

RESEARCH ARTICLE

Experimental Investigation Of Geopolymer Composition For Stabilizing Black Cotton Soil

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This research endeavors to optimize an ambient-cured geopolymer blend for enhancing the stability of Black Cotton Soil (BCS) by incorporating fly ash, sodium silicate, rice husk ash (RHA), and ground granulated blast furnace slag (GGBFS). The primary objectives encompass enhancing the shearing behavior of BCS treated with geopolymer and gaining deeper insights into the engineering attributes of BCS. Through experimental endeavors involving nine distinct samples with varied polymer compositions, the study underscores the substantial promise of this geopolymer amalgam for soil stabilization. Analysis of the trials indicates that sample number 5, featuring 20% fly ash, 30% RHA, and 20% GGBFS, manifests the highest compressive strength along with favorable plasticity characteristics.

Keywords: Geo-polymerisation, Black Cotton Soil, Fly ash, rice husk, Plasticity index, Liquid Limit, Compressive Strength.

1. Introduction

Chemical stabilization of soft soils has become a common approach for enhancing particle bonding by introducing binding materials like Portland Cement (OPC) and limestone into the soil. OPC is favored in geotechnical projects due to its appropriate mechanical properties, availability, and cost-effectiveness, being utilized in various stabilization methods like deep concrete mixing and drilling. However, the heavy reliance on cement has led to significant environmental issues such as high CO2 emissions, depletion of natural resources, and dust generation. OPC production is energyintensive, emitting 0.7–1.1 tons of CO2 per ton of OPC manufactured. Additionally, OPC often exhibits drawbacks like higher plastic shrinkage and reduced strength due to water loss and insufficient moisture, especially problematic in arid regions where large-scale wet curing is impractical. To mitigate these environmental concerns and improve mechanical performance, OPC is partially substituted with materials like fly ash (FA), GGBS, red gypsum, rice husk, and recycled glass powder. While these substitutions enhance moisture resistance and reduce shrinking, the environmental

impact of OPC remains a challenge.

Geo-P has emerged as a viable alternative to OPC by repurposing aluminosilicate-rich contaminants into value-added binding materials. Geopolymers stabilized soils exhibit superior qualities such as compact microstructures, enhanced mechanical properties, and volume stability, meeting the requirements for engineered clayey soil. Various mixing designs have been evaluated to assess the mechanical characteristics of geopolymers stabilized clayey soils, demonstrating significantly lower shrinking strain compared to non-stabilized or OPC-incorporated soils. The reduced shrinkage is attributed to the slower evaporation of pore fluids from the stabilized soil structure. Soil stabilization involves altering soil molecular characteristics to enhance stability and durability, allowing for sustained structural load transfer without failure during its service life. Chemical or mechanical procedures have traditionally been employed to remediate expansive soil, with chemical treatments utilizing soil stabilizers like limestone, concrete, bitumen, and fly ash. However, not all treatments are suitable for all soil types, with recommendations based on soil state, structure, and qualities.

Chemical treatment methods can withstand harsh environmental conditions like acid rain compared to mechanical stabilization methods but are not considered eco-friendly. Therefore, an environmentally acceptable chemical treatment

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method known as "Geopolymer" is sought to reinforce expansive soil for construction industry applications. The primary technique for soil stabilization via geopolymerization involves mixing untreated soil with a liquid activator containing slag or fly ash, along with an alkali activator such as sodium silicate (Na2SiO3), sodium hydroxide (NaOH), or both, resulting in stabilized soil with or without gel formation.

Fig. 1 Shows the basic procedure of Geopolymerization for Soil Stabilization

Various activating agents, such as alkali-silicate or hydroxyl compounds, are incorporated in powdered form along with binders to fabricate solid aluminosilicate components. Scholars have categorized the term "geopolymer" into four classifications: alkalibound ceramics, aqueous ceramic materials, earthy cemented, and biopolymers. The investigation into the reaction mechanism of geopolymer, abbreviated as Geo-P, dates back to 1978, aiming to broaden its application as a synthetic polymer composite in various manufacturing sectors. The molecular structure of Geo-P, depicted in Figure-2, illustrates a complex network of interconnected strings of naturally occurring materials linked by covalent bonds. These interconnected materials encompass Poly Silicone, Poly-Silixo, Poly Sialate, Poly-Silalate-Siloxo, Poly-Phospho-Sialate, Poly-Phospho-Siloxo, Poly-Phosphate, and Poly-Sialate-Disiloxo, all interconnected via covalent bonds, as depicted in Figure-2.

