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MULTISCALE CHEMICAL AND MINERALOGICAL IMPROVEMENT OF VERY HIGH PLASTICITY CLAY STABILIZED WITH NANO MATERIALS AND BIOPOLYMER

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ABSTRACT: Expansive soil, characterized by very high plasticity (CV) with Plasticity Index (PI) of 52.88%, poses a considerable challenge in geotechnical engineering due to its pronounced shrinkage-swelling behavior. It is influenced not only by index characteristics, but also by its chemical and mineralogical reactivity. Although PI has been used as an early parameter for expansive soil identification, but it has not been insufficient to explain the chemical and microstructural mechanisms on volume instability. This study proposes a multiscale chemical-mineralogical approach to complement PI-based evaluation through CV soil stabilization using nano-lime 3%, nano-silica 1%, and chitosan biopolymer 0.05%. Four treatment conditions were considered: untreated soil (CV), soil treated with biopolymer (CV+B), soil treated with nanomaterials (CV+N), and soil treated with a combination of nanomaterials and biopolymer (CV+N+B). The experimental tests were conducted using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), X-ray Fluorescence (XRF), and Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM–EDS). FTIR results revealed Si–O absorption band shift from approximately 1040 cm^{-1} in the untreated soil to approximately 970 cm^{-1} in the nano–biopolymer treatment, indicating a calcium–silicate–hydrate (C–S–H) gel formation. XRD analysis confirmed the montmorillonite minerals' degradation, which was proved by reduced peak intensity in the $2\theta = 5° – 8°$ range. XRF results showed increased CaO content, indicating a pozzolanic reaction at the bulk scale. SEM revealed microstructural densification and pore filling. EDS showed increased Ca/Si ratios, with statistical analysis confirming the nanomaterials–biopolymers combination as the most chemically effective. The study indicates that CV soil shows significant chemical reactivity with the nano–biopolymer system, with Ca/Si–C–S–H correlations functioning as a multiscale indicator enhancing conventional PI-based methods for assessing expansive soil identification.

Keywords: Expansive soil; Very high plasticity clay; Nano-lime; Nano-silica; Chitosan biopolymer

1 INTRODUCTION

Expansive soils represent one of the most problematic geomaterials in geotechnical engineering due to their high sensitivity to moisture variations, which induces swelling–shrinkage behaviour, leading to cracking, differential settlement, and structural damage [1, 2]. Very high plasticity clay (CV), typically characterized by a Plasticity Index (PI) greater than 40 and dominated by smectite minerals such as montmorillonite, exhibits even greater instability due to its strong affinity for water. Although conventional stabilization methods using lime and cement have been widely applied, their performance in CV soils is often limited by incomplete pozzolanic reactions, non-uniform calcium distribution, and long-term durability issues such as carbonation [3, 4]. In addition, increasing environmental concerns related to CO_2 emissions from cement-based materials have motivated the development of more sustainable stabilization alternatives [5].

Previous studies have explored soil stabilization using lime, cement, nanomaterials, and biopolymers through laboratory-based evaluations such as Atterberg limits, unconfined compressive strength (UCS), and swelling tests. These studies generally report improvements in strength and reductions in plasticity and swelling potential. However, most of them focus primarily on macroscopic engineering performance, with limited attention to the underlying chemical, mineralogical, and microstructural processes governing stabilization [6, 15]. As a result, the

fundamental mechanisms responsible for these improvements remain insufficiently explained, particularly for soils with very high plasticity.

Moreover, differences in material composition and experimental conditions across studies make it difficult to establish a consistent and mechanistically grounded interpretation of stabilization behaviour. While recent work has introduced nanomaterials and biopolymers as promising and more sustainable additives, and has demonstrated potential synergistic effects [6, 11], the evaluation is still largely based on empirical macroscopic indicators. A comprehensive understanding that links chemical reactions, mineral transformations, and microstructural evolution is still lacking.

In this context, multiscale approaches that integrate molecular, mineralogical, and microstructural analyses offer a more robust framework for understanding stabilization mechanisms. These analysis series on molecular, mineralogical, and microstructural aspects are conducted using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction-X-Ray Fluorescence (XDR-XRF), and Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS), respectively. However, such approaches are rarely supported by statistical validation to quantify the relationships between key parameters. Recent findings suggest that the Ca/Si ratio and the FTIR band at around 970 cm^{-1} can serve as reliable indicators of hydration products and pozzolanic activity [8, 13], yet their combined application—particularly for CV soils—has not been systematically established.

Therefore, this study aims to investigate the stabilization mechanism of CV soil using a combination of nano-lime, nano-silica, and chitosan biopolymer through an integrated multiscale chemical, mineralogical, and microstructural approach. The main novelty of this study lies in the integration of multiscale analysis with statistical validation to establish a quantitative correlation between the Ca/Si ratio and FTIR response as a reliable indicator for evaluating stabilization in very high plasticity soils. This approach provides a more comprehensive framework beyond conventional PI-based evaluation and contributes to the development of more effective and sustainable stabilization strategies for expansive soils.

