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MINERALOGICAL AND CHEMICAL CHARACTERIZATION OF CEMENT STABILIZED–BLACK COTTON SOIL

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Abstract: The expansive soil found in North–East Nigeria are called Black cotton soils because they are dark in color. This research deals with the effects of ordinary Portland Cement on Black Cotton Soil. This research attempts to investigate the mineralogy of Black Cotton soil, the oxide components of Black Cotton soil and ordinary Portland cement, changes in physical features and chemical components of cement stabilized–black cotton soil. The mineralogical analysis detected that Quartz, Tridymite, Clinoenstatite, Haematite, Kyanite, Corundum, Doyleite and two (2) clay minerals (Montmorilonite and Kaolinite) are the main mineral constituents of Black Cotton Soil. The microstructural analysis showed that there were modifications of the soil particles when cement was added. There were formations of cementitious white coatings and soil flocculation on the surface of the cement stabilized soil mixture. The EDX spectra of the chemical elements characterized natural black cotton soil with three (3) main dominant peaks of Titanium (Ti), Silicon (Si) and Aluminum (Al) in descending order. On addition of cement, the EDX spectrum of the cement stabilized–black cotton soil changed to four (4) main dominant peaks of Titanium, Silicon, Calcium and Aluminum in descending order. The research showed that chemical reactions, formation of new chemical compounds and type of chemical compounds formed have significant importance on the modifications and stability of black cotton soil. The identification and characterization of chemical elements in both soil and stabilizer are necessary for effective soil modification and stabilization.
Keywords: black cotton soil, Portland cement, oxide composition, mineralogy, chemical composition

1. INTRODUCTION

Black cotton soils constitute the predominant soil in North–East Nigeria, Osinubi et al., (2009). They are also found in many other parts of the world, in Australia, Sudan, USA (Texas, California, Montana), India, Argentina, China and Eastern Europe.

Black cotton soil is dark in color. The main characteristics are excessive swelling and loss of strength when it absorbs water. It also shrinks excessively when it dries. It is the main subgrade for the road pavements and foundation of infrastructures in North–East Nigeria. When dry, black cotton soil is very strong with high bearing pressure to support loads. However, when wet, black cotton soil rapidly swell, exhibits a very high swelling capacity, loose its strength, and become very soft with low strength and bearing capacity. This phenomenon and occurrence results in various distresses and deformations on flexible road pavements in Nort–East Nigeria and other parts of the world where black cotton soil is the main natural subgrade, ASCE, (2012); Oghenero et al., (2014); Al–Taiea, (2016).

The high rate of swelling and strength loss when it absorbs water pose different challenges to infrastructures including considerable economic losses. The high swell and strength losses cause road pavement deformations (such as longitudinal cracks and pavement failures) and structural deformations (such as excessive settlement, foundation failures, upheaval of ground floor and uplift of infrastructures), Adams and Adetoro, (2014); Aneke, (2021). Therefore, BCS need to be stabilized to reduce this tendency of swelling and loss of strength.

In all areas of the world where black cotton soil occurs, roads constructed over black cotton soils show similar engineering problems such as road pavement and foundation failures and instability. Dangerous road deformations occur resulting in loss of life, distress to vehicles, road pavements and foundations. Consequently, structures are also subjected to severe distress, destruction, and high maintenance costs. Many authors (Onyelowe et al., 2021; Ashraf et al., 2019; Alayaki et al., 2015; Meshida et al., 2013; Sabat, 2012; Patil et al., 2011; Ali and Korranne, 2011; Osinubi, 2009;) have reported efforts made to stabilize the behavior of black cotton soil with limited success. Despite the great research efforts, more significant improvements are still being pursued.

Globally, limited success is being achieved to improve the behavior of Black Cotton Soil and other expansive soil for road works using cement and lime, and other cost–effective materials like fly ash, Osinubi, (2006) and soil modifications, Nicholson, (2014).

Results of expansive soil stabilization with chemicals have not been as good as with lime and cement. Sodium hydroxide caused abnormal volume increase, Silvapullaiah et al., (2009). Expansive soil stabilization with waste products has shown limited improvement. CBR obtained fall below minimum required, Osinubi et al., (2009). Hence, the obvious need for continuous research efforts, ASCE, (2012).

