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Microstructural properties of lime stabilized naturally occurring acidic soil

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Abstract: Natural Acid occurring soils are very common in regions with high rainfall. KwaZulu-Natal (South Africa) is no exception to this. Agricultural wise, these soils are a major cause of poor yields of crops and vegetables, limiting plant growth by stunting root development and thereby the uptake of water and nutrients. Mixing lime into the topsoil is one of the effective ways of dealing with soil acidity problems. Engineering wise, the use of lime to soil is associated with weak, unstable or unsuitable soils. It is often a norm that when natural or imported natural occurring soil is encountered during road construction for base courses, the need to intervene in improving the structural stability of such soils arises. One of the intervention which has been used and still in use involves the application of lime to soil so as to enhance the stability in the soils.

An experimental program was undertaken to investigate the effects of hydrated lime in studying the microstructural of natural occurring acidic soil samples. Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) tests were used to indent chemical composition and structure of the three soil samples treated with lime. Three (3) natural occurring acidic soil samples were collected at three different locations and treated with lime contents (i.e. 2%, 4%, 6%, 8% and 10% by weight of soil). Further tests such as gradation & consistency limits, were conducted.

The test results indicated that the inclusion of lime to the three soil samples changes the pH (increases) of the tested soil (acidic). SEM and EDS graphics showed that lime treatment changed significantly the soil fabric depending on curing time and water content, this time being 7 days of curing.
Keywords: Microstructural analysis, soil properties, SEM, EDS.

1. INTRODUCTION

1.1Literature Review

Majority of natural occurring soils in the province of KwaZulu Natal (South Africa) are of acidic nature (Diop *et al.*, 2011), and this is due pH below scale of seven (7) and due to high percentage of rainfall the province receives annually. The farming community at all times relies on good quality soil to produce crops and other, hence these soils are a major cause of poor yields of crops and vegetables, limiting plant growth by stunting root development and thereby the uptake of water and nutrients (Iowa State University, 2002; Oosterbaan, 2003; New York state department of transport, 2007; Scientific engineering response and analytical services, 2011). Mixing lime into the topsoil is one effective way of dealing with soil acidity problems. In most of the civil engineering works, the use of lime to soil is associated with weak, unstable or unsuitable soils. The application of lime to soil in trying to increase its strength reduces the acidity of the soil by promoting an increase in its pH, thus enhancing many properties of the soil (Dhakal, 2009; Liu, Pemberton and Indraratna, 2010; Kalantari, 2012; Khattab and Hussein, 2012; Aldaood, Bouasker and Mukhtar, 2014; Jawad *et al.*, 2014). One of these properties is the micro structural properties of the lime stabilized soil.

The microstructural analysis of the soil referred here, relates to examination and the studying of soil sample under magnification so as to determine the properties of soil hence determining how they perform under a given application (Tafffner *et al.*, 2004; Insight, 2013).

Many researches have been carried out to study the changes in the microstructural and mineralogical characteristics of stabilized soils with calcium-based stabilizers (or additives) such as hydrated lime (or lime) (Goegr and Markgraf, 2006; Deneele *et al.*, 2010; Solanki and Zaman, 2012; Buttress, Grenfell and Airey, 2013; Muhmed and Wanatowski, 2013). In the presence of water, the calcium ions released from these stabilizers reduce the thickness of double diffused layer through cation-exchange and flocculation-agglomeration reactions. This is primarily responsible for improvement in workability through reduction of adsorbed water and decrease in plasticity index of the treated soil (Solanki and Zaman, 2012).

The study presented by (Deneele *et al.*, 2010) presents the effect of a saturated Ca (OH)2 solution circulation through lime-treated sample at 60 °C for 3, 6 and 12 months, respectively. According to this study results, the effect of such circulation on the lime-treated Manois argillite (MA) was assessed by petro graphical examination coupled to image analysis and scanning electron microscopy (SEM) equipped with energy dispersive X-ray (EDX) analyzer of soil pieces. Part of the results based on the SEM indicated that lime leads to a modification of the surface state of the soil grains that were tested by the secondary precipitation of amorphous hydrates due to the development and the precipitation of hydrates promoting the collapse among the grains hence modifying the microstructure of the MA.

