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Thermo Mechanical and Microstructural Behavior of Clay Soil Under Elevated Temperatures with CBR Prediction Modelling

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Abstract: ***This study investigates the influence of thermal treatment on the engineering behaviour, mineralogical structure and bearing resistance of a natural clay soil heated up to 600 °C. Comprehensive laboratory testing including Atterberg limits, Proctor compaction, direct shear and soaked CBR was conducted to quantify changes in consistency, density, shear strength parameters and penetration resistance. Results indicate a significant reduction in plasticity and cohesion with temperature, accompanied by a progressive increase in friction angle from 26.1° to 46.1°. The soaked CBR improved markedly from 2.5% for untreated soil to 21.4% for thermally treated soil at 600 °C, demonstrating enhanced load bearing capacity. XRD analysis confirmed the dehydroxylation of kaolinite and breakdown of illite, while SEM images revealed a clear transition from a dense clay fabric to a fragmented granular microstructure, explaining the shift from cohesive to frictional behaviour. Micro structure analysis supported the observed mineralogical transformation with moderate oxide redistribution. A polynomial prediction model ($R^2 = 0.98$) was developed with Python (numPy and Pandas libraries) to estimate CBR at intermediate temperatures, incorporating a 95% confidence interval for reliability. Overall, the study provides a mechanistic understanding of how thermal exposure alters the microstructure and geomechanical performance of clay, offering valuable insights for applications involving fire-affected soils, thermal remediation and high temperature geo thermal material processing.***

Keywords: thermal treatment of clay, micro structural transformation, high temperature soil behavior, bearing resistance

1. Introduction

Clayey soils often exhibit undesirable engineering behaviour such as high plasticity, low bearing resistance, and significant volumetric instability, making them sensitive to moisture and external loading [12]. Thermal treatment has emerged as a promising technique for modifying the physico chemical and microstructural characteristics of clay minerals, influencing their engineering performance [13]. Exposure to elevated temperatures induces a series of mineralogical transformations, including removal of absorbed and interlayer water, dehydroxylation of clay lattices and eventual formation of amorphous phases, all of which significantly alter the soil's mechanical response [18]. Understanding these thermally driven changes is essential for geotechnical applications involving fire affected soils, thermal remediation processes, geo material manufacturing and high temperature energy systems [20].

Previous research has shown that heating clay minerals between 200 to 300°C primarily removes adsorbed and bound water, leading to a reduction in plasticity and weakening of interparticle electrochemical bonds [8]. Kaolinite undergoes complete dehydroxylation between 450 to 650 °C, forming metakaolin an amorphous aluminosilicate with markedly altered structural and mechanical properties [7]. Studies by Locat et al. [11] and Romero et al. [15] demonstrated that such thermal transformations reduce soil cohesion while increasing frictional resistance due to the breakdown of platy clay aggregates into granular fragments. SEM based investigations by Tang et al. [16] and Vardhan et al. [17] further supported these findings, revealing pronounced microstructural changes including pore formation, particle disintegration and loss of clay structure at elevated temperatures. These microstructural shifts have been linked to improvements in penetration resistance and stiffness, as reflected in CBR and shear strength trends.

Despite these advances, the literature contains limited integrated studies that combine geotechnical testing, mineralogical characterization (XRD), microstructural evaluation (SEM) and chemical composition analysis to comprehensively assess soil behaviour under high temperature exposure. Moreover, there is a need to develop predictive models that quantify temperature dependent changes in soil strength parameters for engineering use.

In this context, the present study investigates the thermal modification of a natural clay soil subjected to heating up to 600 °C. The research integrates Atterberg limits, compaction behaviour, shear strength parameters, CBR, XRD, SEM and oxide composition to establish a mechanistic understanding of how thermal exposure affects both microstructure and macroscopic engineering performance. A prediction model for CBR variation with temperature is also developed to support practical interpretation by using Python. The combined findings provide a holistic perspective on the thermo-mechanical evolution of clay soil and contribute to the growing body of knowledge on high temperature soil behaviour.

2. Materials and Methodology

2.1 Soil Collection and Preliminary Classification

The soil used in this study was collected from a depth representative of natural subgrade conditions. Bulk samples were air dried, pulverized and passed through a 4.75 mm sieve prior to testing. Preliminary classification was performed following IS:1498, including grain size distribution and Atterberg limits, to determine the soil's plasticity characteristics and identify the dominant soil type (Table 1). The soil collected location having latitude and longitude 16.470493769865026^0, 80.70503684892776^0 respectively. Now onwards the soil is denoted as VZA since the soil is collected from Vijayawada, Andhra Pradesh.