2 RESEARCH SIGNIFICANCE

The revised statement emphasizes that this study goes beyond conventional approaches by explaining the stabilization mechanism of very high plasticity clay through an integrated multiscale framework. Rather than relying solely on empirical parameters such as the plasticity index, the study identifies the Ca/Si ratio, C–S–H formation, and microstructural densification as key indicators governing stabilization performance. More importantly, the study establishes a statistically validated relationship between the Ca/Si ratio and FTIR response, providing a quantitative link between chemical reactions and microstructural evolution. This allows the stabilization mechanism to be interpreted in a more rigorous and mechanism-based manner. As a result, the proposed approach offers a more reliable evaluation framework and supports the development of sustainable nano–biopolymer stabilization strategies for expansive soils.

3 METHOD OF RESEARCH

3.1 Soil Materials and Samples

The soil samples used in this study came from a naturally occurring expansive soil layer in a tropical region area. They were classified as Very High Plasticity soil based on the plasticity criteria and shrinkage–swelling behaviour of very high plasticity clay soils, as described in the classic geotechnical references [14]. In this classification, CV (Fig. 1) soils are characterized by a very high Plasticity Index (PI > 40), a lot of active clay minerals (particularly the smectite–montmorillonite group), and the potential for significant volume changes due to variations in water content, making them highly challenging materials for infrastructure applications. The results of the Atterberg limit test [17], showed that the test soil had a PI value in the range of 40–80, confirming its very high plasticity and strong expansive behaviour. Before more tests , the soil was air-dried, manually crushed to break up large aggregates, and sieved through a No. 40 sieve [17] to ensure homogeneity of the test particles [8].

All soil properties reported in this study were obtained from laboratory experiments. To further characterize the materials used, the physical and chemical properties of the soil, nano-lime, nano-silica, and chitosan biopolymer are presented in Table 1.

The soil shows high clay content and swelling potential, while the nano materials and biopolymer contribute to stabilization through reactive and binding mechanisms. These combined properties play an important role in improving soil structure and engineering behaviour's optimum mixture, which proved most effective in reducing the PI of CV soil, was determined to be 3% nano lime, 1% nano silica, and 0.05% chitosan biopolymer by dry soil weight. These proportions were adopted from previous studies that investigated the stabilization of high plasticity and expansive clays using nano materials and

biopolymers under controlled laboratory conditions [11, 15].

Table 1. Properties of soil and material

Material/ Property	Symbol	Unit	Value
Soil (Clay Very High Plasticity /CV)			
Specific gravity	G_s	-	2.66
pH			5.90
Gran size :			
Sand	S	%	4.36
Silt	M	%	18.82
Clay (<0.002 mm)	C	%	76.82
Atterberg Limit :			
Liquid Limit	LL	%	80.23
Plasticity Limit	PL	%	27.35
Plasticity Index	PI	%	52.88
MDD	γ_d	gr/cm^3	1.345
OMC	w	%	30.56
Swelling Pressure		kPa	447.83
Nano Lime			
Composition	Ca	%	96.90
	Mg	%	1.47
	Si	%	0.56
Particle Size	D_{10}	nm	86.50
	D_{50}	nm	100.50
	D_{90}	nm	141.50
Nano Silica			
Composition	Si	%	90.10
	Ca	%	5.57
	Mg	%	1.37
Particle Size	D_{10}	nm	130.80
	D_{50}	nm	146
	D_{90}	nm	195.60
Bioploymer (Chitosan)			
Composition	Na	%	83.50
	Cl	%	6.05
	P	%	5.78
	Ca	%	2.43
	K	%	2.25
Particle Size		nm	< 2000

In those studies, soils with similar characteristics, including high plasticity index (PI >40) and dominant smectite–montmorillonite mineralogy, were treated under comparable curing and mixing conditions. Therefore, the selected proportions are considered applicable and relevant for the present study. In addition, preliminary tests conducted in this research confirmed that this combination provided the most significant reduction in plasticity and improvement in soil microstructural stability.

The moisture content and stabilizer dosage were carefully controlled during sample preparation to ensure the reliability of the results. All mixtures were prepared at conditions close to the optimum moisture content (OMC), which facilitates effective interaction between soil particles and stabilizing agents. It is well established that both moisture content and stabilizer dosage significantly influence the degree of chemical reaction, particle bonding, and microstructural development. Therefore, maintaining these parameters within optimal ranges ensures the consistency and accuracy of the obtained results.

Four treatment variations were prepared to compare stabilization mechanisms, namely: (1) untreated CV soil as a baseline, (2) CV + biopolymer (B), (3) CV + nano material (N = nano-lime + nano-silica), and (4) CV + combination of nano material and biopolymer (N+B). All subsequent tests were carried out using the optimum composition to ensure consistency of chemical, mineralogical, and microstructural analyses.

