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Microstructure of Black Cotton Soil Stabilized with Agricultural and Industrial Waste

[1]Nigerian Building & Road Research Institute, Abuja, Nigeria

[2]Department of Civil Engineering, Nile University of Nigeria, Abuja, Nigeria.

Kolawole O [1*] | Amuda A. G[2] |Dayyabu A[2] |Mambo A D[2] |

Corresponding Author's Name:
Amuda A. G
Corresponding Author's E-mail:
akeem.amuda@nileuniversity.edu.ng

ABSTRACT

Black cotton soil (BCS) is known to be expansive and very challenging when encountered during the construction of roads. Conventional stabilizing materials such as cement and lime are expensive. On the other hand, industrial and agricultural waste with pozzolanic properties are littered in factories and difficult to discard. This paper examined the microstructure of BCS soil stabilized with marble dust, MD, and Rice husk ash, RHA. About seven different mix ratios for MD and RHA are considered for 10% replacement in BCS. Pure BCS was also analyzed as a control. Atterberg limits were investigated for various mix ratio to understand the change in index properties. The SEM micrographs for the control samples show a loose pack of natural BCS with a sponge-like and hollow look. Generally, the SEM results for all the other samples show that the hollow perforated structure of the black cotton soil was reduced. The EDX shows the element present in each mix ratio with a mix ratio of 90:10 and 100:0 for MD: RHA as the appropriate mix.

KEYWORDS: BCS, MD, Microstructure, RHA and SEM

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1 | INTRODUCTION

Black cotton soils are also called expansive soils because of the changes in the moisture content which results in changes in volume [1]. There is generally a volume gain while getting moisture and a volume decrease when losing moisture. Swelling characteristics are usually strong in black cotton soil because it is a product of a clay mineral called montmorillonite. Black cotton soil is clay having black or grey color characteristics, it has a stretchy nature with over half of montmorillonite in quantity [2]. The microstructure of montmorillonite consists of a gibbsite sheet sandwiched in between two silica sheets. Water is captivated into the space between the layers [3].

Many clay minerals consist of silica tetrahedrons and alumina octahedrons which are trapped together by ionic bonds; however, montmorillonite clay mineral is trapped together by intermolecular forces. The structure of montmorillonite consists of clay layers that are organized one above another in successive layers bound together by van der Wall forces. These bonds are however weak and easily separated by rift or adsorption of water and other liquids. There is an extensive isomorphous substitution for aluminum and silicon with its grid this gives the clay a net negative charge resulting in the water-absorbing tendencies and an attraction for hydroxyl ions and the water molecules to the clay surface [4] on the other hand, kaolinite is a relatively inactive clay mineral. Its structure is built of repeating layers of basic silica-gibbsite sheets in a 1:1 lattice [5] and also possesses a large surface area with high cation exchange capacity. Consequently, black cotton soil used to be very stiff when dry and very soft when wet. BCS may be described based on geographical location. [6] It is known and found in India, Kenya, and other parts of the world [7] It can be found in other parts of the world including Nigeria. According to [8], the black cotton soil found in Nigeria contains montmorillonite and kaolinite minerals of 70% and 30% respectively. The presence of montmorillonite makes the soil have swelling and shrinking characteristics while the high strength value and ability to take in water is traceable to the presence of kaolinite. It can be concluded that black cotton soil characteristics vary based on geographical location and mineral combination.

In general, BCS is regarded as a geotechnical material with an unstable character that often causes excessive settlement to the structural pavement and cracks in buildings.

Soil stabilisation is soil's alteration of the physical or chemical properties to enhance a better one which can satisfy geotechnical properties [9]. Stabilisation could be through compaction, but it is important to note that stabilisation process alters the soil to improve the properties of the soil [9]. The stabilisation method could be biological, mechanical, chemical, or a combination of any of these. An example of chemical stabilization is the use of cement. Cement helps to improve the strength of the soil, its bearing capacity, and durability [10] It also reduces plasticity [11]. The use of cement in soil stabilization is quite high priced, particularly for developing countries like Nigeria, which necessitates an alternative to the use of cement.