Table 1: Basic and index properties of the soil

Property	VZA
Natural Moisture content	10.4
Liquid limit (%)	55.0
Plastic limit(%)	26.1
Plasticity index	28.9
Specific gravity	2.68
ISSCS	CH
Maximum dry density (g/cc)	1.77
Optimum moisture content (w_{opt}) (%)	19.9
Gravel (>4.75mm)	0
Sand (4.75 mm – 75 µm) (%)	23.5
Silt (75 µm – 2 µm) (%)	72.5
Clay (< 2 µm) (%)	4

2.2 Thermal Treatment Procedure

To evaluate the effect of temperature on soil behaviour, the soil was subjected to controlled heating at 110°C, 200°C, 300°C, 400°C, 500°C and 600°C. Soil was placed in ceramic bowl and heated in a muffle furnace. Each sample was maintained at the target temperature for 3 hours, at a rate of 5°C/min to ensure thermal equilibrium and complete structural transformation. After heating, samples were left in the furnace for 24 hours, cooled to room temperature. Soil samples were kept in a desiccator to prevent moisture ingress prior to testing.

2.3 Testing Methodology

Particle size distribution, Atterberg limit tests were performed in accordance with IS:2720 (Part 5). Standard Proctor compaction tests were conducted following IS:2720 (Part 7). Direct shear tests were carried out as per IS:2720 (Part 13). Samples were compacted to their respective maximum dry density (MDD) and tested under three normal stresses to determine cohesion (c) and friction angle (ϕ) using Mohr Coulomb failure theory. Soaked CBR tests were performed according to IS:2720 (Part 16) on compacted samples. All specimens were soaked for 4 days prior to penetration testing. The CBR values at different temperatures were used to evaluate the effect of thermal treatment on load bearing resistance.

2.4 Micro-Structural analysis

Micro structural analysis conducted for untreated soil and 600°C thermally treated soil. SEM imaging was performed on gold coated samples to examine microstructural evolution. samples were scanned at various magnifications (400×, 25k×). The analysis focused on changes in particle shape, fabric and pore structure. Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDS) to study soil microstructure and elemental composition such as major oxide composition (SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, K_2O) was determined for both untreated and 600°C treated samples. These results were analysed to identify shifts in oxide distribution associated with mineral breakdown and concentration effects due to mass loss.

XRD testing was used to identify mineralogical changes. Diffraction patterns were obtained using Cu-Kα radiation in the range of 5°–70°(2θ). XRD is helpful in observing mineral breakdown and witness the changes in engineering behaviour (Fig. 1).

2.5 Development of CBR Prediction Model

A polynomial regression model was developed with Python using NumPy, Pandas libraries to predict CBR as a function of temperature. The experimental CBR data fitted with a second degree polynomial and model accuracy was evaluated using coefficient of determination (R^2) and root mean square error (RMSE). A 95% confidence interval band was generated to quantify predictive uncertainty. This model supports interpretation of temperature dependent CBR trends across intermediate temperatures.

3. Data Processing and Analysis

All tests were performed in triplicate and mean values were used for analysis. Statistical computations, curve fitting and generation of prediction graphs were conducted using Python NumPy, Pandas and Matplotlib.

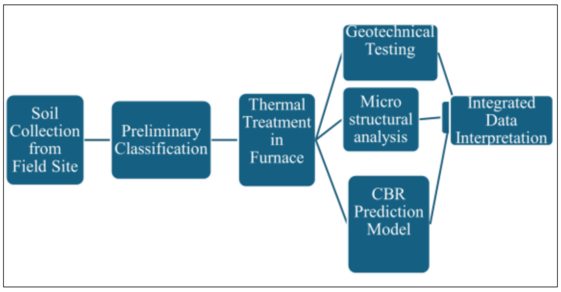

Figure 1: Work flow diagram of this study

4. Results and Discussion

4.1 Particle Size Distribution (PSD)

The natural soil shows a well graded distribution, with gradual reduction in fines toward smaller sieve sizes, indicating a significant fraction of silt size particles. Progressive heating from 110°C to 600°C causes a systematic reduction in percentage passing, especially at finer sizes (<0.6 mm), reflecting thermal degradation, aggregation of clay minerals. The most notable change occurs beyond 300°C, where fines (<0.15 mm) drop sharply, suggesting dehydroxylation of clay minerals and structural breakdown. At 500 to 600°C, the soil becomes much coarser, with fines nearly eliminated. Coarser fractions (4.75–2.36 mm) show only moderate reduction, implying thermal effects predominantly influence finer, clay-rich components. The shift in gradation indicates a transition from a potentially plastic, fine grained soil to a non-plastic, granular material as temperature increases (Fig. 1).