Fig. 1. Clay very high plasticity sample

3.2 Characterization Of Chemicals and Mineralogical

To identify chemical reactions, mineralogical changes, and the degree of pozzolanic reactivity, three main types of laboratory tests—FTIR, XRD, and XRF—commonly used in modern multiscale studies of expansive soils were performed [12,13].

3.2.1 Fourier Transform Infrared Spectroscopy

FTIR testing was conducted using a Shimadzu Prestige-21 with a scanning range of 4000–400 cm^{-1}, The characterization of organic and inorganic functional groups [18]. This analysis aimed to identify chemical changes in the soil by detecting active functional groups such as O–H, Si–O, Al–O, as well as new bonds resulting from stabilization reactions including Si–O–Ca and C–S–H. Significant changes were observed through a shift in the characteristic band from approximately 1040 cm^{-1} (Si–O–T of the original soil)

to 970–980 cm^{-1}, which serves as an indicator of C–S–H gel formation in nanomaterial-stabilized soil [6, 10]. This shift, along with changes in band intensity, provides direct evidence of pozzolanic reactions and chemical transformations in stabilized expansive soils.

3.2.2 X-Ray Diffraction

XRD analysis was performed using a Rigaku SmartLab with a Cu-Kα radiation source (λ = 1.5406 Å) over a scanning range of 2θ = 5° – 60°, a step rate of 0.02° per second, standard for clay mineral characterization [19]. This test was used to identify phase changes in major minerals such as montmorillonite, quartz, feldspar, and new hydration phases, including C–S–H and C–A–H. A decrease in peak intensity within the 2θ = 5° – 8° range, characteristic of montmorillonite, was interpreted as a degradation of the layer structure and a reduction in the soil's expansion capacity following the stabilization process. These changes are consistent with the pozzolanic reactions and mineral transformations report [7].

3.2.3 X-Ray Fluorescence

XRF analysis was performed to measure the composition of the soil's major oxides, including SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, K_2O, and other minor oxides. Testing was performed using a Rigaku High-Power Benchtop Sequential WDXRF Spectrometer [20]. The soil samples were dried, ground to pass a 200-mesh sieve, and then compacted into pellets prior to analysis. XRF provides a quantitative overview of changes in bulk oxide composition after the addition of biopolymers or nanomaterials. Changes in oxide concentration—specifically an increase in CaO and a decrease in SiO_2/Al_2O_3—were interpreted as indicators of chemical reactions or mineral transformations during the stabilization process [10]. Because XRF reflects the chemical properties of the entire sample volume, it provides a robust method for quantitatively assessing macroscopic modifications in mineral composition resulting from stabilization treatments.

3.3 Microstructure and Elemental Analysis

3.3.1 Scanning Electron Microscopy and Energy Dispersive Spectroscopy

Scanning Electron Microscopy testing was performed using a ZEISS EVO 10 at 2000–5000× magnification with an accelerating voltage of 10–20 kV and a resolution of up to 3 nm, following the surface morphology characterization procedure standard [21]. Soil samples were dried at room temperature and subsequently coated with a thin layer of gold using sputter-coating technique to enhance electrical conductivity before being mounted on aluminium stubs. These were carried out on four mixture conditions, namely untreated CV soil, CV+B, CV+N, and CV+N+B, to evaluate changes in intergranular morphology, particle aggregation, pore closure, and gel layer formation due to the pozzolanic reaction.

SEM image interpretation focused on qualitative comparison of microstructural changes among different soil admixtures. The untreated CV soil exhibited a plate-like structure with relatively open interparticle pores, reflecting the dominance of smectite minerals. The addition of biopolymers (CV+B) resulted the formation of a thin organic layer covering the particle surface. However, the pore structure remained relatively visible. In the nanomaterial treatment (CV+N), the formation of more compact aggregates was observed, along with the appearance of hydration gels that began to fill the interparticle space. The combined nanomaterials and biopolymers (CV+N+B) exhibited the most homogeneous and densified microstructure, with pores largely covered by a C–S–H/C–A–H hydration gel matrix and an organo-mineral network, indicating significant microstructural densification [11].

EDS analysis was performed in conjunction with SEM using an Oxford Instruments X-Act (PentaFET Precision) detector, following the energy-dispersive microelement characterization procedure standard [22]. This analysis enabled the identification and relative quantification of major elements such as Ca, Si, Al, Fe, Mg, and Na, on the surface of soil particles. EDS data were used to calculate the Ca/Si ratio as a primary indicator of the degree of pozzolanic reaction at the microscale. An increase in the Ca/Si ratio was interpreted as evidence of the formation of hydration phases such as C–S–H and C–A–H, which consistently correlated with the morphological changes and microstructural densification observed in SEM images. The Ca/Si parameter was subsequently used as the main variable in statistical analysis to compare the effectiveness of each stabilization treatment [13].

