Geotechnical Benign Characterization of Nano-Amended CLS Stabilized Soil

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Abstract. Nanomaterials are gaining acceptance in the geotechnical and geoenvironmental engineering fields due to their non-toxic nature and less energy required for production. This paper deals with the effect of Iron Oxide nanoparticles (nano-Fe_2O_3) inclusion along with Calcium Lignosulfonate (CLS) as a binder in enhancing the strength characteristics of locally available soil. CLS acts as an initiator and enhances the interaction of nanoparticles with soil. The different proportions of nano-Fe_2O_3 and CLS considered for the tests were 0.1%-0.3% and 1%-3%, respectively. Compaction tests and Unconfined Compression Strength tests were carried out at different proportions of nano-Fe_2O_3 and CLS dosages, blended individually and in combination. The desired values of Maximum Dry Density and Optimum Moisture Content were obtained for a combination of 0.2% nano-Fe_2O_3 and 2% CLS. Unconfined Compressive Strength (UCS) test was conducted for samples cured for 7, 14, and 28 days, which were mixed with different proportions of nano-Fe_2O_3, and CLS showed a tremendous improvement in soil strength. There was a significant improvement in the 28 days strength from 183.4 kPa to a maximum of 496.5 kPa. The mechanism responsible for the improvement in the strength characteristics were confirmed by the SEM images of the stabilized soil samples obtained from UCS tests.

Keywords: Iron oxide Nanoparticle, Calcium Lignosulfonate, Compaction, UCS, SEM

1 Introduction

A rapid increase in infrastructural development had driven engineers to suitably modify the properties of undesirable in-situ soils using chemical additives such as lime and cement [1, 2, 3]. Though the use of such chemical additives has significantly improved the properties of soils, they have also substantially contributed to the increase in carbon footprint emissions released during their production and service life. Therefore, a gradual transition from carbon-based additives to sustainable materials is witnessed in soil stabilization and remediation [4]. The emergence of nanotechnology in science and

technology has significantly helped in achieving this transition [5, 6]. Nanotechnology has been applied across many fields such as medicine, engineering, environment, communication, and heavy industry. These materials have significantly enhanced the engineering properties of materials in civil engineering and construction by reducing the energy consumption of structures, environmental impact, and cost associated with structures [7, 8, 9, 10, 11].
The current study investigates the effect of Nano-Iron Oxide (nano-Fe_2O_3) in the enhancement of compaction characteristics, and compressive strength. The use of Calcium Lignosulfonate (CLS) as an initiator along with nano-Fe_2O_3 has also been explored. Nano-Fe_2O_3 and Calcium Lignosulphonate (CLS) have been used in various proportions (0.1-0.3% and 1-3%, respectively) to stabilize the soil. The changes in the microstructure of the soil samples have also been determined for the various nano-soil mixes.

2 Materials and Methods

2.1 Soil and nano-Fe_2O_3 Properties

Soil investigated and evaluated in the current study was stockpiled from a site in Batpalli cheruvu, located in the Telangana State of India. The soil was treated with nano-Fe_2O_3 in the presence of CLS to determine the improvement in the strength of the soil. The nano-Fe_2O_3 was procured from AD-Nano Tech Research Lab in Karnataka along with a characteristics report and contains about 99% iron oxide. Whereas, CLS was procured from Venki Chem, Mumbai, India. Other elements such as chromium, phosphorous, sodium, manganese, sulfur, and calcium, constituted 1% of the powder. The characteristics of nano-Fe_2O_3 used for the study are discussed in Table 1.

Table 1. Physical properties of nano-Fe_2O_3

S. No.	Properties	Value
1	Average Particle Size (APS)	40 nm
2	Specific Surface Area (SSA)	90 m^2/g
3	Molecular Weight	159.69 g/mol
4	Melting Point	1565°C
5	pH	5-7
6	Bulk Density	0.69 g/cc
7	Morphology	Spherical
8	Colour	Red

Wet sieve analysis was carried out in accordance with IS:2720 Part 4-1985 [12], wherein the coarser fraction was observed to be 40.7%, and the finer fraction passing 75 microns was 59.3%. Hence, the soil was termed fine-grained soil. For further classification of soil, other basic tests were conducted. Atterberg limit test was done in accordance with IS:2720 Part 5-1985 [13], and subsequently, Liquid Limit (LL), Plastic Limit (PL), and Shrinkage Limit (SL) of soil were found to be 55.8%, 20.1%, and 12.7%. The plasticity index (PI) of soil tested was 35.6%, and PI, corresponding to A-Line, was 26.1%. Based on Atterberg limits, gradation characteristics, and Unified Soil

Classification System, the soil was classified as fat clay (CH) [14]. The particle size gradation curve is depicted in Fig. 1.

