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Effect of red mud-based geopolymer binder on the strength development of clayey soil

S. Singh[1], A. Kumar[2], and T. G. Sitharam[3]

[1]*Research scholar, Indian Institute of Technology, Guwahati, India, email: surendersingh@iitg.ac.in*

[2]*Associate Professor, Indian Institute of Technology, Guwahati, India, email: abhiak@iitg.ac.in*

[3]*Chairman, All India Council for Technical Education, New Delhi, India, email: proftgs@gmail.com*

ABSTRACT

Disposal of mine tailings is a big concern for mining enterprises as it requires a large part of land for the construction of tailings storage facilities. Utilization of mine tailings as a geopolymer binder for soil stabilization could be a viable solution for its effective disposal. This article investigated the potential of red mud (bauxite tailings) as a geopolymer binder for its use in soil stabilization. Soil mixtures were prepared by partially replacing the soil with 10%, 20%, and 30% of red mud. Further, a mixture of NaOH and Na_2SiO_3 solutions prepared in a ratio of 30/70, 50/50, and 70/30 were utilized for activation. To examine the strength characteristics of stabilized soil, unconfined compressive strength tests were done after a curing period of 3, 7, and 28 days. Additionally, field emission scanning electron microscope images and energy dispersive X-ray spectroscopy were utilized to study the microstructural and elemental level changes occurring during the geopolymerization process. Based on the experimental findings, the compressive strength of bare soil could be increased from 0.35 MPa to 2.14 MPa by utilizing red mud as a geopolymer binder. This is attributable to the formation of N-A-S-H gel structures during geopolymerization which was further confirmed by the microstructural analysis. Furthermore, the highest improvement in compressive strength was achieved with the addition of 20% red mud activated by NaOH + Na_2SiO_3 solution mixed in 70/30 ratio. Overall, the current study shows that red mud can be utilized as a geopolymer binder for its usage in soil stabilization.
Keywords: Red mud, alkali activation, mine tailings, soil stabilization, unconfined compressive strength

1 INTRODUCTION

Red mud (RM) is an industrial waste produced by the aluminum industry during the Bayer process of extracting alumina from bauxite ore (Zhang et al., 2010). Annually, over 120 million tonnes of RM is produced worldwide (Xue et al., 2016), with India alone producing 2 million tonnes (Nath et al., 2015). Due to the addition of caustic soda during Bayer's process, RM is highly alkaline in nature with pH > 11 (Alam et al., 2019), which makes it a hazardous material. Furthermore, the presence of heavy metal ions and excessive alkalinity in RM may pose a risk to the environment and human health (Mudgal et al., 2021). As a result, safe and economical disposal of RM has been a big challenge for mining companies. In most cases, RM is confined in large impoundments (in slurry form) known as tailings storage facilities (TSF). However, this practice is highly expensive and causes serious environmental issues arising due to soil contamination, ground and surface water contamination (Alam et al., 2019). An alternate approach to deal with this problem could be to utilize RM as a geomaterial in various geotechnical construction projects, which can help in minimizing its volume to be stored in TSF. However, the use of RM as a geomaterial is problematic because it may not be environmentally sustainable (Alam et al., 2019). Geopolymer technology can be a green solution to utilize RM as a construction material without creating any environmental troubles. Geopolymers are the materials formed by the reaction of aluminosilicate-rich source material with suitable alkalis (called activators). The reaction between the aluminosilicate-rich source material and alkali results in the development of new aluminosilicates network structures which provide high mechanical strength and high resistance to sulfate and acid attack along with hazardous waste encapsulation properties (Mudgal et al., 2021). Since RM has high silica and alumina content, it can be an excellent material for geopolymer applications. Numerous studies have been carried out in the past which involve the use of RM as a geopolymer binder for its use in various civil engineering projects (Alam et al., 2019; Mudgal et al., 2021; Song et al. 2021;

Occhicone et al., 2021; Li et al., 2022). Alam et al. (2019), for example, investigated the potential of alkali-activated slag stabilized RM as a geomaterial for a variety of field applications. They concluded that RM-based geopolymers not only have great strength and durability properties but are also non-toxic in nature. Mudgal et al. (2021) utilize RM as a precursor material to enhance the strength properties of fly ash-based geopolymers. The experimental study revealed that the addition of 10% RM to fly ash increased the compressive strength of geopolymer from 53 MPa to 65 MPa. On a similar note, Song et al. (2021) investigated the mechanical strength and environmental stability performance of RM and iron tailings-based alkali-activated mortar. The resulting cementitious mortar was found to have excellent compressive and flexural strength properties along with high resistance to acid, alkali, and sulfate attack. Most of the research discussed above has utilized alkali-activated RM for its use as a partial replacement for concrete or mortar. However, to the best of the author's knowledge, there is a dearth of work that utilizes RM in soil stabilization through alkali activation for various geotechnical applications.

