https://doi.org/10.3311/PPci.37270

Periodica Polytechnica Civil Engineering

Characterization of Phosphogypsum Blended Red Mud as Geo-material

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Received: 07 May 2024, Accepted: 21 October 2024, Published online: 30 October 2024

Abstract

Red mud (RM) is a toxic material discarded after extraction of aluminum from the Bauxite and is disposed of in the form of slurry or as low-water content cake as stacking. Either form of disposal is a threat to the environment due to the alkaline nature of RM and also consumes vast tracts of land. Occasional failure of the dyke of the RM pond causes a slurry flooding over a large area, which creates geo-environmental problems. The highly alkaline nature of the RM restricts its bulk utilization, as the high pH value supports the leaching of the heavy metals. On the other hand, phosphogypsum (GYP) is an acidic (pH < 7) material produced from the phosphate industry, blending of which can reduce the alkalinity of the mixture. In the present study, an attempt has been made to use GYP blended RM as a resource geomaterial. The GYP is mixed with RM in 5%, 10%, and 15% on a dry basis to evaluate the chemical, physical, and mechanical properties of the newly developed resource material after 3, 7, and 28 days of curing period. The addition of 5% and 10% GYP lowered the maximum dry unit weight, however, 15% GYP increased the maximum dry unit weight upto 15.47 kN/m^3. The 28-days strength of 5%, 10%, and 15% GYP blended RM was found as 433.57 kPa, 592.44 kPa, and 711.53 kPa, respectively, which further increased to 752.11 kPa, 1120.38 kPa, and 1371.80 kPa, respectively, after alkali activation.

Keywords

red mud, phosphogypsum, microstructure, leaching, geo-material

1 Introduction

The red mud (RM) is a toxic waste material discarded during the Bayers process, and the generation rate as high as 0.8–1.5 tons of RM is produced during the production of 1 tons of aluminum [1]. An approximated quantity of 120 million tons per year of RM is generated throughout the world [2] of which India alone generates 2 million tons yearly [1]. Due to the low utilization rate and high generation rate, most of the RM remains unutilized and stored as slurry in ponds or in the form of dry stacking [3, 4]. In both disposal methods, the storage consumes a vast tract of land and poses serious environmental problems due to the high pH value, which favors the leaching of heavy metals [5]. Also, the occasional failure of the dyke such as Ajka in Hungary causes slurry flooding over a large area [6, 7], and similarly, the slope failure of HINDALCO RM piling in April 2019 creates an environmental problem [8]. Due to the high environmental risk, there is an urgent need to reduce the storage of RM. On the other hand, phosphogypsum (GYP) is a toxic discarded material produced by the phosphate industry, and the generation is as high as 4–6 tons of GYP for every ton of P_2O_5 [9]. The world-wide yearly production of the GYP has been reported as 200 to 250.10^6 tons [9]. In India, Paradeep Phosphate Limited alone generated 1.45 MT GYP in 2020–2021 and disposed in Gypsum Pond; however, some quantity has been used in cement plants and sulfur supplement for farmers [10].

Several studies have been conducted on the utilization of RM for treatment of other waste material, remediation of contaminated soil, improving the geotechnical property of the soil, and additives in the cement [11–13]. Attempts have also been made for bulk utilization of RM [14]. The study on bulk utilization of RM as a binder is limited to a partial replacement of other precursors such as fly ash, lime, ground granulated blast furnace slag, and bio-polymer [15–17]. The reason why RM is used only as a partial

