Experimental study on black cotton soil treated with xanthan gum and palm oil fuel ash

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Abstract: This paper presents the results of the laboratory evaluation of black cotton soil stabilized with Palm Oil Fuel Ash(POFA) and Xanthan Gum(XG), to determine its suitability as reliable construction material. POFA, a major byproduct of the palm oil industry is being dumped into open fields which can cause environmental problems. Using POFA as an additive in soil stabilization can be an eco-friendly and sustainable practice.XG which is generally used as a thickening agent in the food and pharmaceutical industries is used in this study as a binding material. Several influential properties such as UCS, CBR, Compaction characteristics, and Atterberg limits were found for control samples and compared with the samples treated with different percentages of POFA and XG ratio and on different curing periods. The tests are performed by varying the POFA (0-25%) while keeping the XG constant at 1% to the weight of dry soil. The specimens were cured for different curing periods. There is a significant change in the shear strength of the soil as the UCS value increased by 374% and the CBR value increased by 379% when the control sample is compared with those of treated samples with 20% POFA+1% XG at 90-day curing period.

Keywords: Black Cotton soil, Palm oil, Fuel Ash, Microstructural Analysis, Xanthan gum

1 INTRODUCTION

The third major kind of soil in India is black cotton soil (BCS), also referred to as regur soil or tropical chernozems in most other parts of the world. Nearly 25% of the soil in Andhra Pradesh and 15% of the soil in India are black cotton soils. Because of their high moisture retention capacity and problematic swelling and shrinkage behavior, black cotton soils are difficult to work with in civil engineering projects. These soils often include more than 60% clay, which significantly increases the soil's capacity to swell. In order to improve unwanted characteristics, such as shear strength and soil swelling potential, soil stabilization is applied. Lime and cement, two common stabilizers, have lost their economic and environmental viability[6]. Including industrial wastes in construction materials is a good way to get rid of those waste and get a sustainable and economical construction material.

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Innovative soil development techniques that are environmentally friendly throughout the duration of their useful lives are being researched. Due to growing environmental concerns, using natural and synthetic polymers to treat soil has grown more popular. Synthetic polymers are created by the chemical industry, whereas biopolymers are created by living things. The use of polysaccharides in soil improvement outperforms other different collections of biopolymers in the field. In nature, polysaccharides are often exploited as gel-forming and food-thickening agents. Polysaccharide xanthan gum is frequently employed as a viscosity-thickening agent because it absorbs more water and creates hydrogen bonds between particles. Shear strength can be increased by lowering hydraulic conductivity and raising the liquid limit[3].

In recent years, there has been a major increase in the use of alkali-activated binders worldwide. Alkali-activated binders are often used in pavement and geotechnical applications. In order to create these binders, amorphous alumino-silicate source materials like fly ash, palm oil fuel ash, rice husk ash, metakaolin, calcium carbide residue, and ground granulated blast furnace slag are mixed with an alkali solution, usually sodium hydroxide or potassium hydroxide, and alkali earth metals. An amorphous alumina-silicate gel is created by dissolving mineral alumina-silicates, followed by the hydrolysis and condensation of the aluminum and silicon components. The development of these alkali-activated binders was first proposed by Davidovits in 1988 and 1991, and further research was conducted by Yunsheng et al. in 2008. These binders offer advantages such as reduced carbon dioxide emissions and potential cost savings through the use of waste materials as source materials. However, their chemistry and curing behavior can be complex, and ongoing research is being conducted to improve their performance and durability[7].

Xanthomonas campestris secretes xanthan gum, a hydrophilic polymer with a high molecular weight (1.106 g/mol) that is created by the fermentation of carbohydrates. This naturally occurring cellulose derivative has a trisaccharide side chain made up of one glucuronic acid residue sandwiched between two mannose molecules and a cellulose backbone made of four β -D-glucose units connected at the 1 and 4 positions[3]. The nonterminal D-mannose group (near the backbone) may have an acetyl function, whereas the terminal D-mannose group may have a pyruvate function. Depending on the fermentation conditions, different levels of acetylation and prenylation occur.Due to the presence of both glucuronic acid and pyruvic acid groups in the side chain, the polymer has an anionic nature[4]. As a suspending and stabilizing agent, xanthan gum is frequently employed in oral and topical medicinal formulations, cosmetics, and food products. When in contact with moisture, the free Na+, K+, or Ca2+ ions present cause the Xanthan gum to exhibit an anionic activity. Because of its anionic properties, xanthan gum might be a useful alternative to traditional alkaline activators like KOH, NaOH, and CaOH[7].

