

This is a peer-reviewed prepublished version of the paper to be published in **Vol. 26, No. 1, March 2027**.

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26 May 2026

CITE THIS ARTICLE

Kumar, P.S. and Vindula, S.K., 2027. Sustainable Stabilization of NaOH-Contaminated Expansive Soil Using Calcium Lignosulfonate: Mechanical and Microstructural Insights. *Nature Environment and Pollution Technology*, 26(1), B4456.

<https://doi.org/10.46488/NEPT.2027.v26i01.B4456>

EARLY-ACCESS STATEMENT

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PUBLICATION STATUS

PEER REVIEW

Completed

ONLINE VERSION

Prepublished

FINAL ISSUE

March 2027

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Type of the Paper (Original Research)

Sustainable Stabilization of NaOH-Contaminated Expansive Soil Using Calcium Lignosulfonate: Mechanical and Microstructural Insights

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Abstract

The structural stability and long-term performance of any civil engineering infrastructure are contingent on the geotechnical properties of foundation soil. The chemical intrusions into soil alter its properties by tending to its behavioral changes. Industrial alkali spills into the ground are one of the emerging problems which necessitate a broad study to evaluate the impact of alkali induced behavioral changes in a soil. Black cotton soil (BCS) which exhibits weak characteristics within water saturation and deteriorates more with chemical interactions. The present work is focused on the mechanism of the BCS when exposed with alkali solution and amendments in the alkali included BCS treated with Calcium Lignosulfonate (CLS) of 0.5%, 1% and 2%. The alkali solution used in the study was 2N NaOH. A comparative analysis is done between the untreated BCS, BCS added with 2N NaOH solution to identical to that of 2N NaOH-added BCS treated with CLS. The prepared soil specimens were cured for a period of 1, 3, 7, 14, 28, 56 and 90 days and were tested for evaluating the plasticity characteristics and unconfined compressive strength (UCS). Wetting and drying cycles were performed on BCS with same combinations of 2N NaOH and CLS under 7, 28, 56 and 90 days curing to understand the durability performance. XRD and FESEM analysis were performed to analyze the mineralogical and morphological evidences. The BCS added with 2N NaOH demonstrated a progressive tendency in plasticity and reduced durability under wetting and drying cycles. There is a noticeable reduction in the UCS with 2N NaOH. Adding 1% of CLS to 2N NaOH included BCS has improved the plasticity, UCS and durability by enduring more wetting and drying cycles under long-term curing. Microstructure studies revealed clear transformation of montmorillonite-rich BCS into a stabilized geopolymer - matrix by long-term alkali activation under CLS stabilization.

Keywords

Black cotton soil, NaOH, Calcium Lignosulphonate, Unconfined Compressive Strength, XRD and FESEM.

1. INTRODUCTION

The chemical and biological changes in a soil occurred due to soil contamination can lead to the changes in geotechnical properties of the soil and also impacts the structures founded on the same soil (Sivapullaiah and Hari Prasad Reddy, 2009). In India, the expansive soil, usually known to as black cotton soil, covers an area of roughly 800,000 km², extending across the states of Madhya Pradesh, Andhra Pradesh, Maharashtra, Gujarat, Rajasthan, Telangana, and Jharkhand (Kumar et al., 2007; Uppal and Chadda, 1967). Due to seasonal variations in moisture, the volume instability causes stresses in the soil mass that can damage superstructures, especially those that are lightly loaded. Instances of these stresses encompass structural deformation, pavement and flooring upheaval, slab fracture on

grade members, damage to channel and reservoir linings, and distortion of irrigation systems, railways, canals, and subsurface water supply networks (Chen, 1988; Ito, 2013). The primary factor contributing to significant swell and shrinkage is the presence of minerals such as montmorillonite (Katti, 1979). Soil contamination can be caused by both natural and human-made processes (Gratchev and Towhata, 2013). Numerous factors, such as excessive chemical use and accidental spills, can contaminate soil (Mirsal, n.d.). Caustic alkali contaminant generated by numerous industries is a substantial pollutant that can significantly impact soil behavior (Sivapullaiah et al., 2008). Soil alkalinity contamination is a critical concern in numerous regions globally. Globally, over two billion tons of alkaline byproducts are produced. The dust and leachates from alkaline waste pose significant health and environmental risks, both immediate and enduring (Gomes et al., 2016). A multitude of industries, encompassing dye production, ethylene facilities, textiles, food and beverage, paper and pulp, aluminum, ceramics, and refineries, might discharge sodium hydroxide (NaOH)-containing effluents during their operations, which could contaminate soil (Imran et al., 2016; Mechanics and Engineering, 1998; Sivapullaiah and Manju, 2007). Leaks that occur during shipment, storage, or operation can cause high concentrations of alkali pollution in soils, whereas effluent discharge can cause low quantities (Mechanics and Engineering, 1998). The primary application of alkali solutions is as a chemical basis for the manufacture of textiles, potable water, pulp and paper, detergents and soaps, and the refinement of bauxite ore containing alumina. These industries utilize alkali solutions with concentrations of up to 20 N. The solution can range from 1 to 4N as it dilutes and penetrates into the moist earth (Vindula et al., 2018). High-strength alkali is a serious contaminant that alters soil behaviour. In aluminum extraction factories, alkali from the alumina extraction process can leak into foundation soils, causing footings to heave and floor and roof beams to bend. Alkalization causes clay minerals to disintegrate, releasing aluminum ions and generating depolymerized silicic acids. The interaction of clay minerals, acids, and alkali produces irreversible reactions, with the process rate proportional to surface area. In semiarid environments, cyclic swell-shrink movements caused by wet and dry seasons can cause serious structural damage (Lew, 2010). Diverse degrees of contamination result from the spatial dispersion and dilution of concentrated alkali that is accidentally discharged into moist soils. Soils undergo different degrees of transformation depending on the alkali concentration and exposure time (Sruthi and Reddy, 2020). On this basis, various scholars undertook laboratory tests to investigate the impact of alkali on clay minerals (Alshaer, 2013; Bauer, 1999; Bauer and Berger, 1998; Boussen et al., 2015; Cuadros and Linares, 1996; Elert et al., 2015, 2008; Jiang et al., 2008; Taubald et al., 2000; Wang and Siu, 2006). Due to a lack of knowledge about the physio-chemical and chemical interactions that alter fine-grained soils, there are more instances of unexpected soil behaviour. Both swell and non-swell soils may experience unanticipated heave due to high alkali contamination. Water adsorption, which cannot be controlled by reducing the soil's accessible moisture content, is not the cause of this swell brought on by changes in the minerals (Sivapullaiah, 2015). Each soil type can directly or indirectly influence its behavior in both the short and long term, leading to the partial or complete transmission of contaminants into the subsurface. The Scientific-Research Institute of Foundations and Underground Structures has examined the physical and chemical interactions between clay minerals and acids and alkalis (Sokolovieh, 1995). Alkali, such as NaOH solutions, has a notable impact on the red earth's swelling and plasticity behaviour. Red earth when combined with NaOH solution, the soil displayed a notable change in swelling behaviour, resulting in an unanticipated swell with alkali interaction, and the red earth attains more strength with the reach of alkali solutions (Kumar et al., 2024). The soil structure can be changed by a relatively small concentration (0.1 N) of caustic soda solution, but larger alkali concentrations modify the mineralogy of clay and create new mineral forms (Chavali et al., 2017; Mechanics and Engineering, 2008; Mitchell, 2005; Sivapullaiah and Manju, 2007). Clays exposed to alkali exhibit physicochemical changes, such as particle dispersion and mineralogy changes (Sruthi et al., 2020). When clay minerals interact in a highly alkaline environment, precipitation and dissolution occur (Bauer, 1999; Bauer and Berger, 1998; Cuadros and Linares, 1996; Elert et al., 2015; Taubald et al., 2000). Alkali inundation has diminished the unconfined compressive strength of black cotton soil and kaolinite clay. The alkali concentration was demonstrated to greatly influence the lowering of UCS. The results indicated that after 28 days of curing, the UCS values diminished by 46% and 43% for BCS, and by 44% and 36% for kaolinite clay with 2 N and 4 N NaOH contaminations, respectively (Ashfaq et al., 2019a). Thus the alkali contaminated expansive soils throws a lot of scope for soil stabilization approaches. Soil stabilization mainly entails the unification of its particles to obtain improved strength and durability, resulting in transformed fabric and texture (Rahmat and Ismail, 2015). The selection of soil stabilizer is crucial. As an example utilizing industrial by-products such as Waste foundry sand offers a useful recycling technique while simultaneously improving the geotechnical characteristics of soils and reducing waste in landfills (Kumar et al., 2025). Lignosulfonate is a leftover byproduct of the paper and wood processing industries which falls under the family of organic polymers based on lignin. Lignosulfonates are produced annually in the globe in amounts of about 1.8 million tons (Aro and Fatehi, 2017). With a molecular weight ranging from 800 to 100,000 and a molar mass between 4600 and 398,000 g, Lignosulfonate is lignin-based (Lemes and Mei, 2003). Bio-based goods make use of lignin generated from biomass. The chemical and physical processes used to produce energy, as well as most of its applications in the paper industry change the natural characteristics of lignin. The sulfite pulping technique

