: (a) Mix A; (b) Mix B; (c) Mix C

Calcite and gaylussite production was seen in all mixtures, possibly resulting from the interaction between calcium from GGBS and carbonate ions from sodium carbonate. Nonetheless, their relative strengths were diminished in Mix A, indicating reduced carbonation and a more stable geopolymer matrix. The comparison graph is shown in Fig. 11.

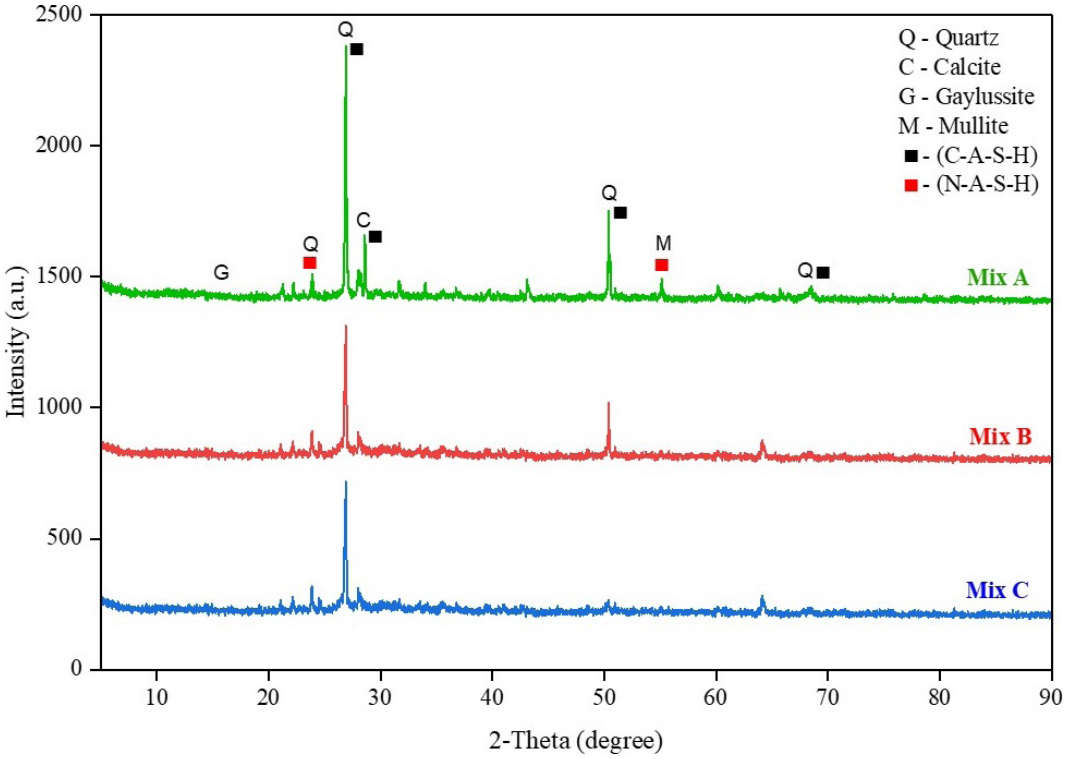


Fig. 11 Comparison of XRD results

4.4 Statistical analysis

4.4.1 ANOVA

The statistical analysis was performed using Design-Expert software (Version 13, Stat-Ease Inc., USA) [33]. The two-way ANOVA study indicated that both curing age and mix proportion significantly affect the material's strength ($p < 0.001$ for both parameters). Strength exhibited a distinct upward trajectory with curing duration, significantly improving from 7 to 28 days, thereby affirming that extended curing improved the material's mechanical capabilities. The mix percentage substantially influenced strength, with Mix A (1:1) surpassing Mix B (1.5:1) and Mix C (1:2) at all evaluated ages. This signifies that the composition ratio is crucial in strength development.

The experimental design, featuring a solitary observation for each factor combination, precluded the assessment of interaction effects between age and mix proportion. Nevertheless, the substantial main effects offer compelling evidence that both factors independently influence strength variation. These findings underscore the necessity of tailoring both the mix ratio and curing duration to enhance performance in real applications, as noted in Table 6 and illustrated in Fig. 12.