Stabilisation of soil can also be achieved by using agricultural throw-away [12]. The residue after processing agricultural products such as vegetables, berry, or animal protein products are familiar as agricultural over. There has been an increase in population globally, leading to an increase in agricultural waste generated annually [13]. Over time, waste products generated from rice husks, bamboo trees, palm oil, locust bean, etc have been utilized in soil stabilization. Each year over 1000,000,000 tons of this waste is developed [13] which could cause a nuisance to the environment. Globally, agricultural spoil is sustainable because of its enhanced pozzolanic capabilities when oxygen is interjected through burning [14].

Many researchers have researched the waste from agricultural leftovers that can be utilized for improving the properties of the soil [14]. Oftentimes, residue from other agricultural produce has been utilized in soil stabilization such as bagasse ash combined with lime helps to improve the properties of expansive soil [15]. According to [16], groundnut shell ash is effective in stabilizing soil when mixed with lime. Also, a study shows that groundnut shell ash on expansive soil is good for soil with a high content of silt as it lowers its plasticity but recommended the addition of step-by-step such as cement [17]. The residue related to olive cake can also be used in stabling soil when combined with other additives [18]. Wheat husk ash when combined with granulated blast slag can stabilize expansive soil [11]. Cassava skin ash also has a good potential to be utilized in stabilisation [19] but must be blended with lime to get the desired result [20]. According to [21], coconut husk ash can improve soil having low CBR and is not ideal for soil with a high liquid limit. Other waste includes millet husk ash, corn cob ash, eggshell powder, cassava peel ash, locust bean, palm oil, banana tree, bamboo tree, and palm kernel which have been widely used to improve the geotechnical properties of tropical soils (lateritic and black cotton soils) and other soils. The report shows that about 998 million tons of agricultural waste are generated every year [13]. Previously mentioned prompted researchers to focus on how these materials can be used for soil improvement. The outcome of the research showed that these wastes must be blended with either lime or cement to improve their stabilisation properties. Since lime and cement are expensive, there is need to think of alternative materials that can be used . This inciate the use of RHA and MD.

Stabilisation of soil can take place using waste generated from industry and agriculture such as marble dust and rice husk ash. These waste materials cause severe disposal issues that can be cut down by using these materials. The marble dust is produced from the cutting and polishing of marble stone. The rice husk ash is a byproduct of burnt raw rice husk. It contains about 67 – 70% silica, about 4.9% aluminum, and 0.95% iron oxide [22]. Marble dust is rich in lime and aids in soil stabilization]23]. Marble dust and rice husk ash were combined at different percentages of 5%, 10%, and 20% by weight.

This work presents the results of the geotechnical test (moisture content, specific gravity, and liquid limit) and microstructure of the black cotton soil conducted in the laboratory. The microstructure of the BCS and the stabilized soil was checked through the scanning electron microscope (SEM) which was linked to the geotechnical properties investigated in the laboratory. This paper aim to understand the BCS fabric modification caused by inter-particle contacts and the resulting bonding when stabilised with RHA and MD

2| SOIL SAMPLING AND SAMPLE PREPARATION

The soil was collected from Adamawa state, precisely Numan local government (latitude of N, longitude of E). The place is occupying the confluence of Rivers Benue and Gongola. The location shown below was from the google map.

Fig. 1: Location of Sampling Point(Source: Google Earth Map)

The sample was gotten from about 1.5m into the subsoil. It was collected through digging. This area was made ready by removing the topsoil and every vegetation to avoid any contamination. A disturbed sampling method was used in the collection of the sample. Also, the RHA and MD were collected from Benue State (Northern centre in Nigeria). The collected soil sample was then placed into rubber containers and transported to the Civil Engineering Laboratory at Nile University, Nigeria. At the laboratory, the natural soil sample was air-dried for about 3 days at room temperature and then pulverized before further tests were conducted. The stabilized black cotton soil is made up of BCS and mixtures of MD, and RHA which are mixed at different percentages and water. Initially, the MD and RHA were mixed in different ratios such as 90:10, 70:30, 50:50, 30:70, and 10:90. The mixed samples were arranged differently in a sealed polythene bag and preserved appropriately. 10% of each of the mixtures of MD and RHA was then fused with BCS. The last sample which is 100% BC serves as control. Table 1 explains the experimental design.

