

Sustainable Soil Stabilization: Strength And Microstructural Analysis Of Calcium Lignosulfonate Treated Black Cotton Soil

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Abstract

The current study considered the calcium lignosulfonate as a stabilizing material to stabilize black cotton soil as it do not release CO₂ upon reacting with soil, unlike traditional stabilizers and can be a sustainable material that helps to achieve Sustainable Development Goals as it is an eco-friendly, non-traditional stabilizer and organic polymer. In addition to that, it is vital to understand the micro-structural properties of treated soil to have a comprehensive knowledge on strength improvement mechanism. To examine the optimum calcium lignosulfonate content, black cotton soil was mixed with different dosages of calcium lignosulfonate ranging from 1 to 5% with an increment of 1% and the optimum content of 4%. The densely packed structure contributed by formation of Calcium Silicate Hydrate in treated soil enhanced the soaked California Bearing Ratio up to 8.13% and Unconfined Compressive Strength up to 203.80 kPa. This enhanced strength characteristics in the black cotton soil appraised by the X-Ray Diffraction, Field Emission Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy analyses. The optimum CBR value and decreased thickness of the pavement made the stabilized BC soil suitable for subgrade material for pavement construction.

Keywords: Black cotton soil, calcium lignosulfonate, industrial waste, calcium silicate hydrate, sustainable stabilization.

INTRODUCTION

Clays with high plasticity and compressibility such as black cotton (BC) soils subjected to excessive volumetric changes during wet and dry conditions [1]. Swelling behaviour is common in BC soil as its mineralogical structure contains montmorillonite that results in low shear strength, bearing capacity and compressive strength. Thus, the stabilization of BC soil is pivotal for majority of engineering works and treating clayey soil with chemical admixtures, including lime, fly ash and cement [2-6], is an available traditional solution for modifying the plasticity, CBR and compressive strength characteristics of the soil [7-11]. Their usage for subgrade soil improvement has usually been limited due to their deficient effects on soil environment such as soil chemistry, ground water [12-14], microstructure and emission of CO₂ [15]. Therefore the application of non-traditional stabilizers, such as organic polymers like lignosulfonates on geo-technical properties of soils have been reported by numerous researchers due to its eco-friendliness and effectiveness [7-10, 11-13]. The CLS is the second most abundant biogenic polymer on the globe [7] and is derived from lignin, which belongs to a family of compounds produced as a sub product during the paper manufacturing process [8] and is approximately 1.8 million tons in quantity annually produced [9]. Various investigations executed on cohesive soils to explore the ability of CLS treatment to increase the strength parameters of expansive soils [8-10]. Few investigations have established that CLS can become a solution by reducing the total amount of water adsorbed from treated soil, thus decreasing the swelling potential [8-10, 12 & 13]. The ability of CLS to modify the shrink and swell capacity, California Bearing Ratio (CBR), Unconfined Compressive Strength (UCS), and compaction characteristics of remoulded expansive soil was evaluated [10, 12-15]. The electrostatic interaction between CLS, water and soil particles increases the stiffness and compressive strength without leading to considerable brittle behaviour contributed by clay mineralogy [11 & 12]. An understanding of the microstructural properties of CLS treated soil is critical in assessing the cause of improvement in soil strength characteristics. CLS is a good

sustainable material compared with traditional stabilizers, as it is an eco-friendly, nontoxic, noncorrosive chemical and does not release CO₂ upon reacting with soil [15]. The present study considered West Godavari District, Andhra Pradesh as a study region where majority of road networks built on BC soil subgrade facing severe pavement failures like fatigue cracks, unevenness and deterioration of service life [17 & 18] due to excessive swell-shrink characteristics, low bearing capacity and compressive strength [16 & 19]. Thus, there is an emerging need to stabilize the subgrade BC soil to improve strength and stiffness as it requires minimal structural support from pavement layers in order to reduce the pavement thickness without losing its performance. Therefore, the current study aims to utilize a non-traditional CLS to stabilize the subgrade BC soil and to undertake multiple laboratory investigations in assessing the microstructural modifications to improve strength parameters feasible to reduce pavement thickness and to explore the optimum CLS content for stabilization. Also focused on providing insight into the index properties and strength characteristics using Atterberg's limits, Free Swell Index (FSI), Cation Exchange Capacity (CEC) measurements, compaction characteristics - Optimum Moisture Content (OMC) & Maximum Dry Density (MDD), soaked CBR and UCS tests [7, 9,10,12 & 13]. All these tests conducted on the BC soil by treating with the optimum percentage of CLS at varying curing periods of 0, 7, 14 and 28 days to find time dependent strength variation. The microstructural properties responsible for enhanced soil strength characteristics observed through X- Ray Diffraction (XRD) for mineralogical analysis, Field Emission Scanning Electron Microscopy (FESEM) for examination of micro structural properties and Energy Dispersive X-ray Spectroscopy (EDS) for analysing elemental composition. The novelty of this study is to explore the potential benefits of CLS in enhancing the time dependent engineering behaviour of stabilized subgrade BC soil and its feasibility to reduce the pavement thickness.

MATERIALS AND METHODS

2.1 Black cotton soil

The current study procured the BC soil sample shown in Figure 1(A) from West Godavari region of Andhra Pradesh, India at a depth 1.0 m below the ground surface under disturbed conditions. After executing the laboratory testing to find soil properties and classify the BC soil sample (Table. 1) according to IS: 1498-1970 the Indian standard classification (ISC) and it is classified as inorganic clay with high plasticity (CH). The soil's elemental chemical composition is shown in Table 2. The soil sample categorized as expansive in nature based on its Free Swell Index (FSI).