Another study by Solanki and Zaman (2012), mineralogical studies namely, SEM and EDS were conducted on raw soils, raw additives powder, raw additives paste and 28-day cured stabilized Carnasaw soil specimens to study the influence of stabilization on microstructure and mineralogical characteristics. The results based on both the SEM and EDS results showed presence of C-S-H, C-A-S-H and ettringite crystals in both CFA-and CKD-stabilized specimens. The XRD results showed a general reduction in all clay minerals' peak intensities particularly in the case of lime-stabilized samples.

Buttress, Grenfell and Airey (2013) paper reports on the dimensional and strength changes of a range of artificial lime stabilized cohesive soils subject to two swell test. In this report, the resulting microstructural composition was analyzed using a combination of Scanning Electron Microscopy and Electron Dispersive X-ray Spectroscopy (SEM-EDX). The results as explained in the paper established theories of crystal formation and subsequent expansion mechanisms in a form of ettringite which exhibit a range of morphologies dependent on the physio-chemical environment in which it forms.

In a paper which aims to investigate the effect of hydrated lime on the strength and microstructure of lime treated clays through a series of laboratory tests conducted, the results indicated that the addition of lime resulted in a reduction in the plasticity of kaolin and an improvement in compaction properties while the SEM analysis showed the presence of the cementious products in the kaolin clay resulted from lime-clay reaction (Muhmed and Wanatowski, 2013).

2. MATERIALS AND METHODS OF TESTING

2.1. Sample preparations

Bags of naturally occurring acid soil samples were collected from Umlazi, a township on the east coast of KwaZulu-Natal, South Africa, located south-west of Durban (29°58′S 30°53′E), Scottburgh is a coastal situated on the mouth of the Mpambanyoni river (30°17′S 30°45′E) and Amanzimtoti, (26.2 km South of Durban in South Africa. Soil samples for laboratory analyses were typically air dried and pulverized to provide a stable homogeneous mixture.

2.2. Tests conducted on soils and testing procedures

Tests such as sieve analysis, consistency limits (Atterberg tests), Maximum Dry Density and Optimum Moisture Content determination, Curing Soaking- for CBR penetration, CBR Penetration were performed. These tests were conducted in relation to South African Technical Methods of Highway 1 (TMH1) under subsections, method A2, A3, and A4 with the prime purpose being to quantify different properties of the soils prior to treating it with lime. Microstructural analysis test on soil was also conducted through micrographs. The micrographs were analyzed to examine the microstructural development of cementitious substances and mineral identification due to stabilization of the three soil samples with different lime contents.

Firstly, soil sample were x rayed from 2° to 70° theta using Cu Kά radiation. The range of 2° to 70° degrees 2-theta was chosen to provide enough X Ray diffraction peaks to identify most common soil minerals on the soil samples that were investigated. Material wise, fine soil grains treated with lime variations and passing 0.425mm sieve size was used for analysis. Secondly, the determination of the types and relative amounts of the minerals present in the soil (soil mineralogy) was determined because of its influence on the soil behavior, its use in soil classification, and its relevance to soil genetic process. Tiny pieces of soil were collected from samples after the UCS tests, oven dried, and then visually observed using SEM/EDS.

3. PRESENTATION AND DISCUSSION OF RESULTS

3.1 Properties of the soil

3.1.1. Consistency limits

Table 1: Consistency index properties of the soil

Property	Sample 1	Sample 2	Sample 3
Liquid limit (%)	32	23.43	22
Plastic limit (%)	21.25	10.61	11.31
Linier shrinkage (mm)	4.21	4.24	3.89
Plasticity index (raw) (%)	10.98	12.82	10.68
Plasticity index (treated) (%)	8.42	10.72	7.61

Soil properties helping in the identification and classification of soil are presented and discussed below. The soils are classified and identified based on index properties. "TABLE 1" shows consistency limits (atterberg limits) results for the three soil samples tested. After lime application, the plasticity decreases (as can be seen in "TABLE 1"-untreated vs lime treated soil samples).

3.1.2. Grading envelop for the three soil samples

Soil gradation/classification ranks the soil based on the different particle sizes contained in the soil for the three soil samples "Fig.1", shows the grading curves for sample 1, 2 and 3 respectively of acidic nature. Based on the grading analysis on the three soil samples indicated that sample one (well graded) is a well graded soil containing particles of a wide range of sizes and has a good representation of all sizes with sample two and three being semi-well graded.

