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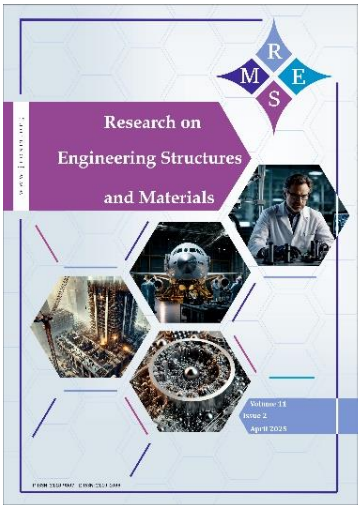


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*Research Article*

# Formulation and optimization of red mud-based geopolymers via simplex-centroid mixture design

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**Abstract**

Red Mud (RM), a byproduct of the Bayer's process, possess significant environmental challenges due to its limited applications beyond landfill disposal. This study investigates the feasibility of developing a novel geopolymer by incorporating Red Mud, Ground granulated blast furnace slag (GGBS) and Metakaolin (MK) mixtures using the simplex-centroid experimental design method and optimize the ternary binder mix with respect to setting time and compressive strength. The role of appropriate mineral proportions presents in all three binders activated with sodium hydroxides were studied to understand their influence on the synthesized geopolymers mixtures. Alkaline activators comprising NaOH and $Na_2SiO_3$ in a 1:2.5 ratio was used in this study. NaOH molarity was varied between 8, 10 and 12 to identify the optimal concentration. Seven mortar mixes for each molarity were prepared with Alkaline-to-Binder (Al/B) ratio of 0.5 and assessed for setting time and compressive strength under ambient conditions (7 and 28 days) and thermal curing (80 °C for 6 hours). Special cubic model equations were developed to quantify component interactions, with model validity confirmed by high predictive accuracy ($R^2$ > 0.95). Quantitative modeling demonstrates ~~that~~ the enhanced effect of the ternary systems compared to binary blends. The mixes with 10M NaOH exhibited better performance across the parameters evaluated. The optimal binder proportions lie within the range of 50–55% GGBS, 20–35% RM, and 20–30% MK. Microstructural studies through X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analyses confirmed a higher degree of geopolymerization, demonstrating a viable alternative to conventional RM disposal as sustainable construction material.



## 1. Introduction

Red Mud (RM) an industrial effluent, produced from Bayer's process which employs excessive alkali to extract alumina, resulting in pH values greater than 11. The elevated pH, combined with heavy metals such as lead, cadmium and arsenic complicate the recycling or appropriate disposal of RM[1]. The storage of RM as dried slabs or in extensive ponds possess significant environmental dangers, including groundwater contamination, soil alkalization, and dam breaches [2,3]. This opened a pathway for the researchers to develop sustainable remedial measures or to convert the highly alkaline RM into sustainable construction material. With significant proportions of silica, alumina, and iron oxides, alkali activated RM mixed with industrial by-products like granulated blast furnace slag, fly ash and rice husk ash are often utilized as binders in binary and ternary geopolymer mortar formulation [4]. Furthermore, silica rich metakaolin is also extensively used as mineral admixture in geopolymer mortar mixes [5-8]. In India, the yearly production of RM reached to 9 million tons, approximately[9]. The manufacturing of iron leads to the formation of byproduct

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i.e., GGBS. As a result of the continuous increase in the need for iron, a significant quantity of GGBS is produced. It has been demonstrated that GGBS can be recycled for use in cementitious materials or for the synthesis of geopolymers [10]. Cement-based mortars, despite their extensive use, exhibit increasingly significant environmental consequences[11,12]. The significant accumulations of industrial land wastes not only consume vast land resources but also present environmental hazards[13-15]. Thus, it is essential to create sustainable alternatives to cement mortars.

Huseien [16] has demonstrated that an increase in the amount of GGBS present in binary mixes with RM was associated with a decrease in the amount of time required for the mixture to set and a rise in its strength. Occhicone [17] has concluded that the compressive strength of RM and GGBS mortars is inversely related to the content of Red Mud. Lemougna [18] has observed that elevated temperatures and prolonged curing are crucial for enhancement of strength as the proportion of RM is increased. Simatupang [19] has used RM along with Metakaolin for the synthesis of geopolymerization. GGBS, characterized by its elevated calcium content, facilitates early strength development, whereas MK offers significant reactivity and a balanced silica-alumina ratio. Concentration of iron oxides is high in RM[20], which may result in inhibitory effects; however, when properly balanced, it offers potential as a sustainable aluminosilicate source. Literature indicates that the processing of materials at ambient temperature progressively enhances strength. Conversely, elevated temperatures provide a rapid enhancement in strength [21,22]. There are limited studies done to study the interaction effects of these three binder materials. This study on ternary mixes containing RM, GGBS, and MK seeks to determine the synergistic properties of these aluminosilicate materials and optimize the mix and effects due to various concentration of alkali solution.

Standards established for cement-based materials are being used for the preparation of geopolymer materials. It is evident that, the existing methods are not entirely applicable to alkali-activated materials[23]. Consequently, determining the ideal ratios of binder materials is essential to reduce the experimental trials needed to assess the performance of binder materials for the desired alkali-activated geopolymer materials. Mixture designs are statistical methods most commonly used in experimental design to evaluate the relationship between many factors and their independent responses[24]. Simplex-centroid design, a type of Mixture design approach, correlates material compositions and performance. It has been successfully used in concrete/binder design to optimize strength [25], dry shrinkage[26], and workability.

This research utilizes the Simplex Centroid Method of Mixture design for performance optimization of ternary composition using RM-MK-GGBS geopolymer mortar. Optimal proportions of each binder material that meet the criteria for strength and setting time under ambient and thermal curing conditions have been established, together with the ideal NaOH concentration. This study provides a systematic design methodology for producing sustainable RM based geopolymer mortars. The complete methodological framework of the present study is illustrated in Figure 1.

## 2. Materials

### 2.1 Red Mud

Red Mud (RM) is obtained from NALCO Damanjodi, Odisha and oven dried for a minimum of 24 hours at 80°C for moisture removal. The dried lumps were ground and passed through 75-micron sieve. As the name suggests the material is red in color and consists mainly of $Fe_2O_3$ and $Al_2O_3$.

### 2.2 Metakaolin

Metakaolin (MK) is a highly reactive, amorphous aluminosilicate pozzolan produced by the thermal activation of kaolinite clay. It is characterized by its off-white color. Silica and alumina constitute the principal oxides in these materials. A low concentration of $Fe_2O_3$ and $K_2O$ has been observed. Commercially available, MK was sourced from Mumbai, India.

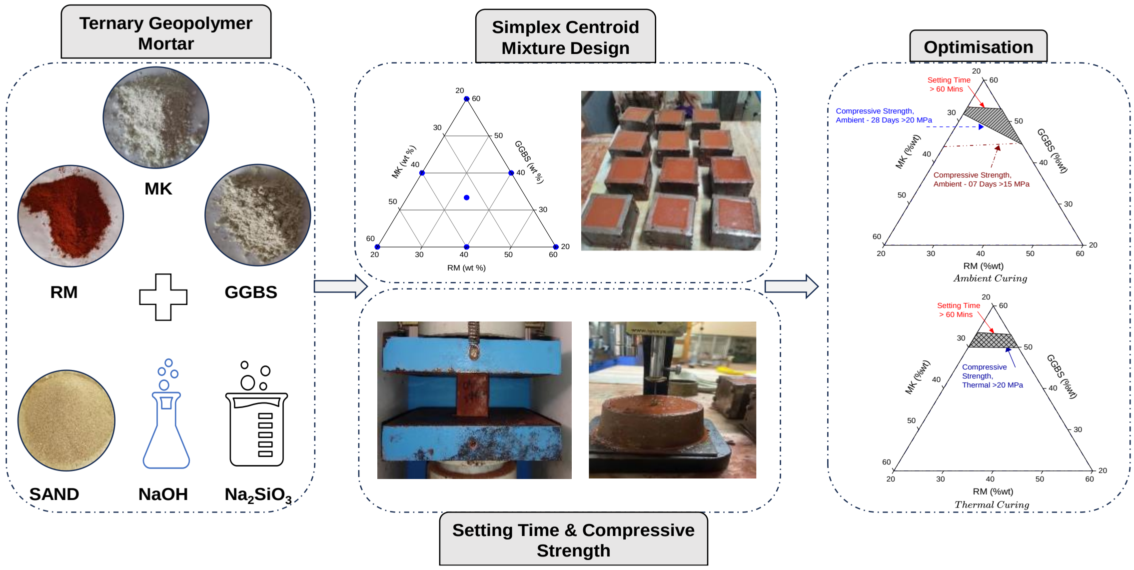



Fig. 1. Overall methodology for mixture design, testing, and optimization of RM-based ternary geopolymer mortar

## 2.3 Ground Granulated Blast Furnace Slag

Ground granulated blast furnace slag (GGBS) was sourced locally at Visakhapatnam, India. It is rich in calcium silicate with off-white color. The incorporation of GGBS expedites the geopolymerization due to the CaO content [27], which generates heat and aids in the development of C-S-H gel alongside N-A-S-H/ N-S-H gel. Chemical compositions of all the three binder materials are shown in Table 1. Particle size analysis of the binder materials are shown in Fig 2.

