

Microanalysis and Compactive Efforts Study of Black Cotton Soil Treated With Cement Kiln Dust

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Abstract-An expansive clay, also known as Black cotton soil (BCS) was treated with up to 10 % cement kiln dust (CKD) using three different compactive efforts; British Standard light (BSL), West African Standard (WAS) and British Standard heavy (BSH) energies. Laboratory tests were performed on the natural soil and CKD treated soil samples in accordance with BS 1377 (1990) and BS 1924 (1990) respectively with the aim of improving the deficient soil to meet engineering requirements. Preliminary evaluations on the natural black cotton soil showed that it fell under A-7-6 (16) using AASHTO classification and CL according to Unified Soil Classification System (USCS). Results of laboratory tests carried out on soil specimens show that the properties of the soil generally improved with CKD treatment. Peak unconfined compressive strength (UCS) values of 357.07, 382.49 and 528.82 kN/m² and California bearing ratio (CBR) of 7, 10 and 19 % as well as resistance to loss in strength of 44, 55 and 55 % were recorded at 10 % CKD treatment, respectively, for BSL, WAS and BSH compactive energies. Reduction in the particle sizes with curing period was observed when samples were viewed through the scanning electron microscope (SEM). The study showed that CKD can be beneficially used to improve the subgrade of lightly trafficked roads and as admixture in lime stabilization during construction of flexible pavements over expansive soil.

Keywords- California bearing ratio; Cement kiln dust, Durability; Expansive soil; Microanalysis, Unconfined compressive strength; Scanning electron microscope.

1 INTRODUCTION

Black cotton soils (BCS), which are among the types of expansive soils, are in the group of problem soils encountered by geotechnical engineers. It was reported that black cotton soils occupy an estimated area of 104×10^3 km² in North-Eastern Nigeria (Salahudeen and Akijje, 2014). The mineralogy of this soil is dominated by the presence of montmorillonite which is characterized by large volume change from wet to dry seasons and vice versa (Adeniji, 1991). Black cotton soils cover an estimated 340 million hectares, or about 3% of world's cultivatable soils, and are found mainly in Africa; in the Sudan's Gezira cotton fields and Southern black soil plains; in South Africa; Ethiopia; and Tanzania. In Asia; they are found extensively in the Indian Deccan Plateau and the Middle Nile valley; it covers large areas of Australia and Russia (Ola, 1983). The swelling and shrinkage properties exhibited by black cotton soils have caused damages to various civil engineering structures constructed with or on these deposits. The costs of these damages have not been properly documented in Nigeria (Salahudeen et al., 2014).

A typical Portland cement is manufactured by feeding materials containing appropriate proportions of lime, silica, alumina and iron into the upper end of a kiln. Burning fuel is forced into lower end of the kiln where it produces temperatures of 1400-1650°C, changing the raw mix to a cement clinker. During this operation a small percentage of the material in the form of dust (i.e., CKD) is collected. The physical and chemical properties of cement kiln dust (CKD) or cement by-pass dust (CBPD) can vary from plant-to-plant, depending on the raw materials used and type of collection process in the plant (Salahudeen and Akijje, 2014). Several factors influence the chemical, mineralogical and physical properties of CKD (Wayne and Donald, 2008).

Due to the high costs of cement and lime, effort is being intensified by researchers (Osinubi, 2000; Ramzi et al., 2001; Alhassan and Mustapha, 2007; Musa, 2008; Suhail et al., 2008; Oriola and Moses, 2010; Osinubi et al., 2011; Akshaya, 2012; Salahudeen et al., 2014; Salahudeen and Akijje, 2014; etc.) to investigate the possibilities of utilizing potentially cost effective locally available industrial and agricultural wastes for the modification and stabilization of deficient soils. Thus, the possible use of industrial waste (such as cement kiln dust) will not only reduce cost of disposal but also reduce or eliminate the environmental hazards caused by such wastes.