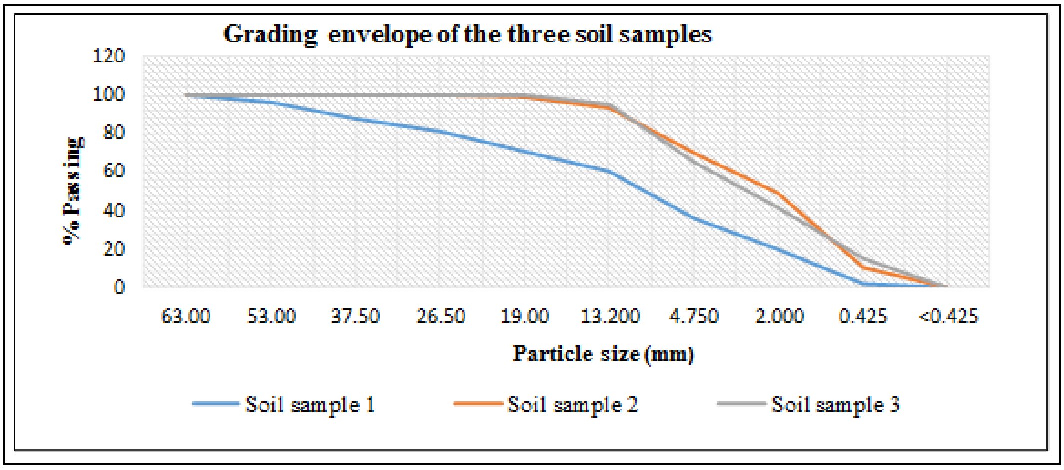


Figure 1: Grading envelop of soil samples

3.1.3 Engineering properties of the soil

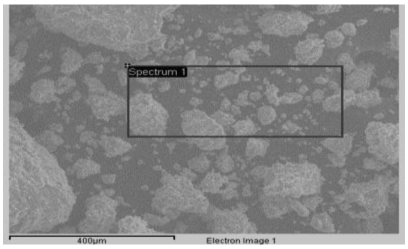
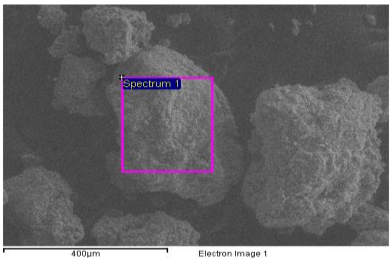
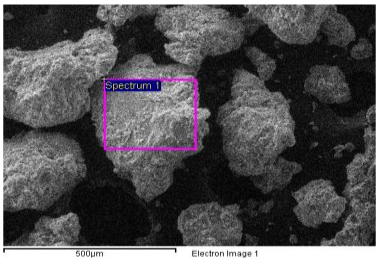
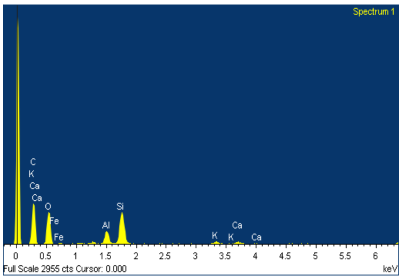
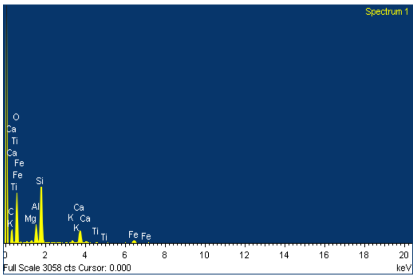
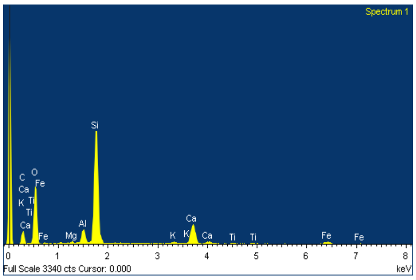
Table 2: Density vs moisture content test results