The engineering properties of a soil are influenced by the chemical composition of the soil and the minerals present in the soil, Ah–Ram Kim et al., (2017). The aim of this research is to investigate, identify and characterize the mineralogical, chemical, and geotechnical changes in cement stabilized–Black Cotton Soil.

2. MATERIALS AND METHODOLOGY

Black Cotton Soil

Samples of Black Cotton soils were taken at Dikwa, along Maiduguri–Gamboru Rd, Borno State, Nigeria. The samples were obtained and used for the experiments in the natural state. The soil is dark grey to black in color. The terrain is flat with occasional trees.

Ordinary Portland Cement

Globally, ordinary Portland cement is commonly used to stabilize soil. Portland cement manufactured by Lafarge in Nigeria was purchased in the Lagos market and used for the investigation.

Oxide Components

The identification of the oxide components of Black Cotton soil and ordinary Portland cement were determined at the Department of Chemistry, University of Lagos, Nigeria using the Atomic Absorption Spectroscopy, AAS. Table 1 shows the oxide components of black cotton soil with ordinary Portland cement

Mineralogy

Identification of minerals in black cotton soil and cement–stabilized mixture was done with the X–ray Power Diffraction, XRD technique. The mineral identification was done at Cornel Center for Material Research, Cornel University, Ithaca, USA. The minerals identified in black cotton soil are shown in Tables 3.

Chemical Composition and Characterization

The chemical composition and characterization were determined, with an Energy Dispersive X–ray spectroscope, EDX, attached to a Scanning Electron Microscope, SEM at Cornell University Center for Material Research, USA. The SEM–EDX equipment has an electron probe micro–analyzer with high resolution scanning electron microscope that can also detect trace elements.

3. RESULTS AND DISCUSSION

Oxide Components

The oxide components of Black Cotton soil and ordinary Portland cement are tabulated in Table 1. The major oxides of Black Cotton soil are the oxides of Silicon, S_iO_2, (34.25%) and Aluminum, Al_2O_3, (30.74%). Black Cotton Soil is pozzolanic, Aminu et al, (2020). The major oxides of ordinary Portland cement are the oxides of Calcium, CaO (58.75%) and Silicon, S_iO_2, (17.73%). The result agrees with the findings of Bediako and Amankwah, (2015) and Alayaki et al., (2015).

Table 1: Analysis of Oxide Components (%)

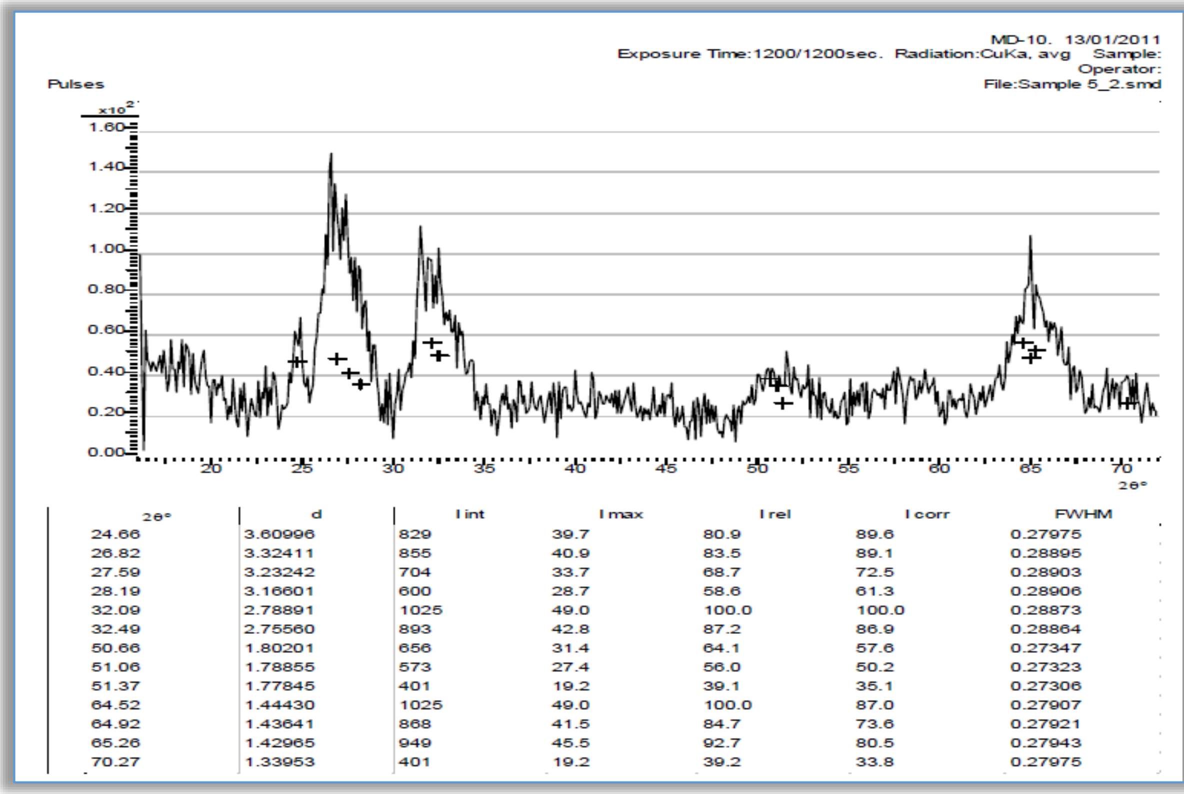
Oxide	Black Cotton Soil	Ordinary Portland Cement
S_iO_2	34.25	17.73
Al_2O_3	30.74	5.19
Fe_2O_3	0.95	De .68
CaO	0.31	58.75
MgO	0.24	2.26
Na_2O	0.07	0.00
K_2O	0.69	0.00
SO_3	0.18	0.44
L.O.I	32.57	12.95
Total	100.00	100.00