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Overall, the soil exhibits thermal induced loss of fines, altered engineering behavior at elevated temperatures.

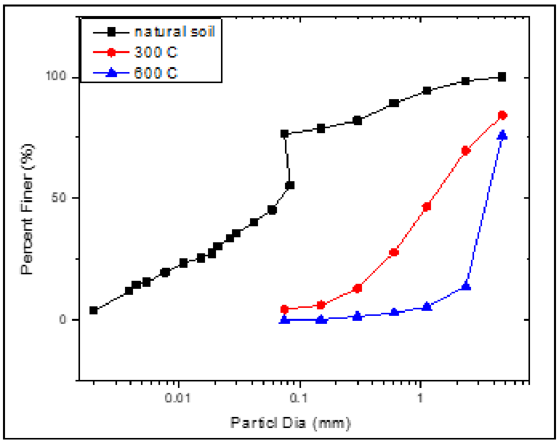
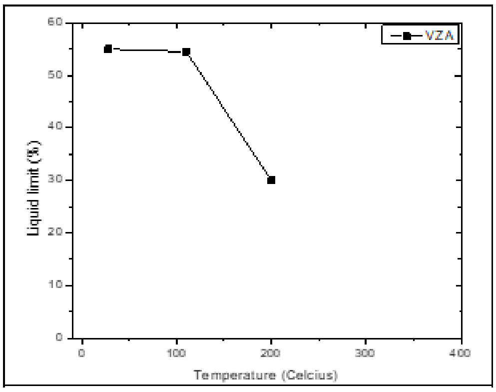


Figure 2: Variation of PSD with thermal treatment

4.2 Plasticity characteristics

The variation of Atterberg limits with temperature indicates a pronounced thermal effect on the soil's consistency behaviour. Untreated soil exhibits a liquid limit (LL) of 55 %, a plastic limit (PL) of 26.1 %, and a plasticity index (PI) of 28.9 %, classifying it as a highly plastic fine grained soil. Heating to 110 °C produces only marginal changes, with LL and PL decreasing slightly to 54.5 % and 25.5 %, respectively, while PI remains essentially unchanged (29 %). This suggests that removal of free water at low heating temperatures does not significantly alter the soil's diffuse double layer. However, upon heating to 200 °C, a substantial reduction in LL (30.1 %) and PL (18.3 %) is observed, with PI dropping sharply to 11.8 %. Such reductions are consistent with the progressive loss of adsorbed water and partial alteration of clay minerals, which diminish the soil's capacity to undergo plastic deformation (Fig. 3 and Fig. 4). Similar trends have been reported for thermally treated clays, where the breakdown of interlayer water and structural modifications reduce plasticity and workability [11]. The observed decline in plasticity at higher temperatures thus reflects a transition towards a more granular and less cohesive material, indicating that thermal exposure can significantly alter the engineering behaviour of clayey soils.