Table 1: Properties of the BC soil samples

S. No	Property of Soil Sample	Numerical Value
1.	Soil pH	7.33
2.	Free Swell Index (%)	57
3.	Specific Gravity	2.62
4.	Consistency Indices-%	
	w _L -Liquid Limit (%)	67
	w _P -Plastic Limit (%)	25.82
	I _P -Plasticity Index (%)	41.18
	w _s -Shrinkage Limit (%)	9.21
5.	Variance in Particle Size	
	Sand -%	8
	Silt -%	24
	Clay -%	68
6.	Soil Classification	CH

Table 2: Chemical compositions of the elements in BC soil sample

Element	Symbol	Atomic No.	Weight %
Oxygen	O	8	55.40
Silica	Si	14	14.42
Carbon	C	6	12.86
Alumina	Al	13	9.53

Ferrous	Fe	26	4.97
Calcium	Ca	20	0.66
Titanium	Ti	22	0.36
Magnesium	Mg	12	1.20
Potassium	K	19	0.60

2.2 Calcium Lignosulfonate

Lignin is the natural bonding material in wood that binds the wood fibres together in plants (Figure 1(B)). It is isolated from pulping liquor via a sulfite process known as lignosulfonate, as shown in Figure 2, and it is available as calcium lignosulfonate produced by using a sulfite salt in the sulfite pulping process [13]. It recognized as a potentially effective stabilizing agent for both cohesive and non-cohesive soils (14, 15 & 20-22). It consists of hydrophobic and hydrophilic groups [24]. Compared to conventional chemical stabilizers, an environmentally friendly, nontoxic, and noncorrosive biopolymer does not alter pH upon soil treatment [19] and does not release CO₂ upon reacting with soil. Various characteristics of CLS [25] mentioned below in Table 3.



Figure 1: (A) Black cotton soil (B) Calcium lignosulfonate

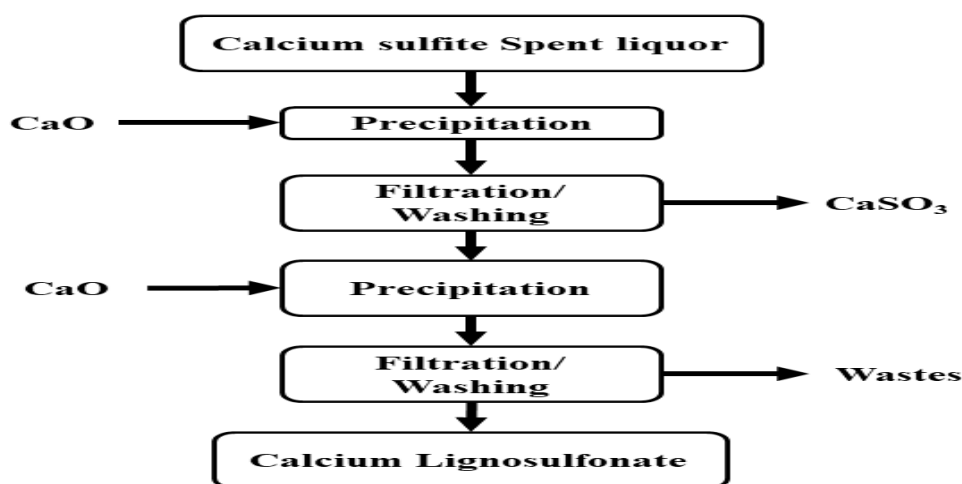


Figure 2: Schematic separation of calcium lignosulfonates from calcium sulphite wasted liquor.

Table 3: Characteristics of Calcium Lignosulfonate

Identification	Detail
Solubility	Soluble in Water, Insoluble in organic solvents
Weight- Avg. Molecular weight	40,000- 65,000
pH	2.7-3.3
Degree of sulfonation	0.3-0.7
Calcium	≤ 5.0%
Sulfite	≤ 0.5%
Total Ash	≤ 14.0%
Arsenic	≤ 1 mg/kg
Lead	≤ 2 mg/kg

2.3 Sample Preparation and Testing Methods

The collected BC soil sample was pulverized, weighed to 150 g of 425 μ size sieved and examined for Atterberg's limits to determine the plasticity of BC soil at different CLS percentage by measuring water content at various transition states as per the IS: 2720-5 (1985) to classify the soil according to ISC. The FSI conducted for assessing the effect of CLS on swelling potential of the BC soil according to IS 2720-40 (1977). The pulverized soil of 4.75 mm sieved and 3 kg weighed, lignosulfonate of 425 μ size sieved was used for determining the OMC and MDD of untreated and CLS-treated BC soils via the IS lightweight compaction test according to the IS code 2720-7 (1980). The optimum CLS percentage for preparing the samples for further test procedures was determined as 1%, 2%, 3%, 4%, and 5% CLS to treat the BC soil by dry weight. The pulverized soil sample of 5 kg which is sieved through 4.75 mm size is used for conducting CBR according to the IS code 2720-16 (1987) and UCS to observe the compressive strength according to IS 2720-10 (1991) of BC soil with different CLS combinations. The BC soil and CLS in dry powder form of various combinations added and mixed uniformly to prepare samples for all laboratory tests. The UCS samples were stored in a desiccator for testing at distinct curing phases from 0, 7, 14 to 28 days. The CLS-treated and 28 days cured soil samples were oven dried and pulverized into fine powders of 75 μ size and tested for CEC, FESEM, EDS and XRD, and their microstructural properties were tested. The CEC determined by method of methylene blue titration (MBT). While this method produces an acceptable CEC value, the conventional ammonium displacement approach misjudges it [34]. The MBT method carried out on 2 g of treated BC soil powder obtained after the test on the 28-day-cured UCS samples by oven drying at 60 °C for 48 hours. The XRD test was conducted with the wavelength - Cu-K α ($\lambda=1.54056$ Å), input voltage - 40 kV, a current - 40 mA, and a continuous scan mode $2\theta = 3^\circ$ to 70° with a step size of 0.02° 2θ at a speed of 10/minute in the laboratory to investigate the mineralogical potential factors. A Quantax 200 EDS used for quantitative microanalysis of untreated and treated soil to estimate the chemical blends and to observe the changes in the compositions of treated soil samples. A set of micrographs was captured with FESEM analysis via Carl Zeiss microscopy equipment, which permitted us to analyse the microstructure of untreated BC soil at 10 μ m and 4% CLS-treated clayey soil at 2 μ m magnification. The assumed hypotheses and testimony obtained for the present study presented in the flow chart Figure 3.