Mineralogical Characterization of Borno BCS Samples

The spectrum of the black cotton soil is as shown in Figure 1. The names of nine (9) minerals detected in black cotton soil, including two (2) clay minerals (montmorillonite and kaolinite) are tabulated in Table 2. The minerals detected are Quartz, Tridymite, Clinoenstatite, Montmorillonite, Haematite, Kyanite, Corundum, Doyleite and Kaolinite. Borno State area of Nigeria is in the Lake Chad basin. It is a very high temperature, humid and flat area, with poor drainage and alternating dry and wet seasons.

The presence of clinoenstatine and corundum the black cotton soil sample may be a confirmation of the speculation / circumstantial evidence (Wright, 1970) that the corundum were weathered out of the extensive basalt sheet in this area in which they are believed to have originated. This is similar to the findings of Onyelowe and Usungedo, (2021).

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665



2θ°	d	I int	I max	I rel	I corr	FWHM
24.66	3.60996	829	39.7	80.9	89.6	0.27975
26.82	3.32411	855	40.9	83.5	89.1	0.28895
27.59	3.23242	704	33.7	68.7	72.5	0.28903
28.19	3.16601	600	28.7	58.6	61.3	0.28906
32.09	2.78891	1025	49.0	100.0	100.0	0.28873
32.49	2.75560	893	42.8	87.2	86.9	0.28864
50.66	1.80201	656	31.4	64.1	57.6	0.27347
51.06	1.78855	573	27.4	56.0	50.2	0.27323
51.37	1.77845	401	19.2	39.1	35.1	0.27306
64.52	1.44430	1025	49.0	100.0	87.0	0.27907
64.92	1.43641	868	41.5	84.7	73.6	0.27921
65.26	1.42965	949	45.5	92.7	80.5	0.27943
70.27	1.33953	401	19.2	39.2	33.8	0.27975

Figure 1: XRD spectrum of the black cotton soil

Table 2: Mineralogical Characterization of Black Cotton Soil

No.	Mineral	Name of compound	Formulae	Structure	Colour
1	Quartz	Silicon oxide	SiO_2	Hexagonal	White
2	Tridymite	Silicon oxide	SiO_2	Hexagonal	Colorless
3	Clinoenstatite	Magnesium silicate	$Mg SiO_3$	Monoclinic	Colorless
4	Montmorillonite	Hydrogen aluminium silicate	$H_{0.32}Al_{0.32}Si_{95.68}O_{192}$	Monoclinic	
5	Haematite	Iron oxide	Fe_2O_3	Monoclinic	Dark brown
6	Kyanite	Aluminium silicate	Al_2SiO_5	Anorthic (triclinic)	Blue
7	Corundum	Aluminium oxide	Al_2O_3	Tetragonal	Not stated
8	Doyleite	Aluminium hydroxide	$Al(OH)_3$	Anorthic (triclinic)	Colorless
9	Kaolinite		$Al_2Si_2O_5(OH)_4$		White