In recent years, there has been growing interest in the research of soil microstructure. From the image of soil microstructure, some micro-parameters such as porosity and soil particle orientation degree could be extracted (Wei, 2010). The new generation scanning electron microscope (SEM), utilizing energy dispersive x-ray spectrometers (EDS), incorporate computer automation and detector technology that allow for rapid elemental analyses of small particles or inclusions.

Automated SEM-EDS analyses remove biases inherently present in manual optical analyses and provide elemental and morphological information (Brian, 2013). The scanning electron microscope (SEM) is designed primarily for producing electron images, but can also be used for element mapping and even point analysis, if an X-ray spectrometer is added. The study was focused on studying the effects of compactive energies on the evaluation of an expansive soil (also known as black cotton soil) treated with up to 10 % cement kiln dust (CKD), for use as a flexible pavement construction material.

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2 MATERIALS AND METHODS

2.1 MATERIALS

Soil: The black cotton soil (BCS) sample used for this study was collected from Deba (Latitude 10°13'N and longitude 11°23'E) in Gombe state, Nigeria, using disturbed sampling method. The samples were taken at depth of 1 m from the natural earth surface to avoid organic matter influence.

Cement kiln dust: The cement kiln dust (CKD) was obtained from Sokoto Cement Factory, Sokoto, Nigeria. The oxide composition of the black cotton soil and CKD was determined by method of Energy Dispersive X-Ray Fluorescence.

2.2 METHODS

Laboratory tests were performed to determine the index properties of the natural soil and cement kiln dust treated black cotton soil in accordance with BS 1377 (1990) and BS 1924 (1990), respectively.

Compaction: The compaction tests were performed on the natural soil and the soil stabilized with up to 10 % CKD treatment by dry weight of soil using British Standard light (BSL), West African Standard (WAS) and British Standard heavy (BSH) compactive efforts. The only difference between BSH and WAS is that while the former is given 27 blows, later receives 10 blows only.

Strength: The strength tests performed were used to determine the unconfined compressive strength (UCS) and California bearing ratio (CBR) values. The UCS test specimens were compacted with BSL, WAS and BSH energies and cured for 7, 14 and 28 days before testing. The CBR tests were carried out in accordance with Nigerian General Specifications (1997) which specifies that specimens be cured in the dry for six days and then soaked for 24 hours before testing

Durability: The durability assessment (which is simulation of some of the adverse field conditions) of the soil sample was determined by resistance to loss in strength of the treated soil specimens when immersed in water. It is expressed as the ratio of UCS of the specimen wax-cured for 7 days and de-waxed top and bottom before being soaked for another 7 days to the UCS of the specimen cured for 14 days.

Microscopy: The properties of the clay sub-fractions in the soil - CKD mixtures were studied by two analytical methods using scanning electron microscope (SEM) and energy dispersive x-ray spectrometer (EDS). The SEM analysis was done using a SEM model "Tecan Vega SEM". Individual analyses from different zones of the sample were performed by EDS. Since the electron probe analyses only to a shallow depth, specimens were well polished so that surface roughness does not affect the results.

3 RESULTS AND DISCUSSION

3.1 PROPERTIES OF MATERIALS

The results of the index properties of the natural soil show that the soil in its natural state is not suitable for the construction of a flexible road pavement. The particle size distribution curve is shown in Fig. 1 while the oxide

compositions of the cement kiln dust (CKD) and black cotton soil (BCS) used in the study are given in Table 1.