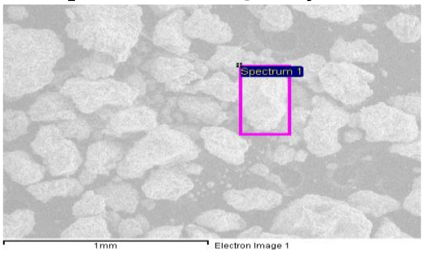
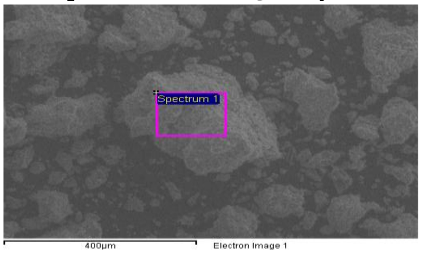
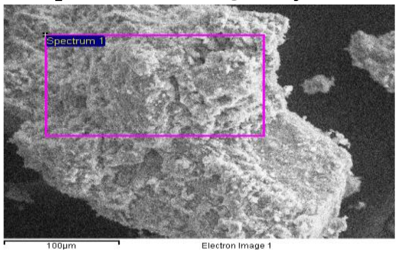
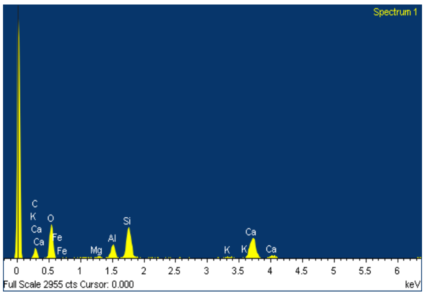
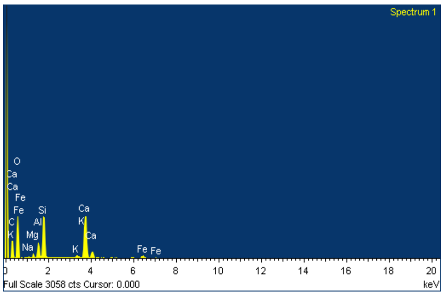
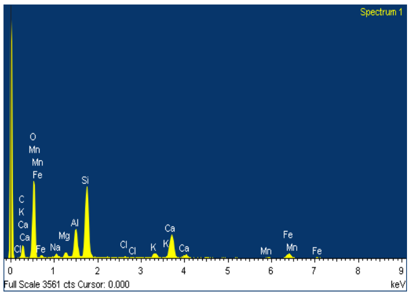
Sample Number	Property (MDD kg/m³) & (OMC %)	2% Lime	4% Lime	6% Lime	8% Lime	10% Lime
1	MDD	1702.8	1702.8	1740.6	1749.2	1672
	OMC	20	20	20	20	19
2	MDD	1667.2	1643.0	1670.1	1677.35	1645.59
	OMC	22	20	20	20	20
3	MDD	1834.4	1937.3	1811.3	1899.9	1792.7
	OMC	15	14	21	14	17

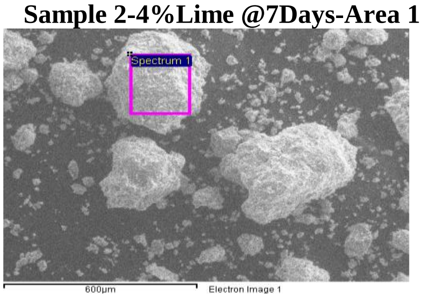
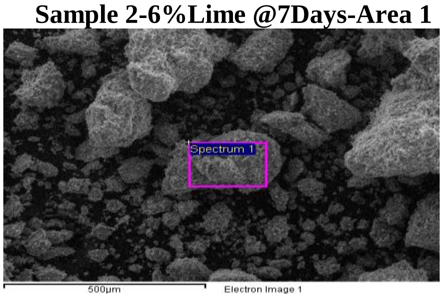
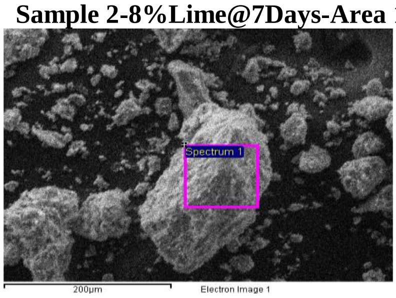
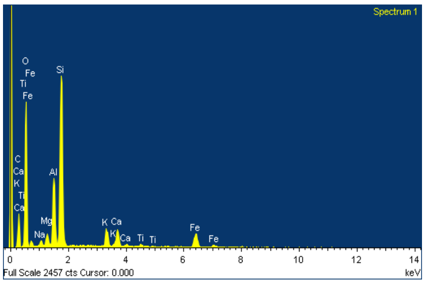
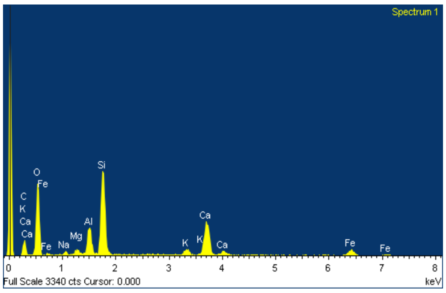
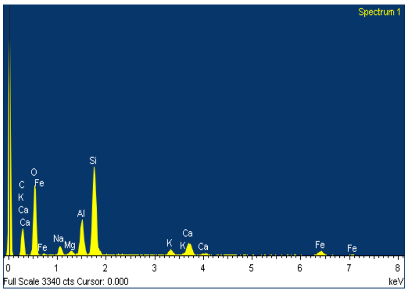
The results of the compaction tests conducted on the soil samples showed that the addition of lime resulted in the improvement in the characteristics of the natural three soil samples. The three soil samples displayed their respective maximum dry densities ranging from 1600 kg/m³ to 1900 kg/m³ and their respective optimum moisture contents ranging from 14% to 20%. With constant comp active effort, the addition of different lime contents indicated highest values of the MDD with corresponding values of OMC for sample three at lime content of 4% by weight of soil. For samples 1 and 2, results showed that further addition of lime decreases the density with constant or increasing moisture content.