Cite this article as: Alam, S., Das, S. K. "Characterization of Phosphogypsum Blended Red Mud as Geo-material", Periodica Polytechnica Civil Engineering, 2024. https://doi.org/10.3311/PPci.37270

replacement of other binders is that RM chemical composition hinders a complete reaction and requires high water demand [18]. The GYP is used in the cement concrete from the very past by several researchers [19, 20]. In the recent era, there is a need for bulk utilization of GYP to reduce its storage and hence the adverse environmental impact. Ghosh [21] studied the mechanical properties of lime and GYP-stabilized pond ash, and Silva et al. [22] utilized the GYP with soil and cement for asphalt pavement to improve the durability. Few researchers worked on the compressive strength of the cement-stabilized GYP [23]. Few researchers have worked on the combined use of GYP and RM with fly ash and cement [24, 25]. However, the study on the development of materials by using GYP and RM is limited [26]. Also, the work on the stabilization of RM using GYP is not available to the best knowledge of authors. So, in the present study, the highly alkaline RM is blended with the GYP to develop a new geotechnical material. This research will be the first of its kind in this direction and will be helpful for the researchers to work in this direction.

2 Materials and methodology

The Indian RM collected from HINDALCO, Muri, has been used in the present study, whereas the GYP is collected from Paradeep Phosphate Limited. Before using the RM and GYP, it has been dried in the electric oven at a temperature of 105 °C to 110 °C for 24 h. Chemical characterization of the GYP and RM has been done using the FTIR, SEM-EDX, leachate analysis, and variation of pH with moisture content. The sample for leachate analysis is prepared by the batch leaching process suggested by EPA test method 1311 [27]. Later, the RM is blended with 5%, 10%, and 15% GYP for geotechnical characterization. The Atterberg limits of the blended RM have been obtained using ASTM D4318-17-e1 [28], whereas the compaction characteristics and the unconfined compressive strength (UCS) have been performed using the method suggested by ASTM D698-12 [29]. The UCS is measured after the ambient condition curing period of 3, 7, and 28 days. Further, it was observed that the GYP blended RM is not durable under the wet-dry cycle, so the blended material is activated by 1 M alkali (NaOH) solution, and the strength is measured after a 28-day curing period.

3 Results and discussion

To understand the functional group present in the raw material, FTIR is performed in this research. The FTIR graphs of RM and GYP are shown in Fig. 1.