4.4.2 RSM analysis

Fig. 13 presents the contour plots generated from the RSM model, illustrating the effects of sodium carbonate (A) and sodium silicate (B) ratios on overall desirability and compressive strength at 7, 14, and 28 days. The desirability plot (top-left) shows a continuous high-desirability region (value = 1.000) across a broad range of activator contents, indicating that the target performance criteria are fully satisfied under multiple mix combinations. The 7-day compressive strength plot (top-right) predicts a peak value of 31.8057 MPa within this optimal range, while the 14-day (bottom-left) and 28-day (bottom-right) plots forecast maximum strengths of 40.4168 MPa and 47.4131 MPa, respectively, reflecting significant strength development with curing time. Across all curing ages, the red-to-yellow regions representing higher strength values align closely

Table 6 Two-way ANOVA summary for compressive strength analysis

Source of variation	Sum of squares (SS)	Degrees of freedom (DF)	Mean square (MS)	F-value	P-value	F_{crit}
Mix ratio	196.28	2	98.14	257.51	0.00005	6.94
Curing age	60.87	2	30.43	79.86	0.000597	6.94
Error	1.52	4	0.38	–	–	–
Total	258.68	8	–	–	–	–

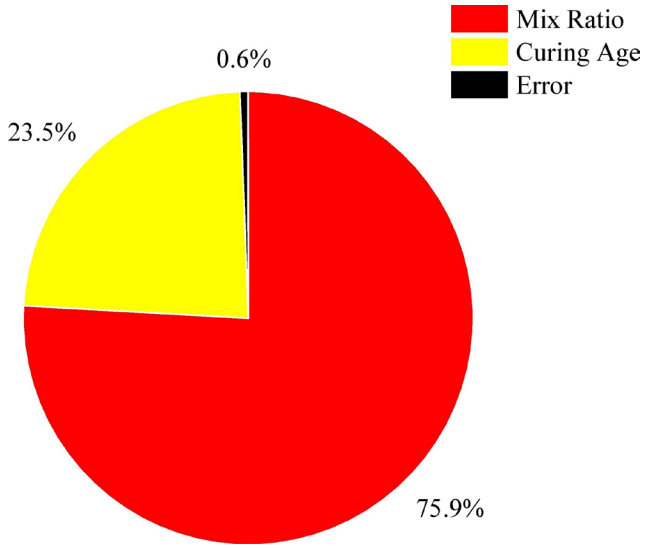


Fig. 12 Statistical analysis result

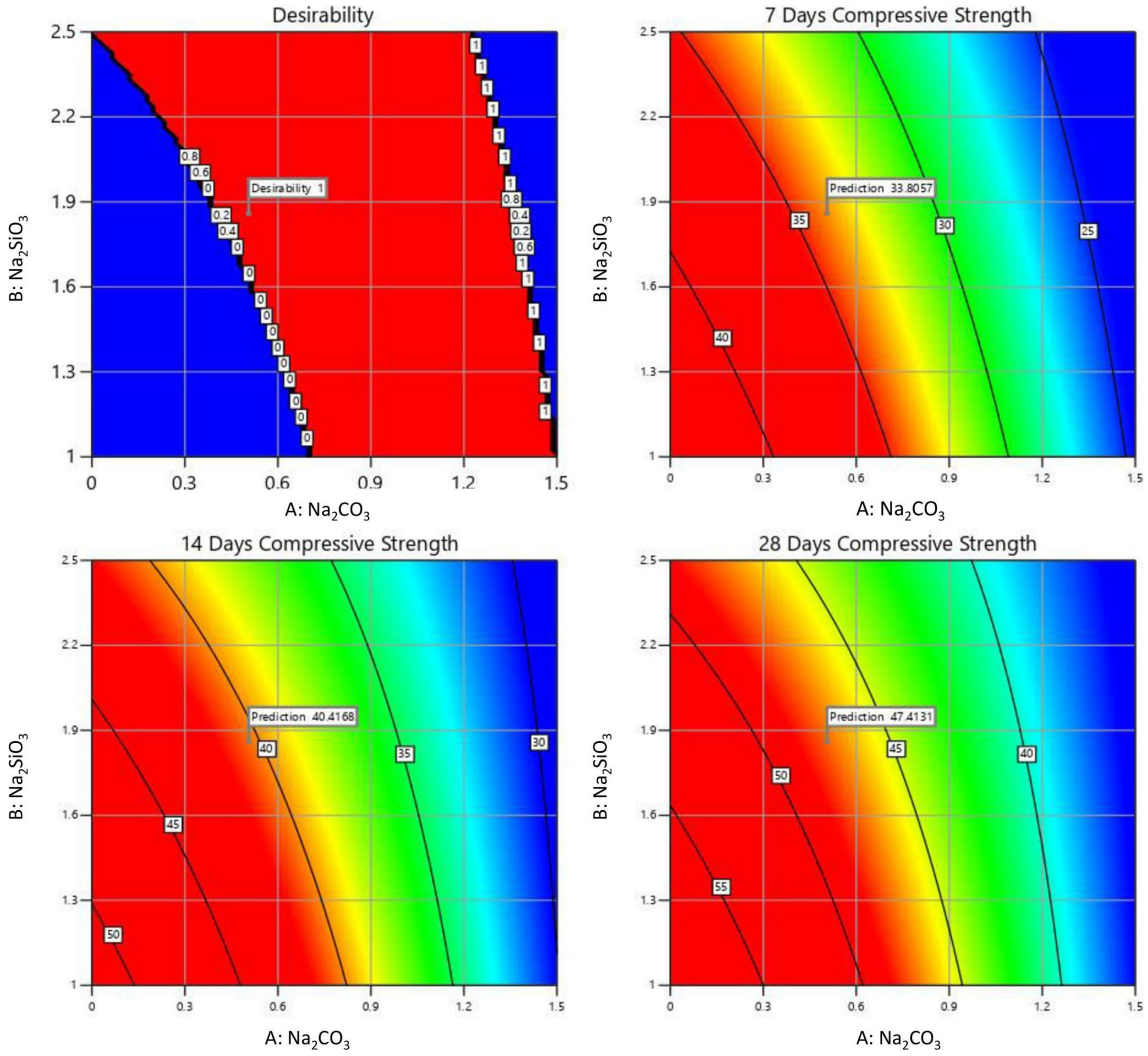


Fig. 13 Contour plots of Na_2CO_3 (A) and Na_2SiO_3 (B) on desirability and compressive strength

with the high-desirability zone, confirming that moderate sodium carbonate and sodium silicate contents yield superior performance. The consistent overlap between peak strength regions and the maximum desirability area validates the robustness of the RSM model in optimizing activator proportions for enhanced geopolymer concrete strength.

Fig. 14 presents the contour plot and numerical optimization results for compressive strength at 7, 14, and 28 days as functions of sodium carbonate (A) and sodium silicate (B) ratios. The optimal region, highlighted in yellow, corresponds to lower sodium carbonate and moderate sodium silicate levels, where the predicted strengths reach 31.8063 MPa at 7 days, 40.4175 MPa at 14 days, and 47.4138 MPa at 28 days. The identified optimum point is located at X_1 = 0.55412 and X_2 = 1.85891, representing the best balance between early-age and long-term performance. Experimental validation confirmed that mixes prepared near these optimum exhibited strengths within the high-performance yellow zone, while mixes outside this region showed comparatively lower values (e.g., 24.6 MPa at 7 days, 35.3 MPa at 14 days, and 48.6 MPa at 28 days). These results emphasize the strong influence of activator ratios on strength development and confirm the RSM model's capability to predict and optimize geopolymer concrete mix proportions accurately.

Although the statistical analysis was performed on a limited dataset due to the controlled scope of the laboratory investigation, all tests were conducted in triplicate to maintain consistency and reproducibility. The results, therefore, provide a reliable indication of the observed performance trends and form a basis for further statistical validation through extended datasets in future studies.