is used in the paper manufacturing sector to extract lignin from fibre or wood. The resulting co-product, lignosulphonate, has molecular weights between 10,000 and 40,000 g/mol and is highly soluble in water. The sulfite pulping method is employed in the paper industry to remove lignin from fiber or wood. The resultant co-product, lignosulphonate, possesses molecular weights ranging from 10,000 to 40,000 g/mol and exhibits great solubility in water. (Meister, 2002). Lignosulfonate is insoluble in organic solvents but is soluble in water at all pH levels (Ding et al., 2018). Since the 1950s and 1960s, civil engineering infrastructure has constantly employed lignosulphonate for diverse uses, like soil stabilization and a dust palliative for roads that are unpaved (Nicholls et al., 1960; Sinha et al., 1957) and also as a set retarding and water-rendering additive of concrete (Mindess et al., 2003). Despite being widely used in many applications due to their non-toxic, non-corrosive, and environmentally friendly properties, lignosulfonates still have a lot of room for widespread use in geotechnical applications (Niaounakis, 2015). A substantial improvement in the engineering behavior of soil treated with lignosulfonate is mostly dependent on the clay mineralogy (Indraratna, 2012). Adding lignosulfonate to expansive soils leads to reduction in swelling (Alazigha et al., 2019) and reduction in swelling pressure (Reza Noorzad and Bahram Ta'negonbadi, 2018). In clayey soils treated with lignosulfonate an increase in unconfined compressive strength and stiffness by being less resilient to moisture with increase in the strength (Indraratna, 2012; Maskell et al., 2012; Ta'negonbadi and Noorzad, 2017). The addition of lignosulfonate lowers soil plasticity, reducing the plasticity index from 36% to 28% at a 1.5% dosage. At this dose, unconfined compressive strength increases by 2.3 times. Curing has little effect on the treatment, while the strength improvement factor maintains between 1.5 and 2 with time. XRD research reveals that the size of montmorillonite crystallites has decreased, demonstrating micro structural stability. Lignosulfonate neutralizes negative clay charges, causing crystallite aggregation and enhanced strength without pozzolanic effect that is dependent on time (B et al., 2021a). (A J Gow, 1961) (A J Gow, 1961) (A J Gow, 1961) Dispersion and cementation were determined to be the main effects of lignosulphonate on soil aggregate mixtures. The water soluble nature and hygroscopicity of the polymer, which slows evaporation, were associated with cementation effects. (Noorzad and Ta'negonbadi, 2021) studied the engineering features of 0.75 percent Lignosulfonate-amended soil and found that the plasticity index has dropped from 29% to 14%. The flaky and uneven structure evident in SEM pictures indicates that this decline resulted from the aggregation of soil particles due to the introduction of lignosulfonate, absent in untreated soil. By adding 2% calcium lignosulfonate, the expansive soil could withstand repeated wetting and drying cycles (Alazigha, 2015). The X-ray diffraction analysis indicated a reduction in the crystallite sizes of the clay minerals in lignosulphonate-amended soil as surface charges diminished. (Zhang et al., 2016). Though many studies were attempted to study the impact of alkali contamination on soil but the work is more focused on kaolinitic soils. All the previous studies were concerned with micro level studies and swell characteristics of kaolinitic soils and very minimal work is attempted on expansive soils. There is a lot of scope for working on the macro and micro level studies of expansive soils exposed to alkali contamination. This paper aims to assess the characterization and remediation of black cotton soil exposed to alkali contamination under different curing periods. Calcium lignosulphonate is chosen as a stabilizing agent to treat alkali contaminated black cotton soil. The prepared soil specimens are subjected to macro level studies in laboratory to assess the plasticity, strength and durability characteristics.

2. MATERIALS AND METHODS

2.1 Black cotton soil (BCS)

The black cotton soil used in this study was procured from Nature and Greens, Gujarat, a commercial geotechnical material supplier. Prior to testing, the soil was processed by air-drying and pulverized to ensure uniformity. The soil was characterized through index properties, compaction, strength, and mineralogical tests for confirming it as highly expansive black cotton soil. With reference to the Casagrande Plasticity chart based on the liquid limit, plasticity index values of soil, we confirmed the soil as CH (Highly Plastic Clay) and even the high amount of percentage of fines, the Colour and the differential free swell index value of the soil confirmed it to consider as black cotton soil.

TABLE 1: BCS Physical Properties

Soil	Soil Group	Specific Gravity	LL (%)	PI (%)	DFSI (%)	Gravel (%)	Sand (%)	Clay (%)	Silt (%)	OMC (%)	MDD (kg/m ³)	UCS (kPa)
BCS	CH	2.63	75	30	75	0.9	39.35	22.80	36.95	17	1450	181.42

TABLE 2: BCS Chemical Composition

Soil	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	MgO (%)	CaO (%)	Na ₂ O (%)	K ₂ O (%)	SO ₃ (%)
BCS	56.90	21	7.78	7.14	1.90	1.82	0.65	2.82

2.2 Calcium Lignosulfonate (CLS)

The CLS used as a soil stabilizer for the study was sourced from an online research materials dealer from Gujarat. The CLS was processed in the laboratory to confirm lump free texture and made air dried to blend with soil specimens. CLS was added at dosages of 0.5%, 1.0%, and 2.0% by dry weight of black cotton soil. Table 3 below present the composition of CLS.

TABLE 3: CLS Composition

Additive	Colour	Density (g/cc)	pH	Moisture at 105 °C	Water Insoluble (%)	Ca (%)	Mg (%)	Fe (%)	S (%)
CLS	Brown	0.42	4.0	6.2	0.16	1.9	1.85	0.05	6.1

2.3. Preparation of 2N NaOH Solution

A 2N NaOH solution was made by dissolving 80 g of sodium hydroxide (NaOH) pellets in distilled water while continuously stirring. NaOH has an equivalent weight of 40 g/equivalent, hence 80 g is required to create 1 L of 2N solution. The pellets were initially dissolved in around 700-800 mL of distilled water, and because the dissolution process is extremely exothermic, the solution was allowed to cool to room temperature. Finally, the volume was increased to 1 L using distilled water, and the solution was properly mixed before use.

2.4. Atterberg Limits (Liquid Limit Test and Plastic Limit Test)

According to the IS Code (IS: 2720 (part 5), 1985), air-dried soil samples were screened through a 425 μ sieve to determine the liquid and plastic limits. The relevant soil samples for testing were mixed into homogenous mixes with alkali solution (2N NaOH) and CLS. The soil specimens were then wrapped in thin polythene sheets and allowed to cure for the specified time period. Following the curing period, the test procedures were applied to the prepared soil specimens.

2.5. Differential Free Swell Index Test

The Differential Free Swell Index (DFSIs) test was conducted confirming with IS standards (Bureau of Indian Standards, 1977). Two 10 g samples of oven-dried BCS, passing through a 425-micron IS filter, were deposited in individual 100 ml graduated cylinders. One cylinder was filled with kerosene (a non-reactive medium) and the other with distilled water (a swelling media), both to a capacity of 100 ml. Following the agitation to evenly disperse the soil, the cylinders were allowed to remain undisturbed for 24 hours. The final measurements were documented, and the DFSI was calculated as the percentage increase in soil volume when immersed in water compared to kerosene. The procedure was reiterated using a 2N concentration of NaOH solution and BCS with varying quantities of Calcium Lignosulphonate (CLS).