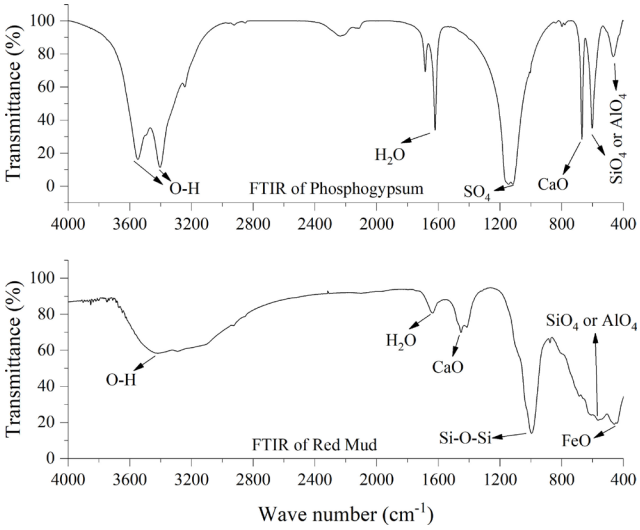


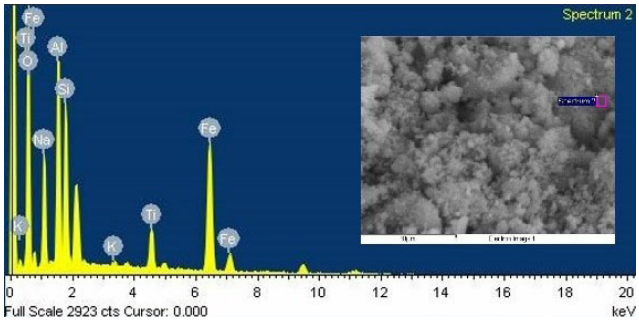
Fig. 1 FTIR of RM and GYP

The functional group corresponding to a particular peak has been identified based on the research published by earlier researchers [30]. The strong broadband is present in between 3400 and 3500 cm^{-1} due to stretching of O-H whereas the H_2O molecule is indicated by the deformation vibration at 1637.09 cm^{-1} in RM and at 1621.11 cm^{-1} in GYP. The stretching vibration of Si-O-Si is indicated by the band at 995.27 cm^{-1} in RM. The peak between 400 and 500 cm^{-1} shows the Si-O or Al-O bend in both RM and GYP which is also confirmed by the EXD result. The peak at 460.24 cm^{-1} in RM shows the stretching vibration of the Fe-O bond, which shows the presence of iron in the RM. In the GYP, a strong peak between 1117.53 cm^{-1} and 669.38 cm^{-1} shows the presence of SO_4 functional group, which is also confirmed by the EDX. The quantitative elemental concentration at the surface of the GYP and RM has been studied through EDX, and the graph is presented in Fig. 2(a) and Fig. 2(b), respectively.

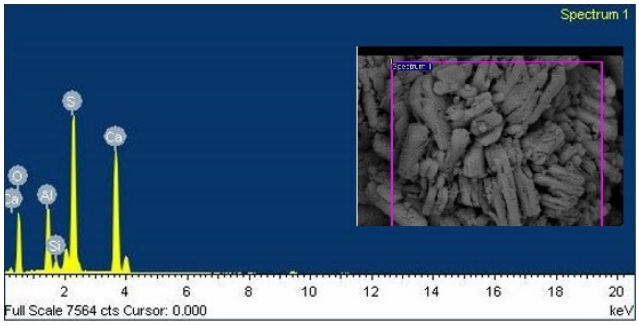
It is observed (Table 1) that the surface of GYP is rich in Ca (23.38%) and S (20.11%), which is in good coordination with the result presented by the previous researchers [19, 31, 32].

However, the RM surface consists of Fe (25.65%) and Al (11.34%) along with 10.44% Na, which makes the RM highly alkaline. To understand the toxicity level of GYP and RM, the concentration (in ppm) of the toxic element is presented in Table 2, along with the pH.

It is observed that the concentration of Ni and Zn is high in the GYP (0.106 and 0.365 ppm) as compared to the concentration in the RM (0.009 and 0.007 ppm). However, the concentration of Cu and Pb in GYP is 0.011 and 0.120. The concentration of Cr is very high in RM (2.59 ppm)



(a)



(b)

Fig. 2 EDX of (a) GYP, and (b) RM

Table 1 Surface chemistry of GYP and RM

Element	GYP (weight %)	RM (weight %)
O	48.90	51.78
Na	–	10.44
Al	6.60	11.34
Fe	–	25.65
Si	1.01	9.34
S	20.11	–
Ca	23.38	0.96
K	–	0.27
Ti	–	4.02

Table 2 Leachate analysis of GYP and RM

Element	GYP (ppm)	RM (ppm) [16]	Level as per EPA
Ni	0.106	0.009	–
Zn	0.365	0.007	–
Cr	0.118	2.59	5.00
Cu	0.011	–	–
Pb	0.120	–	5.00
Hg	–	0.002	0.20
As	–	0.032	5.00
pH	6.18	11.53	–

as compared to the GYP (0.118 ppm), but Hg and As are not observed in GYP. The difference in concentration of toxic material may be due to different pH [5, 33]. In Table 2, it can be observed that the pH of GYP is 6.18, whereas the pH of RM is as high as 11.53. However, in both materials, the concentration of toxic material is observed below the maximum limit suggested by the Environmental Protection Agency (EPA) [27]. Further, the pH of the slurry GYP is studied with different water contents in it. It is observed that the pH value at the water content close to 50% (52.87%) is minimum (4.0) and increases with an increase or decrease in the water content in the slurry GYP (Fig. 3).

