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Mineralogy of Clays from Ikot Ebom Itam, Akwa Ibom State Nigeria

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Abstract: *This study seeks to establish the mineralogy of different coloured clays excavated from one location as a precursor to its identified application.5 kg clay each of four strata colour; Yellow, Brown, White and Grey at different depths of ground level (0.0 m), 3.5 m, 4.5 m and 8m were obtained from the same clay deposit at Ikot Ebom Itam in Akwa Ibom State and were analyzed for their mineralogical properties using X-Ray Diffraction (XRD) Method. The analyses show presence of six minerals; clays kaolinite and illite, and non-clays quartz, goethite, siderite and rutile. Semi-quantitative analysis using High Score Plus show range of quartz as 52-69%, kaolinite 21-30.7%, goethite 4-12.9%, siderite 4-13.1%, rutile as 2% and illite as 27%. Out of the analyzed samples, the White Clay with 69% quartz, 29% kaolin and 2% rutile contains the lowest mineral phase and therefore has the most economic importance as ball clays.*

Keywords: *X-Ray Diffraction (XRD), mineralogy, clay, sieving, mineral*

1. Introduction

Properties of clay are determined largely by their clay mineral composition [1]. The identity of all the clay minerals present in a clay soil must be verified in order to evaluate the pattern behavior of the clay specie[2]. The determination of non-clay mineral composition as well, is also of importance because in many cases, the non-clay minerals can significantly affect the colour and texture of a clay material at its natural state[2].Mineralogy of clay soils helps in better understanding the mineral composition which include both clay and non-clay mineral contents. An understanding of the mineralogy provides a determinant to the behavior of the soil, its class of clay mineral and therefore, the potential engineering applications [3]. Some of the more important analytical techniques that are used include X-ray diffraction, electron microscopy, infrared spectroscopy, and differential thermal analysis [4]. X-ray powder diffraction is an analytical technique widely used for phase identification of unknown crystalline materials (e.g. minerals, inorganic compounds). The principles of X-ray diffraction was postulated by Max Von Laue in 1912 who discovered that crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice [5].X-ray powder diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample [6]. The interaction of the incident rays with the sample produces a diffracted ray when conditions satisfy Bragg's Law which relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. Thereafter the diffracted X-rays are detected, processed and counted. The extension of crystalline structure analysis considers a range of 2 theta angles through which all possible diffraction directions of the lattice can be attained by the powdered samples' unique random orientation. When the diffracted reflections are converted into d-values, the mineral can therefore be identified clearly by its identity d-value through a comparison with standard reference patterns[1] [6].

Akwa Ibom State clay deposits have been investigated in recent times due to the economic potentials of clay to scientists such as geologists, soil scientists, physicists, petroleum and civil engineers as well as industrial designers and ceramists. Ikot Ebom Itam community in Itu Local Government Area (LGA) has had a longstanding history of pottery activities that has opened up the locality for its clay potentials [7].From the mineralogical standpoint, work done on Itu clays in the past has helped to determine its composition, the very recent being [8] who researched on clays sourced from Ikot Ebom and neighboring Ikot Ekwere, Ekim, Obong and Mbak locations all in Itu LGA to determine the chemical, physical and mineralogical properties. Elsewhere in the State, soils from Ikot Ekpene LGA have been analyzed for clay contents using methods of X-ray Diffractometry and seen to be rich in quartz mineral hence its gritty feel [9]. The clay fraction from samples drawn across soils in Akwa Ibom State has been determined to be mainly kaolin[8][9] [10] though montmorillonite content has been reported [11].The reported other non-clay minerals found in minor quantities include goethite, haematite, gibbsite and mica [8][9] [10].

This present research seeks to comparatively evaluate the mineralogy of four coloured clay samples found to co-exist in different strata in Ikot Ebom Itam in Itu LGA of Akwa Ibom State, Nigeria.

