Kochi Chapter

Indian Geotechnical Conference
IGC 2022
15th – 17th December, 2022, Kochi

Characterization of Soil-Jarosite Mixes for Geopolymer Based Soil Stabilization

Surrender Singh[1], Abhishek Kumar[1] and T.G. Sitharam[1]

[1] Indian Institute of Technology, Guwahati, Assam, India, 781039.

Abstract. In this study, an attempt has been made to evaluate the compaction characteristics of soil-jarosite (SJ) mixes with an objective to check the feasibility of jarosite as a geopolymer binder for alkali-activated soil stabilisation. Jarosite (waste from a Zn smelter) is first characterised in terms of its particle size distribution, specific gravity, Atterberg limits, and microstructure in order to draw comparisons with bare soil. Thereafter, two sets of SJ mixes, one with water and one with an alkali activator solution (NaOH), are prepared with varying jarosite content. Results from the experiments revealed that jarosite is a silty soil with high plasticity (I_p = 16.71) having 72.8% silt and 26.6% clay sized particles. Furthermore, the compaction tests revealed a rise in the optimum moisture content (OMC) and a drop in maximum dry density (MDD) with increase in the jarosite content. In addition, SJ mixes prepared with NaOH solution was observed to have lower MDD and higher OMC in comparison to those prepared with water. The findings of this study can be utilised to select a target dry density and OMC for sample preparation required for the strength determination of geopolymerized soil.

Keywords: Jarosite, mine tailings, compaction characteristics, geopolymer binder.

1 Introduction

Jarosite is a waste by product produced by zinc (Zn) smelter during various hydrometallurgical processes, which are performed for the extraction of the metallic Zn from its ore. Jarosite is a hydrous sulphate of potassium and ferric iron with a chemical formula $KFe_3(SO_4)_2(OH)_8$. Due to its hazardous nature, jarosite is mixed with 2% lime and 10 % cement before its disposal into the tailings ponds (TP) (Seyer et al., 2001). Storage of jarosite in TPs adds no value to the mining industries, instead poses a serious threat to the environment and human health due to breaches of tailings storage facilities. As a result, researchers have been investigating different alternatives to minimize the volume of jarosite to be disposed off. For example, Pappu et al. (2006) investigated the potential of jarosite in the development of value-added products such as bricks. Ray et al. (2020) utilized jarosite as a partial replacement of cement in concrete mixtures and obtained satisfactory results in terms of mechanical strength and durability. On a similar note, Gared and Gaur (2020) also employed jarosite as a substitute to cement in concrete mixes for its use in rigid pavement. According to them, with increase in the jarosite content, the mechanical strength of jarosite concrete mixtures was observed to increase, while the porosity and abrasion loss was found to decrease. Beena and Santhosh

(2021) utilized jarofix along with varying percentages of lime to enhance the geotechnical properties of soil to be used in road pavement. Most of the aforementioned studies employed jarofix for soil stabilization or as a cement substitute in concrete mixtures. Since jarofix is made by adding lime and cement to the jarosite, it is not an environmentally sustainable practice due to the high carbon footprint associated with the production of lime and cement. Therefore, it is necessary to seek out alternative methods that not only detoxify jarosite, but also stabilize the soil. Utilizing jarosite as a geopolymer binder for alkali activated soil stabilization (geopolymerisation) can be an alternative way to manage jarosite in an environmentally sustainable manner. In geopolymerisation, aluminosilicate-rich material is combined with an alkali activator to create a new polymeric chain-like structure composed of aluminosilicate networks. Mostly, industrial by products such as fly ash, granulated blast furnace slag, sugarcane baggase, etc. have been utilized as a geopolymer binder for soil stabilization (Pereira dos Santos et al., 2022; Yaghoubi et al., 2018). Since mine tailings (MTs) contains considerable amount of SiO_2 and Al_2O_3 content (Kiventerä et al., 2020), they have recently been studied for their possible use as a geopolymer binder (Xiaolong et al., 2021; Kiventerä et al., 2020; Obenaus-Emler et al., 2020; Manjarrez et al., 2019). For instance, Manjarrez et al. (2019) investigated the viability of a geopolymer binder derived from copper tailings for use in road construction and found that copper tailings has the potential to be used as geopolymer binder. Utilizing MTs in raw material intensive applications, like soil stabilization for road applications, foundations etc. , can be an excellent solution to handle the ever-increasing volume of MTs. To the best of author's knowledge, no study has been carried out till date, which utilizes jarosite as a geopolymer binder for its use in road applications. However, before using jarosite as a potential geopolymer binder, it must be characterized in terms of particle size distribution, specific gravity, mineralogy, microstructure, water-holding capacity, and so on. In addition, the compaction characteristics of soil amended with jarosite, which play a crucial role in alkali-activated soil stabilization (particularly for road applications), must be determined in advance.