Several researchers have also studied the variation of pH of different materials with different liquid-to-solid ratios, and no regular trend of variation is observed [34]. The morphology of the GYP (Fig. 4(a)) shows a plate-like structure with more length and width as compared to its thickness and is very similar to previous research [31]. However, the RM particle (Fig. 4(b)) is irregular in shape and agglomerated, which is very similar to the image presented in the previous research [34].

Later, the RM is blended with 5%, 10%, and 15% GYP to evaluate geotechnical property. For this purpose, three replicas have been studied, and the average value is presented. The liquid limit (LL) of the RM is obtained using the cone penetration method, and the penetration value at different moisture content is presented in Fig. 5. From the plot, the water content at 20 mm penetration is recorded as the LL. The LL of RM with 5% GYP is observed as approximately 41.4%, which is very similar to the LL with 10% GYP (41.5%). However, after mixing 15% GYP, the LL increased to 42.3%. Further, the plastic limit (PL) of the mixture has been obtained, and the variation (Fig. 6) shows almost a linear trend.

The average PL of the RM with 5% GYP is found as 25.33% which increases to 31.77% and 39.15%,

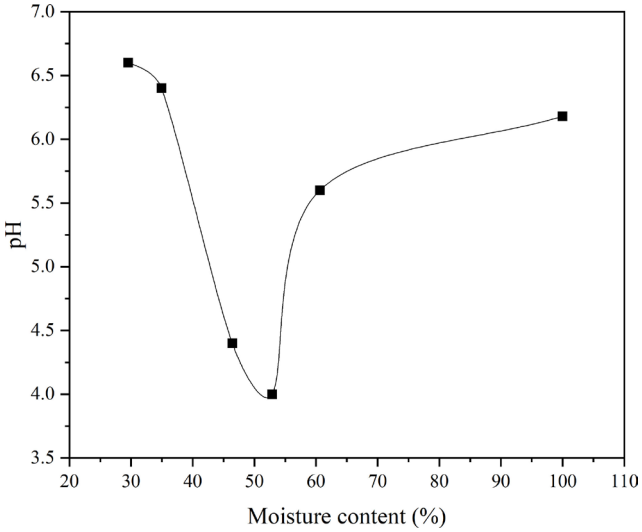
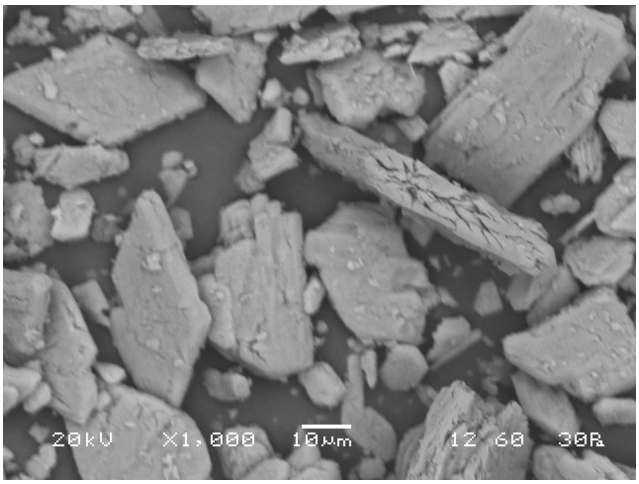
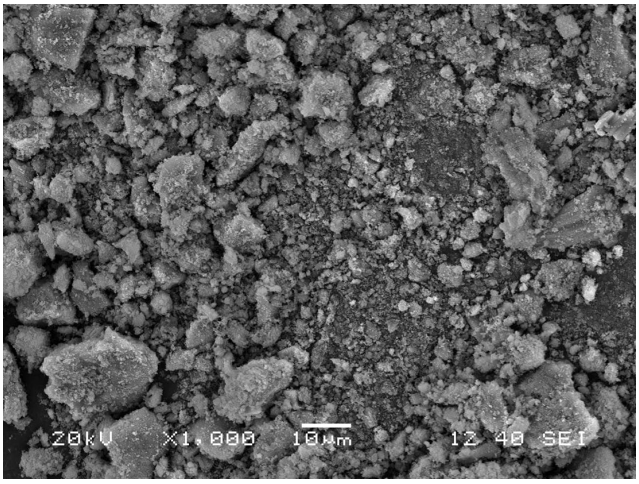


