X-Ray Diffraction And Mineralogical Analysis Of Expansive Soils In Kibaha, Tanzania

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ABSTRACT:- This paper describes the chemical and mineralogical aspects of the expansive soils in Kibaha, Tanzania. It starts by giving general overview of the structure and composition of the expanding clay minerals together with their chemical properties. The identification of the presence of expanding clay minerals by using different x-ray diffraction is explained. Thereafter, soil samples were analyzed for whole-rock and clay sized fractions using XRD. The whole-rock XRD analysis indicated that the rocks are composed of predominantly quartz, albite and montmorillonite. For mineralogical composition, clay size fractions were prepared by sedimentation techniques. The sample was scanned with a 1-s step time at 0.02° 2θ increments between 2° and 32°, thus well-crystallised clay minerals were verified an all particle-sized fraction by the presence of clear peaks in the diffractograms. To determine the major clay mineral components, the samples were then glycolated with a few drops of ethylene glycol and equilibrated overnight and X-rayed. The proportions of kaolinite and smectite were determined by fitting the XRD patterns of ethylene-glycol-saturated samples where *smectite minerals* was calculated to be 57-65% with average of 60%) and kaolinite content with average content less than 10%. Each clay-mineral group has its own particular physical properties and characteristics in the system. A high swell potential was indicated by the sharp maximum in percent smectite and a minimum in percent kaolinite. The vast majority of the expandable smectite clay minerals is attributed to the complex sequence of late mesozoic and early cenozoic sediments.

Keywords:- *Clay minerals, Kaolinite, Smectite, Illites and X-ray diffraction (XRD)*

I.   INTRODUCTION

Expansive soils owe their specific properties to the presence of swelling clay minerals, mainly smectite. The term 'clay minerals' refers to hydrous aluminium phyllosilicates minerals that are fine grained (<0.002 mm) with sheet-like structures and very high surface areas (Cameron, 1992, Velde & Druc 1999 and Uddin, 2008). The clay minerals consist of silcon-oxygen tetrahedral $((Si_4O_{16})^{2-})$ layer and aluminium $(A_{12}(OH)_6)$ or magnesium $(Mg_3(OH)_6)$, the brucite or gibbsite sheet in the octahedral layer (Grim, 1968 and Van Olphen, 1991). The most common clay minerals include kaolinite group minerals (kaolinite, dickite, halloysite and nactrite), chlorite group, illite group (clay-micas), and smectite (pyrophyllite, talc, vermiculite, sauconite, saponite, nontronite and montmorrillonite) and mixed-layer phases (Net *et al*., 2001, Mukherjee, 2013, Guggenheim *et al*., 2006 and Jackson & Richardson, 2007).

The most common smectite characterized by its expandable properties is montmorillonite $(Na,Ca)_x(Al,Mg)_2(Si_4O_{10})(OH)_2 \cdot nH_2O)$. The term smectite (generally used to refer montmorillonite) represents a family of expansible 2:1 phyllosillicate clay minerals having a layer lattice structure in which two tetrahedral sheets are separated by a layer of octahedral sheet (Laird *et al*., 1991 and Diaz *et al*., 2001) as shown in Figure 1. Minerals in the three-layer clays are produced by substitution of Al^{3+} or occasionally Fe^{3+} for Si^{4+} in tetrahedral sheets and substitution of low charge species such as Mg^{2+}, Fe^{2+}, Zn^{2+}, Ni^{2+}, Mn^{2+}, Li^+ etc. for Al^{3+} in the octahedral sheet. Substitution of Si^{4+} in the tetrahedral sheet is up to 15% to the maximum mainly by Al^{3+} ions (Grim, 1968 and Militzer *et al*., 2011). To balance the negatively charged structure stemming from internal substitution of trivalent ions for silica, layers of loosely held hydrated cations such as Ca^{2+}, Mg^{2+}, Na^+ or H^+ (Odom, 1984), separate the structural units (tetrahedral and octahedral sheets). Depending on the ions of substitution, smectite can be either dioctahedral when substitution occurs in alumna (gibbsite) octahedral sheet and silica tetrahedral sheets, or trioctahedral when substitution occurs in magnesium (brucite) octahedral sheet and silica tetrahedral sheets (Kloprogge *et al*., 1999, Barton and Karathanasis, 2002 & Muhammad, 2004).
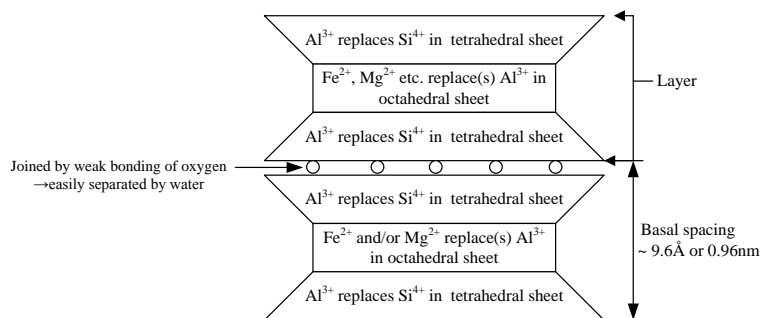
Figure 1: Schematic diagram of smectite (montmorillonite) crystals (Sivakugan, 2001).