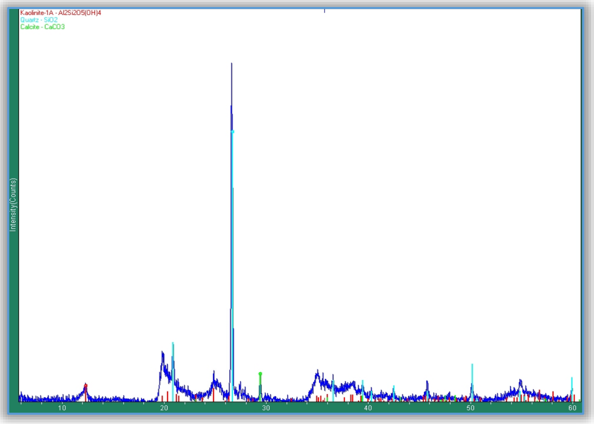


Figure 2: Spectrum of the Black Cotton soil sample

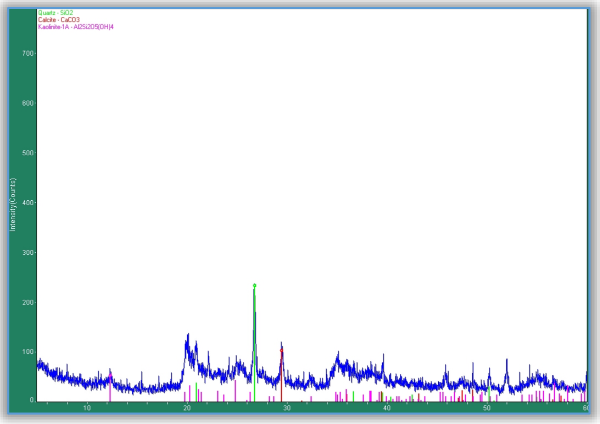


Figure 3: Spectrum of the Black Cotton soil sample with 10%wt. cement

Chemical Composition and Characterization of Cement Stabilized–Black Cotton Soil

— Scanning Electron Microscope Quantitative Analysis

The micrographs of natural and cement stabilized–black cotton soil mixtures are shown in Figures 4 – 7. The micrograph of natural black cotton soil, Figure 4, showed crystalline clay particles that appear amorphous. The particles appear to be loose and rough with visible voids between particles. Figures 5, 6 and 7 are the micrographs of black cotton soil mixed with cement in varying proportions by weight. It shows that the when cement is mixed with black cotton soil, it changed the surface features and properties of black cotton soil. The micrographs show a colour change in black cotton soil from dark grey to whitish grey on addition of cement. The colour change increased with increase in cement content. In addition, large white particles can be seen in the natural black cotton soil, Figure 4, while these appear to have smoothened out and mixed up (flocculated) on addition of cement, (Wu et al., 2019; ElSheikh et al., 2022).

Figures 5, 6 and 7, micrographs of cement stabilized–black cotton soil, also show changes and improvements in the soil characteristics. The voids appeared closer and tighter, particle shapes appear rounder and smoother. There is better cohesion and bonding between the cement stabilized–black cotton soil. This may lead to decrease of Liquid Limit and Plasticity Index, improve workability and engineering behaviour.

The surface of the cement stabilized–black cotton soil also appeared flocculated. Flocculation is an indication of modifications of a soil and changes in the characteristics and properties of the soil. The white gel or coating on the surface may be due to the formation of calcium compounds. Calcium is silvery white and is the main chemical compound in ordinary Portland cement. The colour change indicates the availability of Calcium ions and formation of Calcium compounds, Osinubi, (2006).

— Quantitative Chemical Composition

Figures 8–11 are the EDX Spectrum and quantitative details of the chemical components of the cement stabilized–Black Cotton Soil mixture with varying cement content (0–30%). The distributions of the chemical elements by weight in cement stabilized–black cotton soil is presented in Table 8.