Fig. 2 Geopolymer molecular unit

Duxson et al. [23] introduced a polymerization technique encompassing multiple phases outlined in the conceptual model. Figure 3 illustrates the comprehensive framework of geopolymerization. The model postulates that the geopolymerization process initiates with the dissolution of source materials in alkaline solutions, leading to the rupture of alumino-silicate bonds and the subsequent release of silica and alumina, primarily from the primary sources [24][25]. Alkaline potassium ions, such as potassium, sodium, or limestone, neutralize the negatively charged aluminosilicate chain, influencing the efficacy of geopolymerization. Moreover, as the alkalinity of the solution increases, the dissolution of materials also rises. This dissolution rate determines the time required to achieve saturation, upon which the aluminosilicate solution becomes supersaturated [26][27].

Subsequently, the main condensation process commences, precipitating aluminosilicate gel as oligomers, forming larger and more stable networks. With higher concentrations of Si and Al in the solution, the formation of initial polymers (Gel 1) occurs. As the reaction progresses, a greater influx of Si into the solution leads to increased Si concentrations in the gels (Gel 2). Upon surpassing the dissolution rate, the initial setting phase is triggered as the condensation rate of aluminosilicate species exceeds. Ultimately, polycondensation and rearrangement activities produce increasingly interconnected 3D networks, culminating in the final geopolymer matrix.

Experimental testing procedures on black cotton soil involved the assessment of Liquid Limit, Unconfined Compressive Strength (UCS), Plastic Limit, and Plastic Index using varying ratios of GGBS, RHA, and FA. Nine soil samples were prepared, each subjected to curing periods of 7, 14, or 28 days. The research paper compares the findings obtained from the testing procedure across

different curing durations and compositions of RHA..

2. Materials and Methods

The purpose of this work is to assess how well geopolymer stabilization improves the engineering properties of black cotton soils as a more environmentally friendly alternative to conventional binders. A thorough testing program has been developed to examine the efficiency of soils that have been geopolymer-treated in black cotton. In this chapter, the methods needed to prepare treated soil specimens are covered in detail, along with the raw materials utilized, the geopolymer admixture, the preparation procedure, and the preparation methods. Along with that, it provides details on the experiments that were run and the methodology that was employed. Due to the numerous tests and particular sample preparations needed, it offers indepth insights into the sample preparation procedure for each experiment. After that, the entire testing procedure is carefully examined.

Fig. 3 Flow Chart of the proposed Work

Black cotton soil, also referred to as expansive clay soil, is notable in engineering because of its high clay content and distinctive characteristics. The term "geotechnical properties" refers to a variety of soil and rock traits and behaviors that are crucial for building and engineering applications. These characteristics include soil classification—which classifies soils according to their plasticity and particle sizes—as well as soil composition, which determines the soil's permeability, strength, along with compressibility.

Table 1 Geotechnical Characteristics of Black soil

Geotechnical Property	Value	IS Code		
Shrinkage Limit	$10 - 25$	IS 2720 (Part 2): 1973		
Optimum Moisture Content	12 - 18% (approximately)	IS 2720 (Part 8): 1983		

Engineers use a variety of methods to get around problems with soil stabilization. One noteworthy method is the use of geopolymer stabilization, which entails mixing agricultural waste (RHA - rice husk ash) geopolymer with black cotton soil and materials like fly ash, sodium silicate, agricultural waste (fly ash), as well as ground granulated blast furnace slag (GGBFS). This method will improve the soil's ability to drain, reduce its susceptibility to shrink-swell, and increase its load-bearing capacity.

