MINERALOGICAL CHARACTERISATION OF CLAY DEPOSIT NEAR IGBILE SOUTHWESTERN NIGERIA

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ABSTRACT

X-ray diffraction is a basic tool in mineralogical analysis of sediments, and in the case of fine grained sediments an essential one. Understanding of rocks composition, the nature of clay mineralogy content and provenance is very important in Basin evaluation. This study therefore focuses on the use of X-ray diffraction to study the clay stone unit exposed along Igbile, southwestern Nigeria. Five clay stone samples were collected following fieldwork in the study area and were subjected to X-ray Diffraction analysis. This was done in order to delineate the associated mineral composition in the rock samples, determine the crystal forms and shape of the mineral and probably provenance of the mineral content. Observations from the result of the X-ray Diffraction indicated peaks with d-spacing range of 1.488Å, 1.63 Å, 1.59 Å, 1.62 Å and 7.17 Å indicative of Kaolinite and d-spacing range of 1.541Å, 1.817 Å, 2.282 Å, 2.458 Å, 3.343 Å and 4.26 Å indicative of quartz as the only significant, minor to moderate abundance minerals in the rock samples. The nature of the kaolinite in the rock samples is suggestive of a transformational and neoformational origin with acid tropical environment where the degree of leaching is probably intensive. The d-spacing and the plane axis values of the kaolinite also indicated Dioctahedral, Trioctahedral and Pinacoid crystal shape with range of forms {001},{060}, {133},{134}and {002}. Similarly the quartz d-spacing and plane axis values suggested Orthorhombic and Trigonal crystal shape with crystal forms {101}, {100}, {112}, {211}, {110}and {102}. In conclusion, the clay deposit exposed in the study area is made up of detrital kaolinite and quartz which are probably sourced from low latitude region in acid tropical high leaching environment.

Keywords**:** X-ray diffraction, d-spacing, Kaolinite, Quartz, crystal shape

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Clay minerals are the most common components of all sediments and soils. Clay minerals are found in the soils and sediments from polar to equatorial regions, from sea level to high mountains, from deserts to rain forests and in marine sediments from coasts to deep seas. Clay minerals are also trapped in the ice of mountain glaciers and polar ice caps. Clay minerals are produced by the transformation of parent rocks by physical and chemical disaggregation without chemical modification of the minerals, and by chemical weathering causing a transformation of primary minerals with the formation of secondary clay minerals. These secondary clay minerals make up the weathering complex and result in soil formation. Clay minerals can be eroded, transported and deposited by water and wind. There are several classes of clays such as smectites (montmorillonite, saponite), mica (illite), kaolinite, serpentine, pylophyllite (talc), vermiculite and sepiolite (Shichi and Takagi 2000). Clay is composed mainly of silica, alumina and water, frequently with appreciable quantities of iron, alkalis and alkali earths (Ralph 1968). The development of soils and clay minerals is influenced by climate, vegetation and fauna, lithography, land forms, inter flow water, time, and human activities. Therefore, clay minerals provide clues to their parent rocks and to the climatic conditions during their formation. The wide industrial applications of clays make their identification an inevitable process. This identification includes not only the constituent clay minerals but also the trace impurities (Wilson 1987). Although, there is a variety of modern equipment to study clays: Infrared, Thermal analysis which include Thermogravimetry (TG), Derivative Thermogravimetry (DTG), Differential Thermal Analysis (DTA), electron transmission microscope, electron microprobe, Mossbauer spectrometers, nuclear and isotope technology etc. The X-ray diffractometer remains to be a basic tool for clay studies (Tobia and Sayre 1974).

X-ray diffraction techniques can successfully identify clay mineral groups and subgroups on the basis of their reflections and provide detailed information about the atomic structure of crystalline substances. It is a powerful tool in the identification of minerals in rocks and soils. The bulk of the clay fraction of many soils is crystalline, but clay particles are too small for optical crystallographic methods to be applied (Reynolds, 1989a). Therefore, x-ray diffraction has long been a mainstay in the identification of clay-sized minerals in soils.

The relative abundance and distribution of clay deposit near Igbile, Dahomey basin, Southwestern Nigeria has not been a subject of most research work carried out and with this in mind, this research work seeks to characterize the clay deposit near Igbile using x-ray diffraction and is focused towards identifying the various clay mineral groups and subgroups present in the deposit on the basis of their reflection as well as determining their pattern of distribution within the study area.