Fig. 3 Variation of the pH of GYP with moisture content



(a)



(b)

Fig. 4 SEM image of (a) GYP, and (b) RM

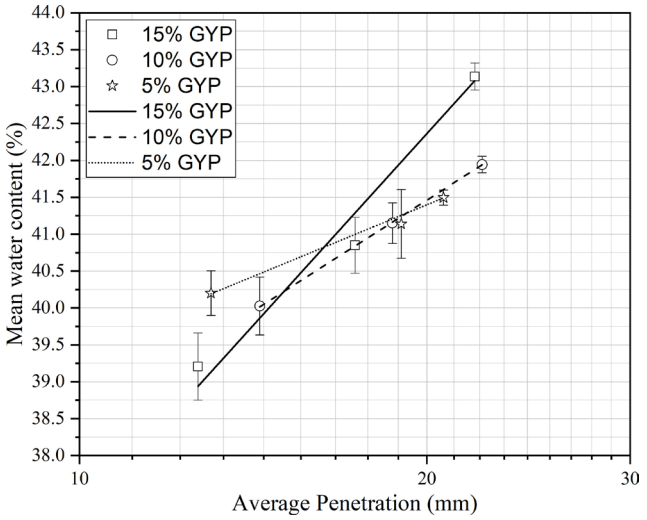


Fig. 5 Determination of LL of RM-GYP mixture

respectively, after adding 10% and 15% GYP. The Fig. 7 shows the variation of the LL and plasticity index (PI) of the mixture. It is observed that the LL of the mixture