This study investigated the utilization potential of RM a geopolymer binder for its use in soil stabilization. Different combinations of soil and RM mixtures were prepared by varying the RM content to 10%, 20%, and 30%. Further, a combination of NaOH and Na_2SiO_3 solutions prepared in 30/70, 50/50, and 70/30 ratios were utilized for activation. Unconfined compressive strength (UCS) tests were conducted after a curing period of 3, 7, and 28 days to examine the mechanical strength of RM-stabilized soil. Additionally, the efficacy of geopolymerization is examined with the help of field emission scanning electron microscope images (FESEM) and energy dispersive X-ray spectroscopy (EDS) analysis. Overall, the current study will help the mining industries to dispose of the RM in an environmentally friendly by using it in various raw material-intensive applications.

2 MATERIALS AND METHODOLOGY

2.1 Material characterization

The soil used in the current study was procured from Guwahati, Assam, India whereas RM was collected from Guru Corporation, Ahmedabad, Gujarat, India. Basic geotechnical properties of soil and RM such as specific gravity, particle size distribution, and consistency limits (liquid limit and plastic limit) were determined as per ASTM D854 (2014), ASTM D422-63 (2007), ASTM D7928-21 (2021) and ASTM D4318-10 (2010) standards respectively. The results of basic geotechnical experiments are summarized in Table 1. Based on the experiments, the specific gravity of soil was determined to be 2.67 while the specific gravity of RM was measured as 2.94. The higher specific gravity of RM can be attributed to the presence of iron compounds which can be verified by the chemical analysis discussed later. Figure 1 shows the particle size distribution curve of soil and RM obtained from dry sieve analysis coupled with hydrometer analysis. It can be noted from Figure 1 that the RM contains a higher finer fraction (particle size < 0.075 mm) in comparison to soil. Based on the basic geotechnical properties both soil and RM were classified as low plastic clay (CL) as per Unified Soil Classification System (USCS) (ASTM D2487, 2011).

Since the efficacy of geopolymerization is highly dependent on the amount of silica and alumina content in raw material. As a result, to determine the silica and alumina content in RM, chemical analysis was performed using a Panalytical Zetium X-ray fluorescence spectrometer (XRF). The results obtained from XRF analysis are summarised in Table 2, which shows a notable alumina and silica content in RM making it a desirable material for geopolymer binder.

Table 1. *Basic geotechnical properties of soil and RM used in the current study*

Properties	Soil	RM
Specific gravity (G)	2.67	2.94
Clay content (< 0.002 mm)	19.05	58.20
Silt content (0.075 – 0.002) mm	51.03	29.40
Sand fraction (4.75 – 0.075) mm	29.81	12.40
Liquid limit (%)	38.51	40.30
Plastic limit (%)	15.14	18.85
Plasticity Index	23.37	21.45
USCS classification	CL	CL

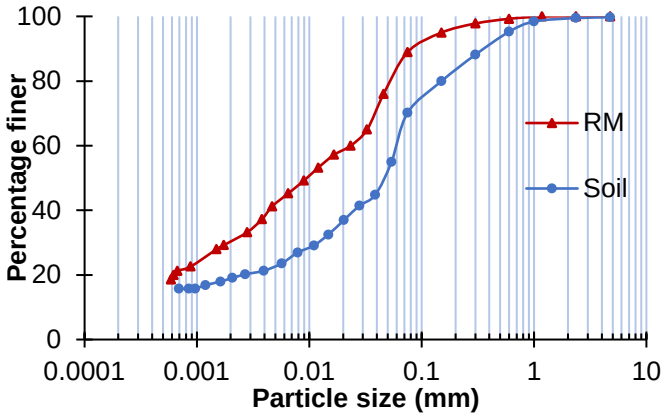


Figure 1. *Particle size distribution curve for soil and RM*

Table 2. *Chemical composition of RM determined from XRF analysis*

Major oxides	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	SO_3	LOI[a]
RM (%)	36.12	23.90	23.11	0.036	0.12	0.18	0.00	0.14	2.67	0.05	0.02	13.65