Lime content at range of 4% - 6% indicates density increase of the stabilized soil samples. The dry density of the soil samples showed a decrease with increase in lime content of 10%. (Ajayi, 2012) once reported that the above situation (i.e. the increase in the moisture content vs decrease in dry density in relation to the addition of lime) results from lower amount of compaction or less compactive effort.

Generally, the addition of lime to the sample increases the optimum moisture content and reduces the maximum dry density as lime content increases.

Sample 1-2%Lime @7Days Area 1	**Sample 1-4%Lime @7Days Area 1**	**Sample 1-6%Lime @7Days Area 1**
 **Figure 2a: SEM-2% Area 1**	 **Figure 3a: SEM-4% Area 1**	 **Figure 4a: SEM-6% Area 1**
 **Figure 2b: EDS-2% Area 1**	 **Figure 3b: EDS-4% Area 1**	 **Figure 4b: EDS-6% Area 1**

Sample 1-8%Lime @7Days-Area 1	Sample 1-10%Lime @7Days-Area 1	Sample 2-2%Lime @7Days-Area 1
 **Figure 5a: SEM-8% Area 1**	 **Figure 6a: SEM-10% Area 1**	 **Figure 7a: SEM-2% Area 1**
 **Figure 5b: EDS-8% Area 1**	 **Figure 6b: EDS-10% Area 1**	 **Figure 7b: EDS-2% Area 1**

Sample 2-4%Lime @7Days-Area 1	Sample 2-6%Lime @7Days-Area 1	Sample 2-8%Lime@7Days-Area 1
 **Figure 8a: SEM-4% Area 1**	 **Figure 9a: SEM-6% Area 1**	 **Figure 10a: SEM-8% Area 1**
 **Figure 8b: EDS-4% Area 1**	 **Figure 9b: EDS-6% Area 1**	 **Figure 10b: EDS-8% Area 1**