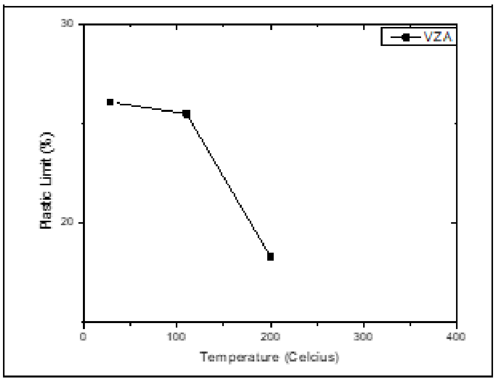


Figure 3: Variation of Liquid limit and plastic limit with thermal treatment

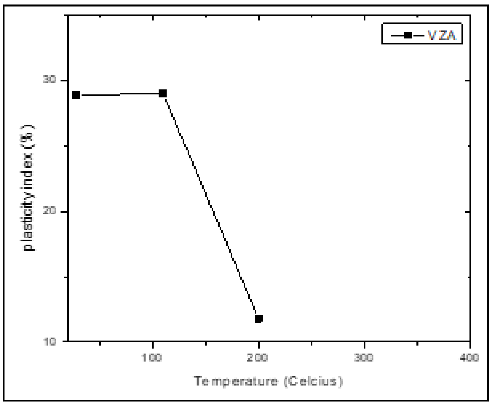


Figure 4: Variation of plasticity index with thermal treatment

4.3 Compaction Characteristics

The influence of heating on the compaction characteristics of the soil reveals a systematic reduction in both optimum moisture content (OMC) and maximum dry density (MDD) as temperature increases. Untreated soil exhibits an OMC of 19.9 % and an MDD of 1.77 g/cc, values that remain unchanged at 110 °C, indicating that removal of free moisture at low thermal exposure does not alter the compaction behavior of the soil. A marked change is observed at 200 °C, where OMC decreases to 15 % and MDD drops to 1.62 g/cc, suggesting structural modification due to loss of adsorbed water and partial alteration of clay minerals. With further heating from 300°C to 600 °C, OMC reduces progressively to 11.5% to 10%, while MDD declines slightly from 1.59 g/cc to 1.55 g/cc. This behavior is consistent with thermally induced breakdown of the clay fabric, reduction in specific surface area and transformation toward a more granular structure, all of which reduce the soil's water holding capacity and compaction potential. Similar reductions in OMC and MDD with increasing temperature have been reported for thermally treated fine grained soils, where dehydration and mineralogical changes lead to decreased plasticity and lower compaction efficiency [1]. The results demonstrate that high-temperature exposure significantly alters the soil's compaction response, with implications for geotechnical applications involving heat affected ground (Fig. 5).

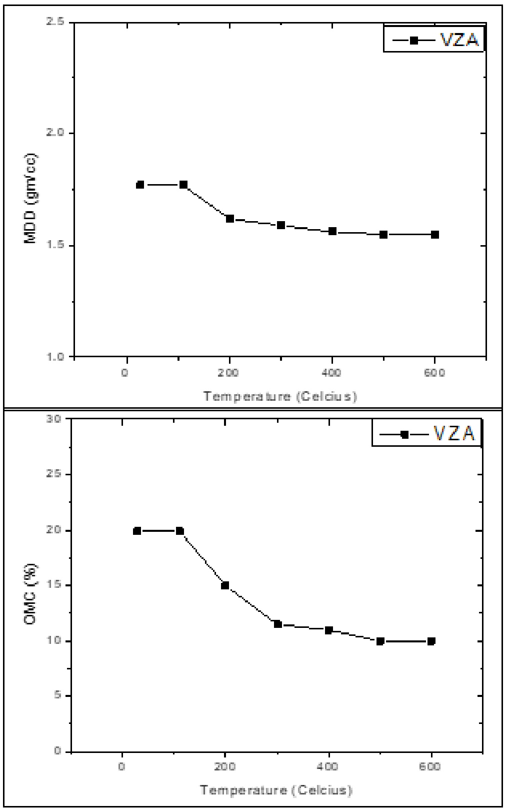


Figure 5: Variation of MDD and OMC with thermal treatment

4.4 Direct shear test

The effect of thermal exposure on the shear strength parameters of the soil shows a distinct transition from a predominantly cohesive material at low temperatures to a friction dominated material at higher temperatures. At ambient conditions (27 °C), the soil exhibits high cohesion (79.4 kN/m²) and a relatively low friction angle (26.1°), values that remain essentially unchanged at 110 °C, indicating minimal alteration to the soil fabric after removal of free moisture. However, heating to 200 °C results in a sharp reduction in cohesion to 37.26 kN/m², accompanied by an increase in friction angle to 36.5°, suggesting dehydration of adsorbed water layers and the beginning of microstructural rearrangement. With further heating to 300 to 600 °C, cohesion decreases drastically 10.51 kN/m² to zero, indicating a breakdown of clay interparticle bonding and loss of electrochemical adhesion. Meanwhile, the friction angle increases steadily, stabilizing at 46° at 600°C, which reflects the transition of the material towards a granular, non-cohesive behaviour. Such trends are consistent with thermally induced mineralogical transformations reported in the literature, where elevated temperatures cause collapse of clay particles, reduction in specific surface area, and increased particle interlocking, resulting in diminished cohesion but enhanced frictional resistance [6]. The observed behaviour highlights that heating fundamentally alters the soil's shear strength mechanism from cohesive soil to frictional making temperature a critical consideration in geotechnical applications (Fig. 6).