Table 1. Oxide compositions of CKD and BCS

Oxide	Concentration (%)	
	Cement Kiln Dust	Black Cotton Soil
CaO	44.28	3.58
SiO ₂	7.23	49.00
Al ₂ O ₃	1.90	15.10
Fe ₂ O ₃	4.47	14.23
MgO	0.82	-
K ₂ O	-	2.25
MnO	0.11	0.23
TiO ₂	0.23	2.09
LOI	39.28	11.1

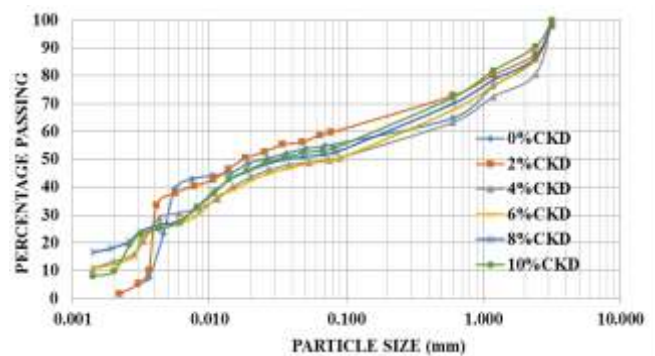


Fig. 1: Particle size distribution curves of BCS-CKD

3.2 COMPACTION CHARACTERISTICS

Maximum Dry Density: Figure 2 shows the curves of maximum dry density (MDD) of black cotton soil with cement kiln dust content for the three compactive efforts used in this study. For BSL and BSH compactions, MDD increased to peak values at 4 and 6 % CKD contents, respectively, and thereafter decreased with higher CKD content. The initial increase in MDD could be due to CKD with a higher specific gravity (2.55) compared to that of soil (2.33) occupying the voids within the soil matrix as well as the flocculation and agglomeration of the clay particles due to exchange of ions (Osinubi, 2000; Oriola and Moses, 2010). Similar trends were reported by researchers like Sadeeq et al. (2015), Salahudeen and Akiije (2014), Salahudeen et al. (2014) and Salahudeen and Ochepe (2015).

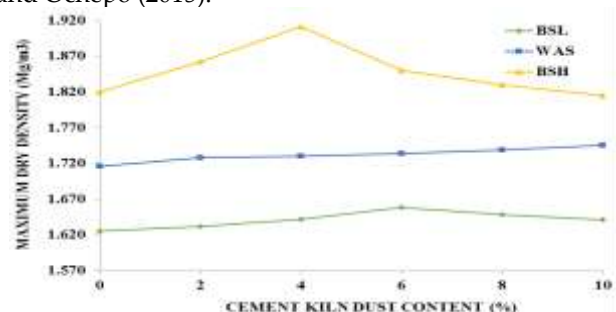


Fig. 2: Variation of MDD of BCS with CKD content

Optimum Moisture Content: Figure 3 shows the curves of optimum moisture content (OMC) of black cotton soil with cement kiln dust content for the three compactive

efforts considered in this study. For BSL and BSH compactions, the OMC increased to peak values at 6 % CKD content and thereafter decreased with higher CKD content. This trend is consistent with the findings reported by Osinubi (1999). An explanation for this trend was the increased demand for water commensurate with the higher amount of CKD required for its hydration reaction and dissociation needed for cation exchange reaction. The subsequent decrease in OMC with increase in CKD content might be due to cation exchange reaction that caused the flocculation of clay particles (Salahudeen and Akijje, 2014).