3.4 Statistical Of Analysis

Statistical analysis was performed to evaluate the effect of stabilization treatments on the chemical, mineralogical, and microstructural characteristics of CV soil. The parameters analysed include the Ca/Si ratio obtained from EDS as an indicator of microscale pozzolanic reactions, the FTIR band intensity at 970 cm^{-1} representing C–S–H gel formation, and the composition of major oxides (SiO_2, Al_2O_3, and CaO) obtained from XRF analysis.

The statistical dataset was constructed from replicate measurements. For each treatment (CV, CV+B, CV+N, and CV+N+B), three independent EDS measurement points were selected to represent the spatial variability of elemental distribution, resulting in a total of twelve data points (n = 12). FTIR intensity values were also obtained from repeated spectral measurements to maintain consistency with the replicate-based dataset. In contrast, XRF data represent bulk composition and were used for comparative interpretation and correlation analysis.

A one-way ANOVA test with a significance level of $\alpha = 0.05$ was applied to evaluate differences among treatments, while paired t-tests were conducted to assess the presence of synergistic effects between the combined nano–biopolymer treatment (CV+N+B) and individual treatments (CV+N and CV+B) [18]. Pearson correlation (r) was used to examine the relationship between multiscale parameters, particularly between the Ca/Si ratio and FTIR intensity at 970 cm^{-1}, where $r > 0.80$ indicates a strong correlation [8]. The magnitude of treatment effects was evaluated using effect size (η^2) based on Cohen (1988), allowing interpretation of both statistical significance and effect strength. This approach ensures that the statistical evaluation is based on representative and reproducible data, providing a reliable framework for interpreting the stabilization mechanism of nano–biopolymer-treated expansive soils [12].

3.5 Multiscale Approach and Data Validation

This study adopts a multiscale–correlative approach to integrate the results obtained from different analytical techniques, enabling a comprehensive understanding of the relationships between parameters across observation scales, from the molecular level to the microstructural level. This approach is intended to ensure that the changes observed in CV soil during the stabilization process are consistent and physically meaningful across different analytical methods. At the molecular level, FTIR analysis was used to identify the formation of new chemical bonds associated with hydration processes, such as Si–O–Ca and C–S–H, indicating the occurrence of pozzolanic reactions. At the mineralogical and bulk chemical levels, XRD and XRF analyses were employed to confirm mineral phase transformations and changes in major oxide composition, particularly the reduction of montmorillonite content and the increase in reactive calcium. At the microstructural level, SEM observations were carried out to evaluate changes in particle arrangement, aggregation behaviour, and pore structure, while EDS analysis provided quantitative information on elemental distribution, with the Ca/Si ratio used as a key indicator of hydration intensity.

Inter-instrument validation was conducted using a data triangulation approach by comparing key indicators across different scales, including: (i) the increase in Ca/Si ratio obtained from EDS, (ii) the shift and increased intensity of the FTIR band at around 970 cm^{-1} associated with C–S–H formation, and (iii) the reduction in montmorillonite peak intensity within the range of $2\theta = 5° – 8°$ in the XRD diffractogram. The consistency among these indicators suggests that the stabilization process occurs coherently across multiple scales, linking chemical reactions to the evolution of soil microstructure.

This integrated multiscale framework follows the correlative validation approach recommended by [7, 8, 13], emphasizing that the combined use of spectroscopic, diffraction, oxide composition, and microstructural analyses is essential to achieve a comprehensive and mechanism-based understanding of expansive soil stabilization.

4 RESULTS AND ANALYSIS

4.1 FTIR Analysis: Changes In Soil Chemistry

After the stabilization treatment, the FTIR spectrum of CV soil indicated significant changes in its chemistry (Fig. 2).

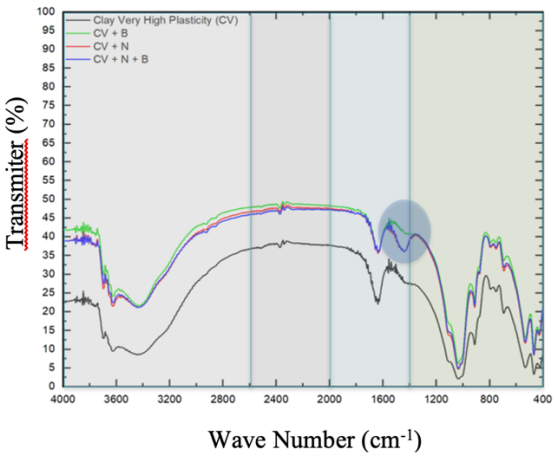

Fig. 2. FTIR test results of CV soil and addition of stabilizer mixture

The untreated soil had strong bands at 3620 cm^{-1} (O–H vibration of montmorillonite), 1040 cm^{-1} (Si–O–T group), and 915 cm^{-1} (Al–OH of illite). After adding chitosan biopolymer (CV+B), a new weak bond formed between 1550 and 1650 cm^{-1}. This band was connected to the $–NH_2$ and –C=O groups and showed that chitosan molecules were sticking to the surface of the soil minerals [15].