In 2009, Tangchirapat et al. investigated the effects of partially substituting POFA for cement on the compressive strength and sulfate resistance of mortars. In comparison to OPC type I, compression strength improved by 102%–104% with 10% POFA. The rate of mortar expansion in the first year of life was greatly reduced when ground POFA (10–40%) was used as a cement substitute, although the compressive strength was lower than for their reference mortars[1]. In the realm of soft soil stabilization, POFA has been employed as a cement substitute to increase Atterberg limits and unconfined compressive strength[4]

There isn't much information in the literature about the strength and durability of soils treated with Xanthan Gum. The purpose of this study is to confirm the engineering

effectiveness and efficiency of soil treated with Xanthan gum biopolymer. To determine the strengthening properties of black cotton soil treated with a mixture of Xanthan gum biopolymer and palm oil fuel ash, experimental tests were conducted. To confirm the viability and sustainability of soil treated with Xanthan gum and palm oil fuel ash, longterm durability was examined. On samples of black cotton soil, the effects of Xanthan gum (1%), based on literature, and palm oil fuel ash (0%, 10%, 15%, 20%, and 25%) were examined. A series of UCS tests were looked at for different curing days (7, 14, 28, 56, 60, and 90). Microstructural investigation using an X-ray diffractometer and a scanning electron microscope.

2 MATERIALS AND METHODS

2.1 Materials

2.1.1 Black Cotton Soil

Black cotton soil, or BCS, was procured for this experimental inquiry fromVaddeswaram in Andhra Pradesh. All samples are taken in a disturbed state at a depth of fewer than 3 meters. The collected soil samples were air-dried for 24 hours, processed in accordance with IS 2720: BIS, I. (1983) (Part I) [25], and then tested in a lab. The unified soil classification system (USCS) classifies black cotton soil as clay with high plasticity (CH).

2.1.2. Xanthan Gum

The fine powder version of xanthan gum was purchased from Urban Platter in west Mumbai. Xanthan gum is a biodegradable, nonflammable, and environmentally beneficial substance [13].Most often, microbes like bacteria, fungi, and algae make biopolymers. Xanthomonas campestris, a bacteria, secretes xanthan gum [14]. This material was selected for this investigation due to its special qualities, such as its pseudo-viscous and bioclogging nature and ease of availability [15].

2.1.3. Palm Oil Fuel Ash

In order to eliminate nutshells and fibers that were only partially burned after the oven, the OPFA was passed through a sieve with a 425μ aperture in line with ASTM C702-98 (2003) for this study. The POFA used in this study was gathered from the Ruchi Soya Industries Limited in the Indian state of Andhra Pradesh's Ampapuram, Krishna district. This LOI score indicates that a significant amount of organic material was not completely burnt during incineration [5]. In order to get rid of the extra unburned carbon that could impact the potential pozzolanic qualities, the heating process was implemented as the second step of the treatment process [8]. The XRF technique was used to conduct chemical analysis. According to Table 1, the three primary pozzolanic oxide compounds (Fe2O3, Al2O3, and SiO2) made up 71.25% of all the oxide compounds in the ground POFA [16]. The pozzolanic material must include at least 70% of the total amount of the three major oxides, per ASTM C 618. (i.e.Fe₂O₃, Al₂O₃, and SiO₂). Such proof supports the treated POFA's appropriateness as a pozzolanic additive for ground improvement applications [2].