2. Materials and Methods

2.1. Sample Collection from Clay Deposit

The clay soil deposit is located in a ravine basin in Ikot Ebom Itam with GPS Coordinates Latitude 5.03N and Longitude 7.59E, in Itu Local Government Area, Akwa Ibom State (Figure 1). It was observed that clay soil colour on the surface was different from the soil colour at different depths descending into the ravine. The clay at ground level obtained was yellow in colour. A section of one side of soil descending into the ravine revealed the soil to be of two different colourations; yellowish-brown above and white beneath (Figure 2). Further into the ravine lying at the bottom were ashy coloured soils. 5 kg of each of the four colours; yellow from the topsoil and three out of ravine (yellow-brown at 3.5 meters, white at 4.5 meters depth and ash at estimated 8 meters depth) were excavated for mineralogical analysis in the laboratory.

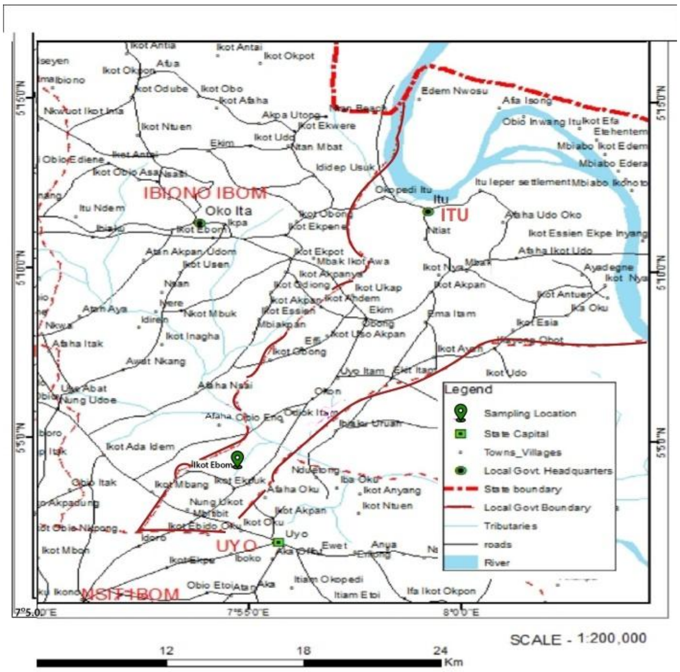

Fig 1: Location of Ikot Ebom, Itu on a Map

Fig 2: Ikot Ebom, Itu LGA clay quarry

2.2. Processing of Clay Soils

The four coloured clay soils obtained from Ikot Ebom Itam were taken to the laboratory for processing. 500 grams of each clay sample were weighed using an electronic weighing balance (Camry model no ACS-30-ZC41). Each clay sample was crushed separately in a ceramic mortar with the aid of a pestle and then subsequently soaked in water for 8 hours and then decanted, in order to, remove organic impurities. Furthermore, the slaked clays were each stirred into a homogeneous solution, poured unto a steel pan 40 mm x 35 mm x 5 mm and dried out in an oven at 110^{o}Cfor 24 hours to eliminate moisture. Thereafter, the dried clay samples were collected and again subjected to pounding into powder and finally sieved through 125 microns for mineralogy characterization.

2.3. Mineralogical Analysis

Qualitative and semi quantitative determination of the mineralogical properties of the clay samples by X-ray diffraction method were carried out using an automated Empyrean pan-analytical X-Ray diffractometer model PW 4030. The diffractometer consists of three basic elements: an X-ray (Copper K α-radiation) tube, a sample holder, and an X-ray detector. A 125 micrometer sieved sample from each of the four collected clays was prepared one after the other by compressing in a sample holder, to create a flat and smooth surface and, then loaded into the chamber of the diffractometer. X-rays were generated in a cathode ray tube by heating a filament to produce electrons. These X-rays were collimated and directed onto each clay sample. As each clay sample and detector was rotated, the intensity of the reflected X-rays was recorded. Each analyzed clay sample was finely ground, homogenized, and then prepared using the sample preparation block by compressing in a sample holder to create a flat, smooth surface that was later mounted on the sample stage in the XRD cabinet.