The main objective of this study is to characterize the jarosite for its use in alkali activated soil stabilisation. Various experiments are performed to examine the basic geotechnical properties such as particle size distribution, specific gravity, Atterberg limits, etc. of jarosite. Furthermore, an attempt has been made to evaluate the compaction characteristics of soil-jarosite (SJ) mixes. Two sets of SJ mixes, one with water and one with NaOH solution (activator), are prepared with varying jarosite content. Comparison are drawn between the compaction curves obtained for bare soil, jarosite and SJ mixes. Finally, target dry density and optimum moisture content (OMC) values were suggested (for different SJ mixes) to be used in sample preparation required for the strength determination of geopolymerised soil.

2 Materials and methods

2.1 Soil and jarosite

Soil used in current study was collected from northeastern state of Assam whereas jarosite was procured from Hindustan Zinc limited (HZL) smelter at Debari, Rajasthan. Basic characterization of soil and jarosite was done following the experiments specified by Bureau of Indian Standards (BIS). Specific gravity (G) of soil and jarosite was determined by density bottle method (IS 2720-3(1), 1980) whereas particle size analysis was done using dry sieve analysis coupled with hydrometer analysis (IS 2720-4, 1985). Liquid limit (LL) of soil and jarosite was measured by Casagrande method whereas plastic limit (PL) was determined by rolling the sample into a thread of 3 mm as per IS 2720-5 (1985). The water holding capacity of soil and jarosite was measured by immersing a known amount of oven-dried sample in distilled water for 48 h. Following that, the wet weight of sample was measured after being gravity drained for 2 h through Whatman grade-1 filter paper. The water holding capacity was then calculated (in percentage) as ratio of wet weight to the initial dry weight of the sample (Patwa et al., 2021). In addition, microstructure analysis was carried out in a field emission scanning electron microscope (FESEM; manufacturer – Zeiss, model – Gemini) which has a Schottky type field emitter system imaging at very high resolution (0.8 nm at 15 kV; 1.4 nm at 1 kV).

2.2 Alkali activator

In addition to water, sodium hydroxide (NaOH) solution (alkali activator) was used to prepare the SJ mixes. NaOH being a strong base activate the geopolymerisation process by dissolving the silica and alumina present in the material. To prepare the NaOH solution, required weight of NaOH pellets was mixed with distilled water to achieve a target concentration. The concentration of NaOH solution in the present study was kept at 5M based on the previous research done on alkali activated soil stabilization (Yaghoubi et al., 2018; Kiventerä et al., 2020; Nwonu, 2021). The dissolution of NaOH in water being an exothermic reaction, generates a lot of heat, therefore the solution thus prepared was allowed to cool down before its use for activation.

2.3 Preparation of SJ mixes

In the present work, jarosite content in the SJ mix was fixed at 15 % and 30%. Two sets of mixes were prepared i.e. one with distilled water and other with NaoH solution. Oven dried soil and jarosite were mixed together thoroughly for 5 minutes to obtain a homogeneous mix. Thereafter, the dry mix thus prepared was blended manually with distilled water /NaoH solution roughly for 10 minutes to devoid the formation of any lumps.