3. Ground Granulated Blast Furnace Slag (GGBS)

GGBS, or ground granulated blast furnace slag, is a by-product of the iron-making procedure in blast furnaces used to manufacture iron and steel. As an

additional cementitious material (SCM), GGBS is frequently used in construction to supplement Portland cement. In order to create GGBS, molten blast furnace slag must be cooled and quenched with water or air, creating a granulated product. To create GGBS, the granulated slag is subsequently pulverized into a fine powder. The slag's cementitious qualities are enhanced and its reactivity is raised during the grinding process. Pozzolanic and latent hydraulic characteristics of GGBS are well recognized. In concrete, when GGBS is used to substitute cement, it combines with the calcium hydroxide that is produced during cement hydration to create more hydration products. The durability and strength of soil are improved by these compounds.

Fig. 4 GGBS

3.1 Fly Ash

Due to its beneficial properties, fly ash is frequently used as a soil stabilizer. Fly ash improves the engineering properties of soil when it is added,

increasing stability and reducing settlement. Fly ash makes a substantial impact through enhancing soil particle cohesiveness.

Fig. 5 Fly Ash

The soil's cohesiveness and strength are increased as a result of the fly ash's fine particles filling up the soil's voids. Further enhancing cohesiveness and stability are cementitious chemicals that can be produced as a result of chemical reactions between fly ash and soil minerals. The flexibility of cohesive soils, like clay, is also decreased by fly ash because it binds to the clay particles. Due to lessening swelling or shrinkage, the soil is less susceptible to volume variations. By making loose or weakly compacted soil denser, fly ash also makes it easier to enhance compaction, which improves stability and loadbearing capacity. Fly ash also has limited permeability, which contributes to a reduction in the amount of water that passes through the soil. This property is especially useful for stabilizing soils that are prone to erosion or places where controlled water flow is necessary.

Property	Approximate Value		
Physical Properties			
Particle Size	2 - 100 micrometers		
Specific Gravity	$2.1 - 2.8$		
Color	Light Grey		
Moisture Content	Typically $<$ 3%		
Chemical Properties			
Silica (SiO2)	$30\% - 60\%$		
Iron oxide (Fe2O3)	$5\% - 20\%$		
Alumina (Al2O3)	$15% - 35%$		
Calcium oxide (CaO)	$1\% - 12\%$		
Magnesium oxide (MgO)	$< 5\%$		
Sulfur trioxide (SO3)	$< 5\%$		
Loss on Ignition (LOI)	Typically $\lt 5\%$		

Table 3 Physical and Chemical Properties of Flyash (IS 3812 (Part 1): 2013)

In specific instances, the activation of fly ash with alkali can lead to notable improvements in strength and thereby augment soil stability. Nonetheless, the efficacy of fly ash in stabilizing soil is contingent upon variables such as soil characteristics, fly ash makeup, blending proportions, and curing circumstances. Hence, it is imperative to undertake laboratory analyses and on-site assessments to ascertain the most suitable blend configurations and deployment strategies to attain the desired stabilization results.

3.2 Rice Husk Ash

An undesired by-product of rice milling is rice husk ash (RHA). An environmentally suitable substitute for final disposal is use as a soil stabilizer. When the organic components and water in the rice husk are burned, about 20% of the mass is left over as rice husk ash (RHA). RHA would be produced in the amount of 20 million tons per year if all rice husks had been burned. This residue's valuation is an environmentally beneficial alternative to its final disposal.

When rice husks are burned, a byproduct known as rice husk ash (RHA) is produced. It has a number of beneficial qualities. With a normal level of over 90%, RHA is abundant in amorphous silica. RHA's pozzolanic reactivity—the ability to combine with calcium hydroxide and generate additional cementitious compounds when there is moisture present—is a result of its high silica concentration. RHA may be used as an additional cementitious material for the enhancement of strength, longevity, and functionality of goods made from cement due to this characteristic. In addition, RHA has a high surface area, that further improves its reactivity as well as potential for use in various applications.

Fig. 6 Ash from rice husks (a) as received; (b) after six hours of burning at 700 °C.

RHA has high thermal insulation properties in addition to its pozzolanic and particle size features. This can help stabilize soil in areas with large temperature swings. It can lessen the amount of heat that is transferred through the soil and aid to mitigate temperature-related problems like frost heave or excessive heat-induced expansion.