[a] Loss on ignition

The role of alkali activators in geopolymerization is to dissolve the alumina and silica in the source material, making free Si- and Al- ions available for the formation of new polymeric chains of aluminosilicate oligomers. In most cases, NaOH has been utilized as an alkali activator since it can readily dissolve alumina and silica species. However, in the present work, a combination of NaOH and Na_2SiO_3 was used for activation since Na2SiO3 also aids in binding aluminosilicate chains during the polycondensation phase (Kiventerä et al. 2020; Nwonu, 2021). The concentration of NaOH solution used in the NaOH+Na_2SiO_3 mixture was fixed at 5M, which was prepared by dissolving the required mass of NaOH pellets (97% purity) in deionized water. Further, the Na2SiO3 solution used in the current work was purchased from the manufacturer having chemical composition as SiO_2 = 28%, Na_2O = 8.5%, and H_2O = 63.5%. In order to examine the most efficient proportion of NaOH and Na_2SiO_3 required for geopolymerization, 3 sets of alkali activator solutions were prepared by varying the NaOH to Na_2SiO_3 ratio as 30/70, 50/50, and 70/30.

2.2 Experimental program

2.2.1 Compaction characteristics of soil-RM mixtures

Compaction characteristics of different soil-RM mixtures were determined using mini compaction tests (Sridharan and Sivapullaiah, 2005) to obtain the target dry density for the sample preparation required for mechanical strength tests. To start the compaction tests, oven-dried soil, and RM are hand mixed thoroughly until a homogeneous mixture was obtained. Thereafter, an alkali activator solution was added to the soil-RM mixtures and blended thoroughly for 5 minutes to obtain a homogeneous mixture with no visible lumps. The mixture thus formed was compacted in three layers in a mini compactor mold by providing 36 hammer blows as per the guidelines given by Sridharan and Sivapullaiah (2005). The procedure was repeated 6 times by altering the amount of alkali activator solution to obtain a smooth compaction curve. 4 compaction tests (including bare soil) were conducted with varying RM content i.e. 10%, 20%, and 30%. Results of compaction tests are presented in the form of dry density and alkali activator content plots as shown in Figure 2. Figure 2 shows that the addition of RM enhanced the maximum dry density (MDD) of bare soil while decreasing the associated optimum alkali content (OAC). For instance, the MDD of soil increased from 1.67 g/cc to 1.77 g/cc when 10% RM was added to it. Similarly, the OAC of bare soil was observed to decrease from 19.20% to 17.80% when it was stabilized with 10% of RM. The reason for the higher MDD of soil with the addition of RM can be attributed to the higher specific gravity of RM in comparison to bare soil which increases the MDD of soil mixes. Moreover, the presence of higher fines in RM as a comparison to soil fills the air voids created by the soil particles further increasing the MDD. On the other hand, the increase in OAC can be related to the higher finer fraction in RM than bare soil which increases the water holding capacity resulting in increased OAC values. However, a slight decrease in the MDD of soil was noticed when RM content was increased from 20% to 30%. This is due to the reason that the addition of RM beyond 20% replaces the soil particles from the Soil-RM mixture creating additional air voids, which decreases the MDD of the

mix. Based on the compaction curves (see Figure 2), the variation of MDD with different RM content was found to be insignificant and was ignored in the current study. As a result, a constant MDD and OAC pertaining to soil stabilized with 30% RM (i.e., MDD = 1.76 g/cc, OAC = 17.80%) were used in preparing the sample for compressive strength tests.