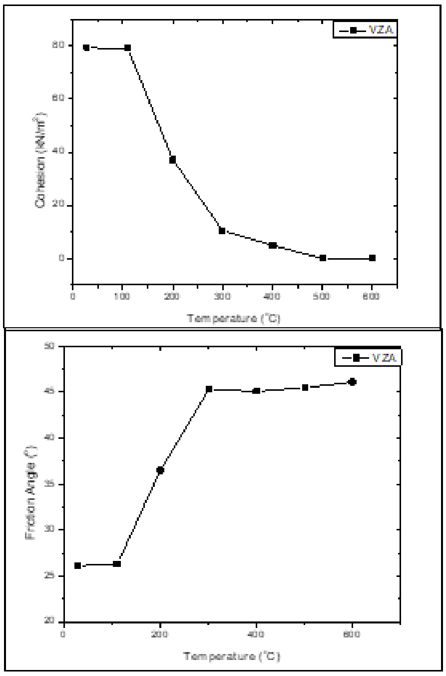


Figure 6: Variation of Cohesion and Friction angle with thermal treatment

4.5 CBR

The soaked CBR values show a pronounced improvement in bearing capacity with increasing temperature, reflecting thermally induced modifications in soil structure. Untreated soil exhibits a low CBR of 2.5 %, characteristic of a highly plastic fine grained soil with weak resistance to penetration. A negligible increase is observed at 110 °C (2.8 %), consistent with the removal of free moisture without significant alteration of clay mineralogy. However, heating to 200 °C results in a dramatic rise in CBR to 14.1 %, coinciding with earlier observations of reduced plasticity, diminished cohesion and increased frictional resistance. This behaviour can be attributed to dehydration of adsorbed water, partial aggregation of clay particles, and consequent densification of the soil matrix. From 300 to 600 °C, the CBR continues to increase, from 14.2% to 21.4%, indicating the development of a rigid, quasi granular structure with enhanced load bearing characteristics. The stable high CBR values beyond 400 °C correspond to reduced cohesion and high friction angles, suggesting that shear resistance is governed primarily by particle interlocking rather than fine clay cohesive bonding. Similar improvements in CBR for thermally treated clays have been documented in the literature, where mineral dehydroxylation and microstructural rearrangement contribute to increased stiffness and bearing capacity [19]. Overall, the results demonstrate that heating significantly enhances the soaked CBR of the soil, making thermally modified soils potentially suitable for subgrade, embankment applications where high strength and reduced plasticity are desirable (Fig.7).

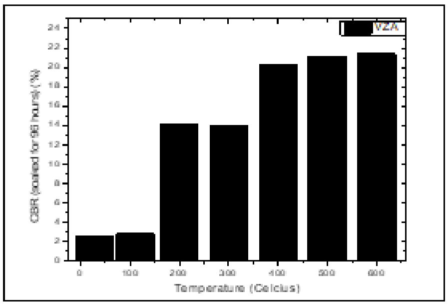


Figure 7: Variation in CBR % with thermal treatment

4.6 CBR Temperature Prediction Model

The relationship between soaked California Bearing Ratio (CBR) and thermal exposure was further investigated using a regression based predictive framework implemented in Python. The experimental dataset was processed and analysed using the NumPy and Pandas libraries, which enabled efficient data handling, statistical evaluation and model fitting. Visualization and model diagnostics were performed to assess prediction accuracy and trend consistency.

Experimental CBR values demonstrated a strong monotonic increase with temperature, rising from 2.5% at ambient conditions to 21.4% at 600 °C. Statistical evaluation indicated that temperature is a dominant predictor of CBR, with a correlation coefficient of r = 0.93**,** confirming a very strong positive association.

To capture the nonlinear strengthening behaviour, a second degree polynomial regression model was developed using the experimental dataset. The resulting prediction equation is

$$\text{CBR} = -5.98 \times 10^{-5} T^2 + 0.07394\, T - 1.0456$$

where CBR represents the predicted soaked California

Bearing Ratio (%) and Tdenotes the thermal treatment temperature (°C).