Composition	BCS	POFA		
SiO ₂	41.25	66.25		
Al ₂ O ₃	37.06	3.5		
Fe ₂ O ₃	11.2	1.5		
CaO	0.2	5.58		
K ₂ O	11.6	5.6		
MgO	0.5	5.4		
TiO ₂	0.88	1.96		
Na ₂ O	1.337	0.25		
MnO	0.2	0.56		
P ₂ O ₅	0.096	4.32		
Cr ₂ O ₃	0.02	N.D		
Rb ₂ O	0.04	0.006		
LOI	N.D	9.5		

Table 1: Chemical composition (% by weight) of BCS and POFA

2.2. Specimen Preparation

The index properties of several mixed samples of BC, POFA, and XG were determined using particle-size distribution, Atterberg limits, and specific gravity tests. These characteristics are used to categorize the different mixtures using the Unified Soil Classification System and the American Association of State Highway and Transportation Officials (AASHTO) soil classification methods[6]. Additionally, the mixtures are subjected to compaction to determine their moisture-density relationships, as per the guidelines provided in the Indian standards IS; 2720. The results of CBR and unconfined compressive strength (UCS) tests, which were performed in accordance with the instructions specified in their respective ASTM codes, were used to evaluate the strength characteristics of the different BC + POFA + XG mixes. All of the treated samples are added with 1% xanthan gum to the mixture ratios, which also include the untreated soil sample and a POFA (additive) addition in a range of 0% to 25% (i.e., 10, 15, 20, and 25%).

2.2.1.Atterberg Limits

The Atterberg limits composed of the liquid, plastic, and shrinkage limits - were calculated for each BC + POFA + XG mix using the material that passed the 0.425 mm screen in order to determine the mix's affinity for water [17][18].

2.2.2. Specific Gravity

The specific gravities of the various BCS + OPFA + mixes were calculated in accordance with I.S. 2720, part 2 by passing approximately 50-100 g of the oven-dried samples through a sieve with a 2.0-mm aperture (2015).

2.2.3. Compaction Characteristics

To evaluate the moisture-density connection of the soil, a variety of BCS + POFA + XG mixtures were put through a compaction test utilizing the I.S. 2720, part 16. This was accomplished by applying 25 evenly spaced blows of a rammer weight weighing 4.5 kg descending through a height of 450 mm to each of the sample's five layers inside the compaction mold measuring 101.6 mm in diameter and 116.43 mm in height [19]. The preselected soil proportion should, in general, be strong enough to support traffic loads, reasonably compressible to prevent major future settling, stable against change in volume as water content or other elements alter, long-lasting and safe against deterioration, and possess desired permeability [6].

2.2.4. California Bearing Ratio

Following the steps indicated in I.S. 2720, part 16, the CBR test was carried out on the compacted samples of the various BCS + POFA + XG mixes under strain control. As a percentage of a solid limestone rock, which is given the value of 100, the measured resistance is stated. To give the highest value under ideal circumstances, samples were tested both after a saturation period (soaked, which creates the worst-case scenario for combinations including clays) and when unsoaked [10].Despite the disparagement of its practical nature, the CBR approach has gained widespread acceptance as the procedure for estimating a soil's or soil-aggregate mixture's strength for use in pavement design calculations in developing nations [1]. This process evaluates the resistance to applied traffic loads displayed by laboratory compacted samples of the various BCS + POFA + XG mixes.

2.2.5. Unconfined compressive strength

The different samples of the LS + OPFA + C mixes were compacted using the BSH compaction energy, removed from the split UCS mold, trimmed to a height of 76 mm with a knife edge, and put through the UCS test for the first day. However, in order to ensure consistent moisture dispersion and to cure at a constant temperature of 25° C, the other trimmed compacted samples were double-wrapped in polyethylene bags and maintained in the humidity chamber. The guidelines for using these indexes for highway purposes are taken from IS:2720 (Part-10):1991.[9]

2.2.6. Microstructural Analysis

In this study, the growth of the polymerization structure on the specimens was examined using XRD, FTIR spectra, and SEM. Phases contained in the specimens were identified using XRD, and various chemical bonds found in the materials at the molecular level were identified using FTIR. The Bruker D2 Phaser was used to do an XRD study from 5 to 80 of 2°. Software called Expert High Score Plus was used to analyze the diffraction patterns. The FTIR Spectrometer Tensor 27 was the tool used for FTIR [13]. 95 mg of potassium bromide and 5 mg of powdered samples were combined (KBr).In order to create the translucent pellets, the powder was hydraulically compacted for 2 minutes at 2.758 MPa before being placed into the infrared spectrometer[11]. Infrared spectra between 550 and 5000 cm-1 in wavelength were captured. Small pieces of the SEM/EDX specimens were broken away from the center. The specimens were then frozen at 195°C by immersion in liquid nitrogen for 5 minutes, and moisture was removed from the solid state by vacuuming at a pressure of 0.5 Pa at 40°C for 5 days [12]. All specimens were coated in gold before

SEM examination. Zeiss's Merlin compact and Aztec's Oxford were used for the SEM analysis[5].