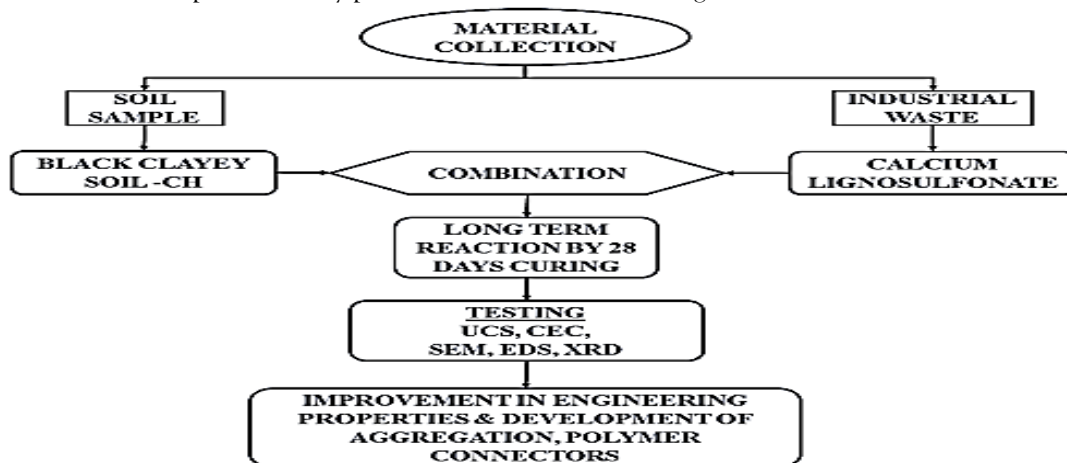


Figure 3: The assumed hypotheses and testimony obtained for the present study.

RESULTS AND DISCUSSION:

3.1 Free Swell Index & Atterberg's Limits

The influence of CLS content on the FSI of BC soil is exemplified Figure 4. The stabilization of BC soil with CLS is greatly reduced the swelling potential and made the treated soil suitable to utilize in pavements by its potency of swelling below 50% as per IRC: SP: 89-2 (2018). The FSI of untreated BC soil noted as 57% in presence of water, it gradually reduced when treated with CLS up to 5%. The observed reduction of FSI is remarkable up to 3% followed by marginal reduction for further increase in CLS content. The liquid limit decreased and plastic limit slightly increased in the CLS treated expansive soil Figure 5. As a result, the plasticity index also slightly decreased. The trend can help to understand the phenomena of enhancement in the soil plasticity characteristics. The improved plasticity characteristics ascribed to the reduction in thickness of the DDL [27] since the adsorption of CLS on the exterior surface of the clay minerals in the BC soil. The lower plasticity index properties can correlate to

decreased swelling and shrinkage potential and denote that the soil is less likely to undergo deformation under changing moisture content.