Also each clay sample was analyzed using the reflection-transmission spinner stage using Theta-

Theta settings. Two-Theta starting position was 4 degrees and ended at 75 degrees with a two-theta step of 0.026261 at 8.67 seconds per step. Tube current was 40mA and the tension was 45VA. A Programmable Divergent Slit was used with a 5mm Width Mask and the Gonio Scan was used as well. The semi-quantitative results were derived from reference intensity ratio of the reflections using the High Score Plus algorithm software. The tests were carried out at the National Geological Research Laboratory located in Barnawa, Kaduna State, Nigeria. Results are reported as reflected positions at 2θ and X-ray counts (intensity) in the form of an X-Y plot.

3. Results and Discussion

3.1. Results

The results of the qualitative diffraction analysis of differing colour samples named Itu Yellow, Itu Brown, Itu White and Itu Grey are presented as X-Ray diffraction spectra in Figures 3-6.

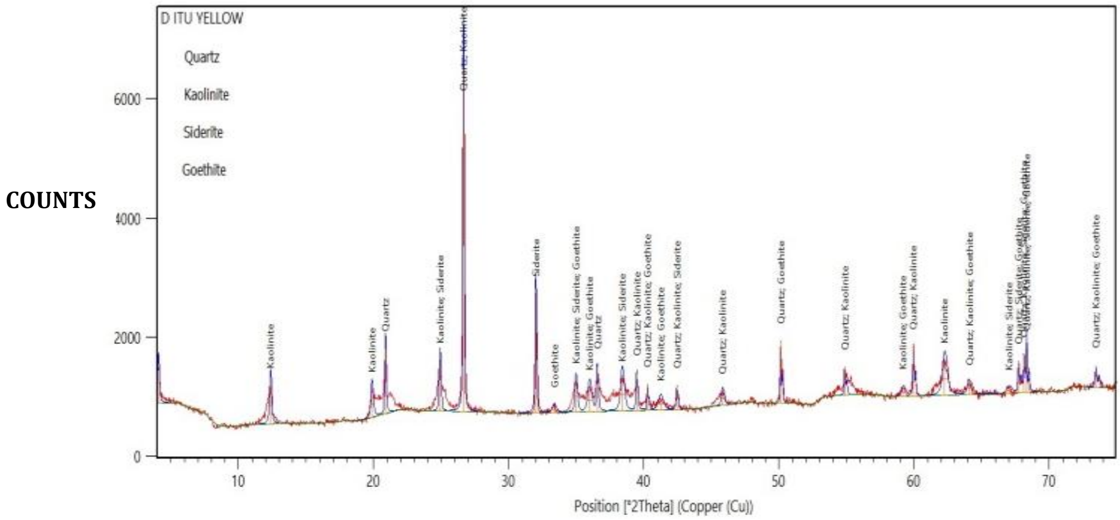

Fig 3: X-Ray Diffraction of Ikot Ebom Itam (Itu) Yellow Soil with Mineral phases present