Usually, the substitution of Al^{3+} by Mg^{2+} in octahedral sheet in montmorillonite creates net negative charges on the clay structure that cause the interlayer space to shrink or expand with change in humidity or moisture content in contact (Tambach *et al.*, 2004 and Burzo, 2007). The three-layer clay mineral as shown in Figure 1 has a structural configuration and chemical makeup, which permits a large amount of water to be adsorbed in the interlayer and peripheral positions on the clay crystalline, resulting in the remarkable swelling of soil (Patrick and Snethen, 1976). As the water molecules are pulled into gaps between the clay plates, the plates are forced further apart, leading to an increase in soil pore pressure and volume. Expanding soils have been a cause for concern in construction because of the damages they cause on structures directly supported on them. Unless identified and rectified before construction, expansive soils may results in significant structural damage over time.

Undeniably, expansive soils comprise expanding clay minerals of varied structural groups and structural varieties coexisting without niche differentiation. Structurally minerals of different composition show characteristic differences in intensities of their X-ray diffraction effects. The presence of various minerals such as montmorillonite in the expansive soils is determined by the use of x-ray diffraction (XRD), cation exchange capacity (CEC), thermal gravimetric analysis (TGA), infrared spectroscopy (IR), specific surface area etc. The basal spacing values (in Angstroms) determined by XRD, specific surface area and cation exchange capacity (CEC) for different clay mineral groups are given in Table 1

Table 1: Some of clay minerals characteristics (Mitchell, 1993).

Mineral	Interlayer bond	Basal spacing	Specific surface area (m^2/gm)	Cation exchange capacity (meq/100 g)
Kaolinate	Hydrogen; Strong	7.2Å	10-20	3-15
Montmorillonite	Oxygen-oxygen; Very weak	9.6 Å	700-840	80-150
Illite	K ions; Strong	10 Å	65-100	10-40
Vermiculite	Weak	10.5-14 Å	870	100-150
Chlorite	Strong	14 Å	80	10-40

II. OBJECTIVE

The objective of this study was to identify both chemical and mineralogical quantitative phases of the expansive soil mineral assemblage in Kibaha, Tanzania. The objective had two targets: (i) whole-rock XRD analysis that allows identification of the mineral because each mineral has a set of unique d-spacings and (ii) basic soil description with identification of major clay forming minerals by sedimentation techniques. Quantitative mineralogical analyses are instrumental in the delineation of the geochemical behaviours of expansive soils in the area. Both whole rock and clay fraction X-ray diffraction analysis provide detailed information about the precise mineralogical information of the sample including the semi-quantitative estimates of the major mineral abundances.