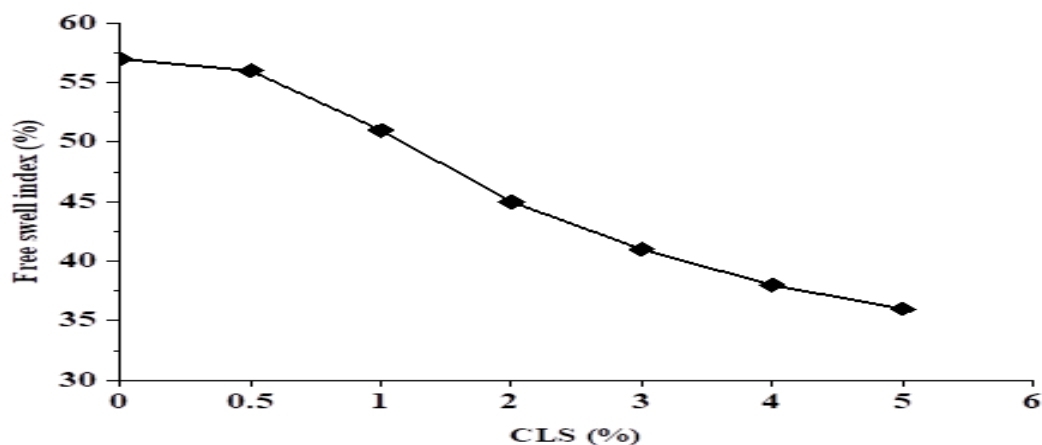


Figure 4: Effect of CLS on FSI

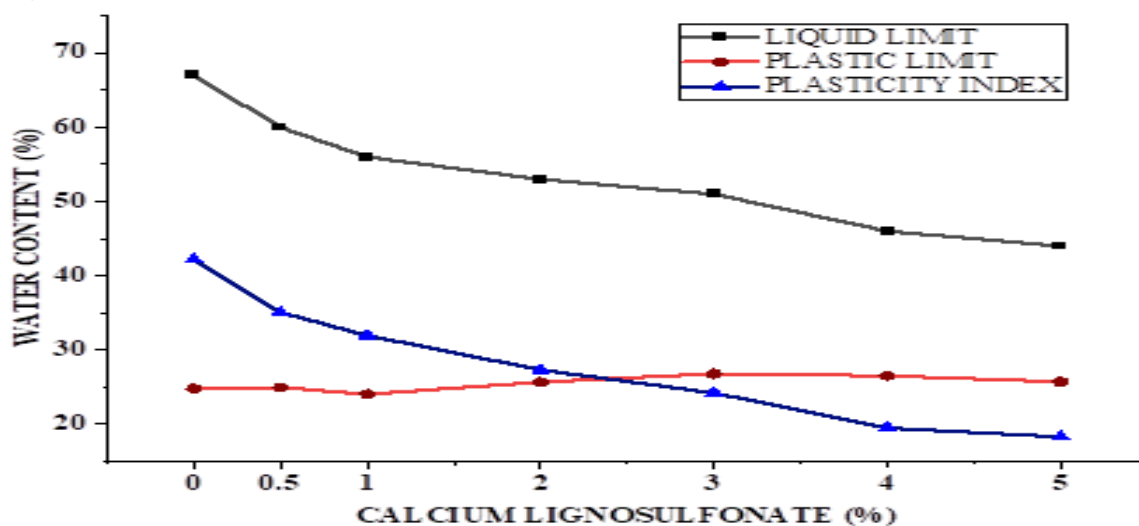


Figure 5: Effect of CLS on Plasticity characteristics

3.2 Standard proctor's test

When CLS added along with water to the soil and compacted, it acts as a lubricant to form flocculated soil structure due to its hydrophilic property that holds water in it. Which also enhance the particle aggregation, increases the cohesion and binding properties of treated BC soil resulting decrease in OMC and increase in MDD exemplified in Figure 6. After observing the results, the optimum CLS content to achieve the optimum results is determined as 4% to get optimal results of OMC as 23.15% and MDD as 19.57 kN/m³ to prepare soil samples for further tests. As adding the CLS content beyond the 4% optimum to BC soil causes holding excess moisture content in the soil and diminishing the results, which leads to increase in OMC (23.59%) and decrease in MDD (18.86 kN/m³).

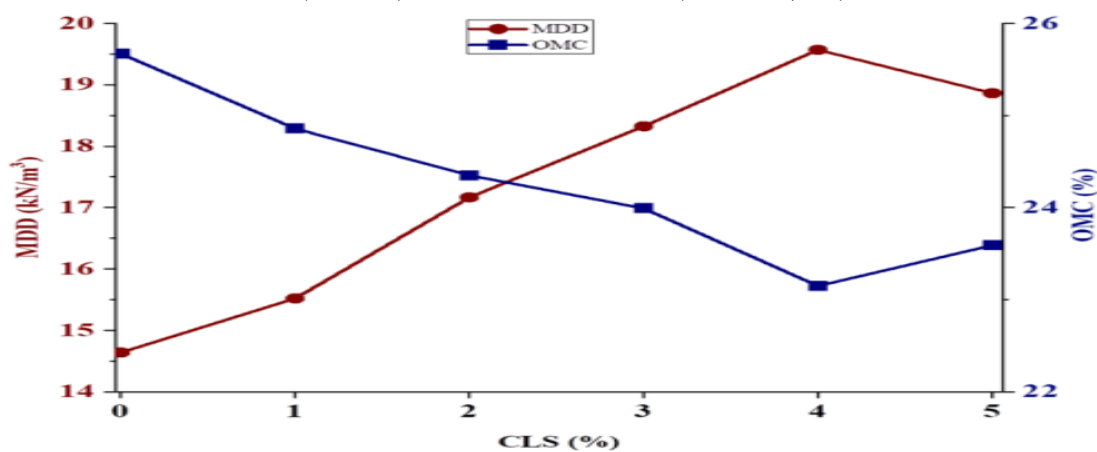


Figure 6: Compaction characteristics of treated BC soil

3.3 California bearing ratio test

As CLS content rose up to 4%, The Soaked CBR values progressively increased from 1.71% to 8.13% later decreased with further increment beyond the optimum CLS content (Figure 7).

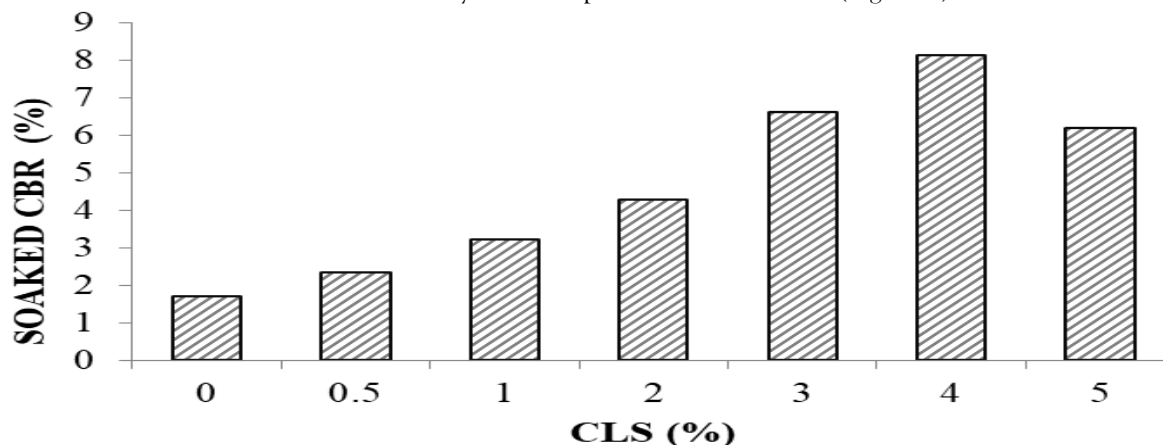


Figure 7: Effect of CLS on Soaked CBR

The enhanced results attributed to the increased cohesion and particle aggregation due to water holding and binding nature of CLS, particle-interconnecting property improved in the BC soil and decreased void space within the soil matrix leading to higher dry density reduces the susceptibility to stress-induced deformation. Beyond the optimum, it acts as degrading element by holding excess moisture content and losing particle-binding nature, which in turn spoils the entire soil structure and leads to decrease in CBR.

3.4 Cation exchange capacity (CEC) test

The capacity of soil to absorb and exchange cations from CLS confirmed by this test on a BC soil [28], which is exemplified in Figure 8. The results revealed that the CLS-treated BC soil had a lower CEC than the untreated BC soil. The values of the CEC decreased gradually with increasing CLS content from 1% to 5%. However, the CEC decreased considerably up to the optimum content of CLS in treatment, thereafter negligible as the mix may reach the saturation limit.