The study area is located between latitude N06°43'00 - N06°45'00" and longitude E003°51' - E003°53'. (Fig.1)

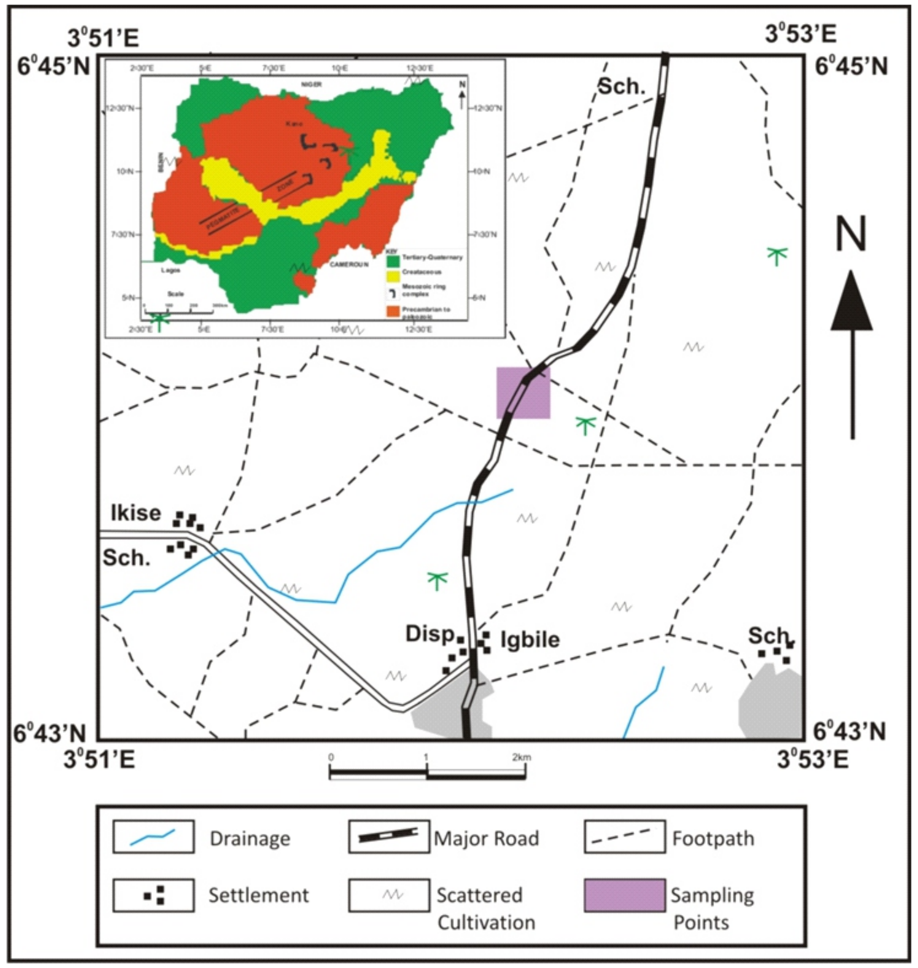


Fig.1: Location Map of the Study Area

The area is accessible by major and minor road networks and the climatic condition is tropical as expressed in alteration of wet and dry seasons.

GEOLOGIC SETTINGS

The study area falls within the Nigeria sector of the Dahomey Basin. The Dahomey Basin is a very extensive sedimentary basin that extends from southeastern Ghana in the west to the western flank of Niger Delta in Nigeria. It constitutes of part of a system of West African peri-cratonic (margin sag) basin (Klemme 1975; Kingston *et al*., 1983) developed during the commencement of the rifting, associated with the opening of the Gulf of Guinea i.e. the separation of African from South American plate, which occurred in the Early Cretaceous to the Late Jurassic Period in the Mesozoic Era (Burke *et al*., 1971; Whiteman, 1982). The basin is thus termed a marginal pull–apart basin (Klemme, 1975) or marginal sag basin (Kingston *et al.,* 1983). It is bounded to the west by the Ghana ridge, which is an extension of the Romanche Fracture Zone; and on the east, by the Benin Hinge line, a basement escarpment which separates the Okitipupa structure from the Niger Delta Basin and also marks the continental extension of the Chain Fracture Zone. The Nigeria portion of the basin extends from the boundary between Nigeria and Republic of Benin to the Benin Hinge Line. The onshore part of the basin covers a broad arc-shaped profile of about 600 km^2 in extent. The onshore section of the basin attains a maximum width, along its N-S axis, some 130 km around the Nigerian–Republic of Benin border. The basin narrows to about 50 km on the eastern side where the basement assumes a convex upwards outline with concomitant thinning of sediments. Along the northeastern fringe of the basin where it rims the Okitipupa high, is a band of tar (oil) sands and bitumen seepages (Ekweozor and Nwachukwu, 1989).