Table 1. Chemical composition of binder materials

| Materials | $Al_2O_3$ | CaO | $SiO_2$ | MgO | $Na_2O$ | $K_2O$ | $Fe_2O_3$ | $TiO_2$ | Others |
|---|---|---|---|---|---|---|---|---|---|
| RM | 15.03 | 1.5 | 7.85 | 2.14 | 4.65 | 2.59 | 49.18 | 4.5 | 12.56 |
| GGBS | 10.39 | 38.12 | 32.41 | 8.96 | 0.35 | 0.5 | 0.69 | 1.23 | 7.35 |
| MK | 41.41 | 0.08 | 52.22 | 0.26 | 0.01 | 1.73 | 0.49 | 0.65 | 3.15 |

## 2.4 Alkaline Activator

A solution of sodium silicate ($Na_2SiO_3$) along with sodium hydroxide (NaOH) is utilized as the alkaline activator, maintaining a ratio of 2.5:1. The pellets of sodium hydroxide (NaOH), with 99% purity, were combined with water for at least 24 hours before the mortar casting process started. Sodium hydroxide (NaOH) pellets were procured from RANKEM–AVANTOR (India), while sodium silicate solution ($Na_2SiO_3$) was sourced from a local commercial supplier.

## 2.5. Experimental Procedure and Testing

Mixture design methodology is used in the design of experiments where the variables are in proportions [28]. The simplex-centroid method originating from the mixture design methodology, aids in determining the correlation between proportions of the mix components and response parameters derived from experimental data. In situations where the number of variable components is represented as n, and the sum of all components equals to 100%, the experimental points are determined as 2n-1. In this study, the total number of binder materials considered is three. The experimental points based on simplex centroid method results in seven points, which include three vertices, three midpoints of the sides, and one center point. The central point experiment is repeated twice for validation purpose. Various responses collected from the seven points are input into special cubic regression model (Eq 1) and the equations relating the proportions of binder materials and responses are determined. Origin Pro Software is used to generate the ternary contour maps. According to the literature, the optimal red mud component for improved compressive strength in geopolymers should range from 20% to 60%. [29]. This study establishes that the utilization range of RM, GGBS, and MK is between 20% and 60%, based on literature and preliminary experiments. Proportions outside this range resulted in either inadequate strength or unfeasibly rapid or delayed setting times.

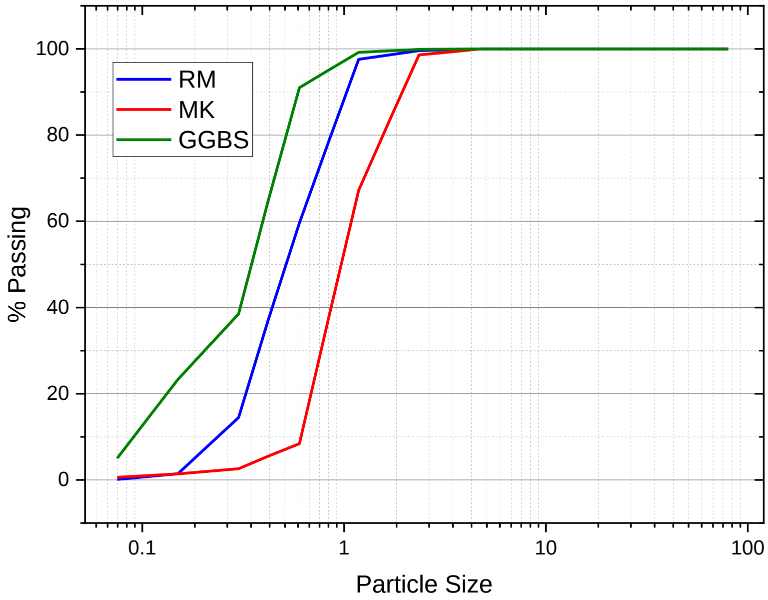



Fig. 2. Particle Size Analysis of the binder materials

The experimental data points are illustrated in Fig 3, while the binder proportions are presented in the Table 2(a) and the alkaline activator compositions are shown in Table 2(b). All mix combinations are designated using a standardized nomenclature; for illustration, R6G2M2 signifies a mixture comprising 60% RM, 20% GGBS, and 20% MK.

$$Y = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 \quad (1)$$

where Y is response data from experiments such as compressive strength and setting time, $\beta_i$ is the performance parameters, $x_1$, $x_2$ and $x_3$ refer to the binder proportions of RM, GGBS and MK respectively.

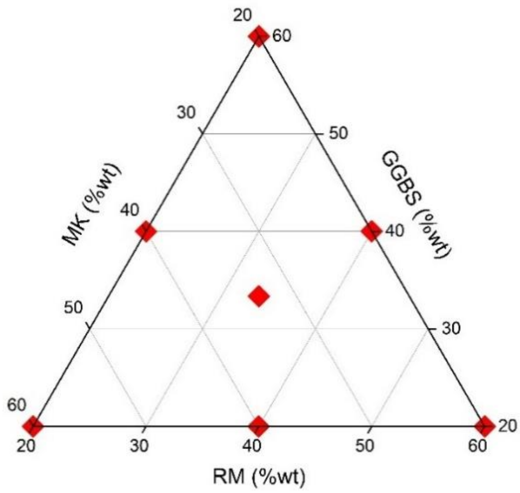



Fig 3. Experimental points for ternary mix using Simplex Centroid Design

## 2.6 Samples Preparation

Binders RM, GGBS, and MK, combined with fine aggregate in a 1:3 ratio, were initially mixed in dry condition using a mortar mixer for 2-3 minutes until thorough blending is achieved, which is followed by an additional three minutes of mixing with alkaline activator. NaOH solution of the desired molarity is prepared 24 hours in advance and combined with $Na_2SiO_3$ at a ratio of 1:2.5 prior to the mixing of binders. The Alkaline to Binder (Al/B) ratio is consistently maintained at a ratio of 0.5 across all mixes. For each NaOH molarity, seven mixes with varying binder proportions were cast for testing under thermal and ambient curing conditions.

Table 2. (a) Mix binder proportions and quantities of GPM mixes

| Mix | Binder Proportions (%) | | | Binders (kg/m³) | | | Fine Aggregate (kg/m³) |
|---|---|---|---|---|---|---|---|
| | RM | GGBS | MK | RM | GGBS | MK | |
| R6G2M2 | 60 | 20 | 20 | 342 | 114 | 114 | 1710 |
| R2G2M6 | 20 | 20 | 60 | 114 | 114 | 342 | 1710 |
| R4G2M4 | 40 | 20 | 40 | 228 | 114 | 228 | 1710 |
| R2G4M4 | 20 | 40 | 40 | 114 | 228 | 228 | 1710 |
| R3G3M3 | 33.33 | 33.33 | 33.33 | 190 | 190 | 190 | 1710 |
| R4G4M2 | 40 | 40 | 20 | 228 | 228 | 114 | 1710 |
| R2G6M2 | 20 | 60 | 20 | 114 | 342 | 114 | 1710 |

Table 2. (b) Alkaline activator composition

| Alkaline Solution (kg/m³) | | | |
|---|---|---|---|
| Molarity | NaOH Pellets | Water for NaOH Solution | $Na_2SiO_3$ |
| 8 | 26.8 | 54.6 | 203.6 |
| 10 | 33.6 | 47.8 | 203.6 |
| 12 | 40.2 | 41.2 | 203.6 |