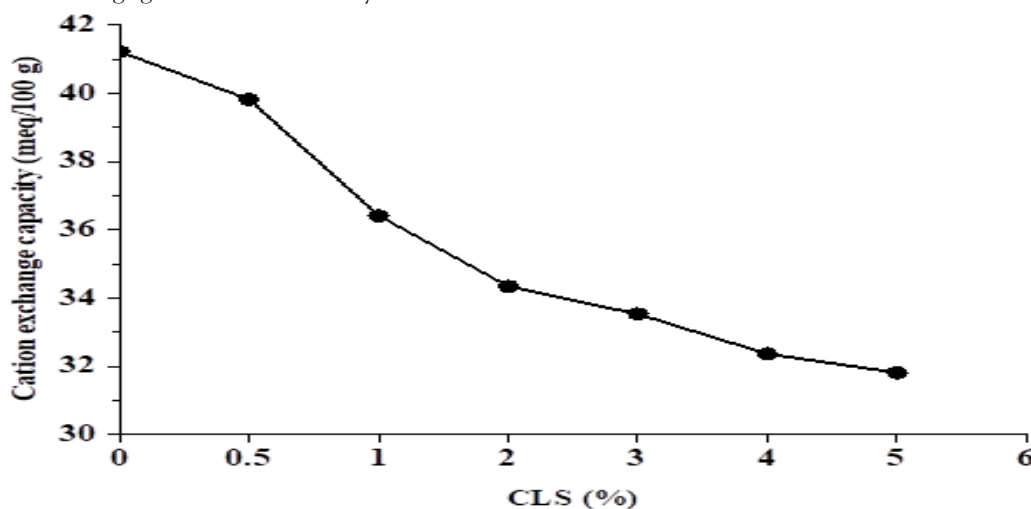


Figure 8: Effect of CLS on CEC of BC soils.

The additional CLS content may excessively cover the surface of the clay particles and fill the pores, which are resulting in reduction of reactive surface area of the soil particle. The negatively charged BC soil particles surface adsorbs positively charged CLS particles in presence of water in the curing period, therefore the reduction in the CEC and thickness of the DDL [8] occurs. The observed XRD phenomenon of reduction in the reflection ability of the clay mineral crystals can attribute to these results.

3.5 Energy-dispersive X-ray spectrometer (EDS)

The CLS facilitates the release of calcium ions upon interaction with soil. It observed in the EDS results that the calcium (Ca) percentage increased from 0.66% to 2.17% Table 4. The Increased calcium availability is vital as it contributes to various pozzolanic reactions [29], which increase soil strength and stability. The percentage of silica (Si) increased from 14.42% to 17.82%, which is a 3.40% improvement in the overall weight due to combination of factors, including the characteristics of lignosulfonate and its interaction with native soil minerals. Hence, the increase in the calcium (Ca) and silica (Si) contents in

the CLS-treated BC soil resulted in increased pozzolanic reactions in the clay particles, which attributed to the amorphous calcium silicate hydrates (C-S-H) formation [30] in the BC soil treated with CLS.

Table 4: Chemical composition of untreated and CLS-treated BC soils

Element	Weight %	
	Untreated	Treated by CLS
O	55.40	53.48
Si	14.42	17.82
C	12.86	8.09
Al	9.53	9.62
Fe	4.97	6.47
Mg	1.20	1.16
Ca	0.66	2.17
K	0.60	0.64
Ti	0.36	0.55

Which is a cementitious element enhances the cohesion among soil particles, creates more robust and dense matrix. As identified in XRD analysis, that the C-S-H effected the crystallization of treated soil elements and decreased with increase in its content [31]. It ultimately results in increased UCS and CBR in the treated BC soil.

3.6 X-ray diffraction (XRD) analysis

The XRD revealed that the black clayey soil comprises Montmorillonite, Illite, Kaolinite and Quartz as clay minerals. The peak strength of the clay mineral Montmorillonite (i.e., the M peak) observed at a 2θ angle of 35.080° in the untreated clayey soil (Figure 10). The peak strength of clay mineral montmorillonite (1057 counts/sec) in CLS-treated BC soil is lower than untreated (1094 counts/sec) BC soil. Figure 10 indicates reduction in the reflection ability of clay mineral crystals [15] as CLS adsorbed by the exterior surfaces of the clay particles of BC soil, which can attributed to the formation of C-S-H. It can also confirmed by the increased elemental percentage weight of Ca and Si observed in EDS. This phenomenon of decreased phase purity attributed to the reduction in the negative surface charge of the clay minerals by enhancing the clay and CLS bonding and affected the phase purity by increasing the Ca/Si molar ratios of solid phase [31].