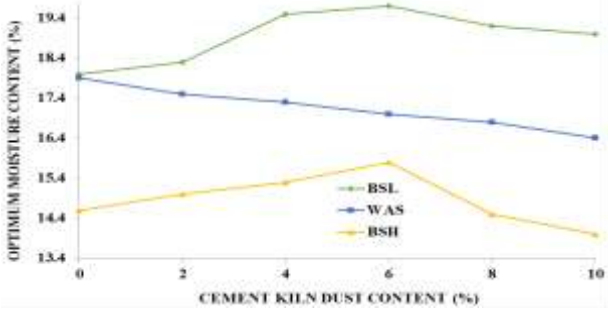


Fig. 3: Variation of OMC of BCS with CKD content

3.3 STRENGTH CHARACTERISTICS

Unconfined Compressive Strength: The variation of unconfined compressive strength (UCS) of black cotton soil with cement kiln dust content for BSL, WAS and BSH compactions at 7, 14 and 28 days curing periods as well as 7 days curing and 7 days soaking are shown in Figures 4, 5, and 6, respectively. The UCS values for the three compactive efforts at all the curing periods generally increased with increase in CKD content. The observed trends can be attributed to ion exchange at the surface of clay particles. The Ca²⁺ in CKD reacted with the lower valence metallic ions in the clay microstructure which resulted in agglomeration of the clay particles (Salahudeen et al., 2014). The increase of the UCS values was primarily due to the formation of various compounds such as calcium silicate hydrates (CSH) and calcium aluminate hydrates (CAH) and micro fabric changes, which are responsible for strength development (Osinubi et al., 2011).

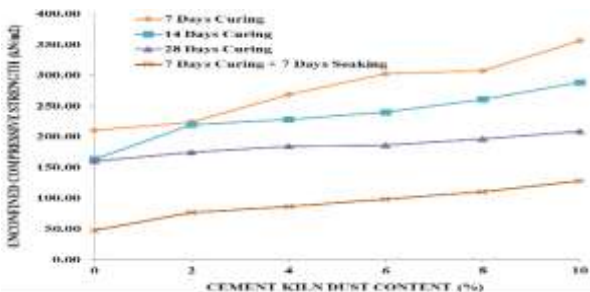


Fig. 4: Variation of UCS of BCS with CKD (BSL)

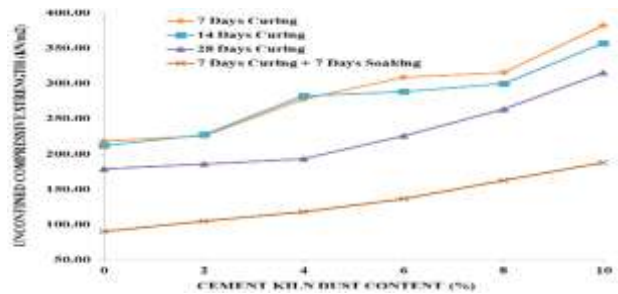


Fig. 5: Variation of UCS of BCS with CKD (WAS)

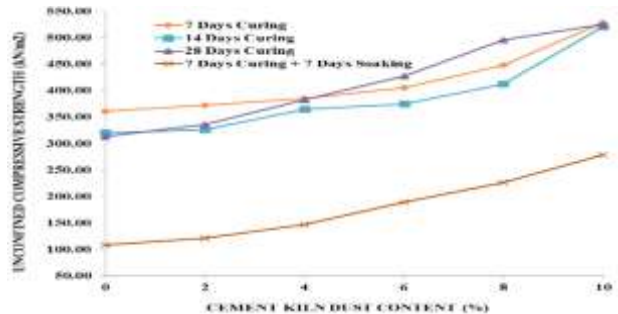


Fig. 6: Variation of UCS of BCS with CKD (BSH)

Although UCS values increased with increase in CKD content for the curing periods considered, however, it was generally observed that strength decreased with curing age. This was primarily due to the high loss on ignition (LOI) value (39.28 %) of the long – dry cement kiln dust used in the study. Usually long – dry kiln dust contains more calcium carbonate with more limited amounts of free lime (Bhatty et al., 1996; Talal and Awad, 1999; Miller and Zaman, 2000; Wayne and Donald, 2008).

The gradual release of the (high content) bound water within the chemical structure of CKD and the high slow-reacting calcium carbonate content was responsible for the decreased unconfined compressive strength values or loss of strength with curing period. In other words, the same mechanism (increased free moisture) that caused the decrease in UCS value when cured for 7 days and soaked for 7 days (compared with 14 days curing period) was responsible for the decrease in strength with curing age (Bhatty and Todres, 1996; Miller and Zaman, 2000; Wayne and Donald, 2008).