Table 3. Test results of GPM mixes

| Mix | Molarity | Final Setting Time(mins) | Compressive Strength (MPa) | | |
|---|---|---|---|---|---|
| | | | Thermal Curing | Ambient 07 Days | Ambient 28 Days |
| R6G2M2 | 8 | 146 | 5.6 | 4.61 | 7.6 |
| R2G2M6 | | 75 | 12.92 | 6.76 | 14.93 |
| R4G2M4 | | 96 | 7.88 | 5.6 | 9.49 |
| R2G4M4 | | 105 | 9.29 | 7.98 | 11.15 |
| R3G3M3 | | 108 | 8.35 | 6.88 | 10.79 |
| R4G4M2 | | 70 | 9.61 | 7.32 | 9.68 |
| R2G6M2 | | 58 | 13.82 | 11.65 | 19.22 |
| R6G2M2 | 10 | 186 | 9.01 | 6.56 | 7.64 |
| R2G2M6 | | 90 | 19.62 | 10.81 | 14.77 |
| R4G2M4 | | 78 | 9.79 | 7.52 | 8.63 |
| R2G4M4 | | 82 | 15.73 | 12.85 | 13.3 |
| R3G3M3 | | 109 | 13.54 | 11.44 | 10.77 |
| R4G4M2 | | 79 | 16.07 | 12.26 | 18.62 |
| R2G6M2 | | 34 | 25.08 | 23.85 | 24.62 |
| R6G2M2 | 12 | 200 | 5.72 | 4.43 | 5.32 |
| R2G2M6 | | 80 | 12.98 | 7.04 | 11.84 |
| R4G2M4 | | 79 | 6.4 | 5.26 | 6.88 |
| R2G4M4 | | 75 | 15.91 | 13.13 | 13.58 |
| R3G3M3 | | 148 | 12.78 | 10.65 | 11.66 |
| R4G4M2 | | 75 | 12.58 | 12.94 | 15.45 |
| R2G6M2 | | 82 | 23.17 | 22.35 | 25.72 |

After mixing, the geopolymer mortar is poured into cube moulds of size 70.6 mm, compacted and vibrated for 2 minutes. Following a 24-hour period, the demoulding process is completed, and the thermal curing cubes are placed in oven set to 80°C for a duration of 6 hours [30]. Subsequently, thermal curing cubes are tested after a 24-hour cooling period. Ambient curing cubes are retained at room temperature until testing. Tests for setting times are conducted in accordance with IS 4031 Part 5 -2018 [31] and Tests for compressive strength are carried out according to IS 516 - 2021 [32]. The test results are shown in Table 3.  Water content represents only the quantity of water used for dissolving NaOH pellets to prepare the NaOH solution. No additional free water was added to the mix.

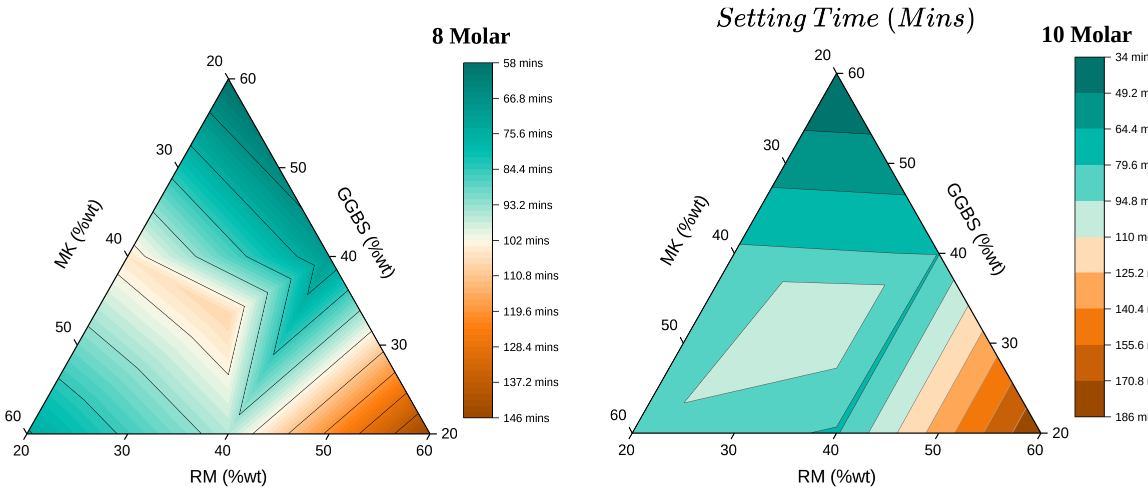
### 2.6 Microstructure Studies

XRD analysis was utilized to investigate the crystalline structure and phase composition. Samples were pulverized to particles smaller than 0.05 mm and uniformly placed on the sample holder. Measurements were performed utilizing a Bruker D8 Advance diffractometer equipped with a Cu-target X-ray source. Phase identification was accomplished by comparing diffraction patterns with standard reference cards. FTIR was employed to examine chemical bonding features using a Bruker ALPHA-II ATR-FTIR spectrophotometer in reflection mode. Dried samples were pulverized, combined with KBr powder, and compressed into pellets for examination.

## 3. Results and Discussions

### 3.1 Setting Time

From the test results, relationship between mix proportions and setting time based on the seven mixes obtained are as follows. Fig 4 illustrates the contour map of setting time as a function of the combination of binder proportions. When the NaOH concentration is constant, the setting time increases with a rise in RM proportion and decrease with a rise in GGBS proportion. GGBS>MK>RM is the arrangement of the contents of the binder that will results in decreasing the setting time of geopolymer mortars. RM demonstrated a significant impact on prolonging the setting periods of ternary mixes, as seen by the consistently elevated setting times recorded for the RM-rich mix (R6G2M2) across all NaOH molarities in comparison to the other ternary mixes. Conversely, the GGBS-rich mix (R2G6M2) exhibited the most expedited setting times of all mixes. Metakaolin (MK) demonstrated a modest effect on setting behavior; the MK-rich mix (R2G2M6) displayed setting periods that were intermediate to those of RM- and GGBS-dominant mixtures, indicating its moderate reactivity under alkaline activation. Moreover, the mix comprising equal ratios of RM, GGBS, and MK (R3G3M3) exhibited a balanced setting behavior, with setting times exceeding those of GGBS- and MK-dominant mixes, although falling short of those in RM-dominant compositions.



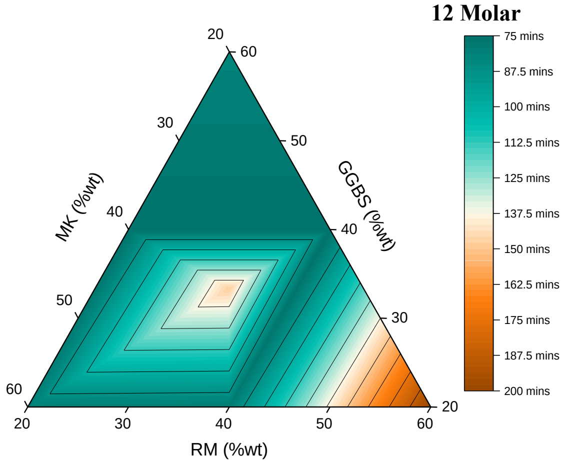



Fig. 4. Contours map of setting time

According to equations 2-4, binary mixtures—RM+GGBS, RM+MK, and GGBS+MK—demonstrate an accelerating effect on the setting time of the geopolymer mortar. In contrast, ternary mixtures incorporating all three components lead to a delay in setting time. This can be attributed to the presence of GGBS, which individually promotes rapid setting; however, in the ternary system, its effect is moderated by RM and MK, resulting in a more balanced and synergistic interaction that extends the setting time.

$$ST_8 = 677\,x_1 + 147\,x_2 + 207\,x_3 - 1944\,x_1\,x_2 - 1919\,x_1\,x_3 - 468.8\,x_2\,x_3 + 6469\,x_1\,x_2\,x_3 \quad (2)$$

$$ST_{10} = 1228\,x_1 + 397.5\,x_2 + 677.5\,x_3 - 3947\,x_1\,x_2 - 4647\,x_1\,x_3 - 2509\,x_2\,x_3 + 15422\,x_1\,x_2\,x_3 \quad (3)$$

$$ST_{12} = 2190\,x_1 + 1475\,x_2 + 1400\,x_3 - 9825\,x_1\,x_2 - 9538\,x_1\,x_3 - 7625\,x_2\,x_3 + 39563\,x_1\,x_2\,x_3 \quad (4)$$

As shown in Fig 5, RM rich mixes, altering the molarity from 8 M to 12 M resulted in a rise in setting time from 150 minutes to 200 minutes, with the longest setting time observed at 12M. In GGBS-rich mixtures, the change in molarity caused in a reduction of setting time from 8 M to 10 M, followed by an increase at 12 M. The maximum setting time was observed at 12 M concentration. In MK rich mixes, an increase from 8 M to 10 M led to an extended setting time, while a further increase to 12 M resulted in a decreased setting time, with the maximum setting time observed at 10 M.