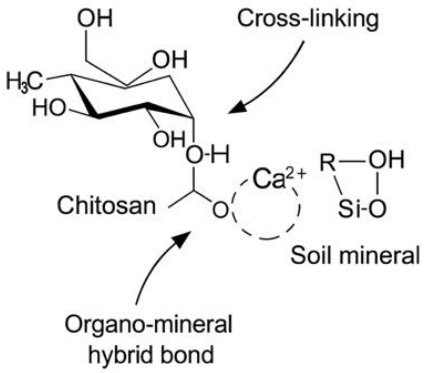

Fig 3. Cross linking mechanism of kitosan – lime – soil minerals

These results indicate that nano-lime, nano-silica, and chitosan reduce hydrophilic O–H intensity and promote organic–inorganic polyelectrolyte bonds (R–NH_2–Ca–Si–O) (Fig. 3). Thus, chemical stability is enhanced, and soil expansiveness is mitigated [10]. The 1040 cm^{-1} band moved to 980 cm^{-1} in the nano-material treatment (CV+N). This shows that Si–O–Ca bonds were starting to form, because Ca^{2+} and silanol groups reacted with each other [7]. In the CV+N+B combination, the main peak expanded higher to 970 cm^{-1}, and the intensity increased noticeably. An additional band about 875 cm^{-1} (Si–O–Al–Ca) connections appeared, showing that stable C–S–H and C–A–H structures developed (Fig. 4.) [6].

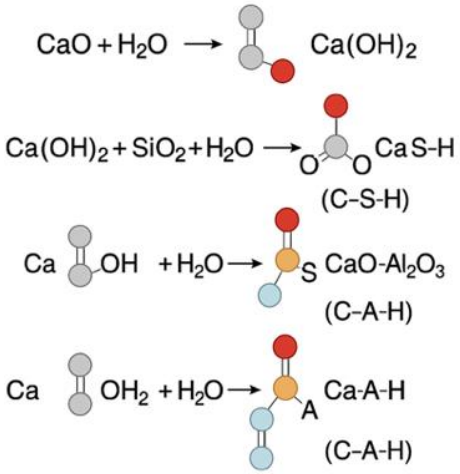

Fig 4. Scheme of C-S-H and C-A-H formation in pozzolanic reactions

4.2 XRF Analysis: The Ratio Of CaO to Sio_2 and The Composition Of Oxides

According to XRF results (Table 2), show a decrease in SiO_2 content and an increase in CaO with the additives. The CaO/SiO_2 ratio increased from 0.084 (CV) to 0.177 (CV+N+B), indicating an active pozzolanic reaction between Ca^{2+} from nano-lime and silanol groups from both soil silica and nano-silica [11]. The increase in the ratio confirms the formation of a more intensive C–S–H gel, consistent with previous observations in nano-silica-lime soil systems [7, 8].

Table 2. Amount of oxides and CaO/SiO_2 ratio

Treatment	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	CaO/SiO_2 ratio
CV	53.0	25.6	10.2	7.77	1.07	0,147
CV + B	53.5	25.7	10.4	6.59	1.21	0,123
CV + N	51.2	26.4	9.93	8.90	1.19	0,174
CV + N + B	51.0	26.2	9.95	9.22	1.16	0,181

4.3 XRD Analysis: Changes in Mineralogy

The XRD diffractogram (Fig. 5) shows changes in the peak patterns of the primary minerals in CV soil following stabilization treatment.

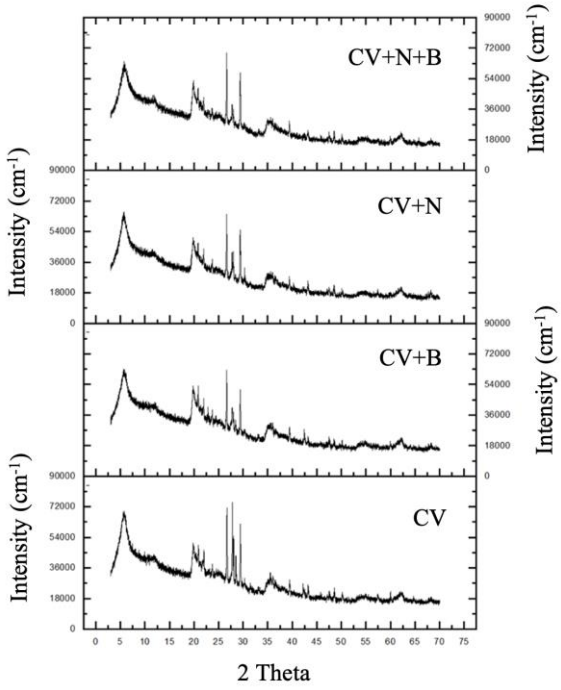

Fig. 5. XRD test results for CV soil and its mixture additions

The untreated soil shows strong peaks corresponding to montmorillonite at 2θ = 5.9° and 19.8°, and quartz at 26.6°. After the +B treatment, the intensity of montmorillonite decreases slightly due to the adsorption of organic molecules between layers. CV+N treatment caused a shift in the montmorillonite peak from 5.9° → 6.1°, accompanied by an increase in the feldspar peak, indicating interlayer dehydration due to the reaction of Ca^{2+} and Si–OH.