Fig. 7 Particle Size Distribution of RHA

3.3 Alkaline Liquid

Alkaline substances play a substantial role in the geopolymerization process, which is crucial for soil stabilization. Similar chemical characteristics are shared by these compounds, such as sodium hydroxide (NaOH) or sodium oxide (Na2O). When they come into contact with acids, they react exothermically, forming salts and water as a result. They result in solutions with pH values greater than 7.0 when dissolved in water. Normal combinations

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of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and potassium silicate (K2SiO3) for geopolymerization. In this experiment, an alkaline liquid was created by combining sodium hydroxide and sodium silicate.

By blending sodium hydroxide solution with sodium silicate solution, the alkaline liquid used in this experiment has been produced. A local supplier was used to get sodium hydroxide, which was bought in the form of flake with a purity of 93–95 percent. To get the desired concentration for the solution, the solid flakes were dissolved in water. In a similar manner, a local supplier provided the sodium silicate solution.

Fig.8Highly Alkaline Liquid

Fig. 9 Pellets of NaOH

4. Experimental Setup and Testing

The soil specimens were fabricated using predetermined mixtures derived from Taguchi analysis. Accurate quantities of fly ash, GGBS, and RHA were incorporated into the soil following meticulous calculations. Subsequent to sample preparation, a battery of tests was conducted on the stabilized soil samples. These assessments encompassed the unconfined compressive strength, plastic limit, liquid limit, and plasticity index of the treated soil. Multiple replicas were generated for each composite blend to ensure precision in the

findings. Statistical scrutiny, including the calculation of mean values and standard deviations, was employed to analyze the data and derive relevant conclusions. The curing regimen was meticulously observed during the experimental phase, with tests conducted at 7, 14, and 21-day intervals to monitor strength evolution. Through this systematic experimental methodology, the study aimed to furnish valuable insights into the optimal formulation for stabilizing black cotton soil using fly ash, GGBS, and RHA.

4.1 Loading Setup

a. Position the soil specimen within the UCT apparatus, which generally comprises a loading framework, a load sensor, and a mobile platen.

b. Verify that the sample is correctly positioned and centralized within the loading framework to prevent any off-center loading.

4.2 Initial Measurement:

a. Adjust the mobile platen to make initial contact with the soil specimen without exerting any pressure.

b. Document the initial measurement from the load sensor to establish a baseline.

4.3 Axial Loading:

a. Employ the loading framework to initiate axial loading on the soil specimen at a consistent velocity (e.g., 1.25 mm per minute).

b. Maintain a continuous observation of both load and deformation (strain) readings during the entire testing process.

c. The application of axial loads induces axial compression in the sample, resulting in shear failure**.**

4.4 Shear Failure:

a. Monitor the response of the soil specimen throughout the test. Typically, there is an initial increase followed by a decline in the applied load after reaching a maximum value.

b. Proceed with loading until either complete failure of the soil sample occurs or until the axial strain attains a predefined threshold (e.g., 10%).

4.5 Calculation of Unconfined Strength:

• Determine the maximum load (P) sustained by the soil sample during the test.

• Calculate the stress (σ) by dividing the maximum load (P) by the cross-sectional area (A) of the sample:

Stress (σ) = Max Load/
\n
$$
\frac{P}{\sigma} = \frac{P}{A}
$$

c. The unconfined strength (qu) is equal to the maximum stress (σ) sustained by the soil sample

Fig. 10 Liquid Limit Flow Curve

The moisture content equivalent to 25 drops must be read from the curve after performing the liquid limit test and figuring out how many drops are needed to close the groove. The soil's liquid limit (wL) will be given as the moisture content value, rounded to the nearest whole number.