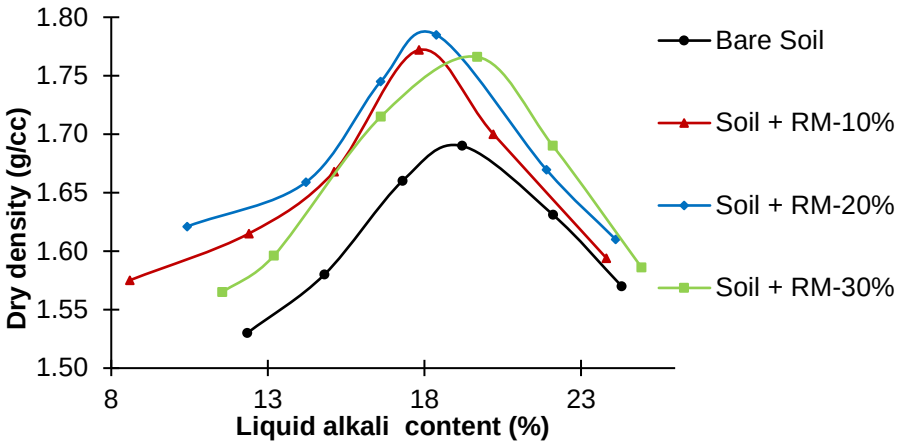


Figure 2. *Compaction characteristics of soil-RM mixtures*

2.2.2 UCS testing

To examine the mechanical response of soil stabilized with alkali-activated RM, UCS tests were conducted following ASTM D2166 (2006) standards. Axial load was applied on soil specimens at a constant displacement rate of 1.25 mm per minute using an automated loading machine with a load capacity of 50 kN. Cylindrical samples of diameter = 38 mm and height = 76 mm were prepared with MDD = 1.76 g/cc and OAC = 17.80% (obtained from compaction tests). The specimens were then wrapped in plastic bags and kept for 3, 7, and 28 days of curing under ambient curing (AC) conditions at (25 ± 2) ℃. For each test, at least three samples were prepared and the average of these three samples was reported as the UCS of the specimen. Results obtained from UCS tests are discussed in the results and discussion section.

2.2.3 FESEM and EDS analysis

To investigate the microstructural and elemental level changes occurring during the geopolymerization reaction, FESEM and EDS analysis were performed on the RM-stabilized soil samples. The microstructural analysis was done utilizing FESEM (manufacturer – Zeiss, model – Gemini) images whereas elemental composition was determined using FESEM with OXFORD energy dispersive X-ray spectroscopy (EDS) (manufacturer – Zeiss, model – Gemini). Samples for FESEM and EDS analysis were taken from the specimens which were already UCS tested. The samples were kept overnight in an oven at a temperature of 60 ℃ before being gold coated for FESEM and EDS analysis. The results of FESEM and EDS analysis are discussed later in the results and discussion section.

3 RESULTS AND DISCUSSION

3.1 Compressive strength

Table 3 presents the summary of UCS tests performed on various soil-RM mixtures after a curing period of 3, 7, and 28 days. The UCS tests revealed that the compressive strength of bare soil could be increased from 0.29 MPa to 2.14 MPa by using alkali-activated RM. Furthermore, the strength improvement in soil was found to be highly dependent on the RM content, curing period, and NaOH to Na2SiO3 ratio, the effects of which are explored in depth in the following sections.

3.1.1 Effect of RM content

Figure 3 illustrates the UCS of soil-RM mixtures activated with different proportions of NaOH/Na2SiO3 after a curing period of 28 days. Figure 3 shows that the compressive strength of soil increases when the RM content in soil was increased from 10% to 20% regardless of the alkali activator used for activation. For instance, the UCS of soil stabilized with 20% RM was found to be 2.14 MPa in comparison

to 1.97 MPa for soil stabilized with 10% RM. The higher compressive strength of soil with 20% RM than 10% RM can be attributed to the increased availability of alumina content resulting in the higher dissolution of Al- ions during the activation phase. This in turn increases the formation of N-A-S-H gel structures during geopolymerization contributing to the increased compressive strength of soil with 20% RM content. However, the compressive strength of soil was found to decrease when the RM content was increased from 20% to 30%. This is due to the fact that the alkali activators employed in the current study failed to dissolve and activate all Al and Si units, resulting in lesser N-A-S-H gel formation during the geopolymerization process. However, increasing the concentration of NaOH solution can aid in the dissolution of additional Al and Si units, the effect of which is beyond the scope of this article. Overall, 20% RM was found to be an optimum content to stabilize the soil via alkali activation.

Table 3. *Summary of UCS tests for alkali-activated soil-RM specimens*

Soil mixtures	Curing period	UCS (MPa)		
		N/S[b]=30/70	N/S=50/50	N/S=70/30
Soil+RM-10%	3	0.41	0.40	0.51
	7	0.49	0.42	0.58
	28	1.27	1.57	1.83
Soil+RM-20%	3	0.37	0.46	0.49
	7	0.42	0.57	0.56
	28	1.24	1.75	2.15
Soil+RM-30%	3	0.35	0.31	0.36
	7	0.38	0.35	0.48
	28	1.19	1.22	1.43