This model exhibited excellent predictive performance, yielding a coefficient of determination $R^2 = 0.98$ and a root mean square error RMSE = 1.06, demonstrating that 98% of the variability in CBR is explained by temperature alone. Predicted CBR values increased rapidly between 200 to 300 °C, followed by a gradual stabilization towards 600 °C, aligning well with measured values. This behaviour reflects the thermal breakdown of clay minerals, dehydroxylation and transition towards a granular microstructure, as observed in SEM images and supported by XRD evidence.

The prediction table generated from the regression model indicates that CBR is expected to increase from 5.8% at 100 °C to 22% at 600 °C, highlighting the significant enhancement in bearing resistance achievable through thermal alteration. The developed Python based model provides a reliable computational tool for estimating soaked CBR at intermediate temperatures, thereby reducing dependence on extensive laboratory testing (Fig. 8).

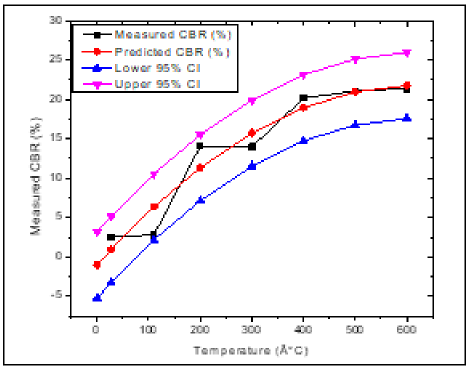


Figure 8: Prediction model for CBR % values

4.7 Color change phenomenon

Visual examination revealed a progressive color change in the soil with increasing thermal treatment temperature. The untreated soil exhibited a dark brown coloration due to the presence of moisture, organic matter and hydrated clay minerals. At 300 °C, the soil changed to a lighter brown shade, indicating loss of adsorbed water and partial oxidation of organic matter. Soil treated at 600°C showed a distinct reddish coloration, attributed to dehydroxylation of clay minerals and oxidation of iron bearing compounds forming hematite. This irreversible mineralogical transformation reflects a transition toward a more granular and stable soil structure. The observed color change is consistent with SEM and XRD results and correlates well with the significant increase in soaked CBR values at higher temperatures (Fig. 9).

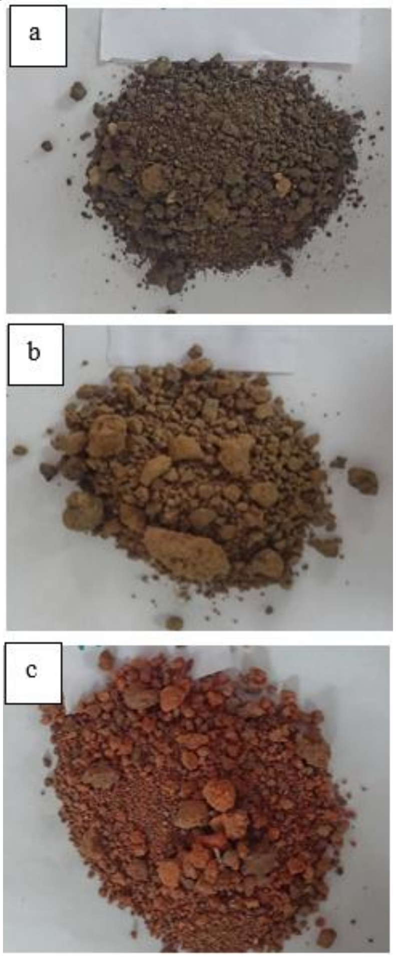


Figure 9: Variation in color change with thermal treatment a)untreated soil b) 300°C c)600°C