The stratigraphic succesions of the Dahomey Basin (fig. 2) have been described by several workers such as Jones&Hockey, 1964; Ogbe, 1970; Omatsola and Adegoke, 1981; Agagu, 1985; Okosun, 1998; Nton, 2001; Elueze and Nton, 2004; Nton *et al*., 2006 among others. Five lithostratigraphic formations were reported which span from Cretaceous to Tertiary ages.

The oldest formation in the Abeokuta Group is referred to as Ise and it uncomformably overlaps the Precambrian basement complex. It has basal conglomerate, gritty to medium-grained loose sand, capped by kaolinitic clay (Omatsola and Adegoke, 1981; Agagu, 1985) but sometimes interbedded within the sand. The maximum thickness of the member is about 1865m and more than 600m of it was penetrated by Ise-2 borehole. The conglomerates are unimbricated and at some locations, ironstones occur (Nton, 2001). The age has been given to be Neocomian to Albian.

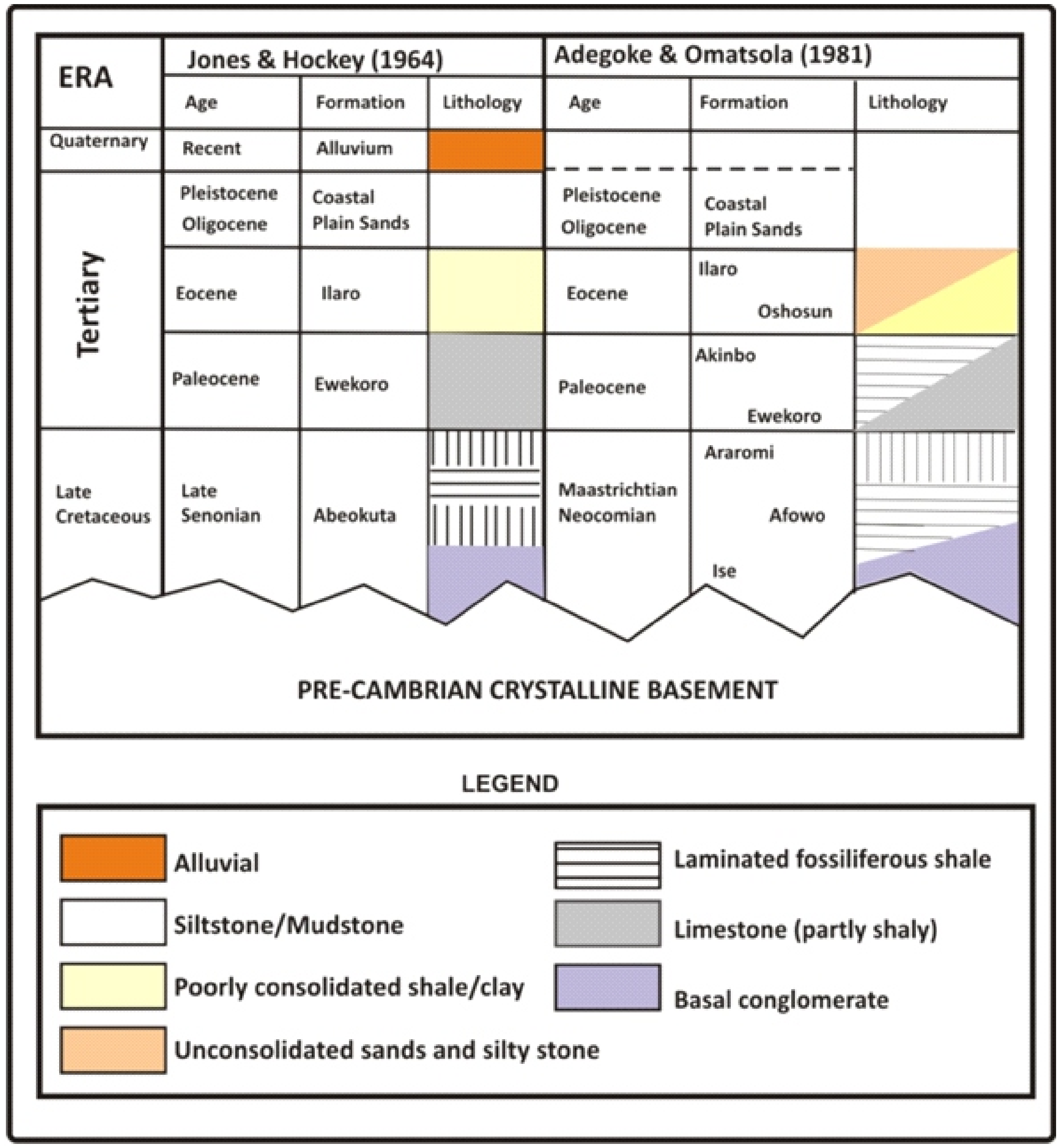


Fig.2. *Stratigraphy of the Eastern Dahomey Basin (After Omatsola & Adegoke, 1981)*