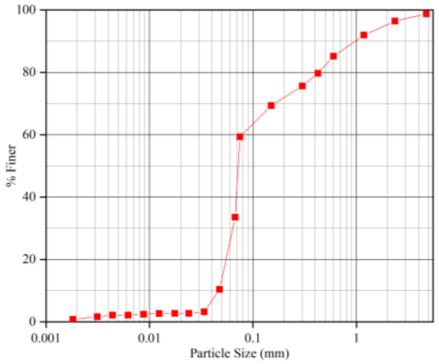

Fig. 1. Particle size distribution curve of the soil used in the study

The samples were prepared at OMC and MDD for various tests. The compaction test was carried out in accordance with IS:2720 Part 7-1980 [15]. After completing the standard proctor test on 16 uncured samples with different proportions of nano-Fe_2O_3 and CLS, OMC and MDD were obtained as 16.5% and 1.835 g/cc, respectively, for a combination of 0.2% nano-Fe_2O_3 and 2% CLS. The other basic properties were also determined in accordance with IS codes. The Free Swell Index (FSI) of the soil [16] was 85.7%, and Specific gravity [17] was 2.685. Table 2 presents the geotechnical properties of the soil.

Table 2. Geotechnical properties of soil

S. No.	Properties	Value
1.	Liquid Limit	55.7%
2.	Plastic Limit	20.1%
3.	Plasticity Index	35.6%
4.	Shrinkage Limit	12.66%
5.	Specific Gravity	2.685
6.	Free Swell Index	85.7%
7.	Optimum Moisture Content	18%
8.	Maximum Dry Density	1.7g/cc
9.	Soil Classification (USCS)	CH

The unconfined compression strength test in saturated clay was determined in accordance with IS:2720 Part 10-1991 [18]. In the current study, samples (76 mm in length and 38 mm in diameter) prepared at optimum conditions and treated with NMs and CLS were tested after a curing period of 7, 14, and 28 days at a loading rate of 1mm/min. The soil fabric for untreated samples and samples treated with different proportions of nano-Fe_2O_3 and CLS was studied using Scanning Electron Microscopy (SEM).

2.2 Sample Preparation

As suggested by Pakbaz and Farzi [19], a dry mixing procedure was adopted for sample preparation. The nano-Fe_2O_3 powder was sprayed over the oven-dried soil to prepare the nano-soil mixture. According to this method, 0.1%, 0.2%, and 0.3% of nano-Fe_2O_3 were taken by dry weight of soil and blended along with 1%-3% of CLS. A homogenous mix was obtained by thoroughly mixing the soil at OMC utilizing a spatula for about 5-10 minutes before placing the soil sample in the mold.

3 Results and Discussions

3.1 Effect of nano- Fe_2O_3 on Atterberg limit

As the proportion of nano-Fe_2O_3 is increased, stabilized soil shows a gain in plastic Limit (PL) and a drop in Liquid Limit (LL), and hence Plasticity Index (PI) of the soil is reduced, as depicted in Fig. 2. The LL and PI of stabilized soil reduced with an increase in nanomaterial concentration, owing to the water adsorbing capacity and non-plastic nature of nano-Fe_2O_3.