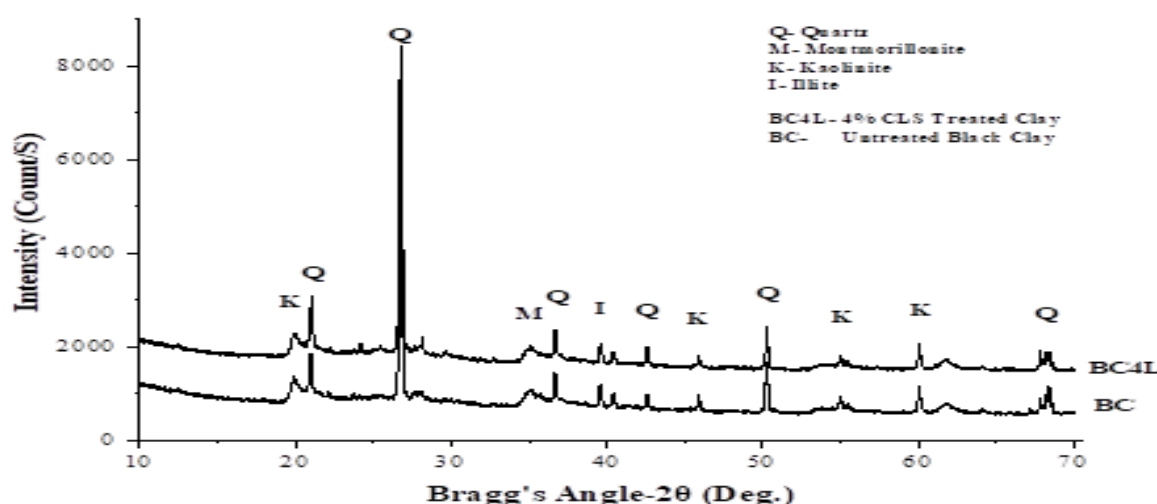


Figure 9: Comparison of the XRD patterns of BC soil and 4% CLS treated BC soil

The M peak value of the soil shifted from 35.08° to 35.13° with CLS treatment Figure 9. The XRD graphs of untreated BC soil and CLS treated BC soil peaks compared to give more visual clarity. There are no new peak intensities are observed in the CLS-treated soil sample via XRD, as C-S-H is an amorphous element and has a poorly crystalline structure [32]. The reduction in the M peak strength attributed to a decrease in DDL by contributing to reduced swelling potential in the treated soil.

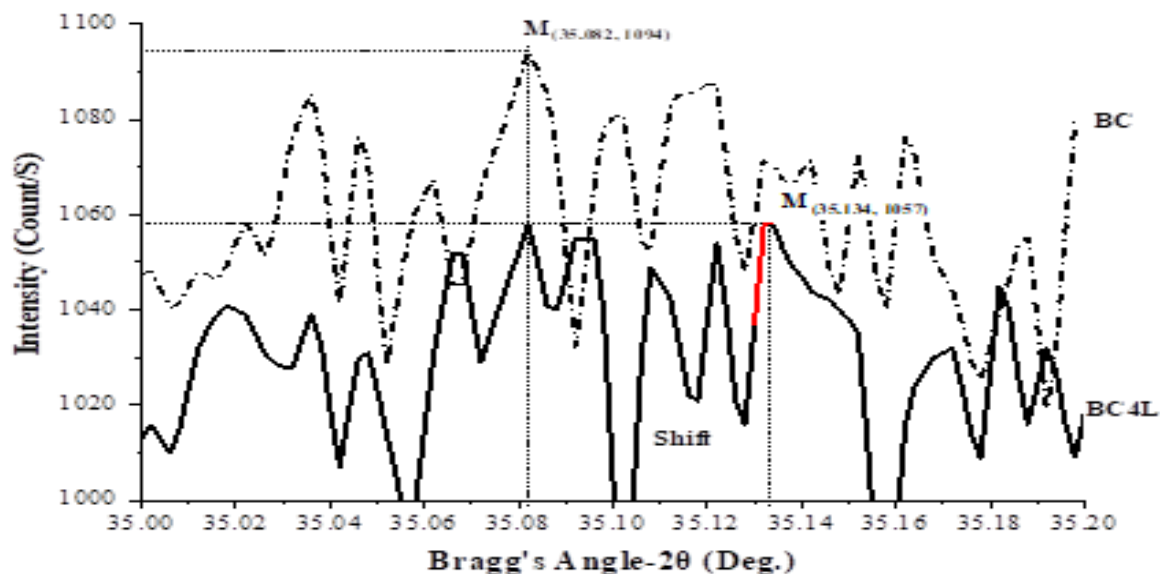


Figure 10: Reduction and shifting of the M- peak strength of untreated and treated BC soil

3.7 Field emission scanning electron microscopy (FESEM) analysis

The FESEM micrographs of black clayey soil revealed that the untreated soil has a rough surface structure with large cracks may be formed due to drying and shrinkage (Figure 11) which make it less effective for engineering purposes with grater void ratio. The presence of surface voids can link to the air spaces between soil particles, which affects water retention, and aeration.