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Properties	Value						
Gravel	-						
Sand	22%						
Clay and silt	78%						
Specific gravity	2.61						
Liquid limit	62%						
Plastic limit	27%						
Shrinkage limit	17%						
Plasticity index	35%						
Type of soil	СН						
Optimum moisture content	16.5%						
Maximum dry density	1.78g/cc						
California bearing ratio							
a. Soaked cbr b. Unsoaked cbr	4.00% 4.60%						
Unconfined compressive test	12.91 KPa						

 Table2:Properties of Untreated Soil

3. RESULTS AND DISCUSSION

3.1 Compaction characteristics

The maximum dry unit weight and ideal moisture content of the -containing biopolymers at different polymer inclusion levels were found. Since the unit weight of specimens will affect mechanical attributes such as strength, cohesion, permeability, and swelling, this research focused on these issues. Utilizing the collected information, compaction experiments were conducted to establish the ideal moisture content and maximum dry unit weight of the treated BCS.

As the percentages of POFA and XG rise, the MDD of soil falls from 1.78 to 1.65g/cc and OMC rises from 16.5% to 22.5%. The agglomeration of the various components of the mixes, assisted by the hydration action of the POFA + XG blends, maybe the cause of the observed MDD/OMC trend of the mixes. As the OPFA and XG fines filled in the wider spaces between the BCS particles and replaced them, the volume of the mixes increased and the dry densities of the mixtures decreased as a result. The increased surface area of the particles in the mixes' greater OPFA and XG fine concentrations, which will require more

water to lubricate the entire matrix of the mixes, may be the cause of the observed OMC trend[11].



Fig. 1: Compaction curves for natural and treated soil

3.2. California Bearing Ratio

The results of the unsoaked and soaked (24 h) CBR of untreated soil and the various BCS + OPFA + XG mixes are presented in Table 3. The results show an appreciable improvement in the CBR values of the various mixes with the addition of OPFA and the C contents to LS. The unsoaked CBR and soaked CBR values for 100% BCS are 4.6 and 3.9 respectively.



Fig. 2: Unsoaked CBR for natural and treated soil

The unsoaked CBR and soaked (24 h) CBR values of the various BCS + POFA + XG mixes do show improvement in CBR values but all of the values for soaked CBR are higher than the unsoaked. These values ranged from 5.7% to 11.19% and 6.13% to 12%, respectively, for a 0-day curing period; 9.4-18.5% and 10.23-20.2%, respectively, for a 14days curing period; 6.13-19.9% and 4.3-22.8%, respectively, for 28dayscuring period; 5.7-22.35% and 4.4-26.53%respectively for 60days curing period and, the

specimens with 90 days curing period, have unsoaked and soaked CBR values of 6-28.2% and 4.6-32.58%.



Fig. 3: Soaked CBR for natural and treated soil

When soaked in water for 24 h and can be used as the base material in highway construction, the peak CBR value of 32.58% (soaked) recorded for the 80% 79% BCS + 20% POFA + 1% XG mix proportion can be adopted for use as a base material for highway construction. According to the Indian Road Congress (IRC:37-2012), a minimum CBR value of 20% and more is often needed for foundation materials, 10% for subbases, 5% for subgrade, and 2% for embankment. According to Hatipoglu et al. (2008) and Li et al. (2009), the uniform distribution of XG and POFA in the mixtures and the agglomeration of the heterogeneous components of the POFA + XG stabilized BCS are the most likely causes of the variation in the CBR. The BCS, OPFA, and C are joined by these substances [8]. The higher soaked CBR values as compared to the unsoaked values may be attributed to strength gain through pozzolanic actions resulting from the complete chemical process of hydration and cation exchange, which leads to the production of Calcium silicate and calcium aluminate hydrates[1].