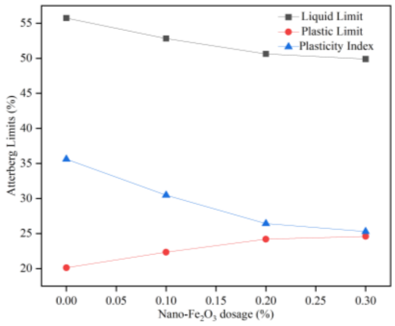

Fig. 2. Change in Atterberg limits with the variation of nano-Fe_2O_3

3.2 Effect of nano-Fe_2O_3 and CLS on Compaction Characteristics

With the addition of nano-Fe_2O_3, a gain in MDD was visible up to 0.3%, and up to 2% of CLS when they were added independently. When the stabilizers were added together, the optimum combination was observed at 0.2% nano-Fe_2O_3 and 2% CLS, where the least OMC and highest MDD were obtained. The increase in dry density can be attributed to the filling of the voids by nanoparticles, and ultimately increasing the unit mass of soil. The decrease in MDD beyond the optimum content is due to flocculation and agglomeration of nanoparticles as a result of cation exchange capacity and low specific gravity of nanoparticles which is generally less than soil [20, 21, 22]. Whereas, Optimum Moisture Content (OMC) decreased due to the ion exchange mechanism during the chemical reaction. In a few cases, there was an increase in OMC suggesting the occurrence of pozzolanic reaction [23]. Variation in OMC and MDD is depicted in Fig. 3-Fig. 5. The mix notations for various combinations of nano-Fe_2O_3 and CLS are presented in Table 3.

Table 3. Mix notations adopted in the study

D	U	0.1%NM	0.2%NM	0.3%NM	1%CLS	2%CLS	3%CLS	0.1%NM+1%CLS	0.1%NM+2%CLS	0.1%NM+3%CLS	0.2%NM+1%CLS	0.2%NM+2%CLS	0.2%NM+3%CLS	0.3%NM+1%CLS	0.3%NM+2%CLS	0.3%NM+3%CLS
M	M0	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	M11	M12	M13	M14	M15

**D-Dosage, M-Mix Notation, U-Untreated soil, NM-nano-Fe_2O_3 and CLS- Calcium Lignosulfonate*

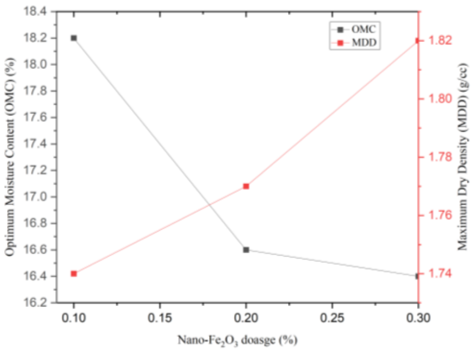

Fig. 3. Variation in OMC and MDD with nano- Fe_2O_3

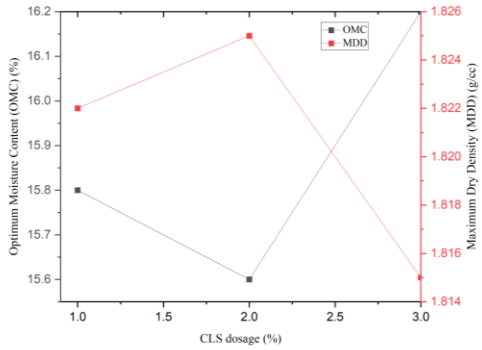

Fig. 4. Variation in OMC and MDD for CLS mixes

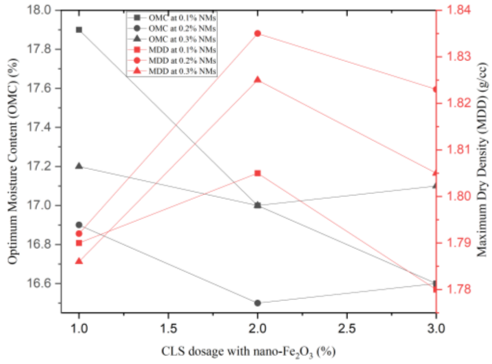

Fig. 5. Variation in OMC and MDD when nano- Fe_2O_3 is blended with CLS

3.3 Effect of nano-Fe_2O_3 on Strength Characteristics

Fig. 6 depicts the variation in UCS upon treatment with nano- Fe_2O_3 and CLS. When nano-Fe_2O_3 and CLS were added independently, the UCS value increased up to 0.2% and 2% respectively, beyond which the compressive strength reduced. For the varying concentrations taken for nano-Fe_2O_3 and CLS, the UCS value increased with an increase in the curing period. Nanoparticles, which are very small in size, have a vast surface area and hence will exhibit high chemical reactivity [24]. When nanomaterial comes in contact with lime, cement, or calcareous compounds, it leads to the genesis of C-S-H gel due to the pozzolanic reaction.

The porosity of soil is reduced as nanomaterial is absorbed onto C-S-H gel, hence aiding in improving the compressive strength [25]. Generally, the compressive strength of montmorillonite soil is constituted by viscous diffused double layer water to the shear formation [26]. As the soil used for the test has low fine contents, it will require a lower polymer chain to bind clay particles; and any additional polymer chain creates reciprocal repulsive force among the charged parts [27], leading to a reduction in UCS.