[b] $NaOH/Na_2SiO_3$

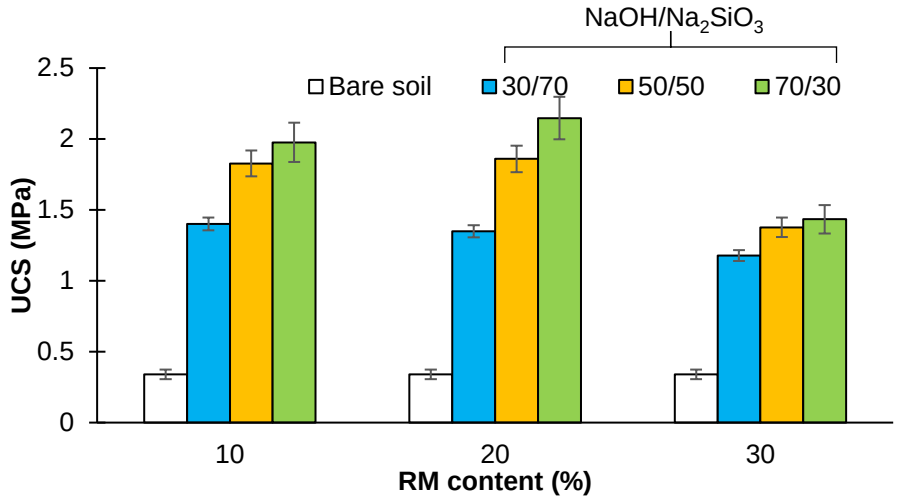


Figure 3. *UCS of different soil-RM specimens after a curing period of 28 days*

3.1.2 Effect of NaOH to Na_2SiO_3 ratio

Figure 4 shows the compressive strength of stabilized soil samples with different proportions of NaOH and Na_2SiO_3 solution. It can be noted from Figure 4 that mixtures of NaOH and Na_2SiO_3 solution mixed in a 70/30 proportion showed the highest strength improvement in comparison to those prepared in 50/50 and 30/70 ratios. For instance, UCS of soil-RM mixture activated with $NaOH/Na_2SiO_3$ = 70/30 was found to be 2.15 MPa as compared to 1.86 MPa and 1.40 MPa for $NaOH/Na_2SiO_3$ = 50/50 and 30/70, respectively. This implies that the efficacy of geopolymerization increases with an increase in the concentration of NaOH in an alkali activator solution contributing to the higher compressive strength of the soil. The reason for this can be attributed to the fact that the dissolution and activation of Al and Si units increase with an increasing proportion of NaOH in an alkali activator solution (NaOH+ Na_2SiO_3), which results in the formation of new aluminosilicate networks.

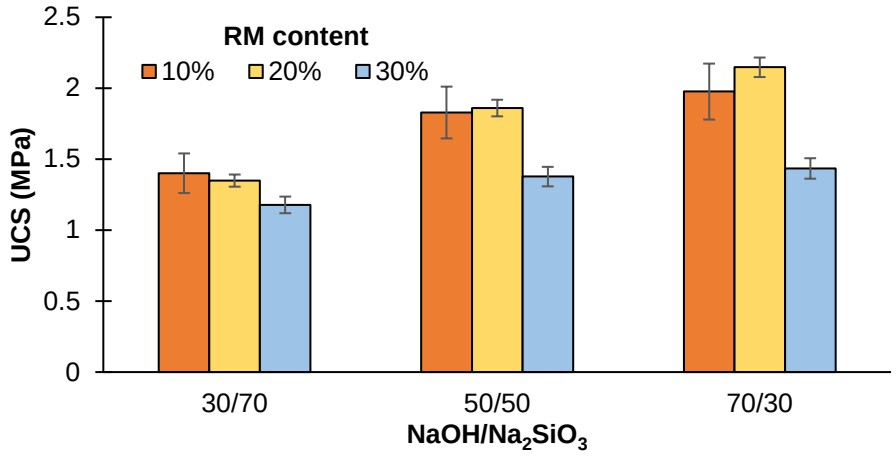


Figure 4. *Variation of UCS determined after 28 days of curing with different NaOH to Na_2SiO_3*