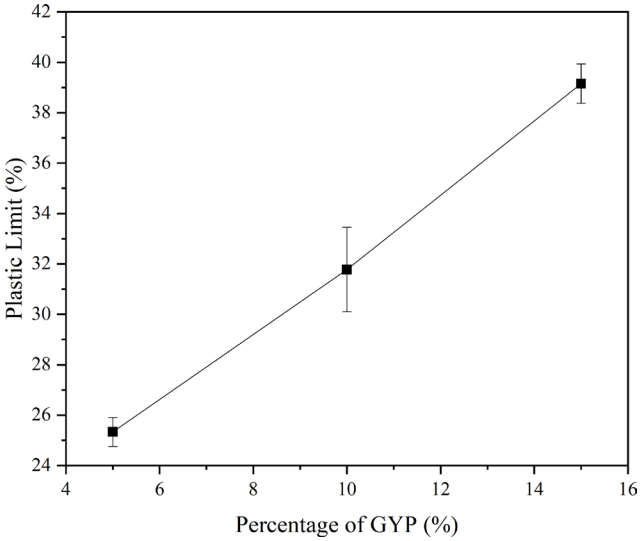


Fig. 6 PL of RM-GYP mixture

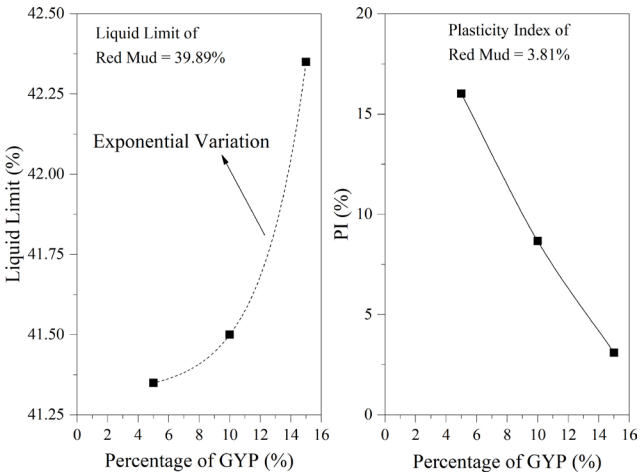


Fig. 7 Variation of LL and PI with percentage of GYP in RM

increases exponentially, and the PI decreases linearly with an increase in the GYP. The PI of RM with 5% GYP is calculated as 16.02%, which decreased to 8.67% and 3.1%, respectively, for 10% and 15% GYP. It is observed from the plasticity chart (Fig. 8) that the RM with 5% GYP lies at the boarder of intermediate plastic clay and silt, whereas, the higher percentage makes the mixture behavior like intermediate plastic silt. From the compaction (Fig. 9), it is observed that the maximum dry unit weight decreased slightly after adding 5% and 10% GYP, however, it increased after adding 15% GYP.

The maximum dry unit weight of the RM is found as 15.05 kN/m^3, which decreased to 14.85 kN/m^3 and 14.94 kN/m^3, respectively, after adding 5% and 10% GYP. However, after adding 15% GYP, the maximum dry unit weight increased to 15.33 kN/m^3 at the optimum moisture content of 30.9%. The result obtained is in well coordination with the results presented in previous research [21, 35].

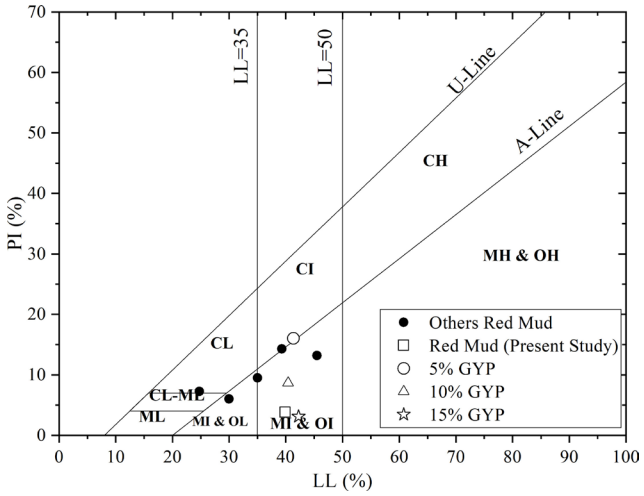


Fig. 8 Classification of RM-GYP mixture

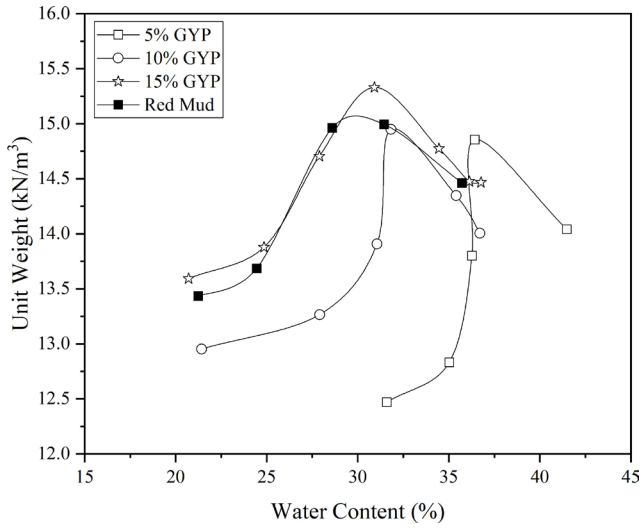


Fig. 9 Compaction curve of RM-GYP mixture

From the UCS (Fig. 10), it is observed that the blending of GYP in the RM increases the compressive strength with curing time as well as with the percentage.