Figure 8, EDX spectrum for black cotton soil, show that the dominant chemical elements, with highest peaks, in the natural soil are Titanium (Ti), Silicon (Si) and Aluminum (Al) in descending order. Figures 9–11, EDX spectra for the cement stabilized–black cotton soil showed that on addition of cement, the dominant chemical elements changed to Titanium, Silicon and Calcium in descending order, In addition, the peaks of Titanium and Silicon became less than that obtained in the natural soil even though in dominion. Calcium displaced the dominion of Aluminum in the natural black cotton soil on addition of cement and reduced the dominion of Titanium and silicon.

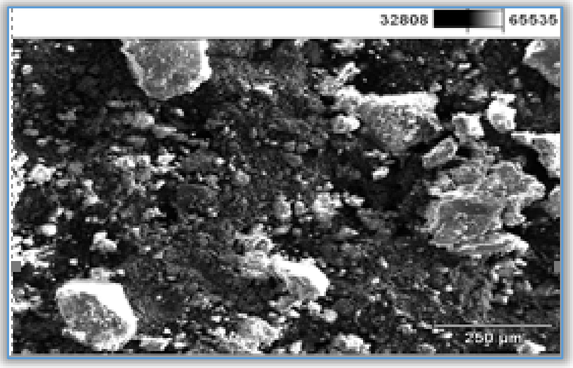


Figure 4: SEM–EDX Micrograph of the Natural Black Cotton Soil (0% Cement)

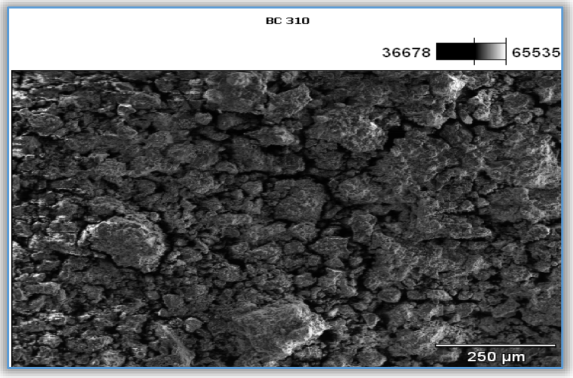


Figure 5: SEM–EDX Micrograph of the cement stabilized–Black Cotton Soil (10% Cement)

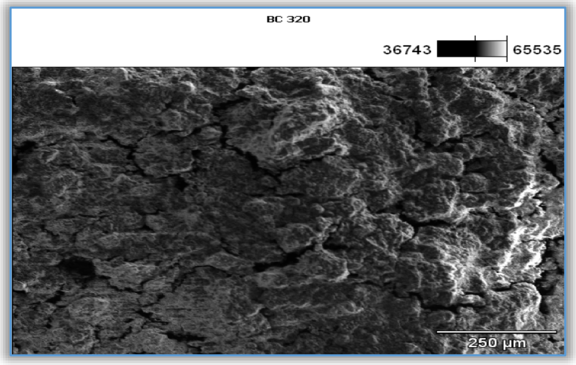


Figure 6: SEM–EDX Micrograph of the cement stabilized–Black Cotton Soil (20% Cement)

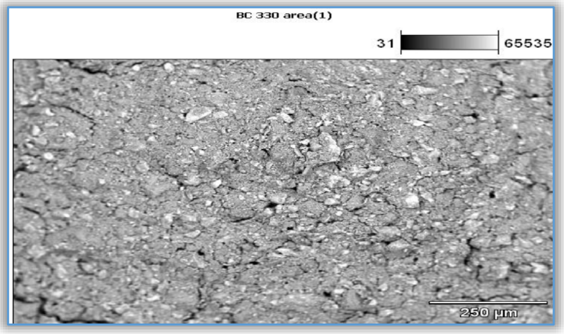
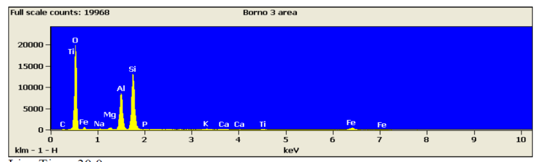


Figure 7: SEM–EDX Micrograph of the cement stabilized–Black Cotton Soil (30% Cement)

The EDX quantitative analysis of the chemical elements of natural Black Cotton soil and of cement stabilized–Black Cotton Soil are also displayed in Figures 8–11 while the change by weight of each chemical is tabulated in Table 7. The variation of each chemical element with cement (0, 10, 20 and 30 % wt. respectively is shown in Figure 12.