The combination of CV+N+B sharply decreased the intensity of montmorillonite and displayed new peaks at 29.4° and 32.2° identified as C–S–H and C–A–H, the result of pozzolanic transformation [10]. This finding is consistent with previous multiscale studies showing that the Ca–Si–Al reaction induced by nano-silica and nano-lime reduce the crystallinity of montmorillonite and generates a non-expansive gel phase [14].

4.4 SEM Analysis: Changes In Microstructural Morphology

SEM images (Fig. 6) were used to qualitatively observe the microstructural evolution of CV soils resulting from biopolymer- and nanomaterial-based stabilization treatments. The analysis focused on changes in particle morphology, aggregation levels, the presence of intergranular pores, and indications of hydration gel formation that fills the interparticle spaces, without performing image-based pore quantification. The untreated CV soil exhibits a typical morphology of highly plastic clay, consisting of a plate-like structure with relatively random particle orientation and open interparticle pores. Irregular micro-voids are clearly visible between the mineral layers, reflecting the dominance of montmorillonite and explaining the high shrinkage potential of this soil. These microstructural characteristics are consistent with classical and recent studies on the behaviour of smectite-rich expansive clays [13].

In the CV+B treatment, SEM images show the particle surfaces are partially coated with a thin organic layer, accompanied by the formation of softer intergranular agglomerates. Although interparticle pores are still identifiable, organic bridges are observed connecting the soil particles. This phenomenon indicates that the chitosan biopolymer primarily functions as a physical-chemical binding agent through adsorption and bridging effects, without producing substantial microstructural densification. This pattern is consistent with previously reported biopolymer-soil interaction mechanisms reports [9, 15].