The rate of increase in the strength is observed to be high at an early age, which decreases with time. On the other hand, the rate of increase in compressive strength is higher in the mixture with a higher percentage of GYP. The rate of change in compressive strength between 3 and 7 days is found to be 20.09 kPa, 26.02 kPa, and 34.39 kPa, respectively, for the mixture with 5%, 10%, and 15% GYP. Whereas, the change in compressive strength between 7 and 28 days is found as 3.69 kPa, 9.07 kPa, and 11.61 kPa, respectively, for mixtures with 5%, 10%, and 15% GYP. It is observed that for the same percentage of GYP in the mixture, the rate of gain of strength is higher during 3 to 7 days and decreases after 7 days. The decrease in the rate of strength gain may be due to the decrease in the

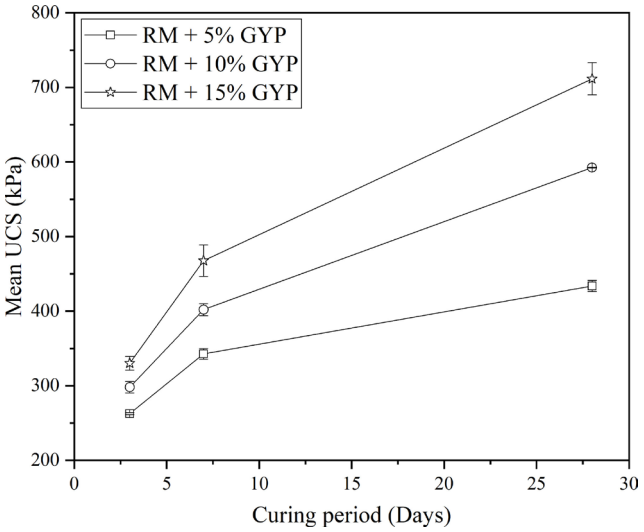


Fig. 10 Variation of the UCS with curing period and percentage of GYP

hydration reaction after 7 days. Further, the increase in the strength with percentage of GYP may be due to the availability of more Ca in the mixture coming from the GYP, as found in the chemical analysis.

Although the strength of the RM blended with GYP increased, it was not found durable under the wet-dry cycle and collapsed when submerged in the water. So, the GYP-RM mixture is activated with 1 M alkali solution (NaOH), and it is observed that alkali activation increases the 28-days compressive strength (Fig. 11). It is observed in Fig. 11 that the rate of increase of UCS from 5% GYP to 10% GYP in the mixture is 368.27 kPa, whereas for further 5% increase in the GYP increases the UCS by 251.43 kPa only, which shows that a further increase in the GYP causes a decrease in UCS. The increase in the strength may be due to the fact that the alkali activation

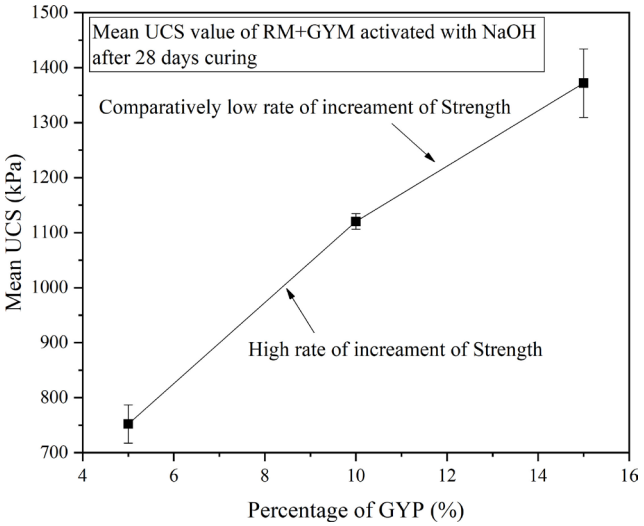


Fig. 11 Variation of 28 days UCS with the percentage of GYP

causes the complete dissolution of the GYP, which makes a solid reactant product [35]. Similar effect on strength after alkali activation of RM-GGBS has been reported in the research [24]. The variation of pH with different moisture content in the GYP has already shown in the Fig. 3. It was observed that the pH of GYP and RM for the liquid to solid ratio of 1:1 is 6.18 and 11.53 respectively.