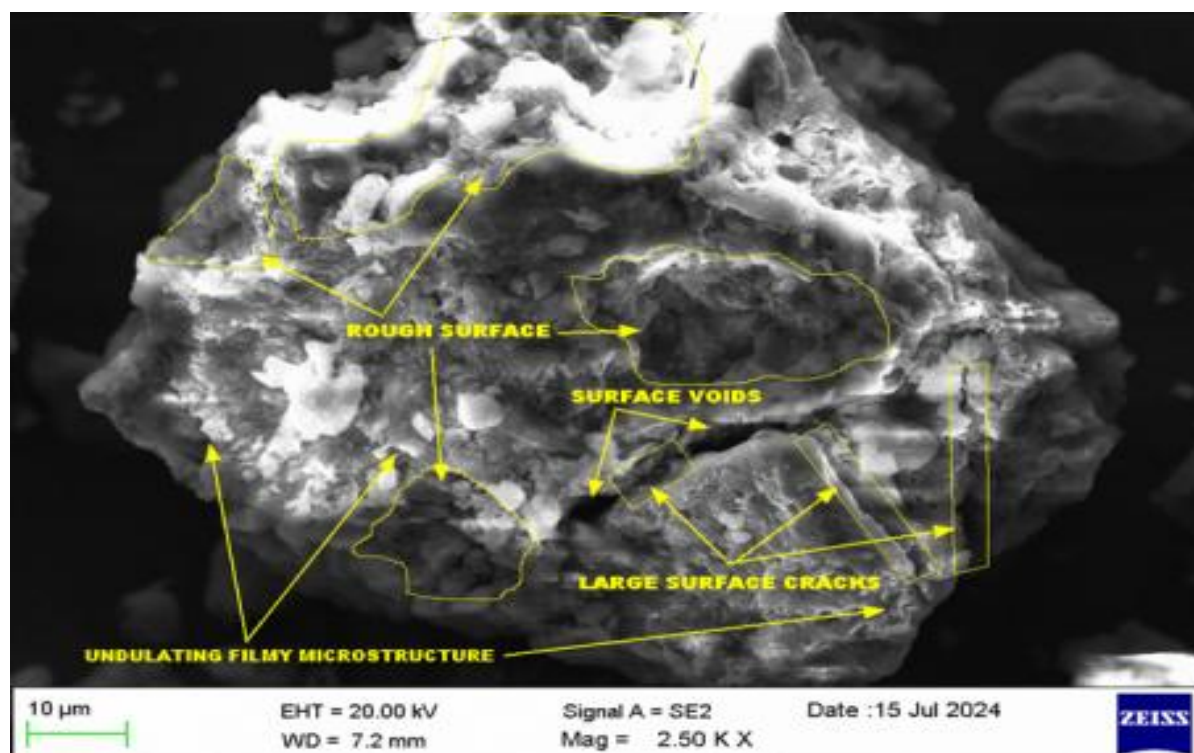


Figure 11: FESEM micrograph of BC soil

Also the montmorillonite clay mineral present in the soil is the cause for these adverse properties in BC soil, which recognized through the existence of microstructures of the undulating filmy particle [33] reflects the layered nature of clay particles, contributing to the soil's ability to hold moisture. As observed in FESEM, the ionic interaction and physical binding with BC soil by CLS lead to the formation of polymeric connectors between individual soil particles, creating stable aggregation and reducing the number of voids as they fill in gaps between soil particles (Figure 12), which increase the UCS, CBR and overall stability of the soil structure.

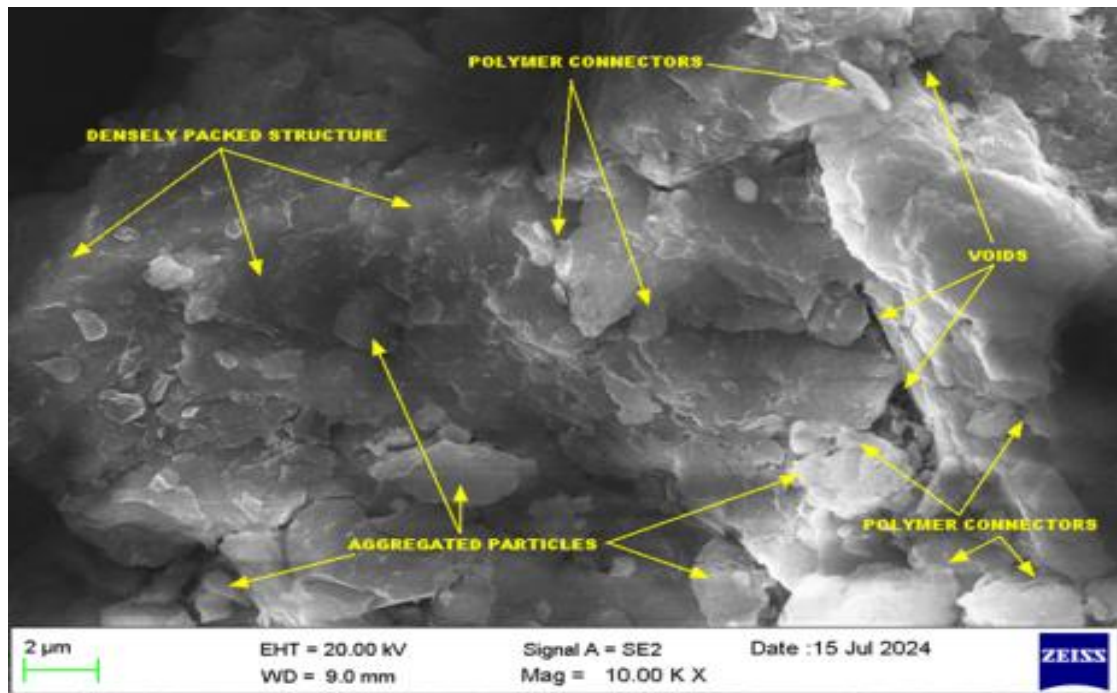


Figure 12: FESEM micrograph of CLS treated BC soil

3.8 Unconfined compressive strength (q_u) test

The UCS of BC soil in untreated condition is 69.09 kPa which escalated to 203.80 kPa with the treatment of 4% CLS with BC soil with curing period up to 28 days, further increment in CLS content reduced the UCS value to 186.52 kPa (Figure 13). This tendency related to the effective cation exchange process due to the adsorption of positively charged CLS particles to the negatively charged surface of BC soil, which usually takes a longer period. It can also attributed to the results observed in FESEM micrographs, which support the formation of a densely packed soil structure implies superior strength under unconfined loading conditions and the aggregation of soil particles enhance the effective load transfer between soil grains which increase the overall structural integrity of the soil matrix. As observed in EDS the increased calcium and silica content may supported to form cementitious compound C-S-H, which contributed to the increase in the compressive strength of the treated BC soil by increasing cementitious bonding between clay particles with CLS.