4.8 XRD

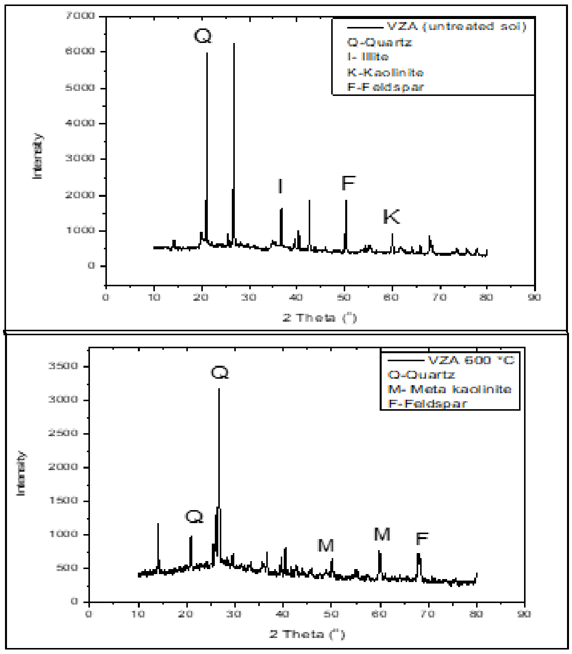


Figure 10: XRD plots showing mineralogical transformation of soil

The X-ray diffraction patterns of the untreated soil and the sample heated to 600 °C reveal significant mineralogical transformations that directly influence the soil's engineering behaviour. The untreated soil displays a well defined assemblage dominated by quartz (SiO_2), illite, kaolinite and minor feldspar peaks. Quartz exhibits characteristic high intensity reflections at 20–27° (2θ), consistent with its crystalline and thermally stable nature. Clay minerals such as kaolinite and illite are present through low to moderate intensity peaks in the ranges 8–12°, 19–22° and 24–25° (2θ), indicating a fine grained, layered silicate structure typical of natural soils.

Heating the soil to 600 °C produces a distinctly modified diffraction signature. While quartz peaks remain sharp and stable reflecting its resistance to thermal decomposition the characteristic peaks of kaolinite diminish substantially. This is consistent with the well documented dehydroxylation of kaolinite at 600°C, which leads to the formation of metakaolin, an amorphous aluminosilicate phase that does not exhibit crystalline XRD peaks. Illite peaks also weaken, indicating structural collapse and loss of interlayer hydroxyl components. The reduction in clay mineral crystallinity results in a lower and broader background intensity, signifying partial amorphization of the soil matrix (Fig. 10).

4.9 SEM

The SEM micrographs provide clear evidence of the microstructural transformations that occur in the soil upon thermal treatment at 600 °C. Both low and high magnification images were analyzed to capture macro and micro scale fabric changes. The untreated soil exhibits a typical clayey microfabric characterized by closely packed morphology. At high magnification (25,000×), the untreated sample shows honeycomb structure, indicative of clay minerals such as kaolinite and illite. These layered structures are associated with physico-chemical interparticle bonding, responsible for the soil's high plasticity, high cohesion and relatively low CBR in its natural state. At lower magnification (200×), the untreated soil presents large, lumpy aggregates interconnected by fine clay bridges, forming a dense and cohesive microstructure (Fig. 11).

In contrast, the soil treated to 600°C shows a markedly altered microfabric. The high magnification SEM image (25,000×) reveals a highly porous, fragmented and granular texture, replacing honeycomb structure observed in the untreated sample. Thermal exposure at 600 °C leads to dehydroxylation of clay minerals, destruction of crystalline clay layers, and formation of amorphous metakaolin, resulting in the breakdown of the clay particles into irregular, flaky particles. The interconnected clay sheets observed earlier are replaced by loosely arranged clusters with abundant micro voids, indicating a substantial loss of cohesive bonds. At lower magnification (200×), the treated soil displays crumbly, granular aggregates and a noticeable increase in grain individuality, confirming the transition from a cohesive clay fabric to a more sand-like, frictional structure (Fig. 12).

Overall, the SEM analysis confirms that thermal treatment at 600 °C fundamentally alters the soil microstructure from a dense, honeycomb clay fabric to a porous, granular and partially amorphous material which directly governs the improved engineering performance documented in the experimental results.