California Bearing Ratio: The variation of unsoaked and soaked (24 hours soaking) CBR values of the soil with cement kiln dust content for the three energy levels used are shown in Figures 7 and 8. Generally, the CBR values recorded increased with higher CKD content. This increase could be due to the presence of adequate amounts of calcium required for the formation of calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH), which are the major compounds responsible for strength gain. It was observed that the unsoaked and soaked CBR values increased with higher CKD content and compactive effort. Peak CBR value of 19 % recorded at 10 % CKD content for BSH compaction is close to the 20 – 30 % requirement for sub base reported by Gidigas and Dogbey (1980), for materials compacted at the optimum moisture content.

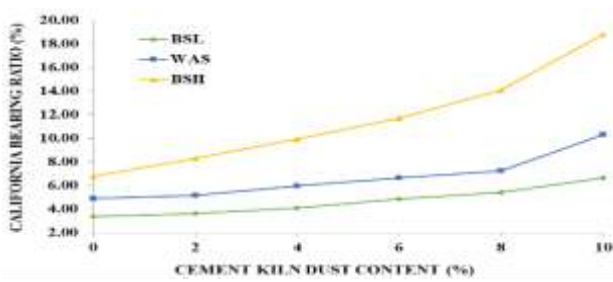


Fig. 7: Variation of unsoaked CBR of BCS with CKD

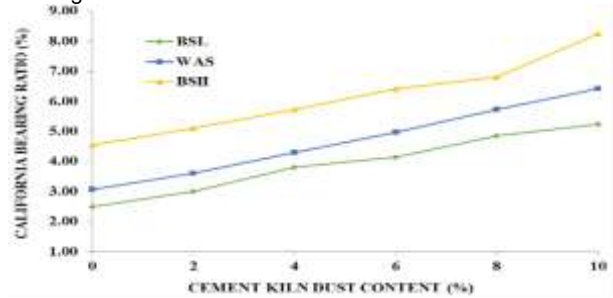


Fig. 8: Variation of soaked CBR of BCS with CKD

Durability: Durability assessment of soil samples involves the simulation of some of the worst conditions that could occur in the field. In this study, durability was evaluated by the immersion of specimens in water to determine resistance to loss in strength which is more acceptable for tropical regions like Nigeria (Salahudeen et al., 2014). Conventionally, an allowable 20% loss in strength (80% resistance to loss in strength) is recommended for a specimen cured for 7 days and immersed in water for 4 days (Osinubi, 1999). The variation of resistance to loss in strength of the soil with CKD content for the three energy levels used is shown in Figure 9. Resistance to loss in strength of the treated soil increased with higher CKD content.

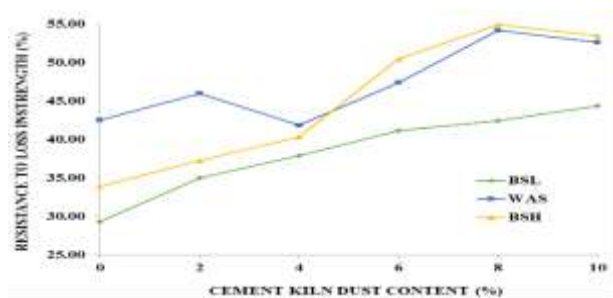


Fig. 9: Resistance to loss in strength of BCS with CKD

Although the peak value did not meet the allowable value, regardless of the harsher condition the samples were subjected to in the laboratory (7 days soaking instead of the conventional 4 days reported by Salahudeen et al. (2014)), the black cotton soil - 8 % CKD mixture compacted with WAS and BSH energies may be used to improve subgrade for the construction of flexible pavements.

3.4 MICROANALYSIS OF SPECIMENS

EDS in the scanning electron microscope is the process of using characteristic x-rays, generated in a specimen by the electron beam, to determine the element composition of the specimen. The Y-axis shows the counts (number of X-rays received and processed by the detector) and the

X-axis shows the energy level of those counts. The EDS is quite good at associating the energy level of the X-rays with the elements and shell levels that generated them (Bob 2013). Each shell of every atom has a unique energy level determined by the atomic configuration for that element. As Z increases the K shell line energy increases. If K-shell is excited then all shells are excited but may not be detected (Nestor 2013).