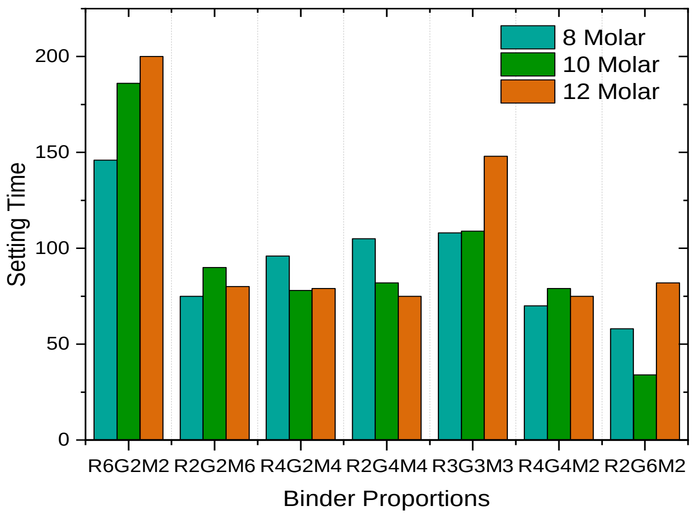



Fig. 5. Relationship between molarity on setting time

The average setting times of 8, 10 and 12 molar NaOH are 94, 94 and 105 minutes respectively. The findings reveal that an increase in NaOH concentration is leading to increase in the setting times which is in line with literature. The fine particle size of red mud (RM), combined with the limited availability of reactive aluminosilicate phases, restricts early dissolution and gel formation, thereby exerting a detrimental effect on setting time. Conversely, ground granulated blast furnace slag (GGBS), owing to its abundant CaO content along with rich $SiO_2$ and $Al_2O_3$, significantly accelerates the setting process. Metakaolin (MK) exhibits a moderating influence due to its high specific surface area and the smallest particle size among the precursors; when combined with its high reactivity, this ensures a controlled and steady geopolymerization reaction which is consistent with literature [28,33].

### 3.2 Compressive Strength – Thermal Curing

As shown in the Fig 6, maximum compressive strength of 25.08MPa was attained by mix R2G6M2 with 10 M alkaline activator. The relation between compressive strength and the mix proportions for thermal curing are shown in equations (5-7)

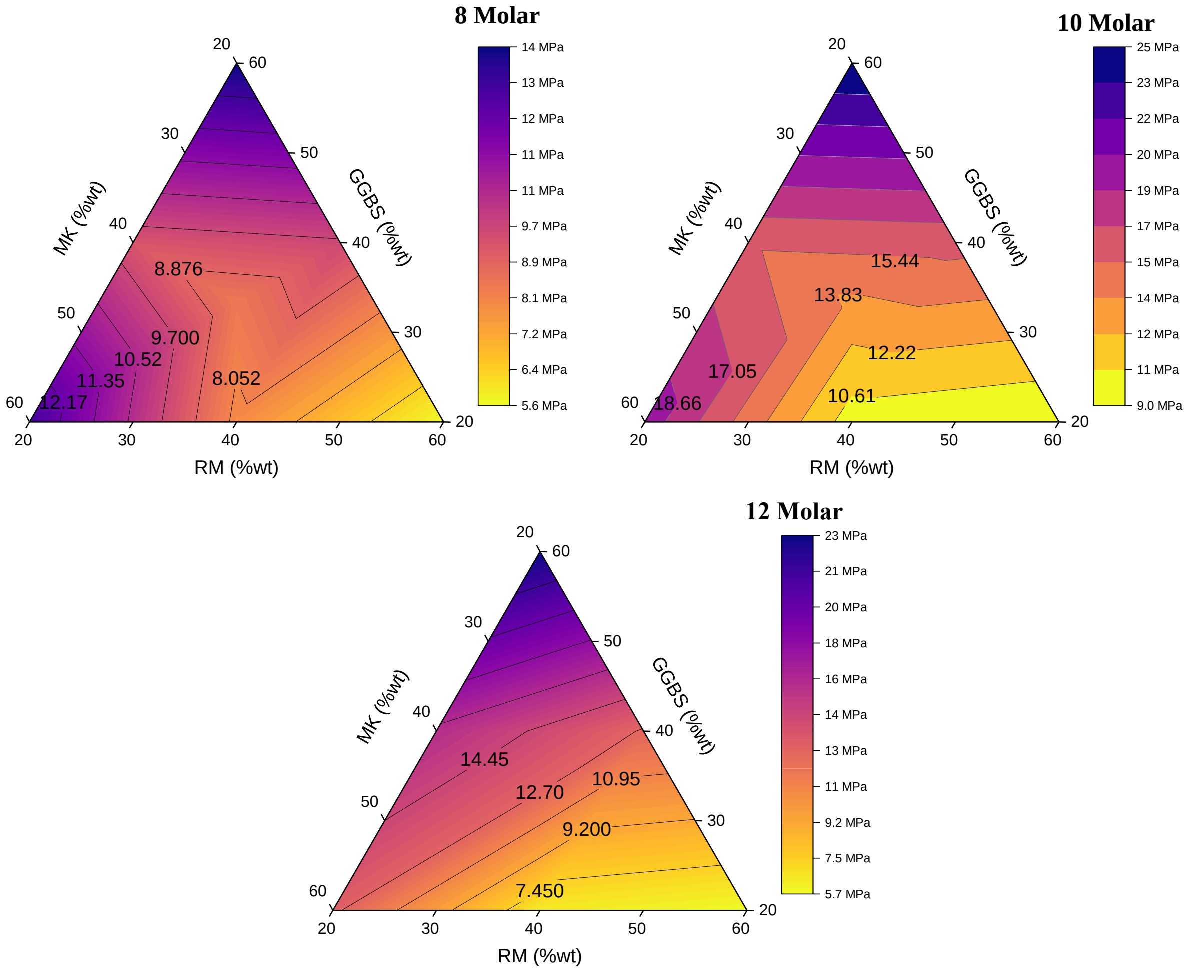



Fig 6. Contours maps for compressive strength – thermal curing

$$CS_{T-8} = -4.695\, x_1 + 25.71\, x_2 + 28.06\, x_3 + 18.91\, x_1 x_2 - 3.594\, x_1 x_3 - 60.09\, x_2 x_3 - 78.28\, x_1 x_2 x_3 \tag{5}$$

$$CS_{T-10} = 22.70\, x_1 + 66.35\, x_2 + 66.85\, x_3 - 108.1\, x_1 x_2 - 169.0\, x_1 x_3 - 199.8\, x_2 x_3 + 406.9\, x_1 x_2 x_3 \tag{6}$$

$$CS_{T-12} = 24.71\, x_1 + 70.21\, x_2 + 54.81\, x_3 - 146.7\, x_1 x_2 - 193.7\, x_1 x_3 - 201.7\, x_2 x_3 + 603.3\, x_1 x_2 x_3 \tag{7}$$

## 3.3 Compressive Strength – Ambient 07 Days

As shown in the Fig 7, the maximum compressive strength of 23.85 MPA was attained by mix of R2G6M2 with 10 M alkaline activator. The relation between compressive strength and the mix proportions for 7 days ambient curing is shown in equations (8-10)