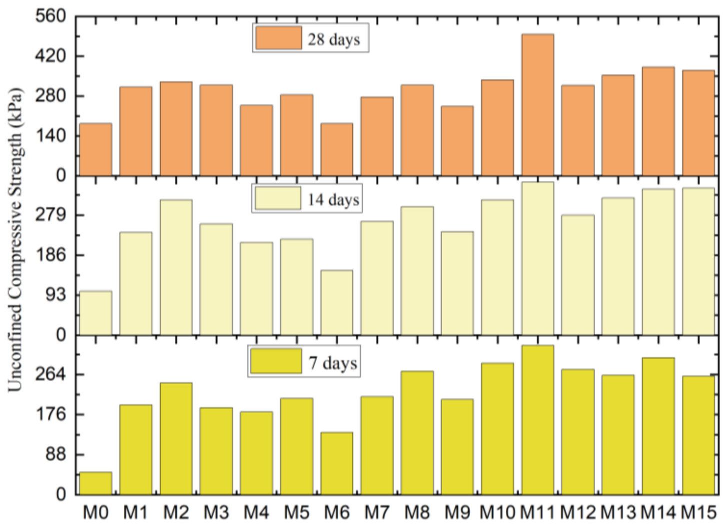

Fig. 6. Variation in Unconfined Compressive Strength upon treatment at 7, 14, and 28 days

3.4 Microstructural analysis

The SEM analysis was conducted on untreated soil, and soils blended with 0.2% NM, 0.2% NM + 2% CLS, and 0.2% NM + 3% CLS to observe the soil fabric. Soil combined with 0.2% nano-Fe_2O_3 contributed to the maximum improvement when blended individually. Similarly, when nano-Fe_2O_3 and CLS were used together in soil, the maximum improvement in soil was observed at 0.2% nano-Fe_2O_3 and 2% CLS. These values were then considered as optimum content. Further addition of CLS beyond 2% led to a negative improvement in strength.

Fig. 7a depicts the SEM image of untreated soil, in which the pores and voids can be clearly observed. The cavities present in the soil are the main reason for the lower strength of the soil. Fig.7b demonstrates the soil blended with 0.2% nano- Fe_2O_3. As the size of nano- Fe_2O_3 is much less than voids of the soil particles, increasing the nanoparticles up to a certain fraction increases the soil strength as it occupies the cavities present in the soil matrix, developing the continuity among particles and acts as filler material.

Fig. 7c depicts an SEM image when 0.2% nano-Fe_2O_3 is blended with 2% CLS. Here CLS addition leads to the formation of a polymeric chain, which occupies the voids, and hence the gain in strength is maximum. The addition of both nano-Fe_2O_3 and CLS creates a more dense structure. With the addition of CLS, there is a reduction in negatively charged clay surfaces with positively charged CLS [28]. Fig. 7d depicts 0.2% nano-Fe_2O_3 blended with 3% CLS and shows a decrement in strength as the polymer chain formed by the nano-soil matrix and CLS particles disintegrates with the addition of CLS or nano-Fe_2O_3 beyond the optimum. Soil particles amalgamate with CLS and nano-Fe_2O_3, but in some portions, excessive stabilizers will accumulate, increasing the soil's porosity. This increase in the porosity will lead to a reduction in strength.

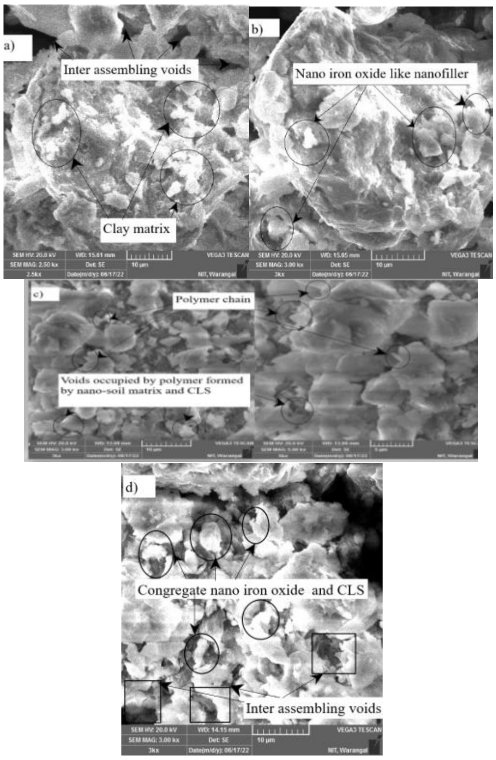

Fig. 7. SEM images of treated and untreated soil
(a) M0 (b) M2 (c) M11 (d) M12

4 Conclusions

In the current study, the combined effect of incorporating nano- Fe_2O_3 and Calcium Lignosulfonate in enhancing the compaction and strength characteristics of a high plastic clay is investigated. The following conclusions are drawn from the study:

- With an increase in nano- Fe_2O_3, LL showed a decrement of 9%, and PI exhibited a decrease of 27%.
- Compaction studies revealed that a notable decrease in optimum moisture content followed by a corresponding increase in maximum dry density was achieved when the clay was blended with 0.2% nano- Fe_2O_3 and 2% CLS.
- Irrespective of the dosage of nano- Fe_2O_3 and CLS, the UCS values increased with an increase in the curing period. The optimum dosage of nano- Fe_2O_3 and CLS was found to be 0.2% and 2% respectively which resulted in a 170% increase in compressive strength at the end of 28 days of the curing period. This increase is attributed to the formation of polymeric chains between nano- Fe_2O_3 and clay particles in the presence of CLS.
- The scanning electron microscopic images revealed a closely packed crystal structure with reduced pore spaces confirming the interaction mechanism responsible for the increase in unconfined compression strength.

This study has corroborated the fact that when nano- Fe_2O_3 is blended with CLS, it can enhance the strength characteristics of high plastic clay. Moreover, the production cost of selected nanomaterials can be reduced with their widespread utilization. Nanomaterials require relatively lower binder dosages compared to other compounds owing to their small size and larger specific surface area.

References

1. Firoozi, A.A., Guney, Olgun, C., Firoozi, A.A., Baghini, M.S.: Fundamentals of soil stabilization. Int. J. Geo-eng. 8, 1-16 (2017). https://doi.org/10.1186/s40703-017-0064-9
2. Moghal, A. A. B., Al-Obaid A. K., Refeai, T. O., and Al-Shamrani, M. A.: Compressibility and Durability Characteristics of Lime Treated Expansive Semiarid Soils. J Test Eval, 43(2), 255-263 (2015). https://doi.org/10.1520/jte20140060
3. Moghal, A. A. B., Ashfaq, M., Al-Obaid, A. A. K., Abbas, M. F., Al-Mahbashi, A. M., and Shaker, A. A.: Compaction delay and its effect on the geotechnical properties of lime-treated semi-arid soils. Road Materials and Pavement Design. 22(11), 2626–2640 (2021). https://doi.org/10.1080/14680629.2020.1784256
4. Moghal, A. A. B., Rasheed, R. M., and Mohammed, S. A. S.: Sorptive and Desorptive Response of Divalent Heavy Metal Ions from EICP treated Plastic Fines. Indian Geotechnical Journal. (2022) https://doi.org/10.1007/s40098-022-00638-8.
5. Harsh, H., Moghal, A. A. B., Rasheed, R. M., and Almajed, A.: State-of-the-Art Review on the Role and Applicability of Select Nano-Compounds in Geotechnical and Geoenvironmental Applications. Arab J Sci Eng. (2022). https://doi.org/10.1007/s13369-022-07036-5
6. Mohammed, S. A. S., Moghal, A. A. B., and Lateef, A.: Strength Characteristics of Nano Calcium Silicate, Fly Ash and Lime Blended Tropical Soils. IFCEE 2018, held at Orlando, Florida, USA ASCE Geotechnical Special Publication No. 296, 105-114 (2018). https://doi.org/10.1061/9780784481592.011
7. Ugwu, O.O., Arop, J.B., Nwoji, C.U., Osadebe, N.N.: Nanotechnology as a preventive engineering solution to highway infrastructure failures. J. Constr. Eng. Manag. 139, 987–993 (2013). https://doi.org/10.1061/(asce)co.1943-7862.0000670
8. Persoff, P., Finsterle, S., Moridis, G.J., Apps, J., Pruess, K., Muller, S. J.: Injectable barriers for waste isolation. Office of Scientific and Technical Information (OSTI) (1995). https://doi.org/10.2172/106544
9. Taha, M.R.: Geotechnical properties of soil-ball milled soil mixtures. In: Nanotechnology in Construction 3. Springer Berlin Heidelberg, Berlin, Heidelberg, pp 377–382 (2009). https://doi.org/10.1007/978-3-642-00980-8_51
10. Mohammed, S. A. S., & Moghal, A. A. B.: Nanomaterials-based solidification/stabilization of metal-contaminated soils. In Nanomaterials for Soil Remediation (pp. 385-407). Elsevier. (2021). https://doi.org/10.1016/B978-0-12-822891-3.00018-9
11. Rasheed R. M., Moghal, A. A. B. (2022).: Critical appraisal of the behavioral geo-mechanisms of peats/organic soils. Arab J. Geosci, 15:1123. https://doi.org/10.1007/s12517-022-10396-9
12. IS: 2720-Part 4.: Method of test for soils: Grain size analysis. Bureau of Indian Standards, New Delhi, India (1985).
13. IS: 2720-Part 5.: Method of test for soils: Determination of Atterberg limits, Bureau of Indian Standards, New Delhi, India (1985).
14. IS: 1498.: Classification and identification of soil for general engineering purpose, Bureau of Indian Standards, New Delhi, India (1970).