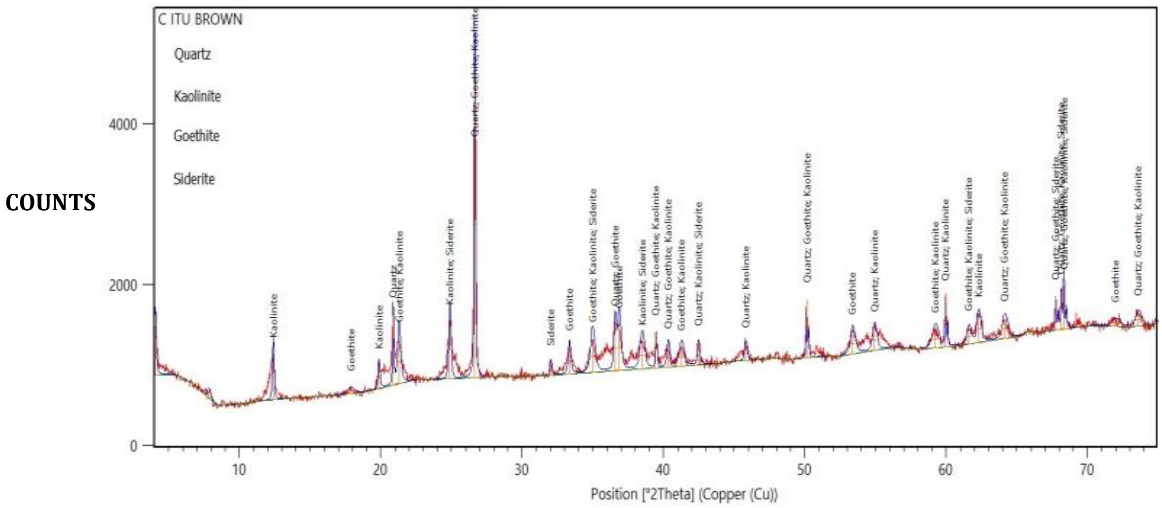

Fig 4: X-Ray Diffraction of Ikot Ebom Itam (Itu) Brown Soil with Mineral phases present

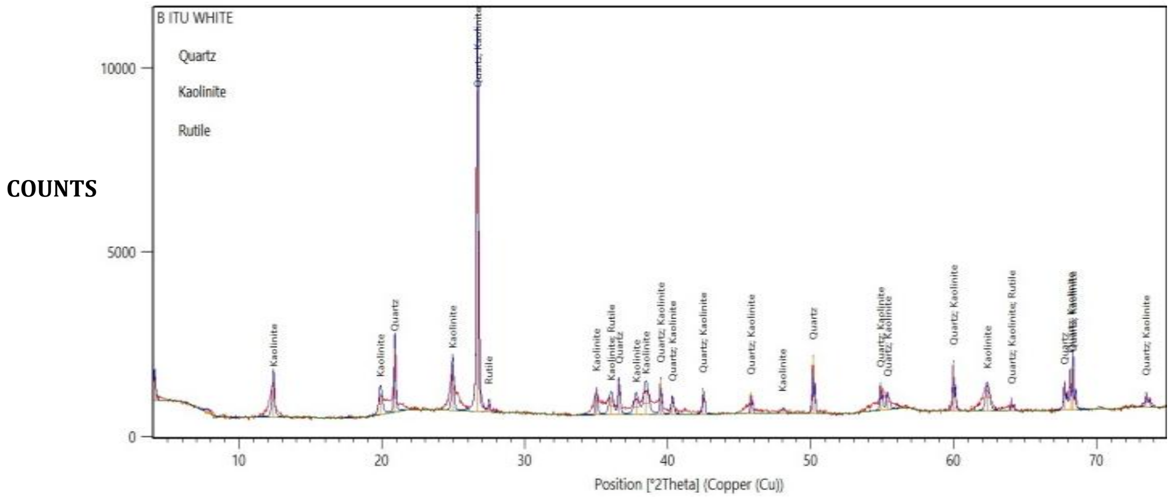

Fig 5: X-Ray Diffraction of Ikot Ebom Itam (Itu) White Soil with Mineral phases present