III. MATERIALS AND METHODS

Clay soil samples were collected from open pits 3 meters deep that were manually dug in Kibaha Township where expansive soils are common. Kibaha is a township located in eastern Tanzania, about 40 km

west of Dar es Salaam (the commercial capital city of Tanzania), along the Dar Es Salaam-Morogoro highway (Figure 2). It's positioned at an altitude of about 155 m above sea level and located approximately by geographic latitude and longitude of 06º46'S and 38º55'E respectively. The region comprises a complex autochthonous and allochthonous sequence of late mesozoic and early cenozoic sediments. The indigenous and non-indigenous sediment fillings are composed of lacustrine, fluviatile, residual, pluvial and alluvial deposits that include micaceous materials (micaceous schists, clay shale, siltstones, silty mudstones etc), calcareous sandstones, limestones, marine marls, shells, organic materials and conglomerates. By the processes of chemical and physical weathering, these conglomerates converted to soils rich in clay. Typically, the deposits are reddish brown, grey brown and grey in colour (Lucian, 2008).

The retrieved samples were wrapped in cellophanes and aluminium foils, logged and transported to the laboratory at African Minerals and Geosciences Centre (AMGC) formerly known as Southern and Eastern African Mineral Centre (SEAMIC). The samples were then ground by hand using a mortar and pestle for whole rock analyses. Clay size fractions were collected from each of the whole rock samples by sedimentation techniques. A portion of each sample was jaw crushed and 25g from each taken. The samples were mixed with the dispersing agent (sodium pyrophosphate) in a 500 ml bottle. 100 ml standard raw water solution was added to the mixture and well shaken. The mixture was sieved through a 63μm sieve while pouring the mixture into a 1-litre cylinder and adding distilled water up to the 1000 ml mark. The mixture was left to settle overnight. A long glass tube was taken and a mark put at 20.9 cm. The tube was then immersed in the measuring cylinder until the 20.9 cm mark touched the water level. The sample was drawn through the tube using a teristatic pump. Thereafter, the sample was dried in an oven at 105°C. 80 mg of the dry sample was weighed and mixed with 1 ml distilled water in a test tube. The test tube with the sample was placed in an ultrasonic bath and agitated for 1 hr so that the clay can be well dispersed. The resulting slurry was drawn, spread on a clean glass slide and left to dry overnight. The sample was scanned with a 1-s step time at 0.02° 2θ increments between 2° and 32° by the use of x-ray diffraction (XRD) patterns following the method of Brown and Brindley (1984).
After recording of the air-dried trace the samples were glycolated for 4 hours using the vapor-glycolation technique to determine the presence of expandable clay minerals. The glycol entered the interlayer spaces, expanded them and replaced the water on the intermellar surfaces, thus aiding the XRD in identification of clay minerals.

IV. RESULTS

Both whole rock and clay-fraction samples were examined for their major mineral phases by XRD. The whole-rock XRD analysis of the samples indicated that the rocks are composed of predominantly quartz, albite and montmorillonite (Figures 3 to 5). The relative abundance of these three predominant minerals varies slightly from sample to sample. Sample S2 (Figure 3) was taken near the regional office block (Kibaha) at the depth of 1 m. The XRD analysis indicates that the sample has high quartz content because of quartize on which the soil was formed. The patterns show that the only present clay mineral is montmorillonite.The next sample S3 (Figure 4) was taken in the vicinity of the Roman Catholic Church in Kibaha down at the depth of 1 metre from the ground level. The patterns show that the sample has high quartz content, similar to that of S3. However, the clay mineral (montmorillonite) present is much greater.Another sample S4 (Figure 5) was taken once again near the Roman Catholic Church (Kibaha). Like sample S3, the patterns confirm the presence of quartz and considerable amount of montmorillonite. Further, the identification indicates a minor composition of albite, calcian-ordered - $(Na,Ca)Al(Si,Al)_3O_8$ in all 3 samples. The presence of albite, calcian-ordered in the sample usually signifies that it is high temperature melt.For mineralogical composition, clay size fractions were prepared by sedimentation techniques as described in the section of materials and methods. Thereafter, clay separate sample was scanned with a 1-s step time at 0.02° 2θ increments between 2° and 32° whereby clear diffraction peaks in the diffractograms provided unabashed evidence for the presence of well-crystallised clay minerals. Usually, the presence of montmorillonite appears at angle ranging between 4˚ and 10˚, and this was the case for all the samples tested.In order to differentiate the mineralogical composition, the samples were then put in the ethylene glycol for overnight. The glycolation treatment was used to determine the expandable clay minerals present. If clay minerals are present, there will be a shift in the d-spacing of the first basal reflection of the clay treated with ethylene glycol, because crystal lattice expands slightly after the treatment. For all the tested samples, the glycolated trace showed a change in the peak positions for smectite and little or no change in the peak positions for nonexpanding kaolinate. The x-ray scans of the samples are shown in Figures 6 to 8.A quantitative estimate of smectite was obtained using the peak-height ratio method by measuring endothermic peak areas. The mineralogical composition determined was expressed in clay fraction. The compositions ranged from smectite content (~60%), to very low kaolinite content (<10%). The smectite clay minerals were vividly identified at the discernible basal spacing of 14 to 15 angstroms. The smectite basal spacing of 14 to 15 angstroms swelled to about 17 angstroms upon glycolation of the samples. In general, the Kibaha samples are very rich in smectite. A sharp maximum in percent smectite and a minimum in percent kaolinite indicate a high