15. IS: 2720-Part 7.: Method of test for soils: Determination of water content dry density relation using light compaction. Bureau of Indian Standards, New Delhi, India (1980).
16. IS: 2720-Part 40.: Method of test for soils: Determination of free swell index of soils, Bureau of Indian Standards, New Delhi, India (2002).
17. IS: 2720-Part 3. Method of test for soils: Determination of specific gravity. Bureau of Indian Standards, New Delhi, India (2002).
18. IS: 2720-Part 10.: Method of test for soils: Determination of shear strength parameters by unconfined compressive strength of soils, Bureau of Indian Standards, New Delhi, India (1973).
19. Pakbaz, M.S., Farzi, M.: Comparison of the effect of mixing methods (dry vs. wet) on mechanical and hydraulic properties of treated soil with cement or lime. Applied Clay Science (105– 106):156–169 (2015). https://doi.org/10.1016/j.clay.2014.11.040
20. Iranpour, B., Haddad, A.: The influence of nanomaterials on collapsible soil treatment. Eng. Geol. 205, 40–53 (2016). https://doi.org/10.1016/j.enggeo.2016.02.015
21. Ghobadi, M.H., Abdilor, Y., Babazadeh, R.: Stabilization of clay soils using lime and effect of pH variations on shear strength parameters. Bull Eng Geol Environ 73:611–619 (2014). https://doi.org/10.1007/s10064-013-0563-7
22. Ferkel, H., Hellmig, R.J.: Effect of nanopowder deagglomeration on the densities of nanocrystalline ceramic green bodies and their sintering behavior. Nanostructured Mater. 11:617–622 (1999). https://doi.org/10.1016/S0965-9773(99)00348-7
23. Krishnan, J., Shukla, S.: The behavior of soil stabilized with nanoparticles: an extensive review of the present status and its applications. Arab J Geosci 12:436 (2019). https://doi.org/10.1007/s12517-019-4595-6
24. Rashad, A.M.: A synopsis about the effect of nano-Al_2O_3, nano-Fe_2O_3, nano-Fe_3O_4 and nano-clay on some properties of cementitious material-a short guide for civil engineer. Mater Des 52:143-157 (2013). https://doi.org/10.1016/j.matdes.2013.05.035
25. Gaitero, J.J., Campillo, I., Mondal, P., Shah, S.P.: Small changes can make a great difference, Transport. Res. Rec J Transportation Research Board of the National Academies, Washington, DC. 2141:1-5 (2010). https://doi.org/10.3141%2F2141-01
26. Sridharan, A., Prakash, K.: Mechanism controlling the undrained shear strength behavior of clays. Can Geotech J 36(6):1030-1038 (1999). https://doi.org/10.1139/t99-071
27. Alazigha, D.P., Indrartna, B., Vinod, J.S., Ezeajugh, L.E.: The swelling behavior of Lignosulfonate-treated expansive soil. Proc Inst Civil Eng 169(3):182-193 (2016). https://doi.org/10.1680/jgrim.15.00002
28. Chavali, R.V.P., Reshmarani, B.: Characterization of expansive soil treated with Lignosulfonate. Int J Geo-Engineering 11(17) (2020). https://doi.org/10.1186/s40703-020-00124-1