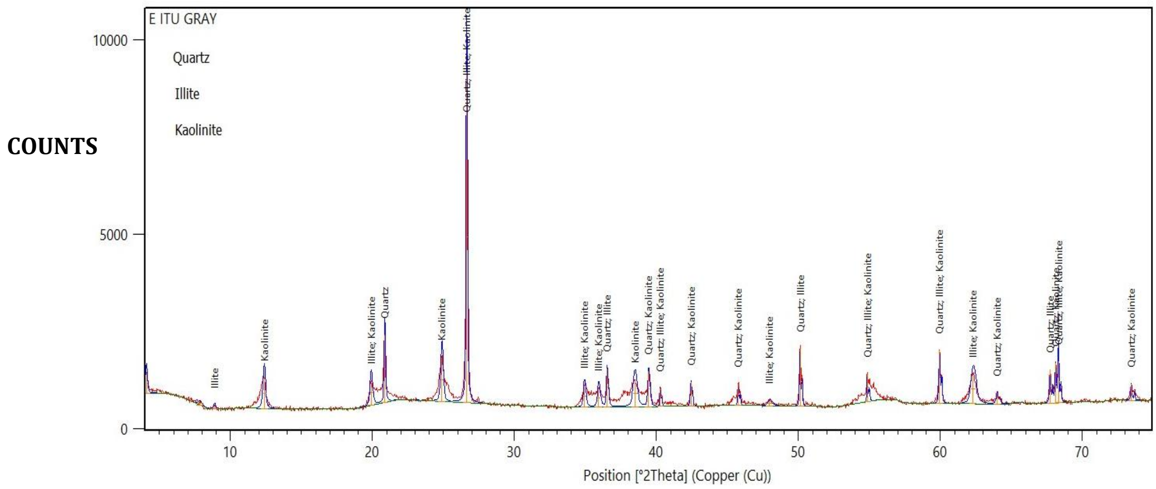

Fig 6: X-Ray Diffraction of Ikot Ebom Itam (Itu) Grey Soil with Mineral phases present

For comparison purposes, the "stick pattern" of two reference minerals quartz from Keokuk, Iowa USA [12]and kaolinite referenced from [13]are presented in Figures 7(a) and 7(b).

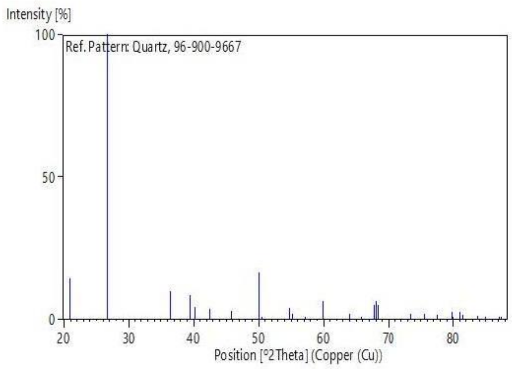

Fig 7a: Stick Pattern references for Quartz [12]

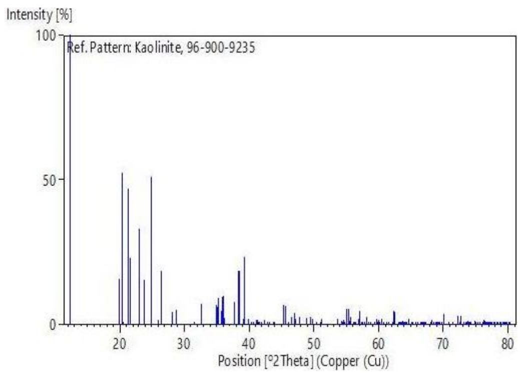

Fig 7b: Stick Pattern references for Kaolinite [13]

From the diffractogram results, it is observed that the major clay minerals present in the location are kaolinite ($Al_2Si_2O_9H_4$) and illite ($K_2Al_4Si_8O_{24}$), while the non-clay minerals are quartz (Si_3O_6), siderite ($Fe_6C_6O_{18}$), goethite ($Fe_{3.72}Co_{0.28}O_8$) and rutile (Ti_2O_4).

Table 1 presents the result of the semi-quantitative analysis of the samples, which further provides the mineral phases present in their weight percentages, in order to better appreciate the dominant minerals and subsequent effect on the soil sample.

Table 1. Semi Quantitative Mineralogical Composition of Clay Colours, in Weight Percentage

Mineral Phase		**Quartz**	**Kaolinite**	**Goethite**	**Siderite**	**Rutile**	**Illite**
Computed Density		2.65g/cm^3	2.61g/cm^3	4.30g/cm^3	3.94g/cm^3	4.25g/cm^3	2.82g/cm^3
Crystal System		Hexagonal	Anorthic	Orthorhombic	Hexagonal	Tetragonal	Monoclinic
Sample	**Depth**						
Itu Yellow	Top Soil	54.5%	28.3%	4%	13.1%	-	-
Itu Brown	3.5 meters	52.5%	30.7	12.9%	4%	-	-
Itu White	4.5 meters	69%	29%	-	-	2%	-
Itu Grey	≥8 m	52%	21%		-	-	27%