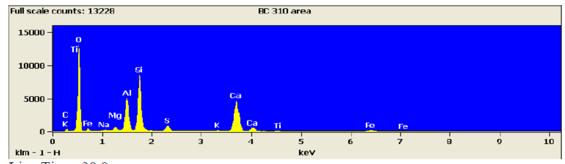


Live Time: 30.0 sec.

Acc.Voltage: 15.0 kV Take Off Angle: 40.0 deg.
Quantitative Results Borno 3 area

Element Line	Weight %	Atom %	Compnd %
C K	0.94	1.66	3.45
O K	48.18S	63.67	---
Na K	0.39	0.36	0.52
Mg K	0.29	0.25	0.48
Al K	13.22	10.36	24.98
Si K	24.79	18.66	53.03
Si L	---	---	---
P K	0.18	0.13	0.42
P L	---	---	---
K K	0.96	0.52	1.15
K L	---	---	---
Ca K	1.14	0.60	1.59
Ca L	---	---	---
Ti K	0.83	0.37	1.39
Ti L	---	---	---
Fe K	9.08	3.44	12.98
Fe L	---	---	---
Total	100.00	100.00	99.99

Figure 8: Chemical Composition of Black Cotton Soil

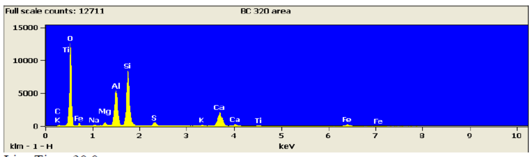


Live Time: 30.0 sec.

Acc.Voltage: 15.0 kV Take Off Angle: 40.0 deg.
Quantitative Results BC 310 area

Element Line	Weight %	Atom %	Compnd %
O K	42.18S	60.66	---
Na K	0.41	0.41	0.55
Mg K	0.45	0.43	0.75
Al K	8.09	6.90	15.29
Si K	16.48	13.50	35.25
Si L	---	---	---
S K	2.45	1.76	6.13
S L	---	---	---
K K	0.70	0.41	0.85
K L	---	---	---
Ca K	23.75	13.64	33.23
Ca L	---	---	---
Ti K	0.47	0.22	0.78
Ti L	---	---	---
Fe K	5.02	2.07	7.18
Fe L	---	---	---
Total	100.00	100.00	100.01

Figure 9: Chemical Composition of BCS stabilized with 10% Cement

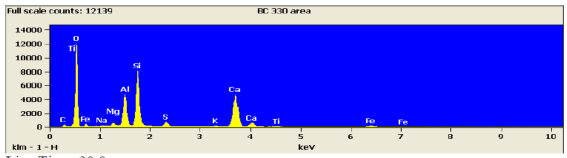


Live Time: 30.0 sec.

Acc.Voltage: 15.0 kV Take Off Angle: 40.0 deg.
Quantitative Results BC 320 area

Element Line	Weight %	Atom %	Compnd %
O K	44.97S	62.26	---
Na K	0.51	0.49	0.68
Mg K	0.38	0.34	0.62
Al K	10.73	8.81	20.28
Si K	20.52	16.18	43.91
Si L	---	---	---
S K	2.05	1.41	5.11
S L	---	---	---
K K	0.83	0.47	1.00
K L	---	---	---
Ca K	13.09	7.23	18.32
Ca L	---	---	---
Ti K	0.79	0.37	1.32
Ti L	---	---	---
Fe K	6.12	2.43	8.75
Fe L	---	---	---
Total	99.99	100.00	99.99

Figure 10: Chemical Composition of BCS stabilized with 20% Cement



Live Time: 30.0 sec.