SOIL	SOAKED CBR (%)				UNSOAKED CBR (%)					
Curing period (days)	0	14	28	60	90	0	14	28	60	90
Untreated soil	3.9	4	4	4.3	6.8	4.6	4.7	5	5.5	6.7
Soil+1%XG	6.13	10.23	12.43	13.16	16.34	5.7	9.4	10.65	12.67	15.68
Soil+1%XG+10 % POFA	6.3	13.7	14.79	17.84	19.55	6.9	12.8	13.97	15.22	18.71
Soil+1%XG+15 % POFA	8.73	16.9	19.21	21.11	27.62	7.9	15.6	16.15	17.86	23.54
Soil+1%XG+20 % POFA	12	20.2	22.8	26.5 3	32.5 8	11.1 9	18.5	19.9	22.3 5	28.2
Soil+1%XG+25 % POFA	9.65	17.9	18.5	22.9	28.36	8.04	13.15	16.24	19.16	22.45

Table.3: California bearing ration for Natural and treated soil

3.3. Unconfined Compression Test

The results of the UCS test of the various LS + OPFA + XG mixes showed a good increase in strength with the increasing percentages of POFA, until the proportion of 1%XG and 20%POFA where the peak values of UCS are observed. Further addition of POFA showed a decrease in the UCS values in every specimen from the first day to the 91st day (see Table 5).For the various LS + OPFA + XGmix for 1st day ranged from 13.121 to 55kN/m2, the 14-day cure ranged from 13.7 to 65.33kN/m2, while the 28-day cure ranged from 13.75 to 76kN/m2,the 60-day cure ranged from 18 to 87.25kN/m2,the 90-day cure ranged from 22.89 to 108.68kN/m2.The peak UCS value of 108.68kN/m2 was recorded for the addition of 1%XG and 20% POFA. The formation of insoluble calcium silicates or aluminates and other pozzolanic products, as well as the agglomeration of the heterogeneous materials of the BC + OPFA + XG mixes, are possible explanations for the general increase in the UCS values with increased curing days [20]. These procedures form a solid matrix that holds the BC, OPFA, and C particles together [5].



Fig. 4: Effectiveness of curing period with respective strength parameters for treated soil

3.4. Scanning Electron Microscopy Analysis

To further understand the strength and increase mechanism, the specimens underwent SEM investigation after 90 days of curing. Figure 11 displays the SEM image of the naturally flocculated black cotton clay. But after therapy, a matrix developed. Fig. 5 displays the SEM images of clays that were treated with 1% of xanthan gum and 20% Palm oil fuel ash. POFA filled the pores between the soil particles whereas Xanthan gum acted as a binder by holding the clay and POFA particles together when there is no curing period.

The ninety days cured specimens were chosen for SEM analysis so that the most byproducts could be seen. The most crucial component of the strength increase, calcium alumina silicates are formed due to flocculation. The increase in strength is then caused by the by-products and their bonding.By-products gradually fill in the gaps and result in an increase in strength, as shown in the SEM study in Fig. 5. When xanthan gum was added, the soil's porosity was reduced. The gels formed in the soil matrix form a hard and nonporous soil matrix.



Fig. 5: SEM images for Natural (A1, A2) and treated soil (B1,B2)

3.5 X-ray Diffraction Analysis

A Rigaku Miniflex diffractometer with Cu-K X-ray radiation at a voltage of 30 kV was used to conduct the XRD investigation. With a continuous scan mode, a 15 mA electrical current was used to scan at a pace of 2.0 degrees/min from 5 to 65 degrees [15]. The peaks were located using the database made available by CDS National Chemical Database Service.

Quartz, kaolinite, and montmorillonite are the primary mineralogical components of the virgin soil, as indicated in Fig. The most typical varieties of crystalline minerals that make up clays include kaolinite, illite, smectite, and montmorillonite [14].Figure 6 displays the powder diffraction patterns from the XRD testing of the soil sample that was used in this investigation (1%XG+20%POFA) that was both untreated and treated.