To understand the effect of GYP percentage and the curing period, the pieces of tested UCS sample and water is mixed in 1:1 solid to liquid ratio and agitated using the magnetic stirrer. Fig. 12 shows the variation of the pH value with the curing period and the percentage of the GYP. It is observed that the addition of 5% GYP in the RM lowers the pH of RM up to 8.87 which further decreased to 8.75 and 8.29 for 10% and 15% GYP, respectively, after 3 days curing and similar changes has been seen for 7 and 28 days of the curing. Further, for the 3 days curing, the rate of change in pH is high for change in GYP percentage from 10% to 15% whereas for 7 days curing, it is almost similar to change in GYP percentage from 5% to 10%.

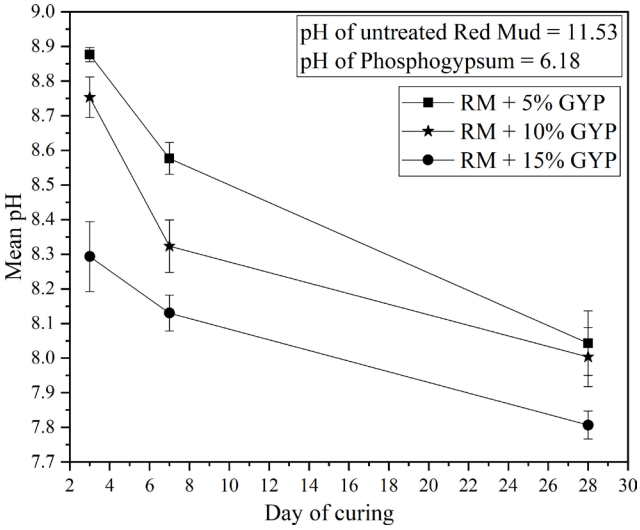


Fig. 12 Variation of pH of RM-GYP with time

4 Conclusions

The present study investigates effect of phosphogypsum blending with the RM to develop a new geo-material. From the extensive laboratory investigations, below mentioned conclusions has been drawn:

- The surface of phosphogypsum is found abundant with Ca (23.38%) and S (20.11%), whereas the surface of RM consists of Fe (25.65%) and Al (11.34%) along with 10.44% Na.
- The pH of RM is found 11.53, which decreases to 8.87, 8.75, and 8.29, respectively, after blending with 5%, 10%, and 15% phosphogypsum.
- The leaching of Zn from phosphogypsum is found maximum with a concentration of 0.365 ppm, and Cr (2.59 ppm) found maximum in RM.
- The average PL of the RM blended with 5% GYP is 25.33% and increased to 31.77% and 39.15%, respectively, after adding 10% and 15% phosphogypsum, whereas, the decreased with an increase in phosphogypsum.
- The maximum dry unit weight of RM is found as 15.05 kN/m^3, which decreased to 14.85 kN/m^3 and 14.94 kN/m^3 for blending of 5% and 10% phosphogypsum, respectively. However, blending of 15% phosphogypsum increased the maximum dry unit weight to 15.47 kN/m^3.
- Three days compressive strength of the material with 5%, 10%, and 15% phosphogypsum is found as 262.46 kPa, 297.97 kPa, and 330.15 kPa respectively, which increased to 433.57 kPa, 592.44 kPa, and 711.53 kPa, respectively after 28 days of curing.
- Alkali activation of mixture with 1M NaOH increased the 28 days compressive strength to 752.11 kPa, 1120.38 kPa, and 1371.80 kPa, respectively, for 5%, 10%, and 15% phosphogypsum.

Acknowledgement

The authors gratefully acknowledge the funding of the Deanship of Graduate Studies and Scientific Research, Jazan University, Saudi Arabia, through project number: RG24-S0168.

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