Every element has a set of characteristic x-rays. A characteristic x-ray has a very specific energy that is unique to an element. Therefore, a characteristic x-ray can be thought of as an element's thumbprint. Incident x-ray of sufficient energy will eject an electron from an atom, leaving a vacancy. The atom will then adjust its electron configuration to be in the lowest energy state; in other words, an electron in a higher shell will drop down to fill the vacancy. The process of an electron filling the vacancy creates an x-ray which is characteristic of a specific electron transition for that element (Amuda et al. 2014).

7 Days Curing Period: The results of microscopic/elemental analysis of specimens of black cotton soils treated with 0 and 10 % CKD and cured for 7 days are shown on Plates 1 (a) – (c) and 2 (a) – (c), respectively. In the energy dispersive x-ray spectroscopy (EDS), when an electron is knocked out from an atom, a vacancy (hole) in an inner shell (say K shell) of the specimen atom is generated by an incident high-energy electron (E_0) that loses the corresponding energy (E) transferred to the ejected electron. The hole in the K shell is subsequently filled by an electron from an outer shell (say L shell). The superfluous (excess) energy is emitted as a characteristic X-ray quantum. The energy of the X-ray is characteristic of the specimen atomic number from which it is derived (Bob, 2013). However, the investigation of the shell from which the electron was ejected as well as the shell that supplied the filled electron is out of the scope of this study. In the EDS plates, the intensity of X-ray emission, which is the number of X-rays received and processed by the detector in count per second per characteristic X-ray energy (cps/eV) was plotted against this superfluous characteristic X-ray energy (in keV) which represents the concentration of electrons at that point (see Plates 1 to 4 b and c). The superfluous (excess) characteristic x-ray energy emitted for each element was found to be equal or around the value of its energy line in the periodic table. The elemental spectra on the EDS plates represent the percentage concentrations of the oxides of the detected elements as given in Table 2. The summation of oxide concentrations of the detected elements are 40 and 50 % (see Plates 1b and 1c).

The EDS elemental analyses of the natural soil shows the usual composition of the aluminosilicates minerals (Reyes et al., 2007) consisting of Si, Al, Fe, with traces of Ca, Mg, Ti and k as shown in Plates 1(b) and (c). On the other hand, due to the cement kiln dust content in sample analyzed in Plate 2 (a), the Si content reduced while the Ca content highly increased (see Plates 2 (b)

and (c)). The presence of elemental Carbon in the EDS is due to the carbon tape at the background of sample holder.

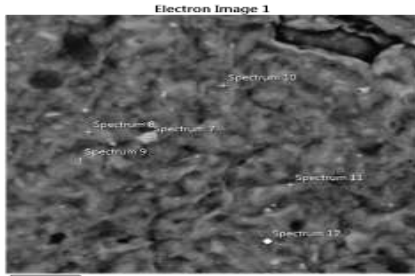


Plate 1 (a)

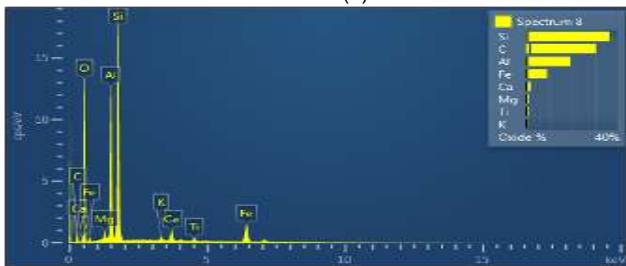


Plate 1 (b)