S1. No.	Observations and Calculations	S ₉	S ₈	S7	S6	S5	S ₄	S ₃	S ₂	S1
1	Mass of empty container (M1)	25 gm	25 gm	25 gm	25 gm	25 gm	25 gm	25 gm	25 gm	25 gm
2	Mass of container $+$ wet soil (M2)	180 gm	182 gm	179 gm	180 gm	183 gm	181 gm	178 gm	184 gm	182 gm
3	Mass of container $+$ dry soil (M3)	159 gm	161 gm	158 gm	159 gm	162 gm	160 gm	159 gm	165 gm	162 gm
Calculations										
4	Mass of dry soil $(MD) = M3 - M1$	134 gm	136 gm	133 gm	134 gm	137 gm	135 gm	134 gm	140 gm	137 gm
5	Water content (w) (6)] x 100 $=$ [(5)	15.79%	15.56%	15.91%	15.67%	15.22%	15.56%	14.39%	13.86%	14.71%
6	Mass of water $(MW) = M2 - M3$	21 gm	21 gm	21 gm	21 gm	21 gm	21 gm	19 gm	19 gm	20 gm

Table 6Moisture content determination of all th 9 samples

5.Plastic Limit Determination

To prepare the soil sample for the plastic limit test, following steps are being followed:

⦁ Select a portion of the soil sample for the liquid limit test, ensuring it weighs at least 20 grams to ensure representativeness of the overall soil composition.

⦁ Continuously spread or mix the soil on a ground glass plate or in a mixing/storage dish to reduce its moisture content. The aim is to achieve a consistency that allows the soil to be formed into threads without adhering to hands.

⦁ Employ an electric fan's airflow during soil sample preparation to expedite the drying process. Position the fan so that it directs airflow onto the soil sample, aiding in the evaporation of excess moisture.

⦁ Monitor the moisture content of the soil closely during preparation. The objective is to attain a workable consistency without excessive stickiness. It is crucial to avoid significant alterations in soil properties or over-drying.

⦁ By spreading or mixing the soil sample on the glass plate or in the mixing/storage dish, uniform exposure of soil particles to air is ensured, facilitating moisture evaporation. While electric fans can accelerate drying, caution must be exercised to prevent excessive drying that could alter soil behavior.

The soil's consistency and attainment of the desired plasticity necessary for the plastic limit test should be consistently monitored throughout the preparation process. By adhering to these guidelines diligently, effective soil sample preparation can be achieved, ensuring accurate plastic limit testing and reliable outcomes.

6. Unconfined Compressive Strength at 7, 14, and 28 Days: Comparative Analysis

Utilizing Rice Husk Ash (RHA) as a stabilizing agent has an effect on soil's unconfined compressive strength (UCS), which is what the comparative analysis seeks to determine. This analysis' primary goal is to compare the UCS values at varied curing times.

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Experiment	FA	GGBS	RHA	Unconfined	Unconfined	Unconfined	
Runs	$\binom{0}{0}$	$\binom{0}{0}$	$\binom{0}{0}$	Compressive Strength	Compressive Strength	Compressive Strength	
				after seven days of	after fourteen days of	after twenty-eight days	
				curing (in MPa)	curing (in MPa)	of curing (in MPa)	
S ₁	10	10	Ω	8.300	8.500	8.700	
S ₂	20	10	15	7.3333	7.6333	7.8333	
S ₃	30	10	30	8.0533	8.2533	8.6533	
S ₄	10	20	15	8.400	8.500	8.800	
S ₅	20	20	30	8.2867	8.6867	8.8867	
S ₆	30	20	0	8.530	8.330	8.430	
S7	10	30	30	7.4333	7.6333	7.9333	
S ₈	20	30	Ω	7.300	7.500	7.400	
S ₉	30	30	15	6.950	7.200	7.450	

Table 7 Unconfined Compressive Strength of Concrete Mixtures with Various Cementitious Materials

The table compares the compositions of various soil samples (labeled as S1 to S9) and their unconfined compressive strengths (UCS) values after 7, 14, and 28 days of curing. The goal is to identify the sample that exhibits better performance based on its composition. Samples have composition of Rice Husk Ash (RHA), fly ash (FA), along with ground granulated blast furnace slag (GGBS).

Fig. 11 Results of UCS with different curing time of the 9 testing samples

The above graph shows that, Sample S5 includes 20% FA, 20% GGBS, and 30% RHA, achieving the highest UCS value of 8.7 MPa with a curing time of 7 days. This sample achieved a UCS of 9.1with

curing time of 14 days and a UCS of 9.3 MPa, indicating the highest strength among all the samples tested at the 28 days of curing period.