swell potential. Furthermore, the analysis showed a stepwise decrease in percentage of smectite and increase in amount of kaolinite with depth.

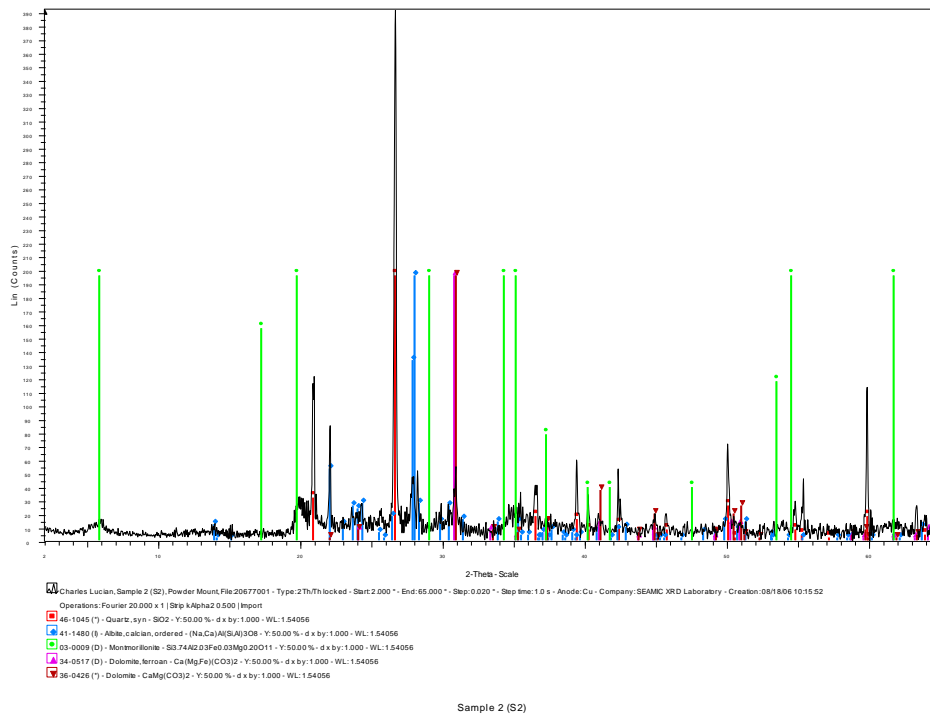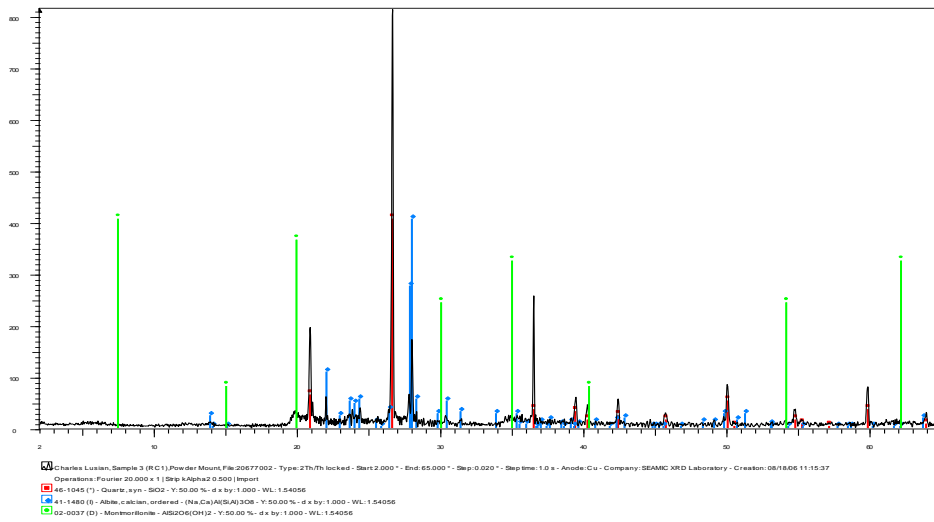