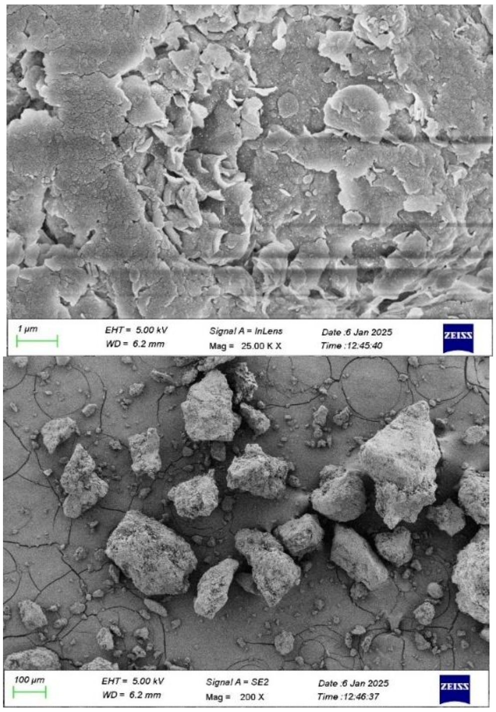


Figure 11: Morphology of Untreated soil

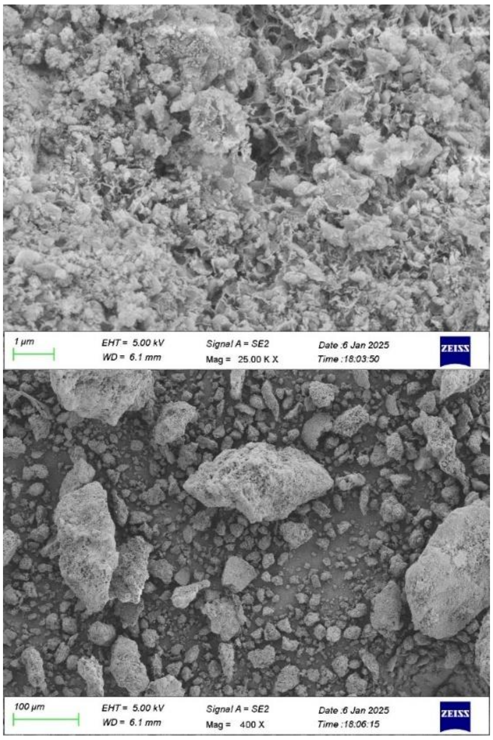


Figure 12: Morphology of 600°C thermally treated soil

4.10 Chemical composition

Thermal treatment at 600°C produced considerable changes in the soil's chemical composition, with SiO_2 decreasing slightly from 61.91% to 59.30%, indicating minor structural disruption of silicate phases. Al_2O_3 remained practically unchanged, reflecting the thermal stability of aluminosilicate minerals. A noticeable increase in CaO (2.52% to 6.12%) and Fe_2O_3 (8.56% to 10.05%) suggests concentration effects due to loss of structural water and partial breakdown of clay minerals. Reductions in MgO and K_2O, along with a slight increase in Na_2O, are consistent with alkali mobility and selective volatilization at elevated temperatures. Overall, the oxide trends support the XRD and SEM findings, confirming partial dehydroxylation and mineral reorganization with compositional alteration. The increase in percentage of CaO may be the possible reason for strength improvement in soil due to thermal treatment (Fig. 13).

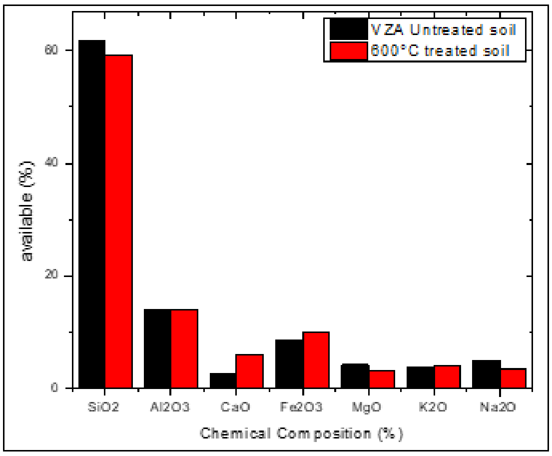


Figure 13: Variation of chemical composition in soil due to thermal treatment

5. Conclusion

This study examined the effects of thermal treatment up to 600°C on the engineering and microstructural behaviour of a natural clay soil. Thermal treatment caused a substantial reduction in plasticity, cohesion and compaction parameters, accompanied by a marked increase in friction angle, indicating a transition from cohesive to friction-dominated behaviour. The soaked CBR increased significantly from 2.5 % in the untreated soil to 21.4 % at 600 °C, demonstrating enhanced bearing capacity due to thermal modification.

SEM and XRD analyses confirmed dehydroxylation of clay minerals, breakdown of crystalline structure, and formation of a granular microfabric responsible for the observed strength improvement. A Python based polynomial regression model accurately predicted temperature dependent CBR variation ($R^2 = 0.98$), providing a practical tool for estimating bearing resistance. Overall, thermal treatment proved effective in improving weak clay soils, with potential applications in fire affected ground, thermal remediation and high-temperature geotechnical systems.

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Declarations
- ***Funding***: No external funding was received for this research
- ***Conflict of Interest***: The authors declare no conflict of interest
- ***Data Availability***: Data available upon request.

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