2.6. Unconfined Compressive Strength Test

An unconfined compressive strength (UCS) test was conducted on cylindrical BCS specimens to investigate the influence of alkali solution and CLS on the undrained shear strength of BCS. CLS was added at specified dosages (as detailed in section 2.2) based on the dry weight of black cotton soil, while the prepared 2N NaOH solution was supplied in an amount equivalent to the optimal OMC and maintained consistently throughout all specimens to achieve uniform moisture conditions during specimen preparation. Cylindrical specimens (diameter 3.8 cm $\times$ height 7.6 cm) were fabricated utilizing the dynamic compaction method to achieve maximum dry density at the appropriate moisture level, as established via the compaction test. The chemically enclosed cylindrical specimens were compacted to verify the optimum moisture content and maximum dry density values obtained from the laboratory compaction test of untreated BCS. The cylindrical specimens were enveloped in thin polythene sheets and sealed to facilitate curing. Subsequent to the curing phase, each cylindrical specimen was evaluated using an unconfined compression testing apparatus in accordance with the prescribed IS Code (IS: 2720 (Part 10):1991, 1991) procedure by applying a loading rate of 1.25 mm/min until specimen failure occurs.

2.7. Wetting and Drying Cycles

The wetting and drying cycles were performed for investigating the durability of BCS contaminated and treated with alkali solutions and CLS. The procedure for soil specimen preparation and curing was adopted same as that of UCS test. Immediate to completion of curing period, wetting and drying cycles were carried out on each soil specimen where the specimens were subjected to 5 hours wetting and 42 hours of drying which makes a cycle. The repetition of cycles till the failure was conducted confirming to the standard ASTM procedure (ASTM D-559, 1996). However, slight adjustments were made to match the characteristics of expanded clay specimens. The brushing intensity and specimen handling were modified to prevent false surface damage while enabling the evaluation of durability by mass loss and visual integrity.

The percentage weight loss was calculated using:

$$\text{Weight Loss (\%)} = \frac{W_i - W_n}{W_i} \times 100$$

where W_i is the initial dry mass and W_n is the dry mass after the n^{th} wetting–drying cycle.

For all laboratory testing, three replicate specimens were prepared and evaluated, and the mean value was reported. The same approach is applied to all results from the experimental investigations conducted in this work.

2.8 Microstructure Studies

2.8.1. XRD Analysis

X-ray diffraction (XRD) was used to study the material's crystalline structure and phase composition. The specimens were pulverised to a particle size of less than 0.05 mm and put evenly on the sample holder. The measurements were taken using a Bruker D8 Advance diffractometer with a Cu-target X-ray source in coupled $2\theta/\theta$ scan mode. Phase identification was accomplished by comparing the resulting diffraction patterns to standard reference cards.

2.8.2. FESEM Analysis

Field emission scanning electron microscopy (FESEM) examination was used to analyse the surface morphology and microstructural features of soil specimens. MURTI-SAIF used a TESCAN MI-RA S6123 FESEM to conduct the analysis. The micrographs collected were utilised to investigate the particle arrangement, surface texture, and morphological changes caused by the treatment procedure.

3. RESULTS AND DISCUSSIONS

3.1. Atterberg Limits

3.1.1. Effect of 2N NaOH and CLS on Liquid Limit and Plasticity Index of BCS

The liquid limit of untreated BCS with distilled water was measured at 75%. Figure 2 shows that adding 2N NaOH to BCS gradually reduced the liquid limit (LL), which went from 74.95% on Day 1 to 68% after 90 days of curing, suggesting a considerable loss of soil flexibility. This decrease was further improved with the inclusion of CLS. Out of all the treatments, 1% CLS showed the most reduction in LL, at 42% after 90 days, whereas 0.5% and 1% CLS decreased LL to 49% and 46%, respectively. These findings suggest that by lowering water affinity and plasticity properties, CLS treatment of alkali-affected BCS can successfully enhance long-term soil stabilization.

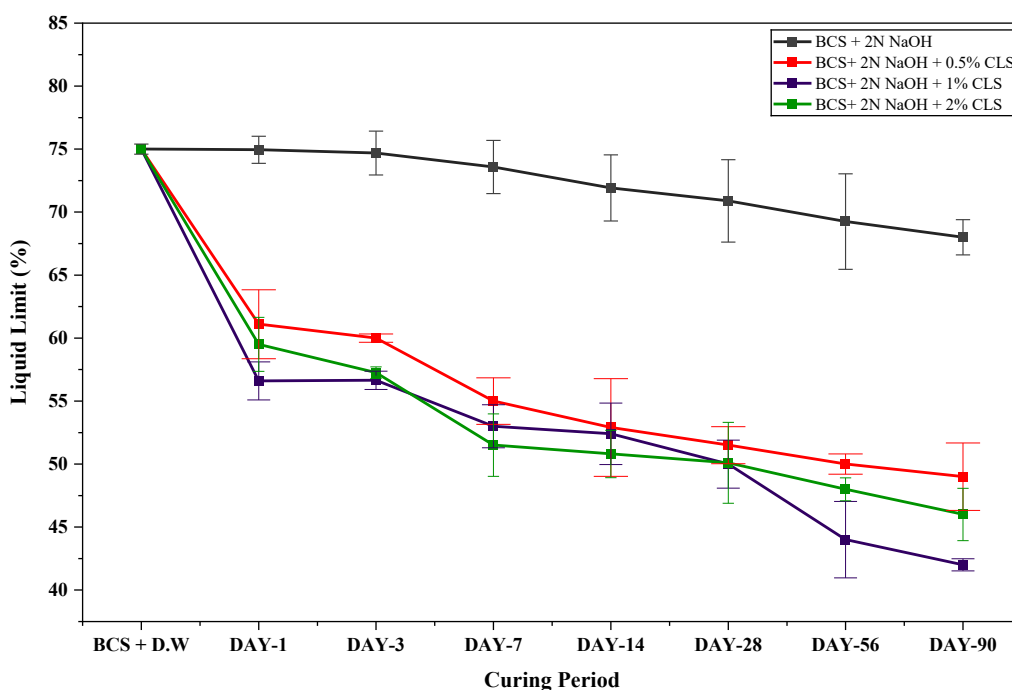


Figure 1. Variation in the liquid limit of BCS treated with 2N NaOH and different dosages of CLS over a 90-day curing period.

According to Figure 3, the plasticity index (PI) of BCS added with 2N NaOH alone first dropped to 20.45% from 30% on Day 1 but significantly increased to 36.7% by Day 90. This suggests that the early stabilization effect was transient and that prolonged alkali interaction may have caused swelling or structural reorganisation in the soil. Although clay particles temporarily flocculate when exposed to NaOH, an excessive sodium concentration eventually weakens the aggregated structure. Over time, clay platelets deflocculate and disperse into finer particles as the edge-to-face linkages between the particles decrease.

On the other hand, the plasticity index (PI) decreased steadily when CLS was added to alkali BCS. Superior long-term plasticity control was demonstrated by the 1% CLS treatment, which reduced PI from 9.4% on Day 1 to 7.5% after 90 days of curing. Similarly post 90 days curing, both the 0.5% and 2% CLS treatments dropped the PI values to 16.5% and 10.0%, respectively, maintaining lower PI levels than NaOH alone. Overall, CLS effectively successfully mitigated the negative long-term plasticity increase brought on by NaOH, with 1% CLS offering the best stabilization performance

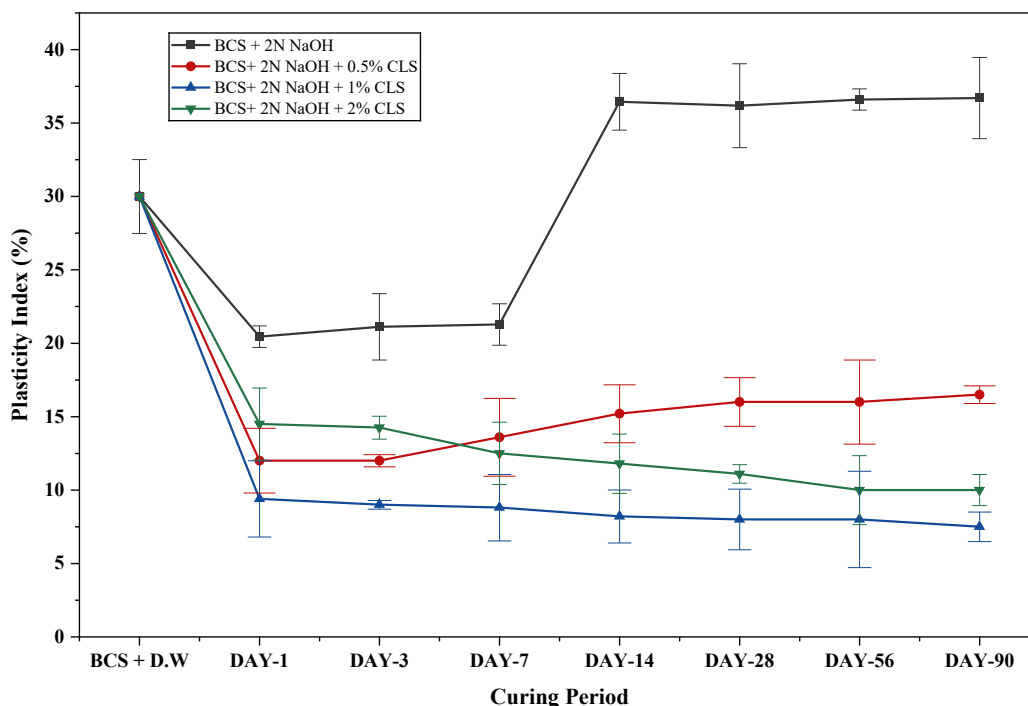


Figure 2. Variation in plasticity index of BCS treated with 2N NaOH and different dosages of CLS over a 90-day curing period.