Fig. 12 Plasticity index of the 9 samples at a curing time of (a) 7 days, (b) 14 days and (c) 28 days

The experimental evaluation of the plasticity index over the different course of during time period are being recorded in the figure (a), (b) and (c). The plots are then accessed to determine the soil behaviour and its strength evaluation.

Fig. 13 Comparative analysis of the plasticity Index with curing time of the 9 samples under testing

Figure refers to a graphical representation of how the plasticity index of a soil sample changes over various curing periods. The plasticity index plot at different curing times is a valuable tool for understanding how soil properties evolve over time with controlled moisture exposure. It has be observed that the curing time significantly improves the plasticity index of the samples.

Fig. 14 Liquid Limit of the 9 samples at a curing time of (a) 7 days, (b) 14 days and (c) 28 days

The various experimental outcomes of the liquid limit for the 9 samples of the experiments are being recorded in the figure. The values calculated at different curing time of the samples with same mix ratios have been represented by plots.

Fig. 15 Comparative analysis of the Liquid Limit with curing time of the 9 samples under testing

Figure depicts the dynamic behaviour of a soil sample's liquid limit as it transmits through several curing durations. The plot comprises of a number of curves or lines, each of which represents the liquid limit at a certain curing time. These lines

demonstrate how the liquid limit changes throughout curing, offering details on how the soil reacts to moisture content and time-dependent processes.

Experiment	RHA	Unconfined Compressive	Unconfined Compressive	Unconfined Compressive		
Runs	$\binom{0}{0}$	Strength after seven days	Strength after fourteen days	Strength after twenty-eight		
		of curing (in MPa)	of curing (in MPa)	days of curing (in MPa)		
S ₁	θ	8.300	8.500	8.700		
S ₆	θ	8.530	8.330	8.430		
S8	Ω	7.300	7.500	7.400		
S ₂	15	7.3333	7.6333	7.8333		
S ₄	15	8.400	8.500	8.800		
S9	15	6.950	7.200	7.450		
S ₃	30	8.0533	8.2533	8.6533		
S7	30	7.4333	7.6333	7.9333		
S ₅	30	8.2867	8.6867	8.8867		

Table 8 UCS with increasing ratios of RHA and different curing time

The table compares the values for Unconfined Compressive Strength (UCS) for soil samples that included and did not contain Rice Husk Ash (RHA). The criteria considered in the comparison are the UCS values obtained with and without RHA, the % of RHA content in the mix, and the curing period in days. The mix design of the soil is also taken into consideration. RHA is added to the soil mixture, and it can be seen that the UCS improves as a result.

7 .Conclusion

 A myriad of soil-related challenges, including soil weakness, instability, inadequate drainage, erosion, and contamination, necessitate the utilization of soil stabilization methodologies. This study underscores the importance of employing appropriate techniques to ensure sound construction practices. The utilization of ambientcured geopolymers incorporating fly ash, GGBFS, and RHA proved highly effective in augmenting the engineering properties of Black Cotton Soil. Experimental findings underscored the substantial potential of this geopolymer blend in soil stabilization.

 Measured unconfined compressive strength (UCS) values from experimental trials demonstrated the progressive development of strength in geopolymer-treated BCS across various curing durations. Optimal UCS of 9.3 MPa, indicating a notable enhancement in strength properties, was achieved at the most effective curing duration. The difference between the liquid limit and plastic limit, quantified as 7.72, was determined through plasticity index analysis. It was observed that the liquid limit, denoting the moisture content at the boundary between liquid and plastic states, stood at 48.00%. Meanwhile, the plastic limit, indicating the moisture content at the transition between plastic and semi-solid states, was identified as 55.72%. These metrics shed light on the plasticity and moisture retention capabilities of geopolymertreated BCS.

 Among the experimental compositions, formulation S5, comprising 20% fly ash, 20% GGBFS, and 30% RHA, exhibited the highest UCS and commendable plasticity attributes. This composition demonstrated enhanced malleability and increased strength, rendering it a suitable option for stabilizing Black Cotton Soil.

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