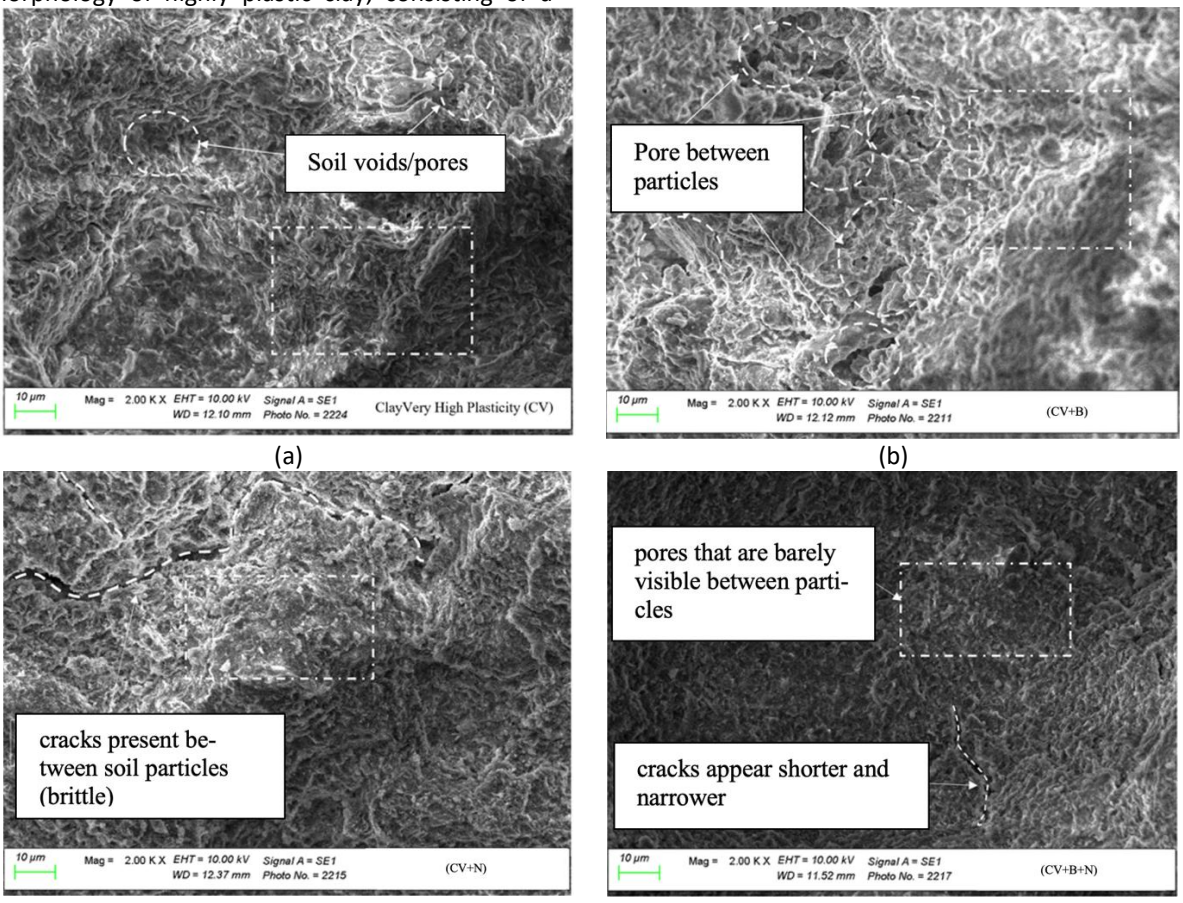

Fig. 6. Test results for CV soil and its mixture additions: (a) Clay Very High Plasticity (CV); (b) Clay Very High Plasticity + Biopolymer (CV+B); (c) Clay Very High Plasticity + Nano Lime + Nano Silica (CV+N); (d) Clay Very High Plasticity + Nano Lime + Nano Silica + Biopolymer (CV+N+B)

The nanomaterial treatment (CV+N) exhibited more pronounced morphological changes. Soil particles appeared to coalesce into more compact aggregates, accompanied by the formation of a gel layer partially filling the interparticle space. Short, discontinuous microcracks were also observed, indicating reduced freedom of deformation between mineral sheets. This morphology indicates an active pozzolanic reaction between CaO and SiO_2 , resulting in initial hydration phases such as C–S–H and C–A–H, consistent with previous reports on nano-lime and nano-silica stabilization of expansive soils [6, 7]. The most significant microstructural changes were observed in the combined nanomaterials-biopolymers treatments (CV+N+B). SEM images revealed a much denser and more homogeneous structure, with intergranular pores barely identified. Particle surfaces were coated by a continuous gel matrix that bound the particles together, while visible cracks tended to be shortened and localized. This morphology indicates the formation of a cross-linked organo-mineral network, where the C–S–H/C–A–H gel from the pozzolanic reaction was strengthened by the interaction of chitosan with Ca^{2+} ions and silanol groups, resulting in stable microstructural densification. This pattern aligns with the synergistic nano–biopolymer model reports [10, 11]. Overall, SEM morphological interpretation confirms that stabilization effectiveness increases sequentially from CV → CV+B → CV+N → CV+N+B. The combination of nanomaterials and biopolymers results in the most extensive microstructural transformation, corroborating the FTIR, XRD, and EDS findings on hydration phases formation and Ca/Si ratio enhancement. Thus, SEM analysis provides strong visual evidence that CV soil stabilization occurs through an integrated and multiscale chemical–mineralogical mechanism.

4.5 EDS Analysis: Distribution Of Elements and Ca/Si Ratio

EDS analysis was conducted using a multi-spot approach (n = 3 for each treatment) to capture the variability of elemental distribution at the microscale. The results show a consistent increase in calcium content accompanied by a relative decrease in silica after stabilization. Consequently, the Ca/Si ratio increases from approximately 0.02 in untreated soil (CV) to about 0.18 in the combined nano–biopolymer treatment (CV+N+B), indicating the formation of calcium-rich hydration products such as C–S–H [15]. This increase reflects the role of nano-lime in supplying reactive calcium, which interacts with silica to form hydration products, while nano-silica enhances the reaction through additional reactive surfaces. The presence of biopolymer further contributes by stabilizing the formed structure. A strong correlation (r = 0.89) between the Ca/Si ratio and the FTIR band intensity at 970 cm^{-1} confirms that the chemical changes observed at the microscale are consistent with hydration processes at the molecular level [8]. In addition to chemical effects, SEM observations reveal a denser and more compact soil structure with reduced pore spaces. This indicates that physical mechanisms such as particle rearrangement, flocculation–agglomeration, and pore refinement also play an important role. Therefore, the stabilization of CV soil can be interpreted as a coupled chemo-physical mechanism, where chemical bonding and physical restructuring occur simultaneously.

4.6 Statistical Analysis: Evaluation Of Mixed Significance

Statistical analysis was performed using replicate data obtained from multi-spot EDS measurements (n = 12), as described in Section 3.4. This dataset allows the variability within each treatment to be quantified and provides a reliable basis for evaluating statistical significance.

The one-way ANOVA results ($\alpha = 0.05$) show a significant difference among treatments, with F = 15.72 and p = 0.002. This indicates that the increase in Ca/Si ratio observed after stabilization is not due to random variation, but is directly associated with the applied treatments. Further analysis using Tukey's post-hoc test shows a consistent order of effectiveness: N+B > N > B > Control

This trend agrees with the EDS results, where the combined treatment produces the highest Ca/Si ratio. Paired t-test results (t = 6.42; p = 0.001) confirm that the nano–biopolymer combination provides a significantly greater effect compared to individual treatments. In addition, the effect size ($\eta^2 = 0.64$) indicates that approximately 64% of the variation in Ca/Si ratio is explained by the treatment factor, demonstrating a strong influence of the stabilization mechanism. Overall, these statistical results are consistent with the findings from EDS, FTIR, and SEM analyses, confirming that the improvement in soil behavior is governed by a synergistic chemo-physical mechanism [10, 15].