Sample 2-10%Lime@7Days-Area 1

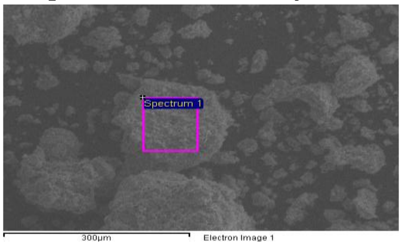


Figure 11a: SEM-10% Area 1

Sample 3-2%Lime @7Days-Area 1

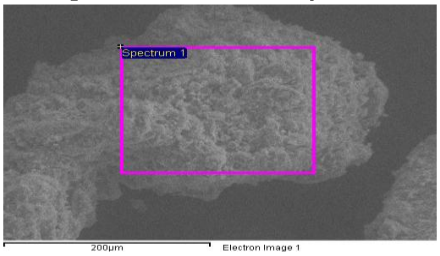


Figure 12a: SEM-2% Area 1

Sample 3-4%Lime @7Days-Area 1

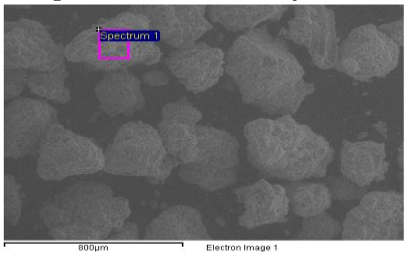


Figure 13a: SEM-4% Area 1

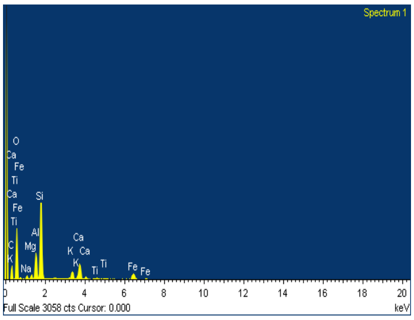


Figure 11b: EDS-10% Area 1

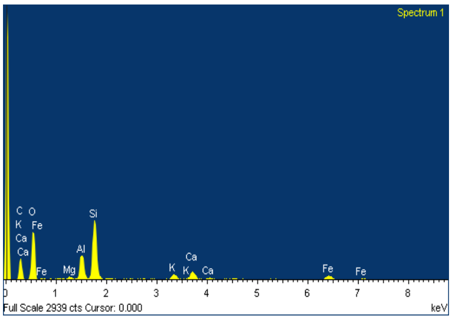


Figure 12b: EDS-2% Area 1

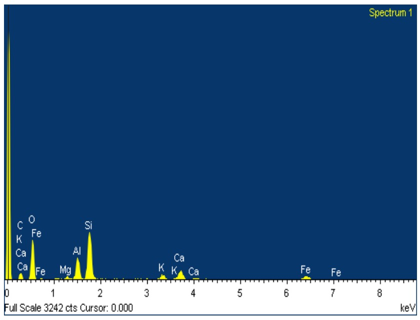


Figure 13b: EDS-4% Area 1

Sample 3-6%Lime @7Days-Area 1

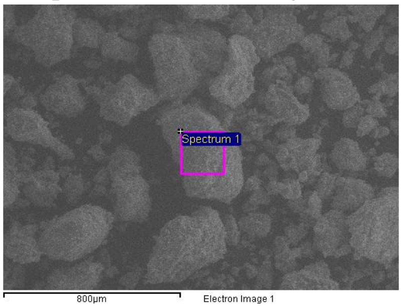


Figure 14a: SEM-6% Area 1

Sample 3-8%Lime @7Days-Area 1

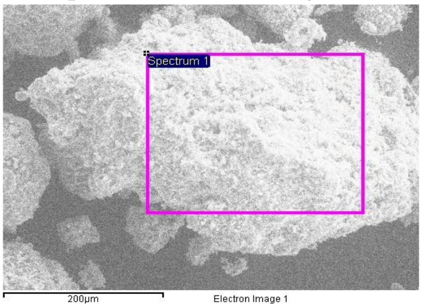


Figure 15a: SEM-8% Area 1

Sample 3-10%Lime@7Days-Area1

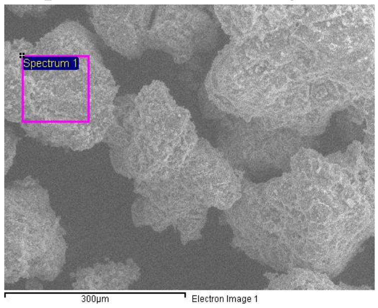


Figure 16a: SEM-10% Area 1

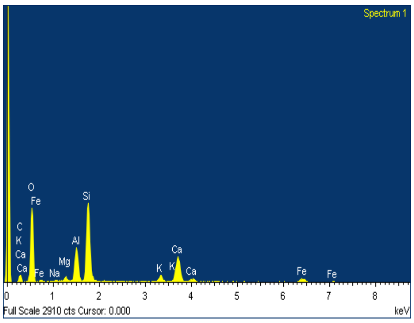


Figure 14b: EDS-6% Area 1

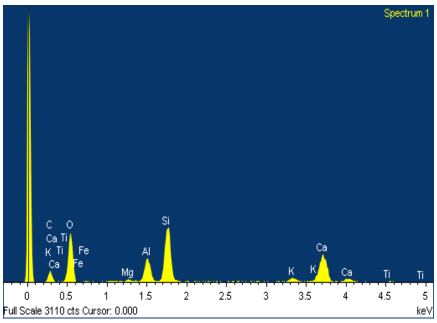


Figure 15b: EDS-8% Area 1

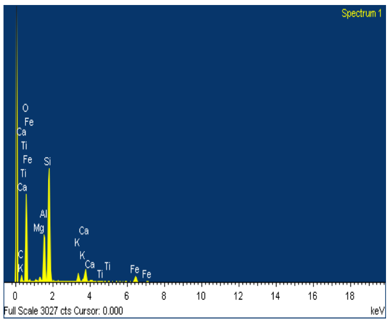


Figure 16b: EDS-10% Area 1

3.1.4 Microstructural analysis
3.1.4.1 SEM & EDS/EDX

SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive Spectroscopy) analysis was carried out on the tiny soil samples after the unconfined compression tests. Lime treatment changed significantly the soil fabric depending on curing time and water content, this time being seven days of curing. Figure 19a to figure 33b shows the SEM and EDS-micrographs of treated soil samples. Images in the SEM/EDS display compositional contrast that results from different atomic number elements and their distribution. As can be seen from "fig." 2a to "fig." 16b, soil particles treated with different content of lime were observed and displayed flaky texture.

Flaky texture confirms the formation of needle-like crystalline formations in the soil sample such as ettringite (The mineral name for calcium substance (Portland cement association, 2001). Further to the above mentioned, the below figures show a reduction in pore spaces or the non-existence of the pores within the soil particles indicating the change in microstructure of the soil on addition of stabilizer (lime) hence promoting the strength of the tested soil samples. During lime soil reaction, hydroxide (OH^{-}) and bicarbonates ($HC03^{-}$) are released, hence reducing the soil acidity (pH increase in the soil sample) .The EDS show elements found in the soil, majority of the soil chemical elements evaluated were significantly influenced by use of lime to the three soil samples. Chemical elements, ordered by their atomic number (number of protons), electron configurations, and recurring chemical properties from the SEM on the soil samples treated with different lime contents were identified on the soil samples tested. These (elements) ranged from Magnesium (Mg), Potassium (K), Calcium (Ca), Titanium (Ti), Iron (Fe), Aluminium (Al), Silicon (Si) Oxygen (O) to Carbon (C).