3.2. DISCUSSION

The sharp reflections of the count readings obtained in the spectra (Figures3-6) clearly indicate the crystalline nature of the clay soil samples. Its wide scattering across the 2Theta range points out the extensive capturing of all minerals present with the dominant mineral species in the clay being quartz and kaolin. From the stick pattern reference of quartz in Figure 7(a), it is clear that the representative reflections for quartz can be obtained from 20 to 60 degrees scattering angles of 2theta with total of thirteen reflections of intensity counted from the figure. The results from sample Itu White clay soil, the comparatively purest mineral obtained in Figure 5, recorded reflected readings at angles 20.88^o with intensity of 1784.18 counts, 26.66^o with 8883.83 counts, 36.55^o with 960.10 counts, 39.47^o with 973.15 counts, 40.29^o with 471.13 counts which matched the structure for quartz (first five reflected positions) published by [12]. For kaolinite stick pattern reference in Figure 7(b), the representative reflections for kaolinite can be obtained between 10 to 30 degrees scattering angles of 2theta with total of twelve reflections of intensity observed. For the obtained results of kaolinite from Itu White clay sample, it can be observed that the kaolinite reflections at angles 12.38^o with 931.57 count, 19.87^o with 563 count, 24.9^o with 1244.83 count and 26.67^o with 8883.83 count matched Keokuk kaolinite from Keokuk, Iowa USA (matching the first, second, seventh and eighth reflected positions) [13]. The result analysis confirms the presence of quartz mineral with computed density of 2.65g/cm^3 and hexagonal crystal system; kaolinite mineral with computed density of 2.61g/cm^3 and anorthic crystal system, respectively.

From the XRD semi-quantitative analysis shown in Table 1, it is observed that the soil in Ikot Ebom Itam, Itu LGA contain six different minerals and the interplay of the different proportions influenced the colouration of the soil in and around the ravine. Itu Yellow and Brown soils are the two clay soils with most mineral phases present while, the soils with the least amount of mineral phases present are Itu White and Itu Grey clay soil. It can be deduced that Itu White soil is the purest clay soil with only quartz and kaolinite minerals present. Quartz and kaolinite are the most abundant minerals in Ikot Ebom Itam, followed by illite, siderite and goethite with a trace presence of rutile. The behavior of the soil can be predicted to display plasticity and retain water but remain non-expanding due to the clay presence of kaolin and illite. Kaolinite contains kaolin clay which is described to be soft, whitish crystalline powder [14]. Kaolin clay is plastic, fires with a high modulus of rupture and to a white or near-white color [15] and these properties make kaolin a very important ceramic raw material. Illite is non expansive clay due to the individual crystal space being filled by poorly hydrated potassium, calcium or magnesium ions that hinder water molecules penetrating the clay structure [16]. In natural form, they are grey to silverish white in color [16] and find application in industrial demands of high potassium in agriculture industry such as crop fertilizers and soil stabilizer, petroleum industry where it is the basis of a K-Ar isotope date used in the control of timing basin heating events [17]. Goethite, a non-clay mineral is an oxidized ferric iron ore [18] common in laterite soil and is known to be a pigment that gives soil its reddish to yellowish brown colour [19]. Siderite, a non-oxide iron bearing carbonate [20] is a ferric mineral ore of value as iron ore due to the absence of sulphur and phosphorus [21]. These minerals being present in the yellow and brown clay samples is worthy of mention in that both goethite and siderite iron ores lie within the shallow range of top and underlying soil in Ikot Ebom

Itam, Itu location and are not present further deep in the soils descending to the base of the ravine.

4. Conclusion and Recommendation

The mineralogical studies conducted on the four clay colour samples obtained at Ikot Ebom Itam in Itu LGA of Akwa Ibom State were successfully carried out. The presence of high contents of quartz and kaolin in the soil to depths of 8 meters prove that the location is rich in kaolinite mineral, which implies that, there will be a strong proclivity to clay behavior in samples collected from the soil in terms of clay plasticity, particle fineness and sintering at elevated temperatures. This gives credence to the site as a location for industrial mining. However, the presence of non-clay impurities in each colour should be a determinant to the end application. Out of all four colours analysed, the Itu White is the purest clay soil with no presence of minor mineral contents except rutile at only 2%. It is recommended that clays sourced from Ikot Ebom Itam be beneficiated and more focus given to Itu White coloured clay for further beneficiation to acceptable industrial standard towards suitability for use as ball clay in production of white ceramic wares and paper.

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