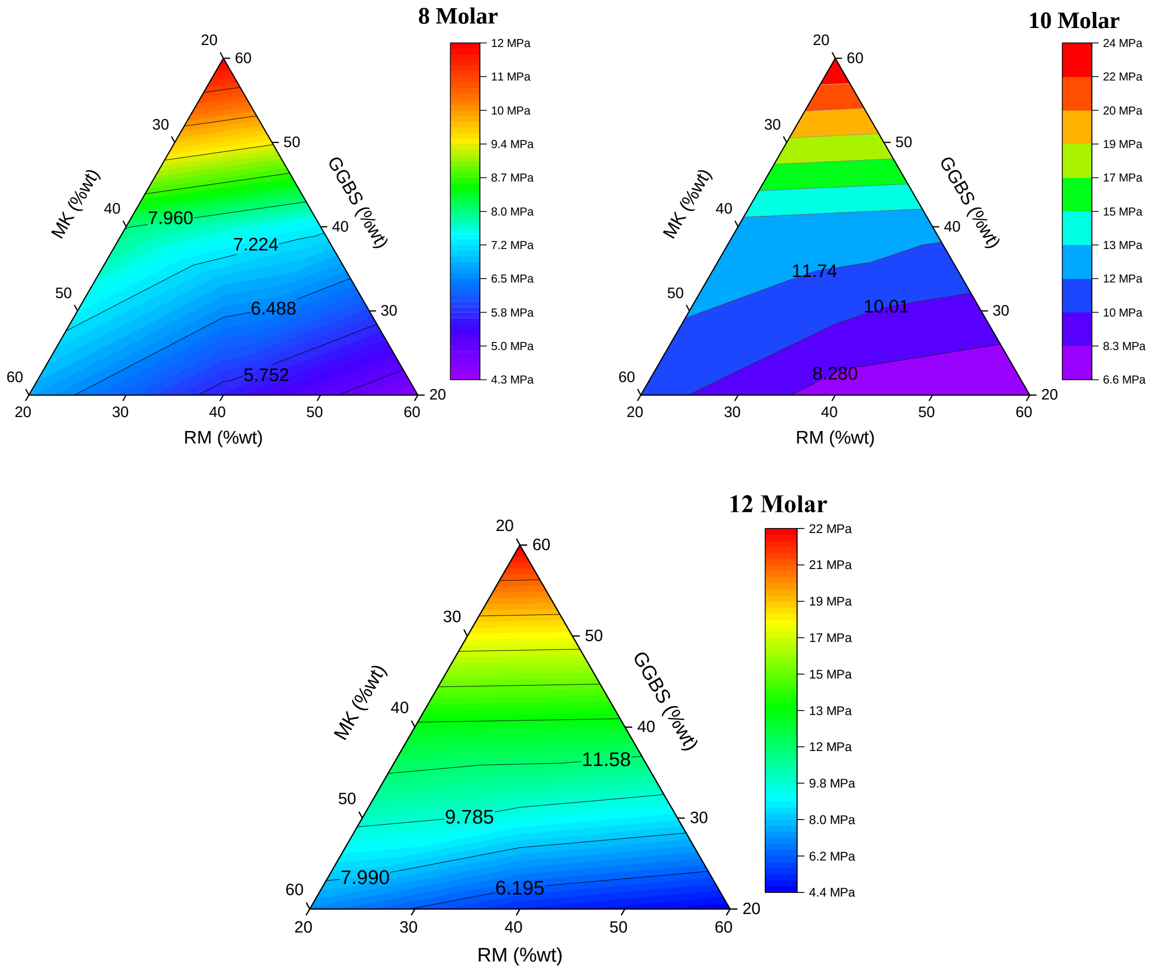



Fig 7. Contours maps for compressive strength – ambient curing – 07 days

$$CS_{7D-8} = 0.2\,x_1 + 21.10\,x_2 + 3.15\,x_3 - 7.813\,x_1 x_2 + 16.19\,x_1 x_3 + 3.437\,x_2 x_3 - 75.94\,x_1 x_2 x_3 \quad (8)$$

$$CS_{7D-10} = 17.29\,x_1 + 74.34\,x_2 + 37.64\,x_3 - 122.8\,x_1 x_2 - 101\,x_1 x_3 - 166.4\,x_2 x_3 + 303.3\,x_1 x_2 x_3 \quad (9)$$

$$CS_{7D-12} = 8.26\,x_1 + 58.31\,x_2 + 21.56\,x_3 - 77.13\,x_1 x_2 - 72\,x_1 x_3 - 104.4\,x_2 x_3 + 236.30\,x_1 x_2 x_3 \quad (10)$$

## 3.4 Compressive Strength – Ambient 28 Days

As shown in the Fig 8, the maximum compressive strength of 24.62 MPA was attained by mix of R2G6M2 with 10 M alkaline activator. The relation between compressive strength and the mix proportions for 28 days ambient curing is shown in equations (11-13). The equations (5-13) indicate that majority of the binary interactions among the alumina silicate materials are antagonistic. The ternary interactions exhibit synergy at 10 and 12 Molarity. The 08 Molar alkaline activator may not provide adequate $Na^{+}$ and $Si^{-}$ ions to effectively activate the ternary mixture. Optimal ternary mix is obtained at 10 M concentration. At 10 Molarity the GGBS supplies the necessary calcium (Ca), MK supplies highly reactive, amorphous alumina (Al) and silica (Si), and RM supplies iron (Fe) and extra alumina [34]. Although 12 M activator produces a synergistic ternary mix, it is not as effective as 10 M alkaline activator.

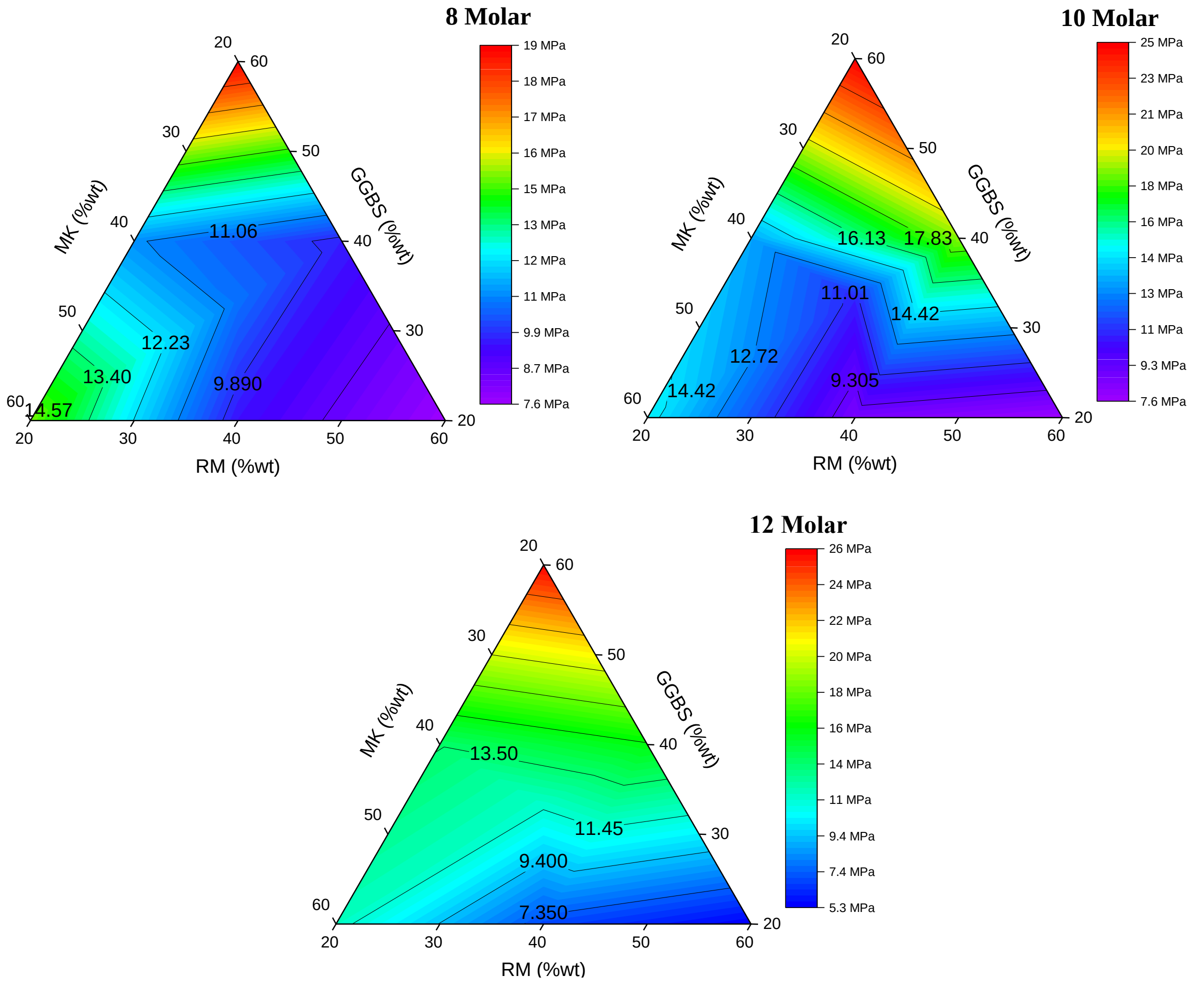


Fig 8. Contours Maps for compressive strength – ambient curing – 28 days

$$CS_{28D-8} = 27.63\,x_1 + 68.18\,x_2 + 48.88\,x_3 - 173\,x_1 x_2 - 128.50\,x_1 x_3 - 198.2\,x_2 x_3 + 483.3\,x_1 x_2 x_3 \quad (11)$$

$$CS_{28D-10} = 25.74\,x_1 + 90.94\,x_2 + 87.19\,x_3 - 151.4\,x_1 x_2 - 269.5\,x_1 x_3 - 375.1\,x_2 x_3 + 952.5\,x_1 x_2 x_3 \quad (12)$$

$$CS_{28D-12} = 0.205\,x_1 + 59.96\,x_2 + 43.61\,x_3 - 17.34\,x_1 x_2 - 82.22\,x_1 x_3 - 146.7\,x_2 x_3 + 105.5\,x_1 x_2 x_3 \quad (13)$$

The effect of NaOH molarity on compressive strength demonstrated a consistent and mix-dependent pattern under all the curing conditions. An elevation in NaOH concentration from 8 M to 10 M led to a significant improvement in compressive strength for the majority of mixes, and in particular in GGBS-rich and balanced ternary blends. This enhancement may be due to the improved dissolution of aluminosilicate species and the expedited synthesis of geopolymer gel at moderate alkalinity[33]. Nevertheless, 12 M NaOH typically resulted in strength decline. While NaOH can facilitate the dissolving of $SiO_2$ and $Al_2O_3$, excessive alkalinity may lead to the precipitation of $Ca^{2+}$ and $Al^{3+}$, thereby impeding the hydration reactions and geopolymerization, subsequently it leads to a reduction of strength [35].GGBS-rich mixtures exhibited greater tolerance to elevated alkalinity and maintained strength improvements even at 12 M, aligning with the findings [36]. This is mostly because there is significant CaO component in GGBS, which generates C-S-H or C-A-S-H gel via hydration processes. This gel exhibits greater compactness leading to improved compressive strength.