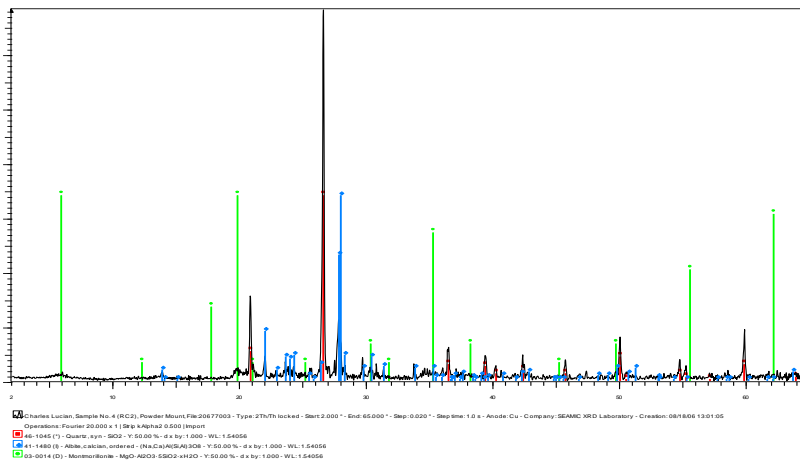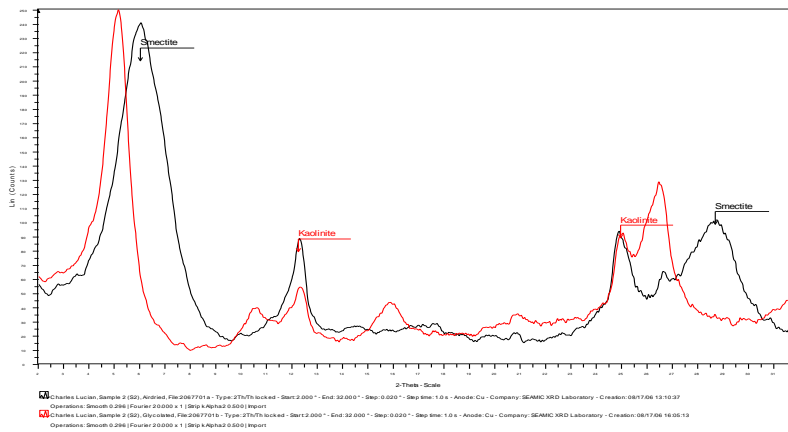**Figure 2:** Location map of Kibaha (Wikipedia, http://en.wikipedia.org/wiki/Kibaha).
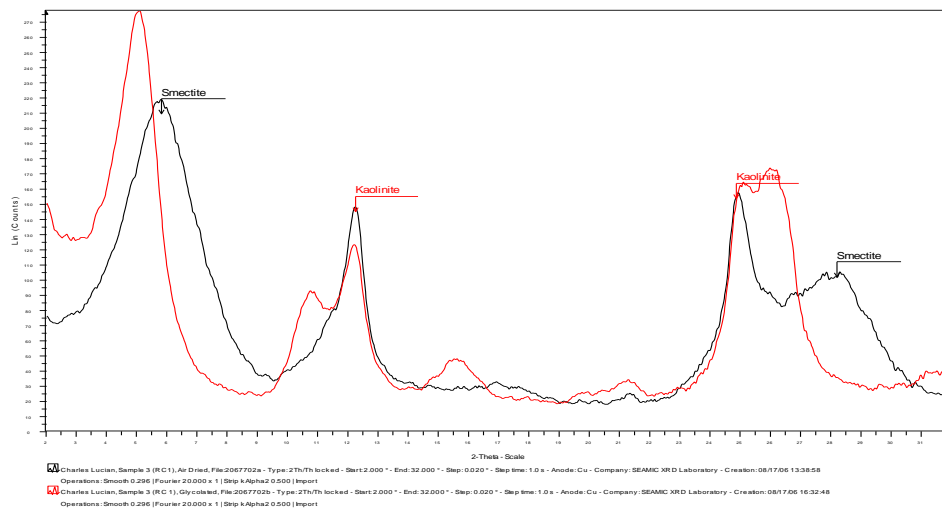

Figure 3: XRD pattern for whole-rock sample S2.