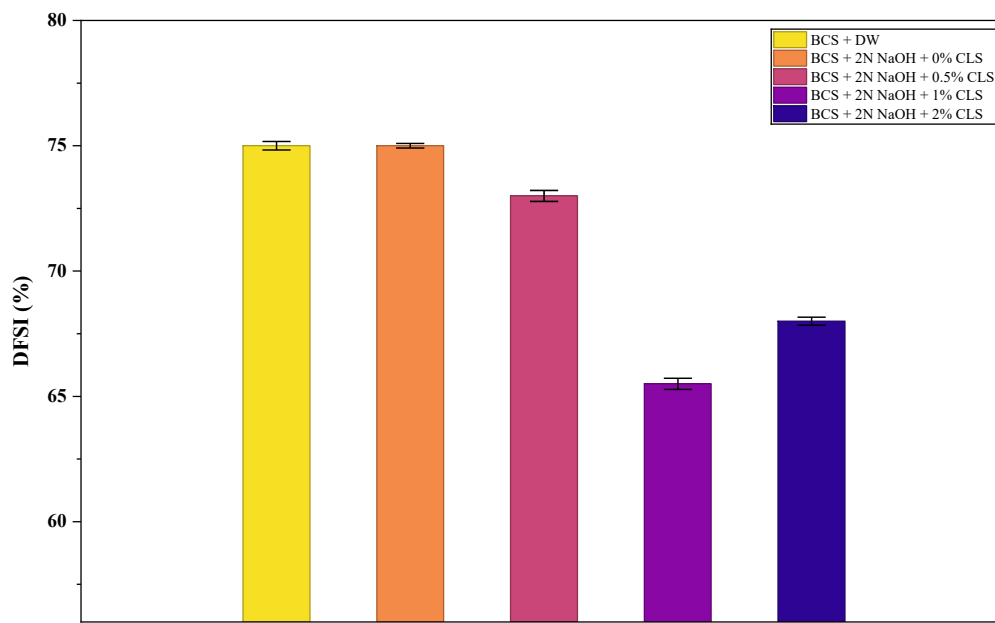
The mechanism for the decremental plasticity characteristics of CLS-treated alkali BCS can be attributed to coupled physicochemical and polymeric stabilization mechanisms. The addition of NaOH causes the clay mineral surfaces to interact with Na^+ and OH^- ions, elevating the soil's pH and enhancing the negative surface charge of the clay particles. This increases interparticle repulsion and expands the diffuse double layer (Brady, 1995). According to Cherian et al. (2018), the extremely alkaline environment further promotes the dissolving of silica and alumina from clay mineral edges. Concurrently, Na^+ ions replace naturally deposited divalent cations in the interlayer gaps of montmorillonite following alkaline treatment, and because of their high hydration potential, they draw enormous amounts of water molecules. This leads in diffuse double layer expansion, increased interlayer spacing, sodium-induced swelling, and clay particle dispersion, all of which increase the soil's long-term plasticity of BCS (Sridha Ran et al., n.d.; Zhang et al., 2014). Addition of CLS mitigates clay particle dispersion through cation exchange and double layer compression. Ca^{2+} ions replace exchangeable Na^+ ions, causing flocculation and aggregation (Chavali and Reshmarani, 2020; WANG Xiu-juan YANG Heng-hui FAN et al., 2017) and additionally, CLS molecules adsorb onto clay surfaces, forming hydrophobic polymeric coatings that reduce water adsorption and swelling. The polymer chains also promote interparticle bridging, resulting in a denser and more rigid soil fabric, which reduces both the liquid limit and the plasticity index.

3.2. Differential Free Swell Index Test

3.2.1. Effect of 2N NaOH and CLS on DFSI of BCS

From Figure 3, it can be observed that the untreated BCS mixed with distilled water (BCS + DW) had a high DFSI value of 75%, indicating its expansive nature. When 2N NaOH was introduced without CLS, the DFSI stayed constant at 75%, demonstrating that NaOH had little effect on minimizing swell. However, adding CLS to NaOH resulted in a steady fall in DFSI: 73% with 0.5% CLS, 68% with 1% CLS and substantially lower at 65.5% with 2% CLS. This

shows that CLS amplifies the action of NaOH by lowering the soil's swell potential, most likely due to enhanced soil structure and decreased clay activity.



BCS with DW, 2N NaOH, & CLS

Figure 3. Variation in the differential free swell index of BCS treated with 2N NaOH and different dosages of CLS.

3.3 Unconfined Compressive Strength

3.3.1 Effect of 2N NaOH and CLS on UCS of BCS

The untreated BCS exhibited a UCS value of 181.42 kPa, which demonstrates the intrinsic weakness and highly expansive nature of clayey soil. It was clear from the figure 4 that UCS values continuously decreased as the curing time increased and showed a declining trend when BCS was added with 2 N NaOH (Ashfaq et al., 2019b). A gradual reduction in UCS value from 177.50 kPa on Day 1 to 117.68 kPa on Day 90 was witnessed after inclusion of 2N NaOH alone to BCS, indicating that extended exposure to sodium ions facilitated clay dispersion and sodium induced expansive nature, which weakened the soil fabric over time. The dissolution of clay minerals in an alkaline environment can be attributed to the decrease in UCS of BCS. (Hari et al., 2011; Sivapullaiah and Manju, 2005). It is observed that, UCS significantly improved at all curing times when CLS was added to alkali BCS. With addition of 0.5%, 1%, and 2% CLS, the UCS rose to 317.97, 451.73, and 408.54 kPa on Day 1, indicating the calcium lignosulfonate's instant stabilizing impact. Because of incremental flocculation, interparticle bonding, and the formation of cementitious reaction products, the strength rose further with longer curing times. On Day 90, the UCS for 0.5% CLS was 431.49 kPa, for 1% CLS it was 738.91 kPa, and for 2% CLS it was 616.84 kPa. Incorporation of 1% CLS to 2N NaOH -BCS showed the greatest strength gain of all the mixes at Day-90, it was more than six times the UCS of 2N NaOH added BCS and over four times the UCS of untreated BCS. The potential stabilization process following the incorporation of CLS into alkali BCS may be ascribed to the production of polymer chains that infiltrate the clay particles, neutralizing the electric charges on the clay's surface and aggregating the particles. The repulsive interactions between clay particles may tend to be mitigated when electric charges on the clay surface are neutralized. The decline in microporosity may be connected to this effect.