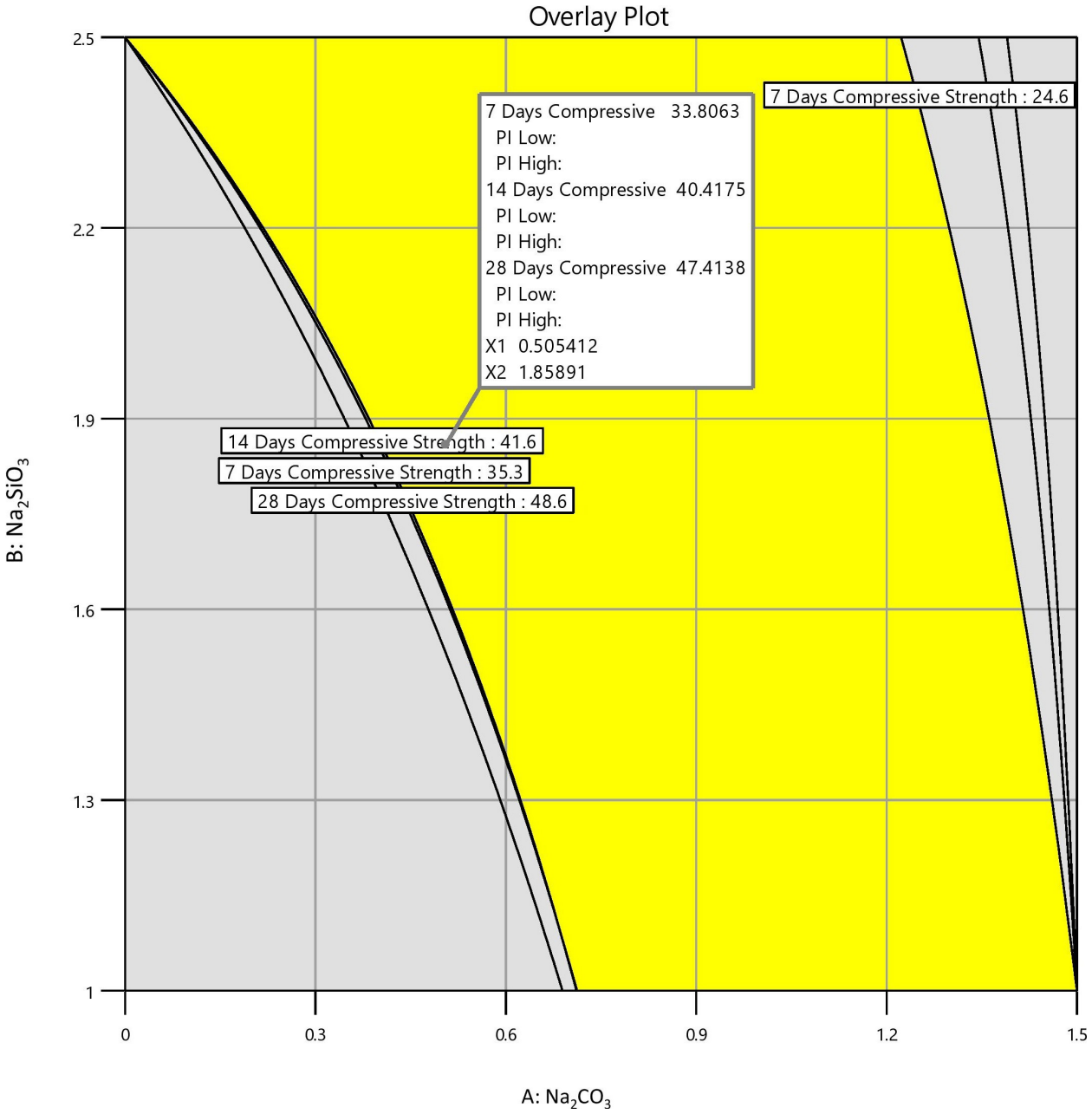


Fig. 14 Overlay plot indicating the optimal mixture region based on compressive strength responses

5 Conclusions

This study examined the mechanical and microstructural properties of alkali-activated geopolymer concrete formulated using a 50:50 mixture of fly ash and ground granulated blast-furnace slag, utilizing sodium carbonate and sodium silicate as alkaline activators in three distinct ratios (1:1, 1.5:1, and 1:2). The 28-day compressive strength findings revealed that Mix A (1:1) demonstrated the maximum strength of 44.1 MPa, followed by Mix C (1:2) at 41.2 MPa, and Mix B (1.5:1) at 36.3 MPa. The control mix activated with NaOH–Na_2SiO_3 (1:2.5) attained a higher strength of 48.6 MPa, highlighting the potent efficacy of NaOH as an activator, albeit with limitations in sustainability and handling safety. Among the carbonate-based mixtures, Mix A provided an optimal alkaline environment for the development of stable N–A–S–H and C–A–S–H gels, as confirmed by SEM–EDS and XRD analyses. Statistical modeling using Response Surface Methodology (RSM) and ANOVA demonstrated that the activator ratio significantly influenced compressive strength ($p < 0.05$) with a high R^2 value of 0.985, confirming an excellent model fit. The desirability and contour plots identified the optimal region at a Na_2CO_3:Na_2SiO_3 ratio of approximately 1:1, predicting compressive strengths of 31.81 MPa (7 days), 40.42 MPa (14 days), and 47.41 MPa (28 days), which closely matched the experimental results. This consistency validates the predictive capability of the RSM model and confirms the strong interaction effect between sodium carbonate concentration and alkaline activator ratio on strength development. The recommended optimum ratio of 1:1 is proposed for producing environmentally sustainable, high-performance geopolymer concrete suitable for rigid pavement applications. Future work should focus on evaluating long-term durability under aggressive environmental conditions such as sulphate, chloride, and freeze–thaw exposure, and exploring hybrid activator systems to enhance early-age strength.

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