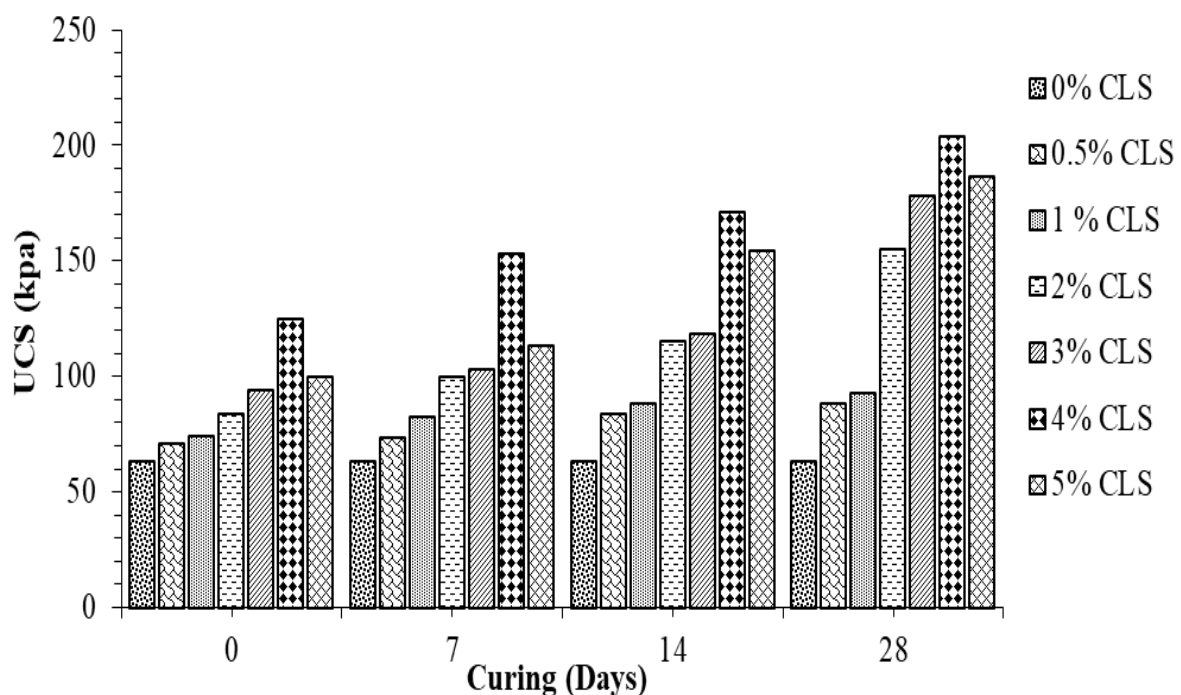


Figure 13: UCS of CLS-treated BC soil samples

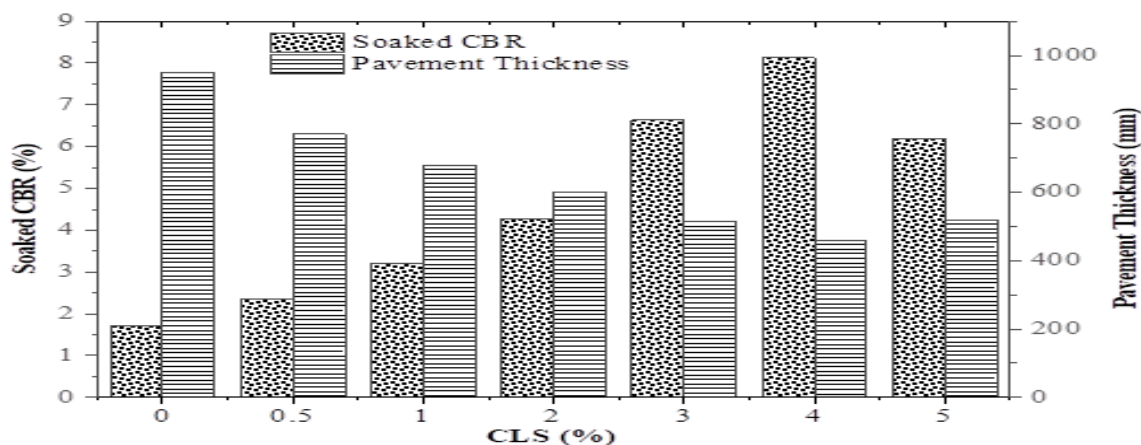


Figure 14: Effect of Soaked CBR on Pavement Thickness

3.9 Effect of soaked CBR on pavement thickness

When BC soils situated as subgrades of the roads, they may cause multiple problems that can affect the pavement service life. The soaked CBR is used as the parameter to design the pavement thickness, which is recommended by IRC- SP- 72, 2007 [16] with a value between 7%-9% is suggested for the selection of good subgrade soil. The increase in CBR value can affect the pavement thickness which helps to reduce the cost of construction and increase the scope for effective utilization of industrial wastes in soil stabilization to improve the properties of poor subgrade BC soil. The soaked CBR value 8.13% of optimum CLS treated BC soil satisfies the criteria as per IRC recommendation. The optimum CBR value and thickness of the pavement achieved by stabilized soil (Figure14) making it suitable for subgrade soil for pavement construction and can bear the loads from vehicles passing on a flexible pavement for a two-lane bypass as considered for designing the thickness with a design traffic of 4.74 msa.

3.10 Proposed stabilization mechanism of CLS-treated BC soil

When the optimum percentage of CLS added to the BC soil, which increased percentage weight of silica (Si) and calcium (Ca) possibly lead to C-S-H formation in the treated BC soil and increase the compressive strength by increasing the bonding between clay particles. The CLS particles serve as glue in soil stabilization by reacting with BC soil particles and can act as dispersing agents; thus, it can destroy the largest soil voids and it also have a dispersing action by supplying required moisture on the soil fines to decreases the OMC, which in turn attain the MDD [32]. CLS intrusion likely decreased the negative surface area of the clay minerals through reaction between the CLS and the clay minerals, decreasing the DDL thickness and increasing the bonding between clayey particles. This process results in formation of stable aggregation and the polymer connectors that bind soil particles together and densely pack the soil structure in BC soil, resulting in increased CBR and resistance to deformation [8, 35].

CONCLUSIONS

This study evaluated the performance of CLS on BC soil strength characteristics. The EDS witnessed formation of higher Ca/Si ratio and C-S-H with increased clay particle binding. The reduced reflection ability of observed XRD graphs strengthening the formation of C-S-H. The phenomenon of the M peak value shifting of the untreated soil from 35.080 to 35.130 with CLS treatment can attribute to a decrease in thickness of DDL. The FESEM micrographs supporting the formation of aggregation and densely packed soil structure ascribed to increase in UCS, CBR and overall stability of the soil structure and decreased thickness of the pavement. Therefore, the eco-friendly CLS stabilized BC soil could utilized as subgrade material, promoting effective utilization of industrial waste for sustainable soil stabilization.

Funding: No funding received for the current study.

Conflict of interest: Authors states no conflict of interest.

Declaration of Generative AI and AI-Assisted Technologies: The authors declare that the Generative AI or AI-Assisted Technologies not used during the preparation of this work.

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