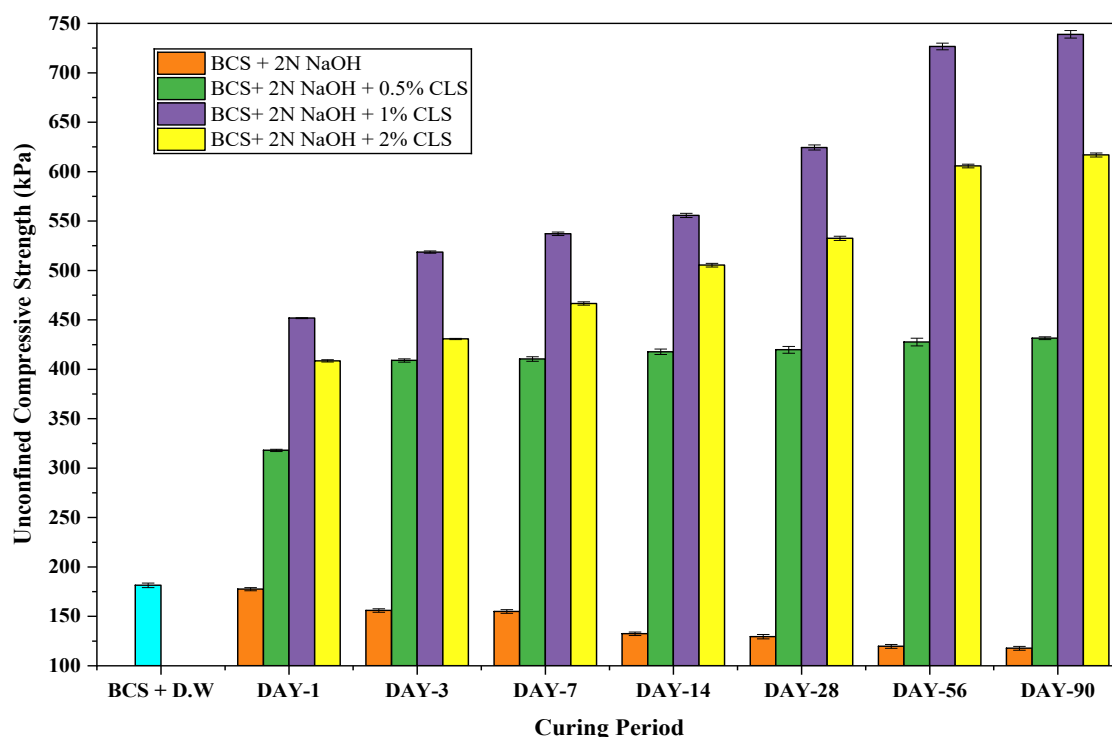


Figure 4. Variation in the unconfined compressive strength of BCS treated with 2N NaOH and different dosages of CLS over a 90-day curing period.

The exceptional efficacy of the 1% CLS dosage illustrates the ideal equilibrium of cation exchange, diffuse double layer compression, and polymeric interparticle bridging. Although 2% CLS significantly increased strength, the decreased UCS compared to 1% CLS shows that too much lignosulfonate may have caused partial coating or oversaturation effects, restricting efficient particle contact and bond generation. Overall, the findings demonstrate that CLS successfully mitigates the negative long-term impacts of NaOH treatment and greatly improves mechanical strength of BCS after curing.

3.4. Wetting and Drying Cycles

3.4.1. Effect of 2N NaOH and CLS on durability of BCS under wetting and drying cycles after 7, 28, 56 and 90 days curing

The findings of the wetting and drying durability tests show that both untreated and 2N NaOH-treated BCS have low cyclic stability. From figures 5,6,7,9 it was evident that the BCS treated with 2N NaOH alone showed total disintegration after the first cycle at all curing times, while untreated BCS failed within two to three cycles. This behaviour is explained by diffuse double layer expansion, sodium-induced clay dispersion, and higher water susceptibility brought on by excess Na^+ ions in extremely alkaline environments. The gradual decrease in UCS of 2N NaOH-treated BCS from 177.50 kPa on Day 1 to 117.68 kPa on Day 90 is consistent with the poor durability. The alkali-treated BCS's resistance to wetting and drying was greatly enhanced by the addition of CLS. The durability gradually rose as the curing time increased; 1% CLS showed the strongest resilience, withstanding up to 7 cycles at 90 days of cure, while 2% CLS withstood 6 cycles. The improvement can be attributed to Ca^{2+} -induced cation exchange and diffuse double layer compression, which reduced sodium-induced dispersion and promoted flocculated soil fabric. In order to reduce water intrusion and shrink-swell damage during cyclic exposure, lignosulfonate polymer chains simultaneously created hydrophobic coats and interparticle bridges. Long-term curing improved interparticle bonding and stabilised the soil matrix, which is consistent with the higher UCS values seen for specimens treated with CLS. In particular, the 1% CLS mix showed the highest UCS of 738.91 kPa at Day-90.

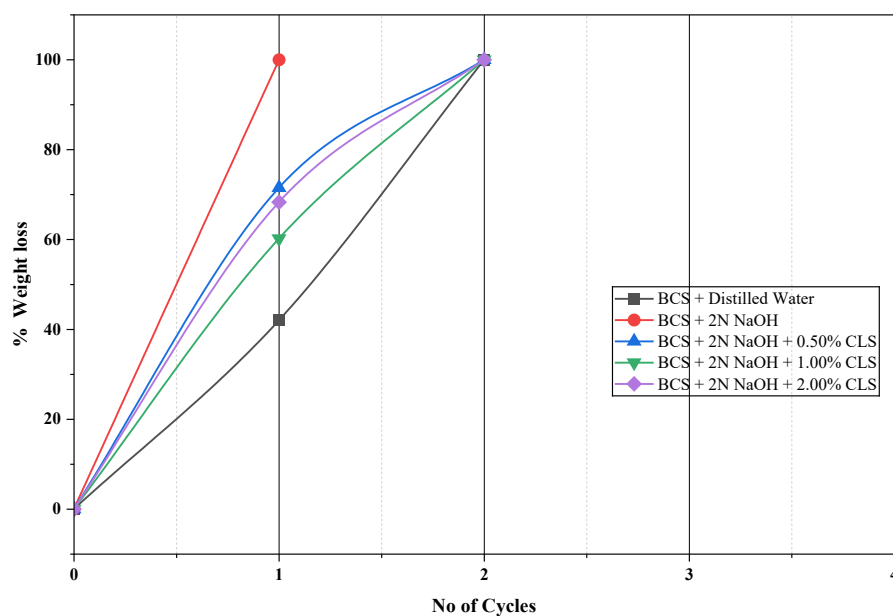


Figure 5. Variation in percentage weight loss of BCS treated with 2N NaOH and different dosages of CLS during wetting–drying cycles after 7 days of curing

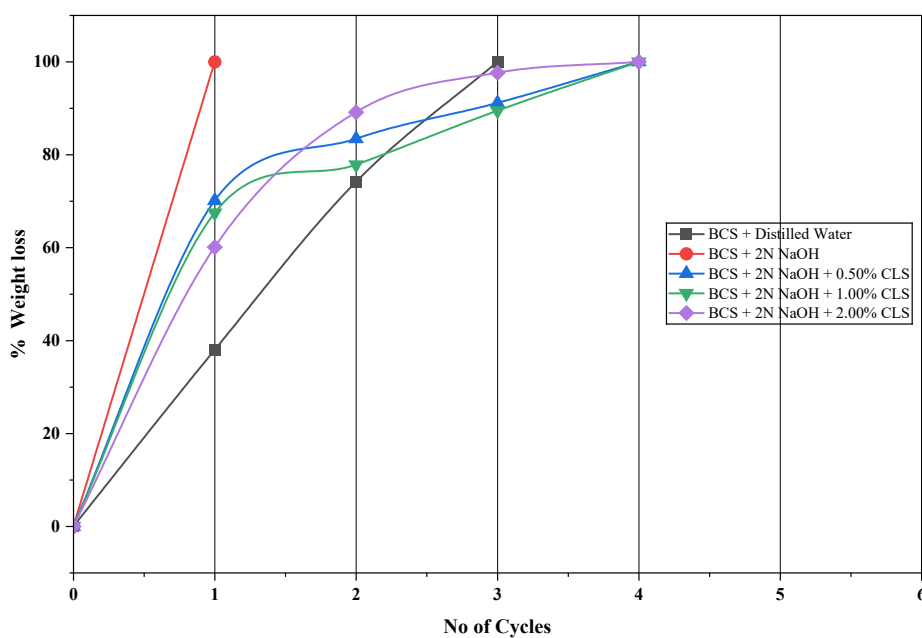


Figure 6. Variation in percentage weight loss of BCS treated with 2N NaOH and different dosages of CLS during wetting–drying cycles after 28 days of curing