2.4 Standard mini compaction test

The compaction characteristics of SJ mixes was determined utilizing standard mini compaction test apparatus (Sridharan and Sivapullaiah, 2005). Mini compaction test not only involves 1/10 volume of soil as required for standard proctor test but can be performed in less time and effort (Sridharan snd Sivapullaiah, 2005). The various parts of mini compaction test apparatus are shown in Fig.1. The test apparatus consists of a brass mold attached with a base plate, removable collar and a steel drop hammer with guide frame (see Fig. 1). The mold has an internal diameter of 3.81 cm and height of 10 cm leading to an overall volume of 114 cc. Weight of drop hammer is 1 kg for light compaction test with a height of fall of 16 cm.

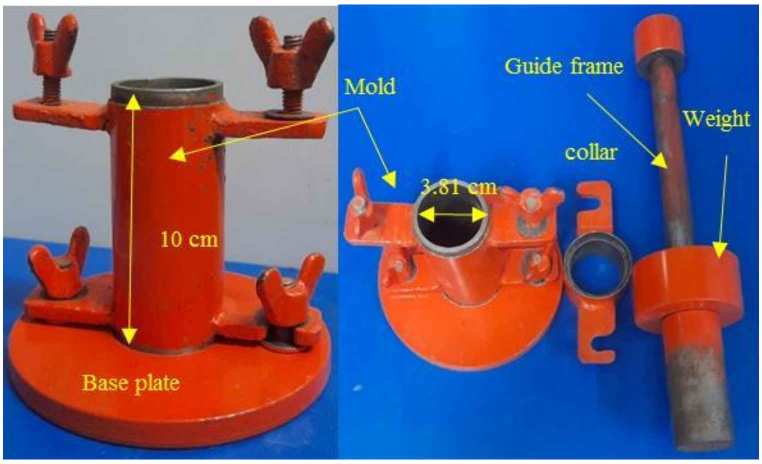

Fig. 1. Mini compaction test apparatus (Sridharan & Sivapullaiah, 2005).

Test procedure. About 200 g dry sample of soil, jarosite and SJ mix was taken for mini compaction test. Soil and jarosite were prepared with distilled water whereas SJ mixes were blended with both distilled water as well as alkali activator solution. The wet mix was then compacted in three layers in the mold by giving 36 blows with 1 Kg hammer. Once the compaction of sample is done, weight of mold with compacted soil and water content was measured to calculate the dry density at respective trials. At least 6 compaction tests were conducted for each mix to obtain an accurate compaction curve. The results obtained from different compaction tests are discussed in the result and discussion section.

3 Results and discussion

3.1 Physical properties of soil and jarosite

Table 1 summarizes the various physical properties of soil and jarosite. The soil has reddish brown physical appearance whereas jarosite has pale yellowish tinge (see Fig. 2). In comparison to soil, the G of jarosite was found to be on higher side. The G of jarosite was measured as 3.01 in comparison to 2.64 for soil. The higher G of jarosite can be attributed to the presence of heavy metals in jarosite raising its G.

Fig. 3 shows the particle size distribution curve obtained from the sieve analysis coupled with hydrometer analysis. It can be observed from fig. 3 that jarosite has greater finer fraction (particle size < 75μ) than the red soil. The sand, silt and clay content in soil was found to be 29.81%, 51.03% and 19.05% respectively. On the other hand, the sand, silt and clay content clay content in jarosite was noted as 1.12%, 72.80% and 26.60% respectively. Based upon the grain size analysis, jarosite has a potential to be used as a filler material for various geotechnical applications.

Table 1. Physical properties of soil and jarosite

Properties	Soil	Jarosite
Specific gravity	2.64	3.01
Clay content (< 0.002 mm)	19.05	26.60
Silt content (0.075 – 0.002) mm	51.03	72.80
Sand fraction (4.75 – 0.075) mm	29.81	1.12
Liquid limit	38.51	66.84
Plastic limit	15.14	50.13
Plasticity Index	23.37	16.71
ISCS classification	CI	MH
Water holding capacity (%)	190.10	140.72

Fig. 2. Physical appearance of soil and jarosite.