Table 1: Experimental Design for 10% stabilizer

STABILISER	AA	BB	CC	DD	EE	FF	GG	HH
MD (%)	0	0	10	90	30	70	50	100
RHA (%)	0	100	90	10	70	30	50	0

3| INDEX PROPERTIES

Index test was administered on the black cotton soil samples and the stabilized sample to determine various properties such as moisture content, atterberg limit, and specific gravity. There was a visual examination that showed dark-colored soil having a smooth texture. The atterberg limit test carried out was Liquid Limit (LL), plastic limit (PL), then plasticity Index (PI) was calculated. The PL also known as the plastic limit is the smallest water content that explains where the soil sample changes from gelatinous form to a plastic state. The liquid limit test was conducted according to ASTM D6951; ASTM 0 43 18. Using the cone penetrometer method, a sieved sample through a 425-micron sieve was used. The test consists of a stainless-steel cone that is positioned to penetrate the moist sample in the stainless cup. The plunger is released and allowed to fall freely to penetrate the sample for 5 seconds. The penetration time is recorded. The plasticity index is the difference between the liquid limit and the plastic limit. The specific gravity is given as the measure of the density of soil compared to the equal density of water. It was done following IS 2720-3-1 (1980).

Table 2: Index Properties of black cotton soil

Index Properties	Value Range	Technic & Device
Moisture Content (%)	16	BS 1377(1990) Part 2
Free Swell Index%	52.38	(ASTM D5890)
Liquid limit (%)	52	ASTM D6951
Specific Gravity (%)	2.37- 2.40	IS:2720(Part 3), Sec.1-1980
Color	Dark color	Sighting
Texture	Smooth	Touching
Dominant Mineral	Montmorillionite	

Table 3: Atterberg Limit

SAMPLES	AA	BB	CC	DD	EE	FF	GG	HH
LL	52	65	63	48	52	50	52	47
PL	33	40	33	26	36	25	32	25
PI	19	25	30	22	16	25	20	22

4| MICROSTRUCTURAL ANALYSIS

The samples used for this experiment were crushed and then transferred to the Disc Milling Machine which reduced the samples to pebbles before taking them to the vibrating cup milling machine which will pulverize the fine sample to a powder of 150 microns. The pulverized sample was released to the analytic unit for analysis

Fig. 2: Setup for SEM Analysis

4.1 Oxides of MD and RHA

A common abbreviation for the X-ray diffraction test to spot the composition of minerals in materials is XRD. It was used to point out the mineralogical constituent of marble dust and rice husk ash used as a pozzolana for black cotton soil. A Rigaku sixth-age Miniflex benchtop XRD apparatus available at the National Steel Raw Materials Exploration Agency, Kaduna was employed for the analysis. The powder sample was conveyed to the analytical unit for XRD analysis to be carried out on them. For every mix, two grams of powder sample is apportioned into the sample holder and with the use of a glass slide, the sample is pressed down to give an even surface area. The sample holder was then placed in the XRD multi-sample holders' chamber. The machine is then gauged i.e. set to begin the analysis. The analyzed sample was immediately saved into a directory folder which was further uploaded on Studio Smart Lab 1 jointly with ICDD PDF-4 to perform an automatic/ manual search match. The results of the analysis are then produced and saved for reference intent. The elements present in marble dust and rice husk ash in the presence of water react such as with CaO, Al_2O_3, and SiO_2. The silicate and Aluminum combined with the available Calcium produce cementitious compounds [24]. The chemical characterization of the composition of the element in the materials used was done, Table 4 below shows the oxides present in MD and RHA used for the experiment.