Figure 4: XRD pattern for whole-rock sample S3.
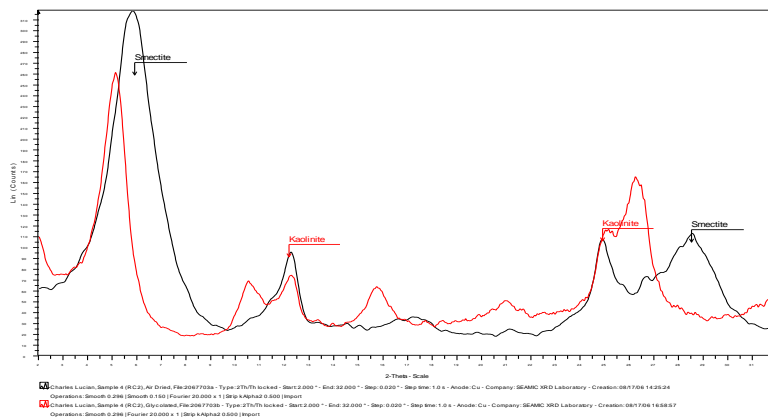

Figure 5: XRD pattern for whole-rock sample S4.

Figure 6: X-ray diffractograms of a soil sample S2 for clay content.

Sample 3 (RC1)

Figure 7: X-ray diffraction scans of a soil sample S3 for clay content.

Sampl 4 (RC2)

Figure 8: X-ray diffraction scans of a soil sample S4 for clay content.

V. CONCLUSION

X-ray diffraction (XRD) technique is widely used to identify whole rock mineralogy and clay mineralogy through interaction of the x-ray beam with a sample. The method is based on identifying the pattern of basal peaks and their corresponding relative intensity values occurring in the x-ray diffraction pattern (Sachan and Penumadu, 2007 and Flogeac *et al*., 2005). The quantitative analysis of clay minerals in soils involves working out of peak intensity (area or height) ratios based on standard powder diffraction of the International Powder Diffraction File. In this study, the identification of the specific soil mineralogical composition was obtained by the use of x-ray diffraction (XRD) patterns following the method of Brown and Brindley (1984). X-rays were collimated and directed onto specimen of finely ground powder (normally less than 10 micrometers or microns) and the diffracted rays were collected.

The mineral species present and their relative abundance were interpreted from an x-ray diffraction trace using a set of standards and models at African Minerals and Geosciences Centre (AMGC). The relative presence of the clay minerals was estimated from the maximum peak intensity or area from the representative clay mineral XRD patterns retrieved from the profile. Based on this approach, it was revealed that the clay mineral assemblage in the profiles contains both smectite and kaolinite. Based on the x-ray pattern, the expandable clay, smectite is the dominant mineral component present in the samples and only a small amount of quartz, dolomite, albite and kaolinite occured in these soils. The amount of smectite minerals was calculated to be 57-65% with average of 60%) and of kaolinite content was indeed very low (<10%). The dominance of clay species (smectite) indicate that expanding lattice type of clay predominates. Markedly, the quantity of clay content present in the soil does not matter very much because the presence of even minute quantity (5-10%) of montmorillonite produces sensitive clay (Grim, 1948). The vast majority of the expandable smectite clay

minerals are attributed to the complex sequence of late mesozoic and early cenozoic sediments mixed with fluviatile sediments of the alluvial plain such as micas, calcareous stones, marine marls, shells, mud and organic matter. The presence of expansive materials sends a strong signal for prevention measures to control the swell-shrink behavior of these soils prior to construction.

VI.   ACKNOWLEDGEMENT

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