Acc.Voltage: 15.0 kV Take Off Angle: 40.0 deg.
Quantitative Results BC 330 area

Element Line	Weight %	Atom %	Compnd %
O K	41.76S	60.37	---
Na K	0.43	0.43	0.58
Mg K	0.41	0.39	0.68
Al K	7.86	6.74	14.86
Si K	16.33	13.44	34.93
Si L	---	---	---
S K	2.10	1.51	5.24
S L	---	---	---
K K	0.67	0.40	0.81
K L	---	---	---
Ca K	24.95	14.40	34.91
Ca L	---	---	---
Ti K	0.63	0.31	1.06
Ti L	---	---	---
Fe K	4.86	2.01	6.94
Fe L	---	---	---
Total	100.00	100.00	100.01

Figure 11: Chemical Composition of BCS stabilized with 30% Cement

Table 7: Distribution of Chemical Element in BCS by Weight with Cement from SEM test

Element Line	Standard Formulae	Cement Content %						
		BCS	BC310 (10%wt. cement)		BC320 (20%wt. cement)		BC330 (30%wt. cement)	
		0%	Weight, %	Percentage increase	Weight, %	Percentage increase	Weight, %	Percentage increase
C K	CO_2	0.94	0.00	−100	0.00	−100	0.00	−100
O K	SiO_2	48.18	42.18	−12.45	44.97	−6.66	41.76	−13.33
Na K	Na_2O	0.39	0.41	5.1	0.51	30.77	0.43	10.25
Mg K	MgO	0.29	0.45	55.17	0.38	31.03	0.41	41.38
Al K	Al_2O_3	13.22	8.09	−38.80	10.73	−18.84	7.86	−40.54
Si K	SiO_2	24.79	16.48	−33.52	20.52	−17.22	16.33	−34.13
S K	SO_2	0.00	2.45	245	2.05	205	2.10	210
P K	P_4O_{10}	0.18	0.00	−100	0.00	−100	0.00	−100
K K	K_2O	0.96	0.70	−27.08	0.83	−13.54	0.67	−30.21
Ca K	CaO	1.14	23.75	1983.33	13.09	1048.25	24.95	2088.6
Ti K	TiO	0.83	0.47	−43.38	0.79	−4.82	0.63	−24.1
Fe K	Fe_2O_3	9.08	5.02	−44.71	6.12	=32.60	4.86	−46.48
Total		100.00	100.00		99.99		100.00	

The natural black cotton soil has a very low percentage of ions of Sulphur (0%), Phosphorous (0.18%), Magnesium (0.29%), Sodium (0.39%), Titanium (0.83%), Potassium (0.96%) and Calcium (1.14%), Table 7.

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

On addition of cement, the quantities of these ions with low percentages increased: Sulphur (2.45%), Magnesium (0.45%), Sodium (0.41%), Calcium (23.75%) while the quantities of following elements reduced: Phosphorous (0%), Titanium (0.47%), Potassium (0.7%). Sulphur, Magnesium, Sodium and Calcium which increased in quantities in the cement stabilized–black cotton soil are major positively charged ions.

There is no Calcium ion inside natural Black Cotton Soil. The mineralogy of Black Cotton soil detected the presence of montmorillonite, a clay mineral. Clay minerals have a net negative charge on their surface. On the addition of cement to black cotton soil, negatively charged clay minerals attract the positively charged ions in cement (sodium, magnesium, Sulphur and calcio). Hence when cement is added to Black Cotton Soil, there is a rapid increase in the Calcium content from 1.14% to 23.75% (at 10%wt. cement). This is a result of an exchange of ions (cation exchange) where positively charged Calcium ions replaces other positively charged ions in the black cotton soil to form new chemical compounds. The increase in calcium signifies the absorption and exchange of positively charged ions from cement (Table 7), while carbon and phosphorous contents were not noticeable. The quantity of exchangeable cations (sodium, magnesium, Sulphur and calcium) increased with the addition of cement. Sodium content increased by 5%, magnesium content by 55%, calcium content increased by over 1900% while Sulphur content increased by 245% at 10%wt. cement content. Quantity of Aluminum, Silicon, Phosphorous and Ferrous decreased with addition of cement. The adsorption of positively charged Calcium ions will lead to formation of calcium compounds in the cement stabilized–black cotton soil. Calcium compounds are very stable thereby making the mixture stable. The result show that a good stabilizer must be able to react with the clay minerals in the soil, form new chemical compounds that are very stable and have strong bonds in order to effectively stabilize weak soil. The result also indicate the need study and determine the chemical composition and characteristics of a soil, the clay minerals and possible positively charged chemical ions that will form chemical compounds with the soil that are stable, with high strength and very strong bond to effectively stabilize and strengthen weak soil, ElSheikh et al., (2022).