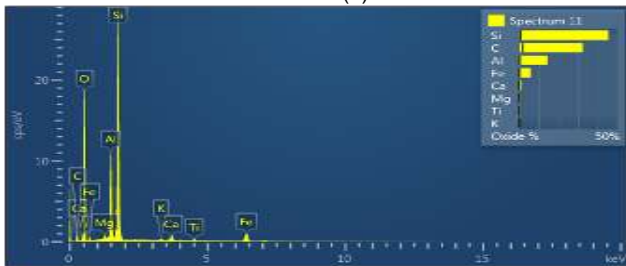


Plate 1 (c)

Plates 1 (a) – (c): Micrograph and diffractographs for BCS - 0 % CKD mixture (7 days curing)

The untreated black cotton soil (see Plate 1 (a)) formed aggregates of particles with smaller sizes distribution than the soil – CKD mixture (see Plate 2 (a)). The more active and higher valent cation (i.e., Ca^{2+}) in the mixtures replaced the weakly bonded ions in the clay structure. The larger particle sizes formed in the soil treated with 10 % CKD were responsible for the higher strength values recorded.

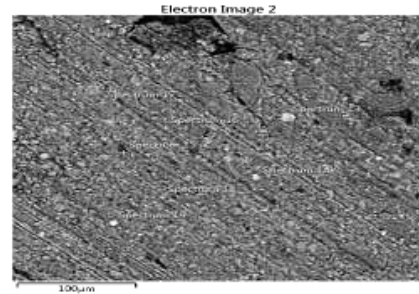


Plate 2 (a)

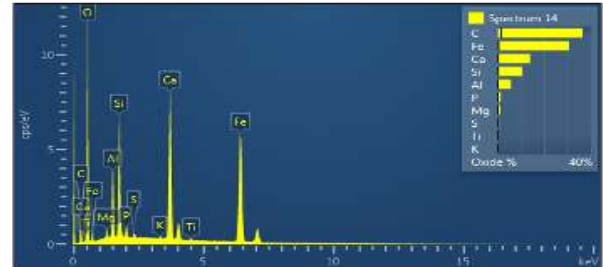


Plate 2 (b)

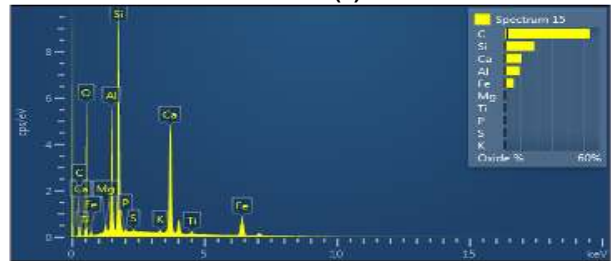


Plate 2 (c)

Plate 2 (a) – (c): Micrograph and diffractographs for BCS -10 % CKD mixture (7 days curing)

28 Days Curing Period: The coarseness of soil – CKD mixture specimens reduced after 28 days curing (see Plates 3a and 4a) when compared with the images of specimens cured for 7 days. This was probably due to the high loss on ignition value of cement kiln dust used that liberated bond water at the outer layers. Consequently, the unconfined compressive strength values reduced at 28 days curing period when compared to the 7 days curing period values. The EDS elemental analysis patterns for specimens cured for 28 days (see Plates 3 (b) - (c) and 4 (b) - (c)) are similar to those recorded for specimens cured for 7 days curing period discussed above.

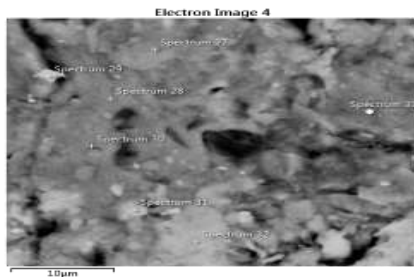


Plate 3 (a)