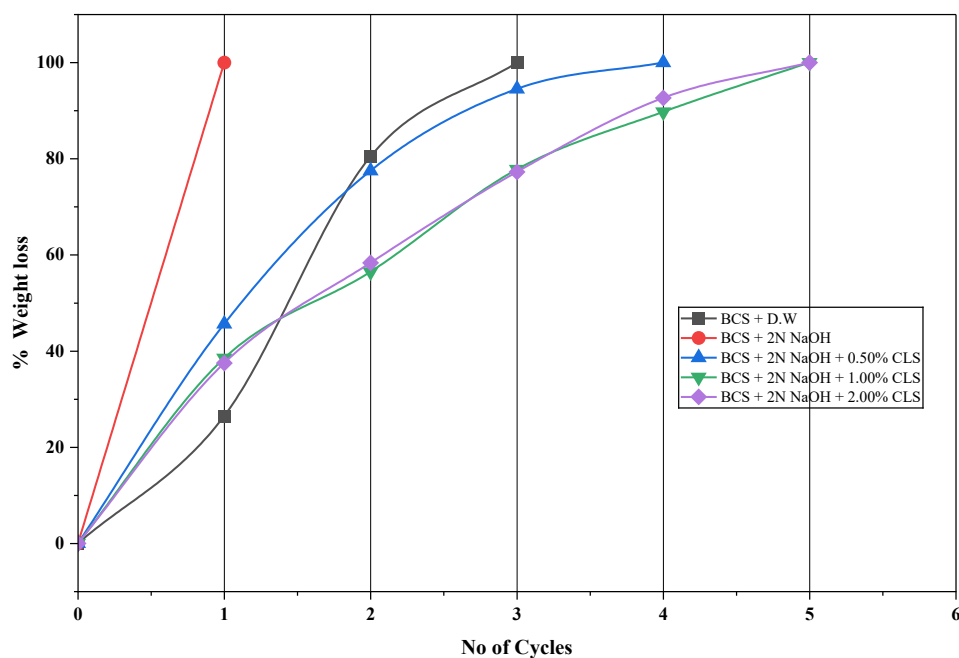


Figure 7. Variation in percentage weight loss of BCS treated with 2N NaOH and different dosages of CLS during wetting–drying cycles after 56 days of curing.

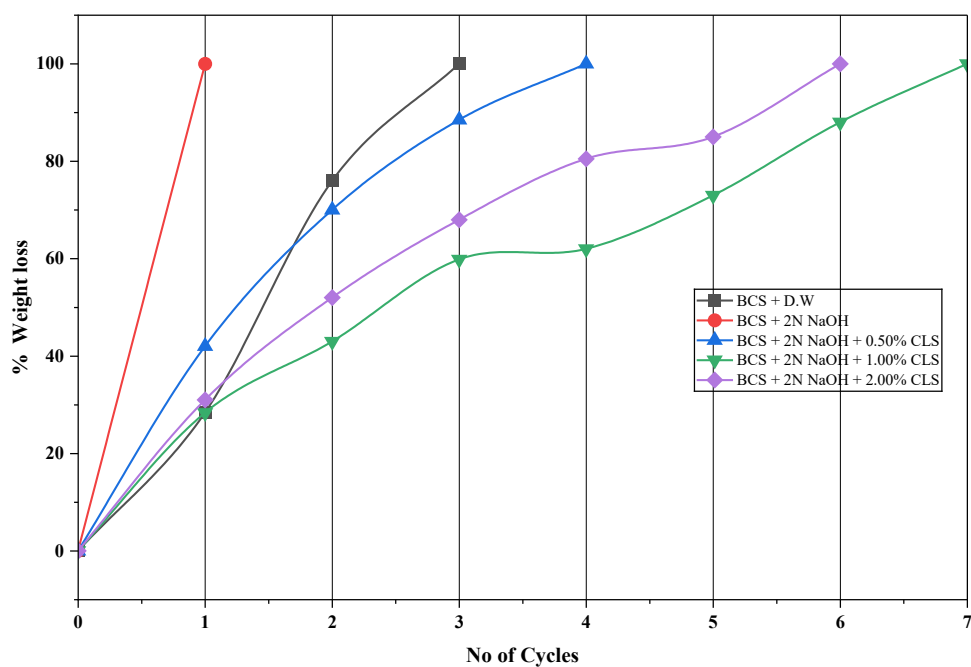


Figure 8. Variation in percentage weight loss of BCS treated with 2N NaOH and different dosages of CLS during wetting–drying cycles after 90 days of curing.

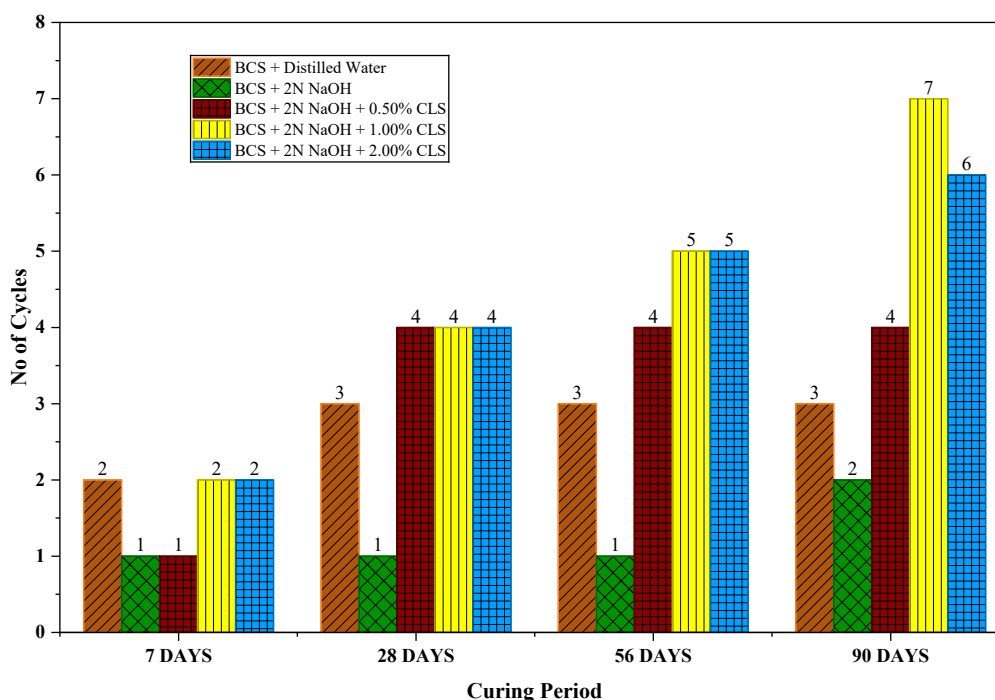


Figure 9. Variation in the number of wetting-drying cycles of BCS treated with 2N NaOH and different dosages of CLS over a 90-day curing period.

3.5 XRD Analysis

XRD study was performed on untreated BCS, BC+ 2N NaOH, and the combination of BCS+2N NaOH+1% CLS, which had a significantly greater effect on stabilization than other CLS percentages.

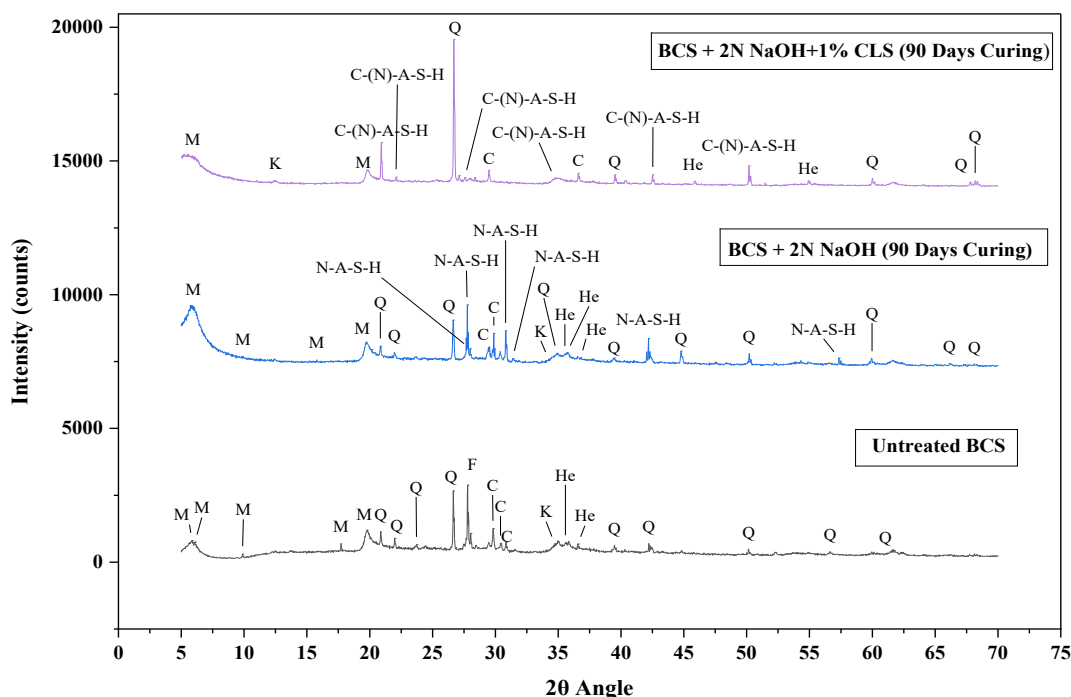


Figure 10. XRD Patterns of Untreated BCS, BCS+ 2N NaOH + CLS (90 days curing) & BCS+ 2N NaOH + 1% CLS (90 days curing).