As shown in the Fig 9, GGBS & MK dominant mixes show higher compressive strength with 10 M alkaline activator, followed by 12 and 8 M in decreasing order. RM-rich mixes had higher compressive strength with 10 M alkaline activators, followed by 8 and 12 M.

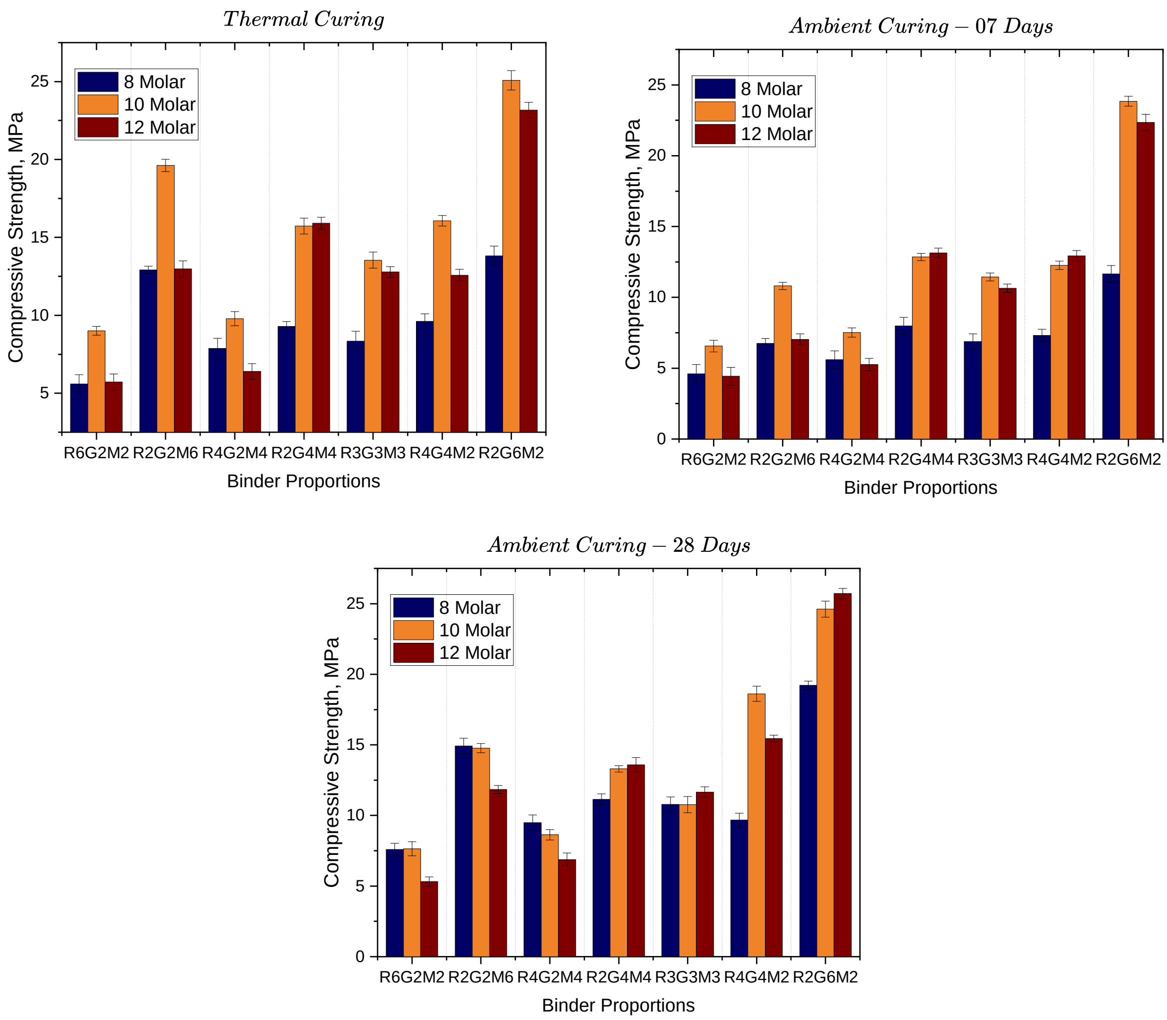



Fig. 9. Relationship between molarity and compressive strength

## 4. Verification

A fitting analysis to compare the actual and predicted values derived from the equations was conducted to further assess the feasibility of the experimental design method of using simplex-centroid method in the design of proportions of ternary mixtures. The fitting results shown in Fig 10 a-c, demonstrate strong predictive performance for all response variables, as evidenced by $R^2$ values exceeding 0.95. The experimental design method is effective for optimizing the mix proportions of ternary blended geopolymer concrete.

## 5. Optimization

The performance optimization of the ternary mix range, which satisfies the criteria of a setting time exceeding 60 minutes and minimum compressive strengths of 15 MPa and 20 MPa at 7 and 28 days under ambient curing, is depicted in the shaded area of the Fig 11a. The optimal mixture should consist of GGBS proportion between 50% to 55%, RM proportion between 20% and 35%, and MK proportion between 20% to 28%.

The Fig 11b, illustrates in shaded area proportions of geopolymer mortar, tested 24 hours after thermal curing, results in a compressive strength that is greater than 20 MPa and a setting time that is greater than 60 minutes. The recommended percentages for GGBS, RM, and MK are 50% to 55%, 20% to 30%, and 20% to 30%, respectively.