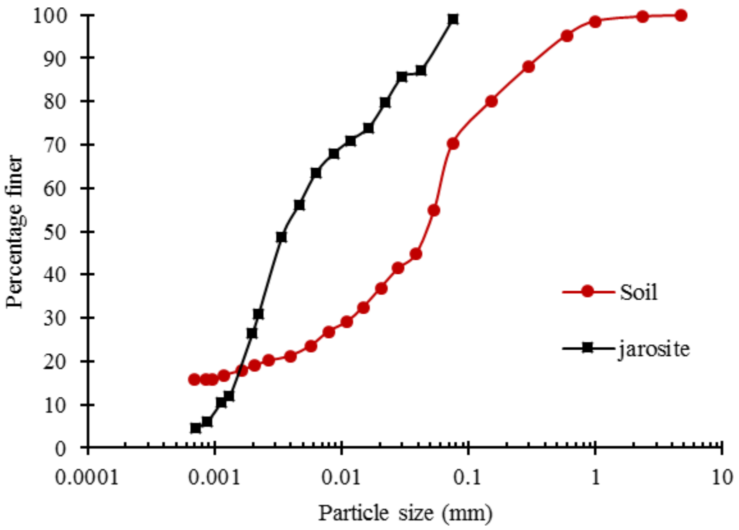

Fig. 3. Particle-size distribution curve for soil and jarosite.

LL of soil was determined to be 38.51% as opposed to 66.4% for jarosite. Similarly, PL of soil and jarosite was found to be 15.14% and 50.13% respectively. As per Indian Standard Soil Classification System (ISCS) (IS-1498, 1970), soil was classified as clay with intermediate plasticity (CI) while jarosite was categorized as silt of high plasticity (MH).

The water holding capacity of jarosite was observed to be higher (190.10%) as compared to soil (140.72%). This might be due to the fact that jarosite contains a much higher finer fraction (particle size < 75μ) than soil, which increases its specific surface area and consequently, its capacity to hold water.

3.2 Microstructure of soil and jarosite

Fig. 4 depicts the microstructure of soil and jarosite obtained from FESEM analysis. It can be seen from fig. 4 (a) that soil particles have irregular structure without any particular shape. On the other hand, jarosite particles have trigonal crystal structure as evident from fig. 4 (b). Similar observations regarding the crystalline structure of jarosite has been also reported by Cruells and Roca (2022) in their study.

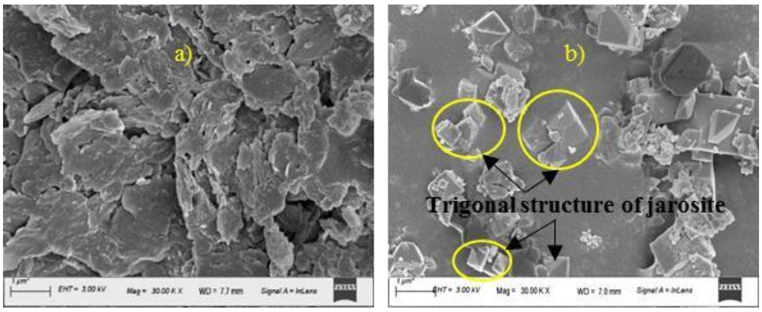

Fig. 4. FESEM images showing the microstructure of a) soil, and b) jarosite particle.

3.3 Compaction characteristics of soil, jarosite and SJ mixes

The MDD and OMC of different materials obtained from mini compaction test are summarized in Table 2. The MDD of jarosite (1.24 g/cc) was determined to be significantly lower as opposed to soil (1.78 g/cc), while the OMC was noticed to be higher than soil. The lower MDD of jarosite can be attributed to its smaller particle size as compared to soil, contributing to it increased air void content. On the other hand, the proportions of sand, silt, and clay in soil are such that the voids left by coarser fractions are filled by finer fractions (see Fig. 5), hence increasing the MDD. Since, jarosite has a higher water-holding capacity than soil, a greater quantity of water is required for lubrication, resulting in a higher OMC compared to soil.