Afowo Formation succeeds the Ise Formation. The lower part of this formation is transitional with mixed brackish to marginal horizons that on in a transitional environment after the entire basal and conalternate with well sorted, sub-rounded sands indicating a littoral or estuarine near-shore environment of deposition. Afowo Formation thus indicates the commencement of deposititinental Ise Formation. The sediments are composed of interbedded sands, shales and clays, which range from medium to fine grains in sizes (Omatsola and Adegoke, 1981; Agagu, 1985). The sandy facies are tar-bearing while the shales are organic - rich (Enu, 1990). This bituminous content has been found to be in both surface and sub-surface sections. Using palynological assemblage, Billman (1992) assigned a Turonian Age to the lower part of this formation, while the upper part ranges into the Maastrichtian Age.

Araromi Formation, the topmost unit of the Abeokuta Group. Sediments of the Araromi Formation represent the youngest topmost sequence in the group and as thus been described as the youngest Cretaceous sediment in the eastern Dahomey basin (Omatsola and Adegoke, 1981). The formation is composed of fine to medium grained sandstone at the base, overlain by shales, siltstone with thin interbeds of limestone, marl and lignite bands (Omatsola and Adegoke, 1981; Agagu, 1985). It is an equivalence of a unit known as Araromi shale by Reyment (1965). The shales are grey to black in colour, marine, and rich in organic matter. This Formation is highly fossiliferous containing abundant planktonic foraminifera, ostracods, pollen and spores. Omatsola and Adegoke (1981) assigned a Maastrichtian to Palaeocene age to this formation based on faunal content.

The Imo group consists of the two lithostratigraphic units which are: Ewekoro Formation (oldest) and Akinbo Formation (youngest). Ewekoro Formation directly overlies the Abeokuta Group as it has been observed from the sections at Ewekoro and Sagamu quarries as well as the cored sections at Ibeshe. It is an extensive limestone body, which is traceable over a distance of about 320km from Ghana in the west, towards the eastern margin of the Dahomey basin in Nigeria (Jones and Hockey, 1964). Elueze and Nton, (2004) has reported that the limestone is of shallow marine origin owing to abundance of coralline algae, gastropods, pelecypods, echnoid fragments and other skeletal debris . It is made up of grayish white and occasionally greenish limestone which is sandy toward the base and having a thickness that varies between 15-30m. This formation is dated Paleocene age. Akinbo Formation is mostly found in the western part of the Imo Group, directly overlying the Ewekoro Formation. It constitutes the upper part of the Imo Group. It is made up of shale and clayey sequence (Ogbe, 1972). The claystones are concretionary and are predominantly kaolinite (Nton and Elueze, 2004).The base of the formation is defined by the presence of glauconitic band with lenses of limestones (Ogbe, 1972; Nton, 2001). It is essentially greenish, highly fossiliferous and thickly laminated. The age of Akinbo Formation is considered Paleocene to Eocene.

Oshosun Formation overlies the Akinbo Formation and consists of greenish – grey or beige clay and shale with interbeds of sandstones. The shale is thickly laminated and glauconitic. According to Okosun (1998), the basal beds consist of any of the following facies; sandstones, mudstones, claystones, clay-shale or shale. This formation is phosphate- bearing (Jones and Hockey, 1964; Nton, 2001).

Ilaro Formationoverlies conformably the Oshosun Formation and consists of massive, yellowish, poorly consolidated, fine to coarse, cross-bedded sandstones, clays and shales with occasional thin bands of phosphate beds being observed at Ilaro. The formation is Eocene in age.

The coastal plain sand overlies the Ilaro Formation but evidence for this is lacking (Jones and Hockey, 1964). This coastal plain sands consist of very poorly sorted clayey, pebbly sands, sandy clay and rare thin lignite. They are the basal continental beds of the Abeokuta Group. The sands show transitional to continental characteristics. The age is from Oligocene to Pleistocene.

Recent Alluvium is the youngest unit in the Eastern Dahomey basin. It has been thought to overlie the Ilaro Formation, but convincing evidence for this is lacking (Jones and Hockey, 1964). The exposure at the road cuttings between Ofada and Mokoloki on the Ogun River reveals coarse clayey sorted sands with clay lenses and occasional pebble beds.