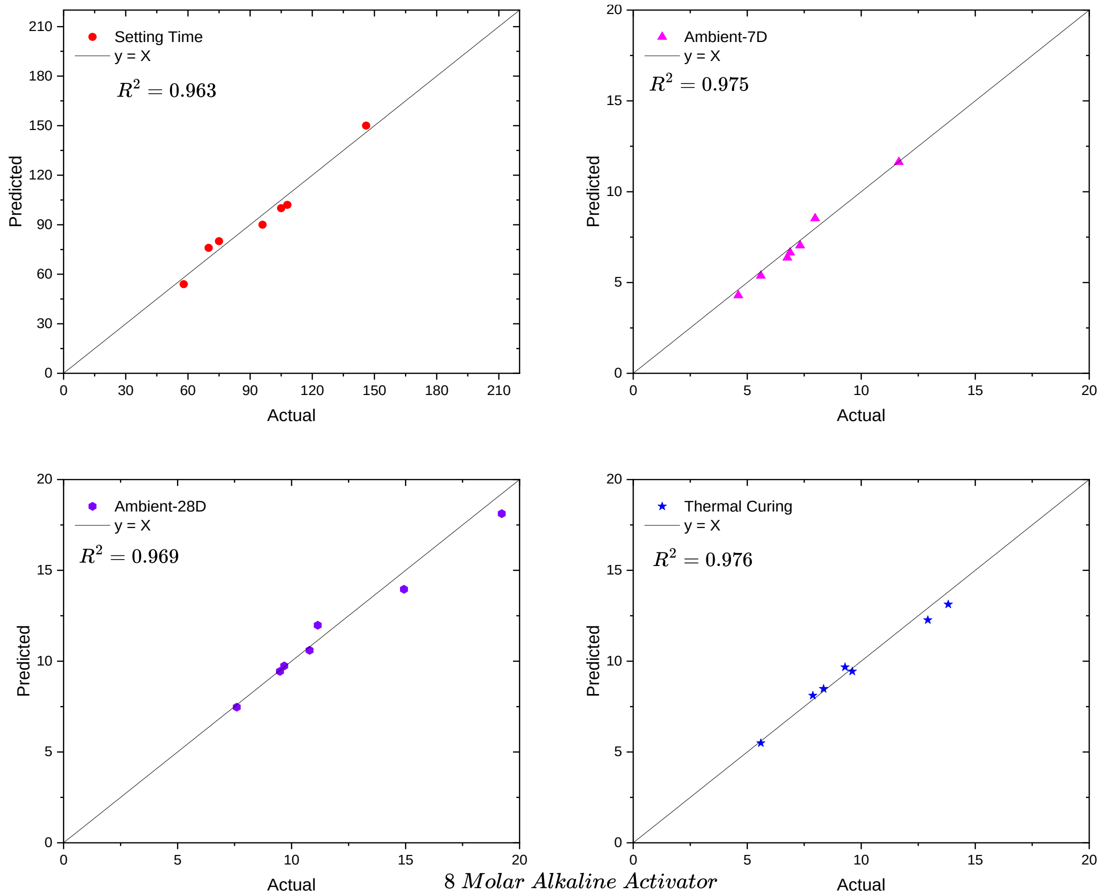



Fig. 10. (a) Predicted vs actual for 08 molar alkaline activator

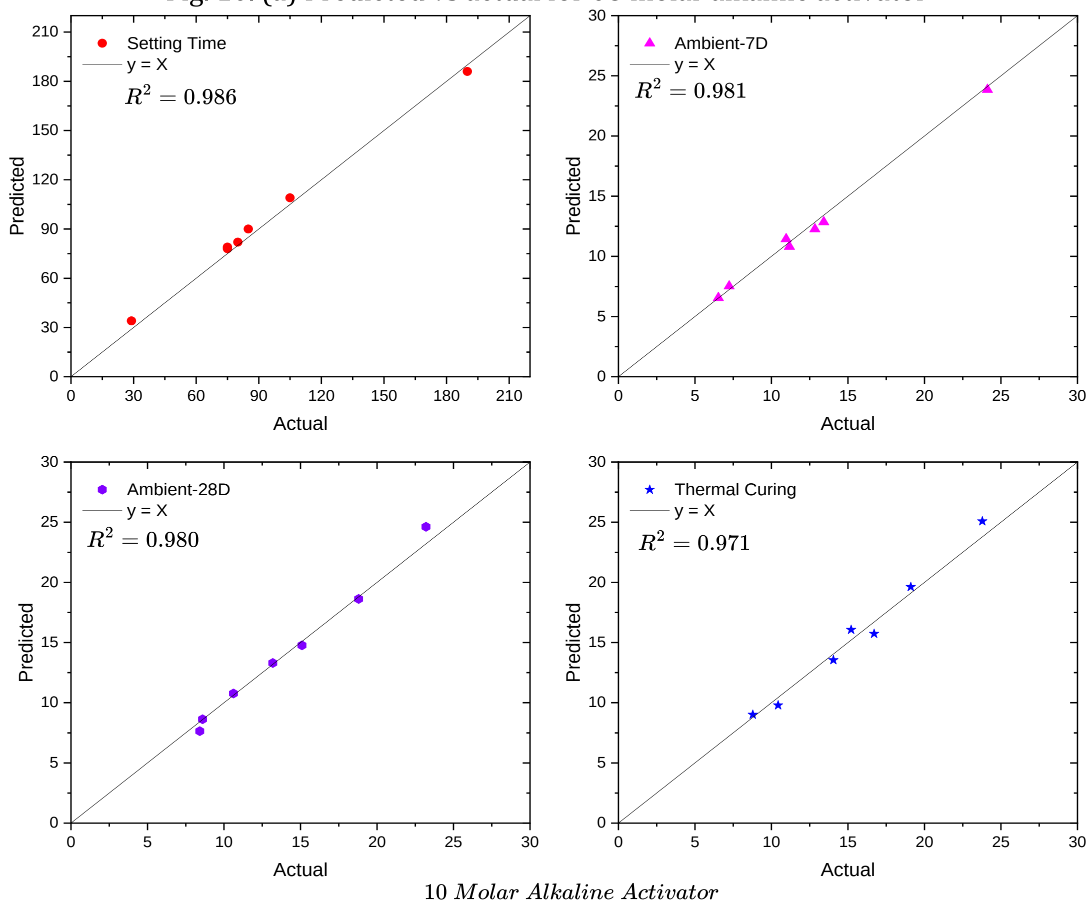



Fig. 10. (b) Predicted vs actual for 10 molar alkaline activator

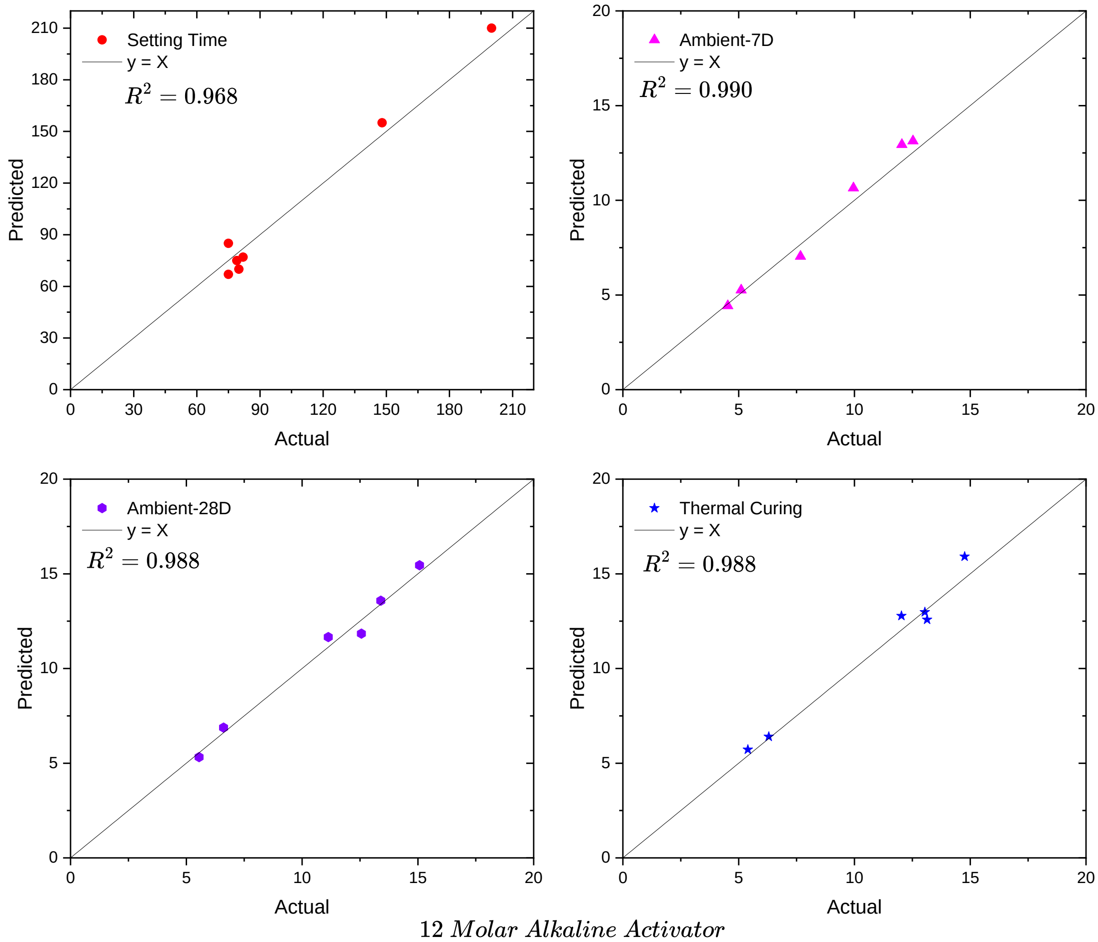



Fig 10. (c) Predicted vs actual for 12 molar alkaline activator

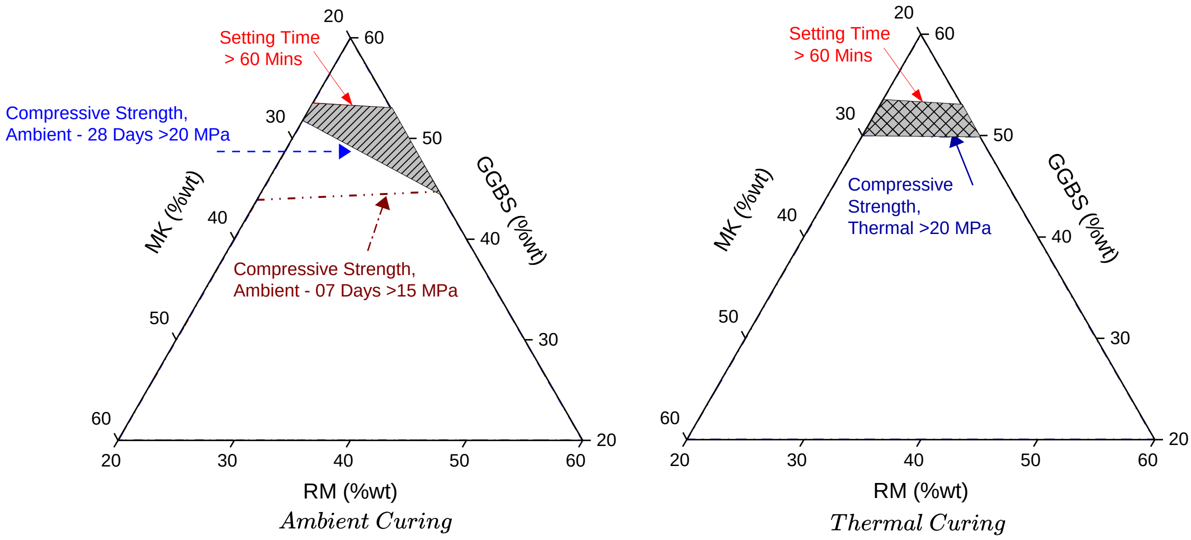



Fig 11. Optimum mix proportion range – ambient and thermal curing

## 6. Microstructure Studies

Selected geopolymer mortar samples were subjected to XRD and FTIR investigations in order to investigate the microstructural evolution and determine whether crystalline and amorphous phases contributed to the mechanical performance. The XRD patterns of the selected mixes of R2G6M2-T, R2G6M2-A28, R6G2M2-T, R6G2M2-A28 as shown in Fig 12, exhibit significant quartz reflections (Q; 2θ ≈ 26.6°), minimal calcite (C; 2θ ≈29.4°) suggesting modest carbonation, and hematite (H) peaks indicative of iron-rich components. Kaolinite (K) is mostly present in the RM-rich R6G2M2 mixtures, but subtle characteristics between 32° and 34° are compatible with calcium-silicate-hydrate-type products formed from slag. All patterns display a wide amorphous peak in the 25–35° range, indicating the production of an aluminosilicate gel that influences strength. Comparative analysis indicates that the GGBS-dominant R2G6M2 mixtures have diminished crystalline peaks and a more prominent amorphous background, while the RM-dominant R6G2M2 mixtures maintain more robust hematite/kaolinite signatures. Calcite reflections are more pronounced in ambient-cured specimens compared to thermally cured ones, aligning with increased carbonation due to exposure. The diffractograms indicate geopolymerization, with increased amorphous gel content in GGBS-rich formulations, while residual crystalline phases remain prevalent in compositions with greater RM.

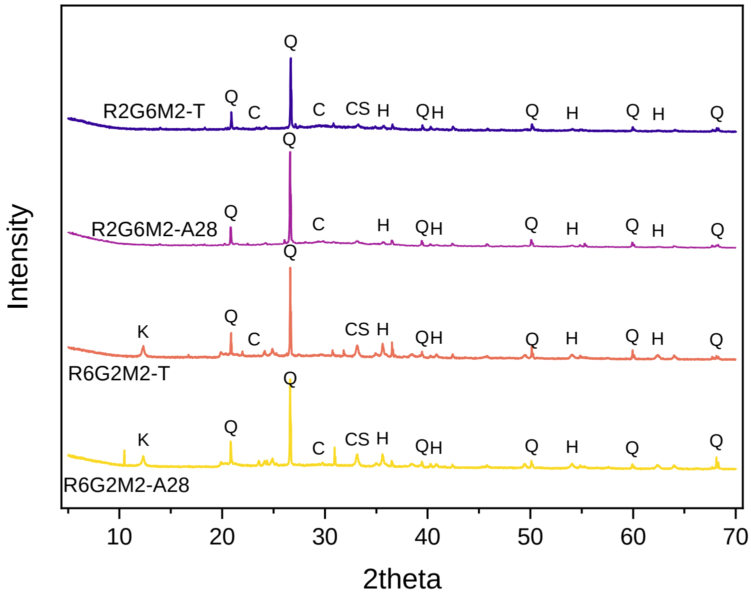



Fig 12. XRD spectra of GPM mixes

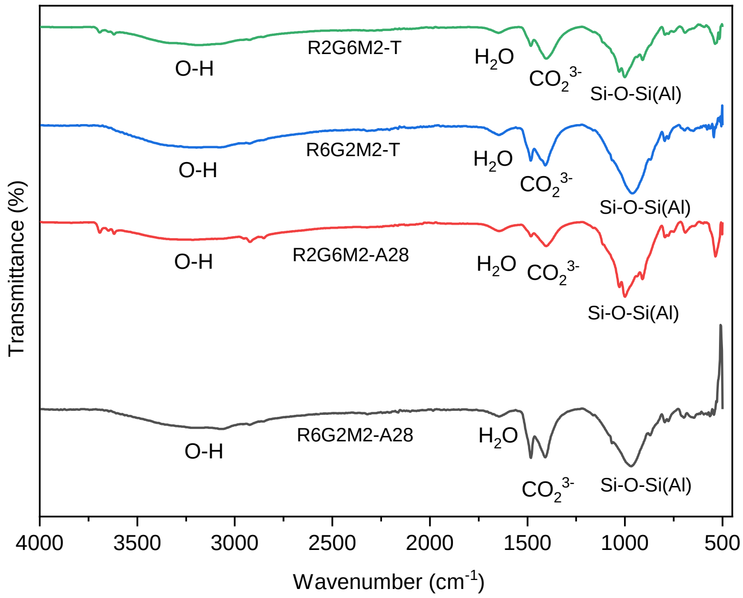



Fig 13. FTIR spectra of GPM mixes

The FTIR spectra of the geopolymer mixes as shown in Fig 13, exhibit bands that validate the development of reaction products due to geopolymerization. A prominent absorption band within the range of 1000–1020 cm$^{-1}$, attributed to the asymmetric stretching of Si–O–Si(Al), was observed in all mixtures, indicating the formation of geo-polymeric gel. The bands at 450–470 cm$^{-1}$

correspond to Si–O–Si bending vibrations, whereas the peak near 780 $cm^{-1}$ signifies the presence of Al–O connections within the matrix. Carbonate groups were detected by peaks in the range of 1415–1425 $cm^{-1}$, indicative of mild carbonation, while molecular water was represented by bands about 1635–1640 $cm^{-1}$. A broad band at approximately 3450 $cm^{-1}$ was attributed to O–H stretching vibrations from bound water. The spectra illustrate the gradual polymerization of aluminosilicate species into stable Si–O–Al networks, with the prominence of Si–O–Si(Al) bands indicating the production of continuous geopolymer gels that enhance strength development. XRD and FTIR analyses indicate that RM-rich mixes show residual crystalline phases indicative of limited geopolymerization, while GGBS-rich mixes demonstrate improved amorphization, increased aluminosilicate polymerization, leading to a denser and mechanically superior matrix.