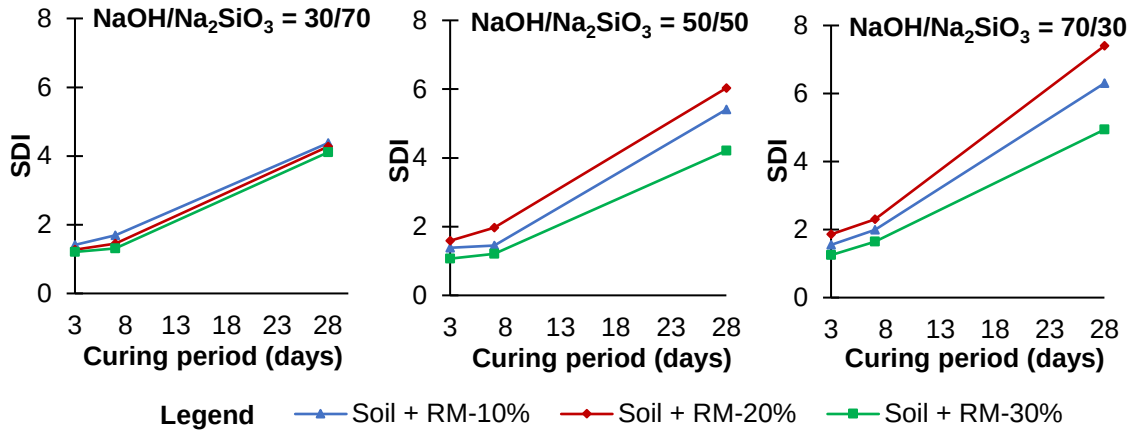


Figure 5. *Variation of strength development index (SDI) with curing period*

3.1.3 Effect of curing period

The strength development index (SDI), which is the ratio of UCS of soil after stabilization to the UCS of bare soil, was employed in the current study to examine the impact of the curing period on strength development (Abdullah et al., 2020). The variation of SDI with the curing period is depicted in Figure 5 for different soil-RM mixtures. It can be noted from Figure 5 that for all soil mixtures, SDI increases with an increase in the curing period indicating a progressive development of compressive strength in the soil matrix. This increase in the SDI with an increasing curing period can be related to the formation of new aluminosilicate networks during geopolymerization, which imparts strength properties to soil specimens. Furthermore, independent of the RM content, the rate of SDI increase showed an increasing trend with an increasing proportion of NaOH in the alkali activator solution (refer to Figure 5). This may be because an increase in the amount of NaOH leads to a greater dissolution of Al and Si units, which in turn enhances the formation of N-A-S-H gel structures. Moreover, soil stabilized with 20% RM exhibits the highest SDI at all curing periods, which can be related to its higher UCS (see Table 3) than other soil-RM mixtures.

3.2 FESEM and EDS analysis

Figure 6 presents the results of the FESEM analysis performed on soil stabilized with different percentages of RM after a curing period of 28 days. It can be seen from Figure 6 that N-A-S-H gel was formed in all soil mixtures indicating the geopolymerization reaction between the alkali activator and Al- and Si- units. Furthermore, the highest response to geopolymerization reaction was observed in soil stabilized with 20% RM, which can be confirmed by Figure 6b showing the development of more N-A-S-H gel structures than other mixes. The development of more N-A-S-H gel structures in soil stabilized with 20% RM results in a much denser soil matrix than other soil mixes (refer to Figure 6). Furthermore, microcracks were noticed in the soil specimens stabilized with 10% and 20% RM (refer to Figure 6),

which may have contributed to their lower compressive strength when compared to soil stabilized with 20% RM.