4.7 Multiscale Integration and Mechanistic Interpretation of CV Soil Stabilization

The integration of FTIR, XRD, XRF, SEM, and EDS results shows a consistent multiscale pattern in explaining the stabilization mechanism of nano–biopolymer-treated CV soil. A strong correlation (r = 0.89) between the Ca/Si ratio obtained from EDS and the FTIR band intensity at 970 cm^{-1} confirms that the increase in reactive calcium at the microscale is directly associated with the formation of C–S–H gel at the molecular level [8,13]. In addition, the agreement between changes in oxide composition from XRF and the reduction of expansive mineral phases observed in XRD indicates that the pozzolanic reaction affects both local and bulk properties of the soil. This is further supported by SEM observations, which show a denser and more homogeneous microstructure, reflecting pore reduction and improved interparticle bonding. These findings demonstrate that the chemical, mineralogical, and microstructural responses are strongly interconnected.

Based on this multiscale evidence, the stabilization mechanism can be interpreted as a synergistic process. Initially, nano-lime undergoes hydration to produce $Ca(OH)_2$, releasing Ca^{2+} ions. These ions react with silica from both soil and nano-silica to form C–S–H gel through pozzolanic reactions. At the same time, chitosan interacts with Ca^{2+} and silanol (Si–OH) groups through its functional groups ($–NH_2$ and –OH), forming an organo-mineral network. This interaction results in a cross-linked hybrid matrix that integrates inorganic hydration products (C–S–H/C–A–H) with the biopolymer structure.

The formation of this hybrid matrix leads to pore filling, particle bonding, and improved microstructural compactness, as observed in SEM analysis. Therefore, the stabilization of CV soil is not governed solely by conventional pozzolanic reactions, but also by organo-ionic interactions that enhance the stability of the soil structure and reduce its susceptibility to volumetric changes.

These findings are consistent with the statistical results presented in Section 4.6, which confirm the significant and synergistic effect of the combined nano–biopolymer treatment. The overall interpretation supports a coupled chemo-physical mechanism, where chemical bonding and physical restructuring occur simultaneously [10, 11].

From an engineering perspective, this mechanism implies that the nano–biopolymer stabilization approach has strong potential to improve subgrade performance by reducing swelling potential, increasing soil stiffness, and enhancing long-term volumetric stability, particularly for highly plastic expansive clays. This study was conducted under controlled laboratory conditions, with the assumption that the soil samples were homogeneous and representative of very high plasticity clay. Mixing, curing, moisture content, and stabilizer dosage were maintained under controlled conditions to ensure consistent interaction between soil particles and stabilizing agents.

However, some limitations should be acknowledged. The study focused on a single soil type, which may limit the finding generalization to other soil conditions. In addition, the experiments were conducted at a laboratory scale and did not account for long-term environmental effects such as cyclic wetting–drying or field loading conditions. Therefore, further studies are needed to evaluate the performance of the proposed stabilization method under more varied and realistic conditions. Despite these limitations, the results provide a clear and consistent understanding of the coupled chemical and physical mechanisms governing the stabilization of expansive soils.

5 CONCLUSION

Expansive soils with very high plasticity pose significant challenges in geotechnical engineering due to their high swelling potential and sensitivity to moisture variations. It requires effective stabilization strategies to improve their engineering performance. This study investigates the stabilization mechanism of CV soil using a combination of nano-lime, nano-silica, and chitosan biopolymer through an integrated chemical–mineralogical–microstructural approach supported by statistical analysis.

The results demonstrated that CV soil stabilization could not be adequately explained solely by the plasticity index, as substantial chemical reactions and microstructural transformations occur. FTIR and XRD analyses confirmed the formation of C–S–H gel and the reduction of montmorillonite activity, while XRF results indicated the occurrence of pozzolanic reactions. SEM–EDS observations further revealed pore closure, microstructural densification, and an increase in the Ca/Si ratio, confirming enhanced hydration processes. These findings indicate that stabilization is governed by a coupled chemo-physical mechanism that involves simultaneous chemical bonding and physical restructuring.

Statistical analysis verified that mixture variation significantly influences the performance of CV soils. The nano–biopolymer combination (N+B) showing the highest effectiveness in CV soil stabilization due to its synergistic interaction.

The proposed Ca/Si–C–S–H–based framework provides a more comprehensive evaluation

approach beyond conventional PI-based methods and offers a reliable basis for improving subgrade performance by reducing swelling potential and enhancing volumetric stability.

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