Fig. 6 illustrates the compaction curves obtained for soil, jarosite and SJ mixes. In case of SJ mixes, with increase in jarosite content, a decrease in MDD and increase in OMC was observed. For instance, the MDD of SJ mix (prepared with water) with 15% jarosite content decreased by 3.9% in comparison to bare soil. On the other hand, a 20% increase in OMC was observed with the addition of 15% jarosite to the bare soil. Similarly, for SJ mix with 30% jarosite content, a 5% decrease in MDD and 24.24 % increase in OMC was observed as compared to bare soil (see Fig. 6). Since, jarosite contains a greater proportion of finer particles than soil, air voids existing in soil should be filled with jarosite particles. However, in this case, the amount of jarosite added (i.e. 15% and 30%) was substantially higher than the volume of air voids present in the bare soil, resulting in the reduction of MDD values. Higher OMC of SJ mixes (in compared to bare soil) can be attributed to the high water holding capacity of jarosite (i.e. 190.12%).

Table 2 Maximum dry density (MDD) and optimum moisture content (OMC) of mixes

Material	MDD (g/cc)	OMC (%)
Bare soil	1.78	16.5
Jarosite	1.24	45.5
Soil + jarosite (15%), with water	1.71	19.8
Soil + jarosite (30%), with water	1.69	20.5
Soil + jarosite (15%), with NaOH solution	1.60	23.8
Soil + jarosite (30%), with NaOH solution	1.48	27.8

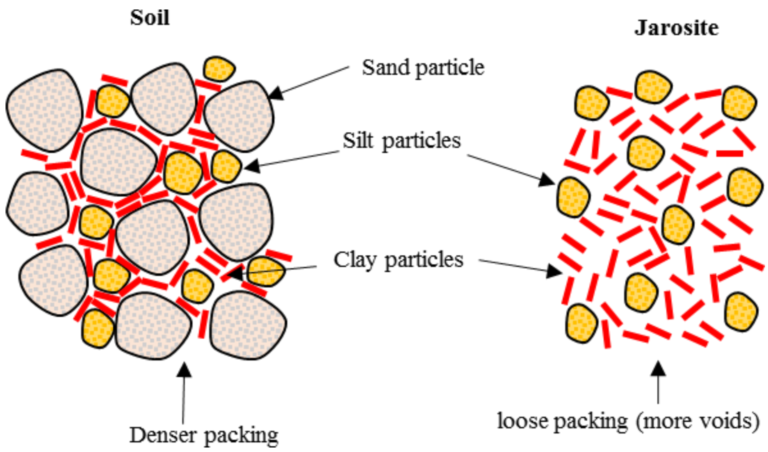

Fig. 5. Particles arrangement in soil and jarosite

Furthermore, the MDD of SJ mixes blended with alkali activator i.e. NaOH solution was observed to be significantly lower than SJ mixes prepared with water (see Fig. 6). For example, MDD of SJ mix (with 15% jarosite content) prepared with NaOH solution decreased by 6.43% in comparison to SJ mix blended with water. On a similar note, a 12.42% decrease in MDD was observed for SJ mix (with 30% jarosite content) prepared with NaOH solution as compared to that prepared with water. This is due to fact that a portion of soil and jarosite particles in SJ mix are replaced by Na_2O solids present in NaOH solution (see Fig. 7), reducing the MDD of SJ mixes. Furthermore, the OMC of SJ mixes prepared with NaOH solution was found to be higher than that prepared with water. Because NaOH solution has a higher viscosity than distilled water, a larger amount of solution is required to lubricate soil and jarosite particles, increasing the OMC values (see Fig. 7).