## 7. Conclusion

This study establishes the optimal mix proportions for geopolymer mortar using a ternary blend of red mud (RM), ground granulated blast furnace slag (GGBS), and metakaolin (MK). The Mixture Design methodology is employed to systematically determine compositions that satisfy performance requirements for both setting time and compressive strength under thermal and ambient curing conditions. The average setting times of 8 M, 10 M and 12 M NaOH are 94, 94 and 105 minutes respectively. Compressive strength improves with increasing molarity up to 10 M, beyond which it declines. As the molarity increases from 8 M to 12 M, the setting time decreases, indicating accelerated geopolymerization. Under thermal curing, the average compressive strength at 10 M was 61.30% higher than that at 8 M and 21.57% higher than that at 12 M. For ambient curing at 7 days, the compressive strength at 10 M exceeded that of 8 M and 12 M by 67.76% and 12.46%, respectively. Similarly, under ambient curing at 28 days, the average compressive strength at 8 M and 12 M was 18.66% and 8.75% lower, respectively, compared to 10 M. Thus, 10 M NaOH is identified as the optimal concentration, offering a balance between workability and mechanical performance. The special cubic equations coefficients confirm strong positive ternary interaction coefficient and antagonistic behavior (negative coefficients) of binary blends. The ideal proportions for GGBS are between 50% and 55%, for RM between 20% and 35%, and for MK between 20% and 30%. Mixes within these proportions have been found to achieve a minimum compressive strength of 15 MPa at 7 days and 20 MPa at 28 days under ambient curing conditions. Furthermore, under thermal curing, the mixes yield compressive strength exceeding 20 MPa along with a setting time exceeding 60 minutes. The XRD spectra of the geopolymer samples showed crystalline peaks that matched those of quartz, calcite, hematite, and kaolinite. This showed that the raw binders had partially reacted. The GGBS-rich mixes showed better geopolymerization, as shown by a stronger amorphous hump that was linked to the creation of C–S–H and N–(C)–A–S–H type gels. FTIR examination corroborated these results, revealing a significant change of the asymmetric stretching vibration of Si–O–T (T = Si or Al) bands towards lower wavenumbers in the 800–1200 $cm^{-1}$ range, indicating the increasing polymerization of the aluminosilicate framework. The simplex-centroid method, a type of mixture design methodology, effectively identifies the relationships between binder proportions and response parameters based on experimental data. This approach provides an efficient framework for optimizing the performance of ternary geopolymer mixes.

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