With limestone being the source of Ca and Mg and in the presence of water the carbonates dissolve and the Hydroxide, some of the elements listed above form part of the chemical composition making up the hydrated lime used for this experiment. Among these are Calcium, Magnesium, iron, and silicone as can be seen on the EDS images.

Furthermore to the above listed elements, carbon listed relates not to the element that was found in the soil but the non-porous carbon double sided adhesive tape which was used for SEM or EDS tests. The double sided adhesive permitted quick mounting of samples without using liquid or colloidal adhesives.

Due to chemical reactions occurring to the soil particles treated with lime, cementitious substances of the soil samples resulting from the use of hydrated lime are enhanced by the changes in soil properties.

4. CONCLUSION

Significant increase in the soil pH due to use of lime contents in the soil samples leading to the reduction of soil acidity was observed and discussed through the microstructural analysis of the soil samples tested for the experiment. In studying the soil samples under magnification so as to determine how they perform under a given application, in this case under the application of lime gave the following conclusion.

The behavior of treated soils shown by the SEM-micrographs indicated the formation of flaky texture of cementitious form. This flaky texture which confirms the formation of needle-like crystalline formations in the soil sample were shown in the micrographs of the lime treated soil samples due to reactions which contributed to the increase of the strength of stabilized soil samples.

According to EDS, certain elements were found in the soil. These (elements) ranged from Magnesium (Mg), Potassium (K), Calcium (Ca), Titanium (Ti), Iron (Fe), Aluminium (Al), Silicon (Si) Oxygen (O) and Carbon (C). Majority of the soil elements evaluated were significantly influenced by use of lime and other factors to the three soil samples. Elements such as Ca and Mg relating to limestone being the source of these elements in the presence of water during manufacturing.

Based on the experimental results, it can be concluded that the use of hydrated lime for the experiment effectively enhanced the soil properties as can be justified by the SEM/EDS micrographs of lime treated soil sample indicating cementitious substances of the particles due to chemical reactions occurring to the soil particles treated with lime.

5. ACKNOWLEDGEMENTS

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