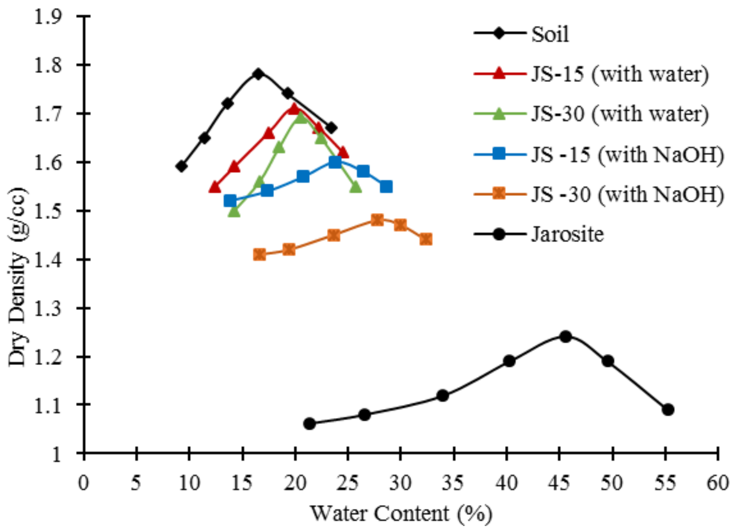

Fig. 6. Compaction curve for soil, jarosite and SJ mixes.

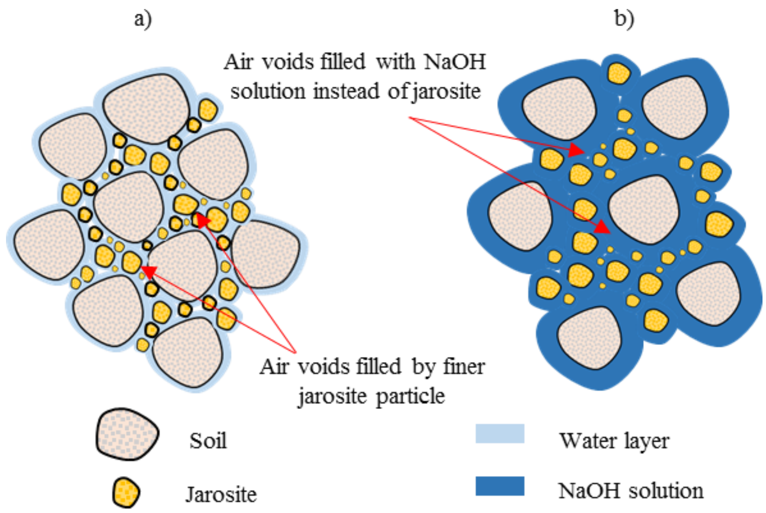

Fig. 7. Mechanism of particles lubrication in SJ mix prepared with a) distilled water, and b) NaOH solution

3.4 Practical applicability of current study

The construction of the pavement layer of a road necessitates an understanding of the compaction characteristics of the materials employed. To achieve desirable strength and durability properties, a specific pavement layer must be compacted on-site to its maximum possible density. On a similar note, the compaction properties of SJ mixes must be evaluated prior to their use as a paving material. The MDD and OMC of SJ mixes varies considerably with NaOH solution as compared to water. Consequently, using compaction properties of SJ mixes determined with water can result in an overestimation of the MDD for SJ mixes prepared with NaOH solution, which in turn results in an overestimation of their strength characteristics. Moreover, determination of compaction characteristics of SJ mixes (with alkali activator) gives a rough estimate about the quantity of NaOH solution required during geopolymerization to obtain a maximum strength. In addition, the determination of compaction characteristics of SJ mixes (with alkali activator) provides a rough estimate of the quantity of NaOH solution required during geopolymerization to achieve maximum strength. In a nutshell, the potential of jarosite as a geopolymer binder cannot be evaluated without knowledge of its fundamental physiochemical and compaction properties.