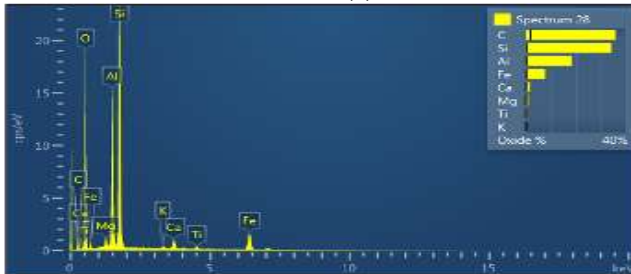


Plate 3 (b)

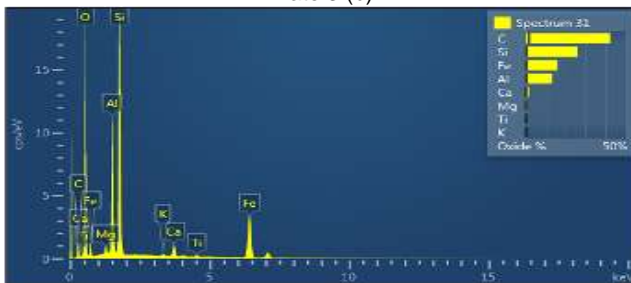


Plate 3 (c)

Plate 3 (a) – (c): Micrograph and diffractographs for natural BCS (28 days curing)

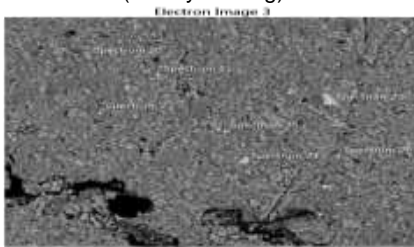


Plate 4 (a)

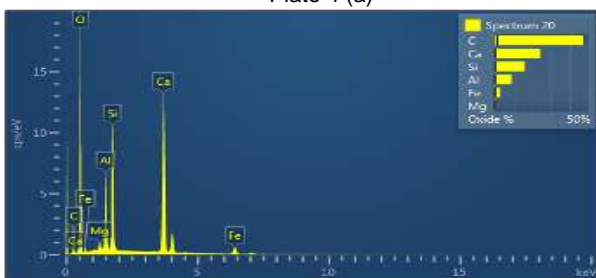


Plate 4 (b)

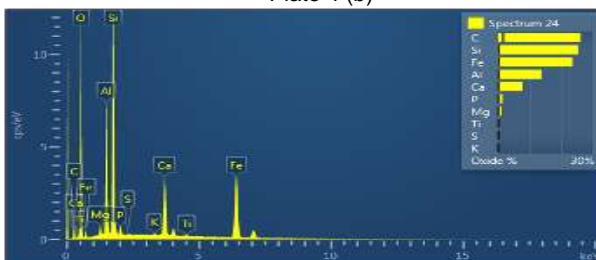


Plate 4 (c)

Plates 4 (a) – (c): Micrograph and diffractographs for BCS – 10 % CKD mixture (28 days curing)

4 CONCLUSION

The black cotton soil (expansive soil) used in the study classifies as A-7-6 (16) and CL soil using the AASHTO classification system and USCS, respectively. Although the properties of the natural soil improved, peak UCS, CBR and resistance to loss in strength values of 357.07 kN/m², 7 % and 44.37 %, for the BSL, 382.49 kN/m², 10 % and 54.17% for WAS and 528.82 kN/m², 19 % and 54.90% for BSH compaction, respectively, did not meet the requirement for its use as a sub base material. However, the black cotton soil - 8 % CKD mixture compacted using WAS or BSH energy will be effective when used in combination with other cementitious materials such as Portland cement after the determination of their optimum content for sub-base stabilization. The microscopy/ EDS elemental analysis results of the 0 and 10 % CKD treated UCS specimens cured for 7 and 28 days showed reduction in particle sizes with higher curing period. The study showed that CKD can be beneficially used to improve the subgrade of lightly trafficked roads during construction of flexible pavements over expansive soil. However, CKD with high LOI should be avoided in the treatment of expansive soils.

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