Table 4: Oxides of MD and RHA

Elemental Oxides	**Chemical Composition**	
	RHA (%)	**MD (%)**
SiO_2	92.75	2.24
Al_2O_3	1.47	4.47
Fe_2O_3	0.58	0.89
CaO	1.19	85.96

4.2 SEM and EDX Approach

The procedure for scanning electron microscope (SEM) involves mixing the powdered sample thoroughly to achieve a homogenized representation which was placed on the sample stub with the help of carbon double adhesive and slotted into the machine, ready for analysis. The machine was then calibrated. The machine is then powered in the scanning mode to generate the overview of the microstructure of the samples under study. This is further zoomed in to achieve the desired magnification, and then focus to have a sharp image. The image captured is saved in the archive for reference purposes. Furthermore, Energy Dispersive Xray (EDX) analysis was used for chemical characterization of the already mixed samples. EDX shows the element and their concentration in a soil sample. The test procedure for EDX is similar to that of XRD earlier explained in this paper.

5 | RESULTS AND DISCUSSIONS

The SEM was depleted to inspect closely the morphology of the soil sample. Figure 3 shows the SEM images for untreated BCS and both images (Fig.3a and 3b) show similar structures. Taken at 500x and 1000x magnification respectively. It was discovered that BCS is not formally organized which is due to the presence of montmorillonite minerals. Hence, the microscopic analysis shows an amorphous solid with a hollow cavity. The microparticles are like a mixture in structure, the surfaces are fixed together by pore water with a spongy look.

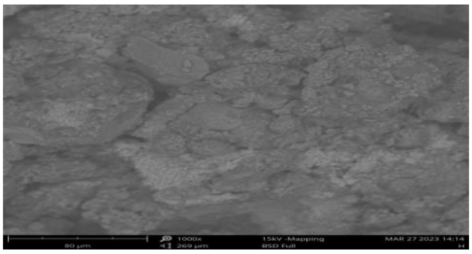

Fig. 3(a): Untreated BCS at 500x

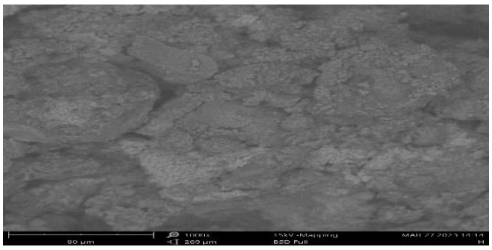

Fig. 3(b): Untreated BCS at 1000x

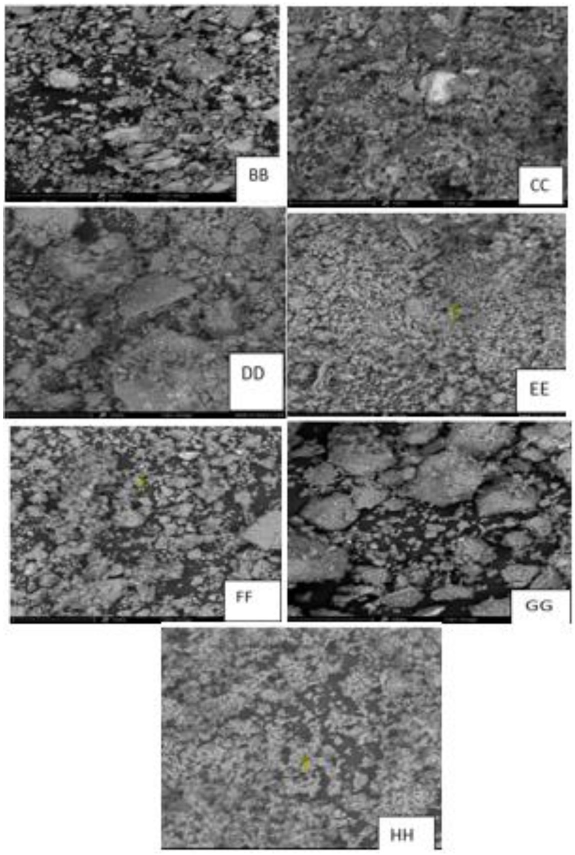

Fig. 4: Elemental Images of Stabilized BCS at different percentages

The energy dispersive Xray (EDX) was carried out to spot the chemical components of the stabilized BCS when MD and RHA were added. Table 5 explains the EDX results of the pure BCS and BCS treated with MD and RHA. Figure 3 shows the elemental images of BCS stabilized with varying percentage of MD and RHA. It was seen that the main mineral present in untreated BCS is Si with a 50.84 concentration by weight.