From fig 10 the XRD examination demonstrated a considerable mineralogical alteration of untreated BCS after alkali activation with 2N NaOH and CLS stabilization over a 90-day curing period. The figure 10 shows that untreated BCS

was dominated by expansive montmorillonite(M) clay minerals, with basal reflections at $2\theta = 5.844^\circ$, 6.10° , 17.723° , and 19.785° , indicating high potential for swelling and plasticity. Kaolinite(K) peak at 34.726° , as well as the dominant quartz(Q) reflection at 26.657° , verified the presence of clay and silica-rich mineral phases, whereas feldspar(F), calcite(C), and haematite(He) reflections revealed minor aluminosilicate, carbonate, and iron oxide constituents. Following 90 days of curing, inclusion of 2N NaOH to BCS caused partial dissolution of aluminosilicate clay minerals, resulting in reduced montmorillonite crystallinity which is indicative to the production of new semi-crystalline sodium aluminosilicate hydrate (N-A-S-H) phases. Soil alkali contact may result in the creation of a novel chemical called NASH(Sivapullaiah and Manju, 2005). Intensified peaks at 27.776° , 27.992° , 30.819° , 31.389° , 42.187° , and 57.345° are indicative to alkali-induced dissolution-precipitation and geopolymeric reaction product(N-A-S-H) production during long-term curing. However, the presence of residual montmorillonite peaks indicated that expanding clay behaviour had not been completely suppressed.

Further application of 1% CLS to the NaOH included BCS for 90 days boosted calcium-assisted stabilization and accelerated mineralogical change. Weak residual montmorillonite reflections at 5.922° and 19.785° showed a significant reduction in swelling clay activity. The maximum strength of calcium lignosulphonate-treated montmorillonite was inferior to that of pure montmorillonite, suggesting that calcium lignosulphonate is adsorbed onto the external surface of montmorillonite minerals, hence diminishing mineral crystal reflection. The XRD diffractograms show a decrease in the crystallite size of the montmorillonite mineral, which indicates a stabilizing action at the microstructural level(B et al., 2021b). The appearance of peaks at 20.923° , 22.102° , 27.580° , 34.707° , 42.521° , and 50.179° can be indicative to the presence of calcium-modified aluminosilicate hydrate (C-(N)-A-S-H) gel phases. Possible Calcite-related reflections at 28.365° , 29.484° , and 36.592° may be attributed to carbonate-assisted cementation and interparticle bonding. Overall, the XRD results revealed a gradual transformation of expansive montmorillonite-rich BCS into a stabilized geopolymeric - matrix by long-term alkali activation, calcium-mediated flocculation, cementation, and aluminosilicate gel formation with the addition of CLS.

3.6 FESEM Analysis

FESEM analysis revealed a progressive morphological alteration of the BCS following 2N NaOH (alkali)inclusion and 1% CLS stabilization. Fig 11 reveals that, untreated BCS has exhibited a dispersed and porous lamellar morphology, consisting of loosely packed flaky and plate-like montmorillonite particles with large interparticle voids indicating poor particle bonding and a strong swelling tendency. After treatment with 2N NaOH, the soil microstructure changed into a partially aggregated and rough-textured matrix as a result of alkali-induced dissolution of aluminosilicate minerals and indicative to the production of semi-crystalline N-A-S-H gel phases. The flaky clay structure became less apparent, but remaining layered particles and microvoids revealed that montmorillonite activity had not been completely suppressed. With the addition of 1% CLS, the morphology transformed into a dense, compact, and well-cemented matrix with clustered agglomerates embedded in continuous cementitious gel products. The significant reduction in pores and disappearance of dispersed clay platelets indicated efficient calcium-assisted flocculation and can be possibly attributed to the production of C-(N)-A-S-H and calcite-based cementitious phases. Overall, the SEM observations are highly consistent with the XRD results, revealing the progressive transformation of the BCS into a stabilized geopolymeric and calcium-cemented matrix.

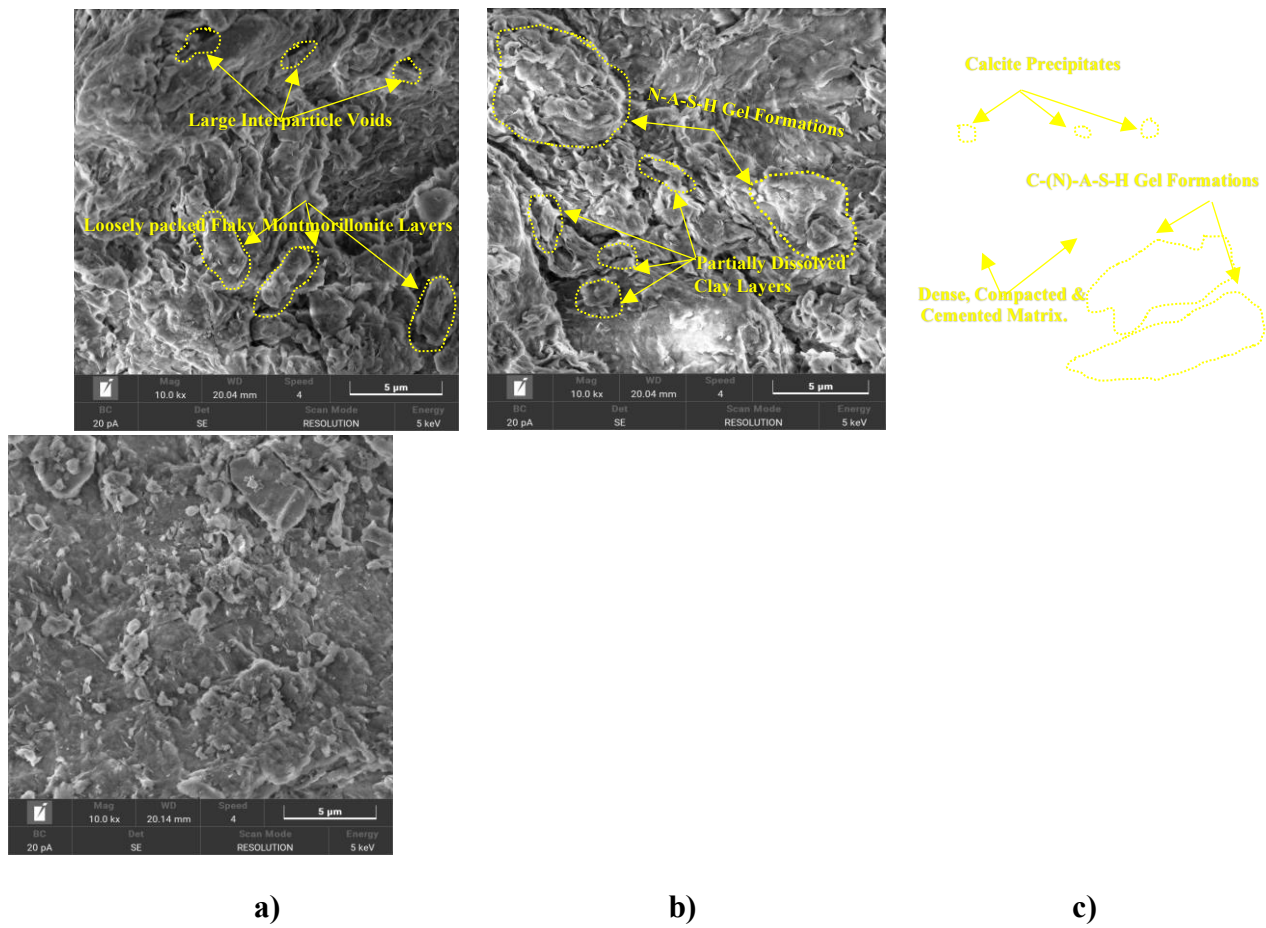


Figure 11. FESEM images of a) Untreated BCS, b) BCS + 2N NaOH + CLS (90 days curing) & c) BCS + 2N NaOH + 1% CLS (90 days curing).