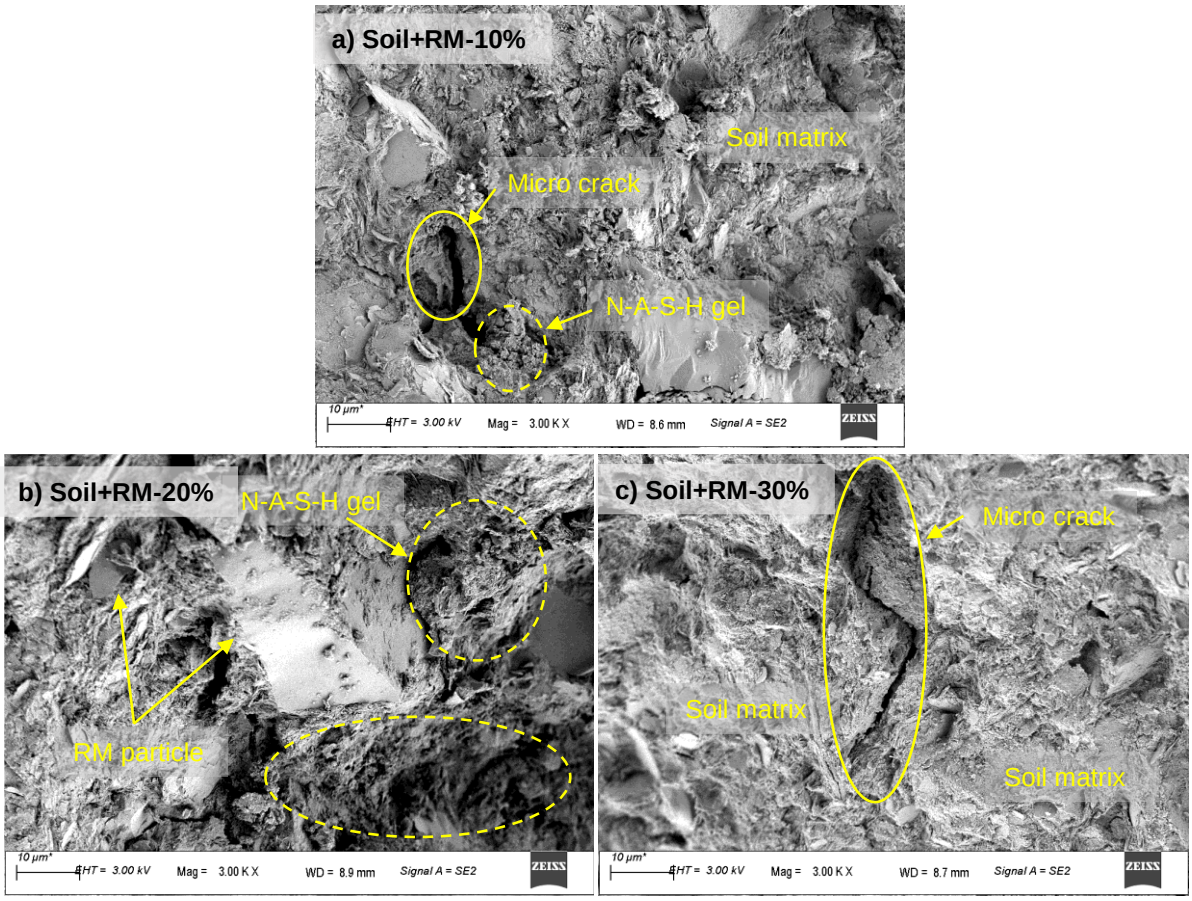


Figure 6. *FESEM images of different soil-RM mixtures activated with NaOH/Na_2SiO_3 = 70/30*