4 Conclusions

The main objective of the present study was to characterize the jarosite for its potential use as a geopolymer binder for soil stabilization. Basic physical properties of jarosite including particle size analysis, consistency limits, microstructure analysis, etc. were initially investigated. Thereafter, compaction characteristics of SJ mixes prepared with the addition of 15% and 30% jarosite to soil was examined with an objective to find the target dry density and OMC required for the strength determination of geopolymerized soil. Following conclusions were drawn from the study;

- The finer fraction (particle size <75μ) in jarosite was found to be higher than soil. Jarosite was classified as silty soil with high plasticity (MH) whereas soil was categorized as clay of intermediate plasticity as per ISCS system.
- MDD of jarosite was observed to be significantly lower than the soil, however the OMC was found to be higher than bare soil.
- A decrease in MDD and increase in OMC was noted for SJ mixes with increase in the jarosite content.
- In contrast to water, SJ mixes blended with NaOH solution was found to have lower MDD and higher OMC.

References

1. Beena, K. S., Santhosh, G.: Transport geotechnical properties of lateritic soil stabilized with Jarofix and lime. Environment, Development and Sustainability 23(2), 1601–1622 (2021)
2. Cruells, M., Roca, A.: Jarosites: Formation, Structure, Reactivity and Environmental. Metals 12(5) (2022).

3. Gared, O., Gaur, A. Feasibility study of Jarosite as cement replacement in rigid pavement. Materials Today: Proceedings 44, 4337–4341 (2020).
4. IS 1498: Classification and identification of soils for general engineering purposes, Bureau of Indian Standards, New Delhi (1970).
5. IS 2720-Part 3 (1): Methods of test for soils – Determination of specific gravity, Bureau of Indian Standards, New Delhi (1980).
6. IS 2720-Part 5: Methods of test for soils – Determination of liquid limit and plastic limit, Bureau of Indian Standards, New Delhi (1985).
7. Kiventerä, J., Perumal, P., Yliniemi, J., Illikainen, M.: Mine tailings as a raw material in alkali activation: A review. International Journal of Minerals, Metallurgy and Materials 27, 1009–10202020 (2020).
8. Manjarrez, L., Nikvar-Hassani, A., Shadnia, R., Zhang, L.: Experimental study of geopolymer binder synthesized with copper mine tailings and low-calcium copper slag. Journal of Materials in Civil Engineering, 31(8), 04019156 (2019).
9. Obenaus-Emler, R., Falah, M., Illikainen, M.: Assessment of mine tailings as precursors for alkali-activated materials for on-site applications. Construction Building Materials 246, 118470 (2020).
10. Patwa, D., Chandra, A., Ravi, K., Sreedeep, S.: Influence of Biochar Particle Size Fractions on Thermal and Mechanical Properties of Biochar-Amended Soil. Journal of Materials in Civil Engineering, 33(9), 04021236 (2021).
11. Pereira dos Santos, C., Bruschi, G.J., Mattos, J.R.G., Consoli, N.C.: Stabilization of gold mining tailings with alkali-activated carbide lime and sugarcane bagasse ash. Transportation Geotechniques 32, (2022).
12. Ray, S., Daudi, L., Yadav, H., Ransinchung, G. D.: Utilization of Jarosite waste for the development of sustainable concrete by reducing the cement content. Journal of Cleaner Production, 272, 122546 (2020).
13. Seyer, S., Chen, T. T., Dutrizac, J. E. Jarofix: Addressing iron disposal in the zinc industry. Jom 53(12), 32–35 (2001).
14. Sridharan, A., Sivapullaiah, P. V.: Mini compaction test apparatus for fine grained soils. Geotechnical Testing Journal 28(3), 240–246 (2005).
15. Xiaolong, Z., Shiyu, Z., Hui, L., Yingliang, Z.: Disposal of mine tailings via geopolymerization. Journal of Cleaner Production 284, 124756 (2021).
16. Yaghoubi, M., Arulrajah, A., Disfani, M. M., Horpibulsuk, S., Bo, M. W., Darmawan, S.: Effects of industrial by-product based geopolymers on the strength development of a soft soil. Soils and Foundations 58(3), 716–728 (2018).