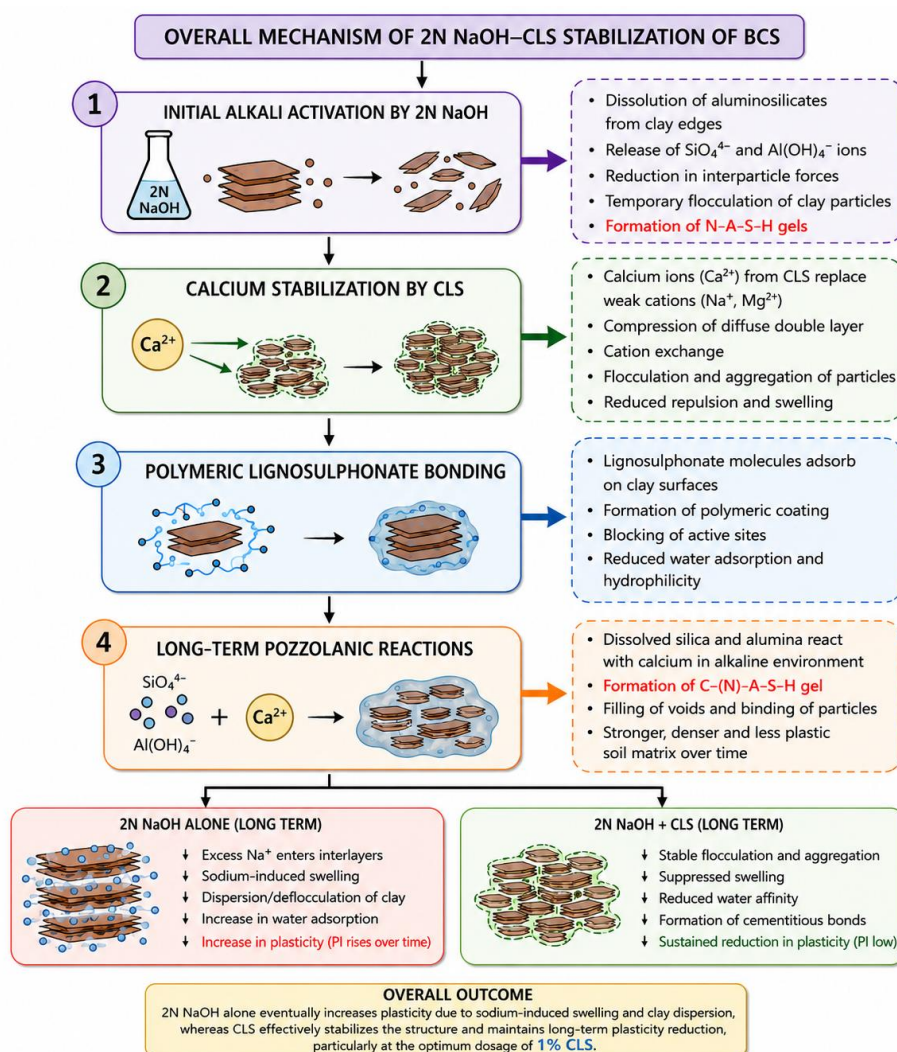


Fig 12. Mechanism of long term curing effect of 2N NaOH and 1% CLS on the BCS stabilization.

3.7. Environmental Implications of CLS and Alkali Treatment on BCS sites

From an environmental standpoint, the alkalinity of the soil system may initially rise due to NaOH treatment, potentially elevating soil pH and soluble salt content for a finite duration. However, the later incorporation of CLS improves calcium-mediated cation exchange and the generation of stable calcium-modified aluminosilicate reaction products, thereby reducing the availability of free sodium ions in the stabilized matrix. CLS is a biodegradable by-product of the pulp and paper industry, which can be recognized as a sustainable soil stabilizer. The development of cementitious reaction products is anticipated to immobilize a significant fraction of the dissolved materials, hence potentially decreasing their mobility. Nevertheless, the criteria such as long-term soil pH, electrical conductivity (EC), salt and calcium leaching, and environmental durability of the treated soil were not evaluated in the current study. Consequently, thorough leaching and ecotoxicological assessments are recommended to fully ascertain the environmental safety of the proposed stabilizing method prior to extensive field application.

4. CONCLUSIONS

From the comprehensive experimental analyses conducted on alkali-activated BCS treated with CLS, the subsequent conclusions can be derived.

- The expansive BCS treated solely with 2N NaOH exhibited poor performance over time due to sodium-induced clay dispersion. The 2N NaOH treatment first decreased the liquid limit and plasticity index causing temporary flocculation. After 90 days, the plasticity index rose to 36.7% from 30%, while the UCS decreased to 115.09 Kpa from 181.42 kPa, indicating soil fabric deterioration resulting from prolonged alkaline conditions. Prolonged

curing facilitated Na^+ transport into montmorillonite interlayers, leading to diffuse double layer expansion, higher interparticle repulsion, and deflocculated clay platelets.

2. The addition of 1% CLS significantly enhanced the physicochemical stability of alkali included BCS through a combination of cation exchange and polymeric stabilizing mechanisms. CLS-supplied Ca^+ ions substituted exchangeable Na^+ ions on clay surfaces, leading to double layer compression and the flocculation/aggregation of clay particles. Lignosulfonate polymer chains concurrently adsorb onto clay surfaces, creating hydrophobic interparticle bridges that diminish water adsorption and clay mobility. After 90 days of curing, this process decreased the liquid limit to 42% and the plasticity index to 7.5% for the 1% CLS mixture.
3. CLS treatment substantially improved the strength and durability of alkali-infused BCS by facilitating the development of a thick and cemented soil matrix. The 1% CLS dosage yielded optimal results, achieving a maximum UCS of 738.91 kPa and withstanding 7 wetting-drying cycles after 90 days of curing. The enhancement is ascribed to the synergistic effects of calcium-mediated flocculation, decreased microporosity, polymeric interparticle bonding, and the generation of cementitious reaction products, all of which augmented soil stiffness and resistance to cyclic moisture-induced degradation.
4. XRD investigations revealed considerable mineralogical modification of montmorillonite-rich BCS under alkali and CLS stabilization. The addition of 2N NaOH to BCS resulted in the dissolution of aluminosilicate minerals which may be attributed to the formation of semi-crystalline sodium aluminosilicate hydrate (N-A-S-H) phases through dissolution-precipitation mechanisms. The incorporation of 1% CLS improved calcium-assisted geopolymeric stabilization, which is indicative to the formation of calcium-modified aluminosilicate hydrate (C-(N)-A-S-H) gel phases and calcite-based cementitious materials. The simultaneous decrease in montmorillonite peak intensity can be possibly validated the inhibition of expansive clay action and mineralogical stability.
5. SEM analysis confirmed that stabilised BCS evolved from a porous lamellar clay fabric to a solid, compact, and well-cemented matrix. NaOH-treated specimens displayed partial aggregation with persistent micro voids, whereas CLS-treated soils formed clustered agglomerates embedded inside continuous gel phases with dramatically reduced pore gaps. The observed reductions in plasticity, significant increase in UCS, and wetting-drying durability were all substantially connected with microstructural densification, supporting the efficiency of 1% CLS as the most effective stabilizer for long-term stabilization of expanding BCS.

5. ACKNOWLEDGMENTS

Funding: “This research received no external funding”

Author Contributions

Conceptualization, Padam Siva Kumar and Sai Kumar Vindula.; methodology, Padam Siva Kumar and Sai Kumar Vindula.; software, Padam Siva Kumar and Sai Kumar Vindula.; validation, Padam Siva Kumar and Sai Kumar Vindula.; formal analysis, Padam Siva Kumar and Sai Kumar Vindula.; investigation, Padam Siva Kumar.; resources, Padam Siva Kumar.; data curation, Padam Siva Kumar and Sai Kumar Vindula.; writing—original draft preparation, Padam Siva Kumar.; writing—review and editing, Padam Siva Kumar.; visualization, Padam Siva Kumar and Sai Kumar Vindula.; supervision, Sai Kumar Vindula.; project administration, Padam Siva Kumar and Sai Kumar Vindula.; funding acquisition, Padam Siva Kumar . All authors have read and agreed to the published version of the manuscript.” Authorship should be restricted to individuals who have made significant contributions to the research.

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BACKGROUND INFORMATION FOR RECORD

Institutional Review Board Statement: “Not applicable.”

Informed Consent Statement: “Not applicable.”

Conflicts of Interest: “The authors declare no conflicts of interest.” “The funders had no role in the design of the study; in the collection, analysis, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.”