The application of MD and RHA has affected the chemical components of soil samples depending on the proportion of the MD and RHA added. It was observed that chemical reactions occurred in the mixtures and it affected the weight concentration of the minerals present in each mix. The percentage of silica increases on mix DD, BB, CC, EE, FF, and GG with CC having the highest increase in silica content. The CC sample consists of 10:90 for MD: RHA. The next is EE which consists of 30:70 for MD: RHA. Sample HH which is 100% MD with no RHA has the lowest silica content. This result means that MD cannot improve silica content, instead, it reduces the content. It was also observed that no matter the proportion of RHA in the mix, the silica content increased. However, the increase in silica content does not follow any orderly pattern with the increase in RHA. Also, the aluminum content in the mix reduces for all the mix, this reduction does not follow a specific trend as CC which is 10:90 for MD: RHA has the lowest aluminum content.

The content of the Iron element is reduced for some of the mixes but increased for GG which contained 50:50 MD: RHA. Although the untreated BCS contains Fe which is responsible for the dark color noticed in the sample, it is not certain if the change to a lighter color may reduce the iron content. There was a reduction of iron content in sample DD, which is 90:10 for MD: RHA, followed by BB, which contains 100% RHA without MD. The concentration of calcium was reduced in all the mixes except in the mix of HH and GG which increased considerably. The very high percentage noticed in HH which is 100% MD confirms that MD is mainly calcium in nature. The concentration of magnesium increased for all the mix. From HH to GG, to BB, to FF then EE, and CC which is 90% RHA with 10% MD. The highest concentration was noticed in DD which is 90:10 for MD: RHA.

Comparing the EDX, SEM, and Atterberg limit results, it was clear that liquid limit data reduces with an increase in Magnesium. Murat, 2014 also observed that increased Magnesium Chloride decreases the liquid limit. Comparing Table 3 with Table 5, it was observed that the liquid limit value was the lowest for DD with a high content of magnesium of 1.06.

Table 5: EDX results for samples mix

Element	Weight Concentration							
	AA	BB	CC	DD	EE	FF	GG	HH
Si	50.8	56.2	75.6	56.3	65.7	56.6	60.3	46.4
Al	16.1	12.7	5.99	12.9	10.1	13.5	8.12	7.82
Fe	14.2	12.3	3.43	12.8	10.0	12.4	16.4	6.55
Ca	3.86	2.32	2.86	2.55	2.31	2.10	4.32	14
Mg	0.27	0.44	0.73	1.06	0.62	0.50	0.43	0.4I

6 | CONCLUSION

Index properties and Microstructural tests such as SEM and EDX were carried out on the BCS stabilized with the mixture of MD and RHA at different proportions. Seven different mixtures were used to stabilize BCS and a control sample, which is a pure BCS was also analyzed. During stabilization, 10% of each mixture was added and thoroughly mixed with BCS. The SEM for BCS control shows some organic content, as is seen in the internal structure which is loosely packed. Generally, the SEM results for all the other samples show that the hollow perforated structure of the black cotton soil is not as large as the control. The particles are closer and appear to be denser.The following has been concluded;

- The soil structure has become more compact and the passage of water in between the arrangement of particles will be reduced.
- The changes in the stabilized BCS from EDX shows different elements were found in different mix ratio used.
- The study showed there are changes in the microstructure arrangement in BCS when mixed with industrial waste and agricultural waste nevertheless the environmental effect of RHA needs to be evaluated.
- The Atterberg limit tests revealed that the combination of more marble dust and little rice husk ash improves the liquid and plastic limits of BCS
- Comparing the SEM results and Atterberg limit test, mix ratio from DD and HH which are 90:10 and 100:0 for MD: RHA respectively could be considered as appropriate mix ratio.

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