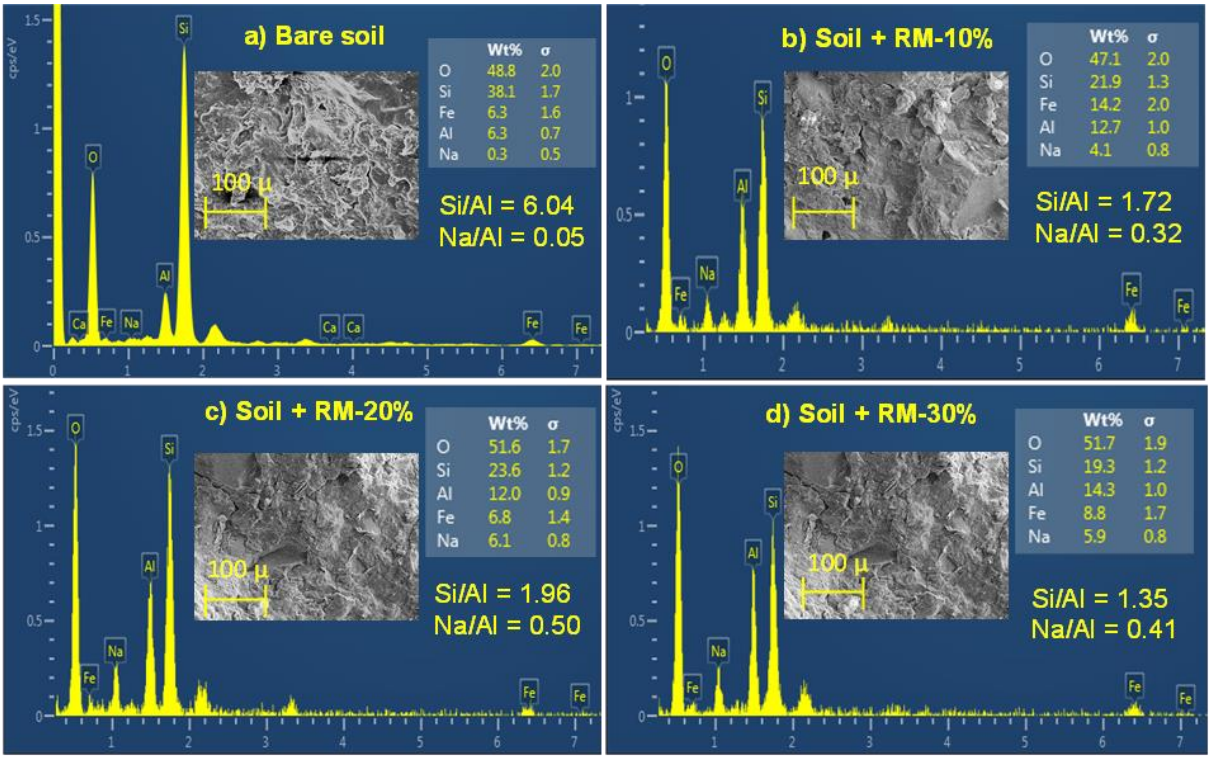


Figure 7. *Results of EDS test performed on different soil mixtures after 28 days of curing*

The rate and efficacy of geopolymerization is highly dependent on the Si/Al and Na/Al ratios of the stabilized soil matrix. In general, the microstructural and strength properties of geopolymer specimens improve with an increase in the Si/Al and Na/Al ratios (Wang et al., 2021). To further investigate the effect of Si/Al and Na/Al on the geopolymerization reaction and thereby on the overall development of compressive strength, EDS tests were conducted on different soil mixtures after 28 days of curing. Results of EDS analysis (obtained in terms of wt.% of elements such as Si, Al, Na, etc.) performed on different soil mixtures are presented in Figure 7. Of all geopolymer specimens, the highest Si/Al ratio (i.e. 1.96) was observed in soil stabilized with 20% RM promoting the formation of more -Si-O-Si- networks than -Si-O-Al- or -Al-O-Al-. Since, the chemical bond strength of -Si-O-Si- bonds is much stronger than -Si-O-Al- and -Al-O-Al- bonds (Wang et al., 2021), higher compressive strength is achieved by the soil mixtures with greater Si/Al ratio. Similarly, the compressive strength of geopolymer specimens increases with an increase in the Na/Al ratios. This is due to the reason that higher dissolution of Si- and Al- ions occurs with an increase in the Na/Al ratio, leading to the formation of more N-A-S-H gel structures. As a result, the soil specimen with 20% RM acquired the maximum compressive strength since it has the highest Na/Al ratio (i.e. 0.50) among all soil combinations (refer to Figure 7).

4 CONCLUSIONS

This study investigated the feasibility of RM as a geopolymer binder in soil stabilization for various geotechnical applications. The mechanical strength of soil stabilized with different percentages of RM and activated with different proportions of NaOH to Na_2SiO_3 ratio was determined by UCS tests. Further, FESEM and EDS analysis were performed to study the microstructural and elemental changes occurring during the geopolymerization reaction. The following conclusions were drawn from the current experimental study:

- The compressive strength of bare soil could be increased significantly from 0.29 MPa to 2.15 MPa with the use of alkali-activated RM as a geopolymer binder.
- The compressive strength of soil first increases and then decreases with an increase in the percentage of RM content. The highest compressive strength of soil was achieved with the addition of 20% RM followed by 10% and 30% RM.
- The compressive strength of soil-RM mixtures was observed to increase with an increase in the proportion of NaOH in the alkali activator ($NaOH+Na_2SiO_3$) solution. This is due to the higher dissolution of Al and Si units in a highly alkaline environment created by the addition of additional NaOH to the alkali activator solution as confirmed by FESEM and EDS analysis. Overall, the highest improvement in compressive strength of soil was achieved with the activator solution having $NaOH/Na_2SiO_3$ = 70/30.
- Overall, a geopolymer binder combination of 70% NaOH, 30% Na_2SiO_3, and 20% RM was recommended as a suitable geopolymer binder combination to stabilize the soil for various field applications.

In conclusion, the current study can assist mining enterprises in the clean disposal of RM by utilizing it as a geopolymer binder for various field applications such as deep soil mixing, highway embankment material, pavement material, etc. Furthermore, utilizing RM in various raw material-intensive applications will help the mining sector in minimizing RM volume to be stored in TSF thereby reducing the operational cost involved in its management.

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