The calcium aluminate silicate hydrate peak (gismondine, C-A-S-H) and calcium sodium aluminum silicate hydrate peak (Gismondine, C-(N)-A-S-H) both emerge at about 20.98 and 29.52°2 and 21.06 and 30.07°2°2, respectively[2], confirming the presence of the calcite peaks. Patimapon Sukmak et al. (2017) reported similar reflections.



Fig. 6:XRD graphs for Untreated and treated soil

3.6 FOURIER Transform Infrared Spectroscopy

The FTIR data for the raw soil, XG, POFA, and treated BCS cured at 90 days at the ideal additive concentration are shown in Fig.7 According to ul Haq et al. (2014), FTIR spectra for materials with an FA: POFA ratio of 100:0 at room temperature show the vibration bands at 1,444 cm⁻¹that correspond to an O-C-O group in terms of carbonate[16]. It is possible to determine the functional groups of soil minerals by using Fourier transform infrared spectroscopy to examine the absorption bands at various wavelengths of bond formation. The black cotton soil's post-treatment spectrum is shown in Fig. The graph's values illustrate the appropriate band allocations and places. Detectable quartz and silica bands from the material may be detected at 511, 1002, and 1004 cm⁻¹in addition to the conventional band assignments for montmorillonite. The 3736 and 3620cm⁻¹ bands were attributed to the -OH (alcohols) stretching group[5]. The bands at 1002, 1004, and so forth, demonstrate C-O stretching. A band at 3736 cm-1 in the treated black cotton soil's FTIR spectrum shows stretching of the -OH group, indicating hydration.

This is due to a pozzolanic reaction between the POFA and free calcium and potassium ions in Xanthan gum consequently, attributed to calcium sodium aluminum silicate hydrate (gysmodine).In POFA, this significant asymmetric vibration band moves from 1,013 to 1,059cm⁻¹ towards 966-989 cm⁻¹, indicating the development of sodium aluminum silicate hydrate (N-A-S-H), one of the hydration products. According to Madavarapu (2014), the higher wavelength number denotes a greater concentration of C-A-S-H bond vibrations, which leads to a more networked or dense structure and greater compressive strength.



Fig. 7: FTIR Graphs for natural, POFA, Xanthan gum, and treated soil

4. CONCLUSION

Extensive mechanical and microstructural tests were conducted in this experimental investigation to evaluate the impact of two different additives, xanthan gum, and palm oil fuel ash, on the geo-polymerization process. The varied XG, POFA, and BCS mix and curing time were evaluated as effect factors. From this study, the following findings can be made:

- 1. By raising the quantities of xanthan gum and palm oil fuel ash inclusion, the maximum dry unit weight decreased and the ideal moisture content rose. By adding more additives, the treated soil's dry unit weight dropped from 1.78 g/cc to 1.68 g/cc and its ideal moisture content rose from 16.5% to 22.1%.
- 2. The UCS of treated specimens rose at all degrees of additive inclusion and during all curing times. The specimens made at 79 percent BCS plus 20 percent POFA plus 1 percent XG showed the highest strength values. The maximum UCS was confirmed to be 108.68 kPa after 90 days of curing.
- 3. The peak CBR of 32.58 (soaked), with 90-day UCS of 108.68 kN/m2, and, observed for the 79% BCS + 20% OPFA + 1% C mix proportion, can be employed as a base material in road building.
- 4. The UCS value rose for all additive percentages as curing days increased and 90 days of cure time yielded the highest CBR and UCS values.
- 5. As curing time increased, the peak intensities of the XG, POFA moistened paste underwent considerable modifications. This explains why some substances, such as lime and portlandite, are absorbed during the hydration processes, resulting in the production of new substances and a mineral called in addition to the calcite created by

free radicals from xanthan gum. We can infer that the latter substances contributed to the stabilization of BCS with XG and POFA.

- 6. The FTIR spectroscopy shows that the peaks have been moderately influenced by the selected proportions of the POFA and XG due to the formation of C-A-S-H and C-N-A-S-H gels.
- 7. The SEM images depict that the gel was formed in the soil sample by interlocking the soil particles and stabilizing the soil which can bear more load which occurs by the flocculation and agglomeration.

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