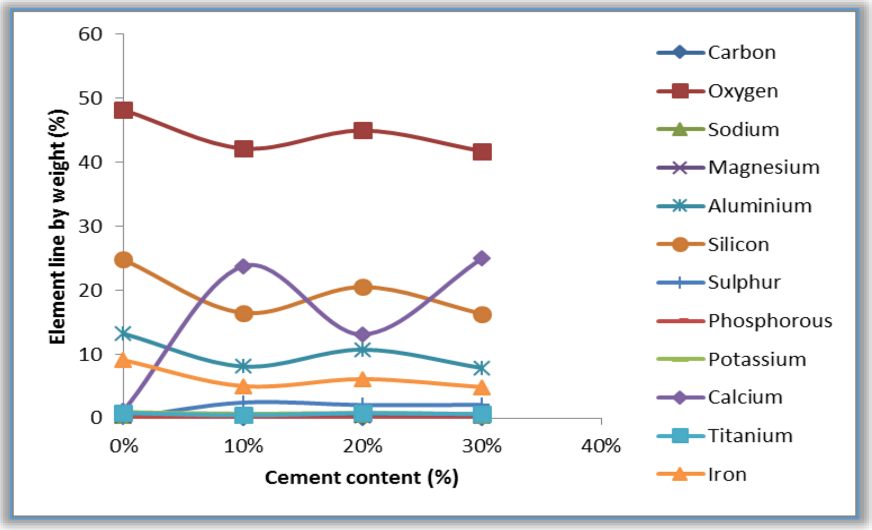


Figure 12: Variation of Chemical Element by Weight, with Cement Content, from SEM test

4. CONCLUSION

- The research characterized the mineral constituents of black cotton soil as an expansive clay containing clay minerals (montmorillonite, and Kaolinite), Quartz, Tridymite, Clinoenstatite, Haematite, Kyanite, Corundum and Doyleite.
- The research characterized the oxide components of Black Cotton Soil and ordinary Portland cement. The major oxides in black cotton soil are silicon oxide, S_IO_2, (34.25%) and aluminum oxide, Al_2O_3, (30.74%). It can be classified as a pozzolan. The major oxides in ordinary Portland cement are calcium oxide, CaO (58.75%) and silicon oxide, S_IO_2, (17.73%).
- The SEM microstructural analysis established the modification of the structure of the clay minerals.

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

- The microstructural analysis also established the flocculation and formation of cementitious white coatings on the surface of the soil mixture on the when cement is added.
- The EDX spectra of the chemical elements characterized natural black cotton soil as having 3 main dominant peaks of Titanium (Ti), Silicon (Si) and Aluminum (Al) in descending order. On addition of cement, the EDX spectrum of the cement stabilized–black cotton soil changed to four (4) main dominant peaks, the dominant chemical elements changed to Titanium, Silicon, Calcium and Aluminum in descending order. In addition, the peaks of Titanium and Silicon became less than that obtained in the natural soil. Calcium displaced the dominion of Aluminum.
- The quantitative investigation of the chemical elements characterized the natural black cotton soil as having very low quantity of ions of Sulphur (0%), Phosphorous (0.18%), Magnesium (0.29%), Sodium (0.39%), Titanium (0.83%), Potassium (0.96%) and Calcium (1.14%), The addition of 10%wt. cement increased the quantities of Sodium, Magnesium, Calcium and Sulphur by 10%, 41%, 2090% and 210% respectively. These are major positively charged ions.
- The addition of cement to black cotton soil causes the release of major positively charged ions, Sodium, Magnesium, Calcium and Sulphur, which reacts with negatively charged ions in the black cotton soil clay minerals. The rapid increase in calcium content and dominion indicates absorption and formation of strong calcium compounds and modifications to the black cotton soil.
- The research showed that chemical reactions, formation of new chemical compounds and type of chemical compounds formed have significant importance on the modifications and stability of black cotton soil. The identification and characterization of chemical elements in both soil and stabilizer are necessary.
- The changes in the mineralogy, chemical composition and behaviours of cement stabilized–Black Cotton soil have significant effects on the engineering properties. These will be investigated and published in subsequent publications.

Acknowledgment

The authors appreciate the support of the Management and staff of Cornell Center for Materials Research; Management and staff of Chemical Department and Civil Engineering Department, University of Lagos for their facilities, contributions and support.

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ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665