METHODOLOGY

Five fresh samples were selected and analyzed for clay minerals present using X-ray diffraction at ACME laboratories, Vancouver, Canada. The analyses procedures include; mixing with acetone the predetermined sample to produce a thin slurry. Afterwards, the mixture was then applied onto a glass slide which was scanned for determination by XRD. The X-ray diffraction was carried out using a Siemens D500 Diffractometer (using MDI Data Scan and JADE 8 software to plot the diffractograms.

Production of X-rays for XRD analysis is accomplished using an X-ray tube consisting of a filament electron source and a metal target. The tubes are evacuated to minimize absorption of electrons accelerated from the filament (cathode) to the target (anode). Activation of the tube entails passing a current through the filament to establishing a current (e.g., 10–30 mA) under high

voltage (e.g., 30–50 kV) between the filament and target. X-rays generated from the target during the rapid deceleration of electrons from the filament emerge from windows in the tube. The material comprising the window has a minimal tendency to absorb X-rays.
X-ray diffraction analysis uses monochromatic radiation. Intense X-radiation at a specific wavelength can be produced when electrons from a source (such as tube filament) dislodge inner shell electrons from the atoms of the metal target. Instantaneous replacement of the dislodged electron by an electron from a specific lower energy shell results in the quantum release of a packet of energy corresponding to a specific wavelength and termed characteristic radiation. Characteristic radiation is element specific and relates to the atomic number of the element selected for the target in the X-ray tube. Hence, tubes can be selected based on the wavelength (λ) that is most advantageous for the material being analyzed by XRD.

RESULT AND DISCUSSION

The result of the XRD analysis is displayed in figure 3 through figure 7. Observations from the result and interpretation of the claystone samples subjected to XRD analysis indicated Quartz and Kaolinite peaks (figure 3 to figure 7). The kaolinite peaks is the only main indicated peaks of clay mineral in the samples. The kaolinite is important in the manufacturing of ceramics and porcelain as well as fillers for paint, rubber and plastic. Recently, kaolinite has found the greatest use in the paper industry in the production of glossy paper used in most magazines.
Observations from the result of the X-ray Diffraction indicated peaks with d-spacing range of 1.488Å, 1.63 Å, 1.59 Å, 1.62 Å and 7.17 Å indicative of Kaolinite and d-spacing range of 1.541Å, 1.817 Å, 2.282 Å, 2.458 Å, 3.343 Å and 4.26 Å indicative of quartz as the only significant, minor to moderate abundance minerals in the rock samples.

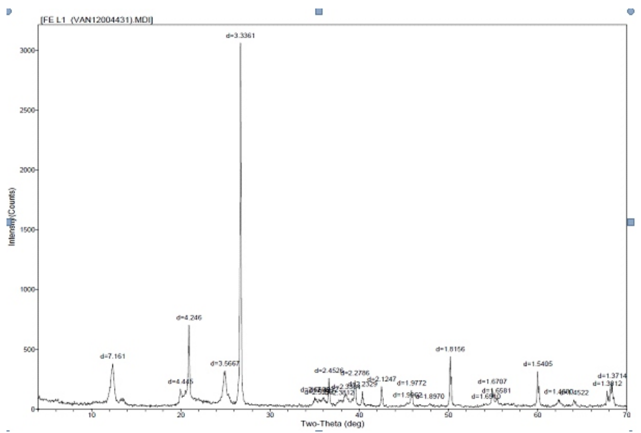
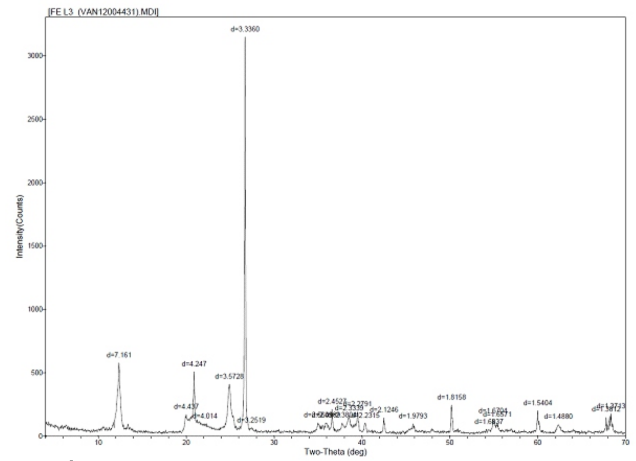


Fig.3: X-ray diffraction pattern for sample FEL1



&B) Ỳ8 -ray diffraction pattern for sample FEL3

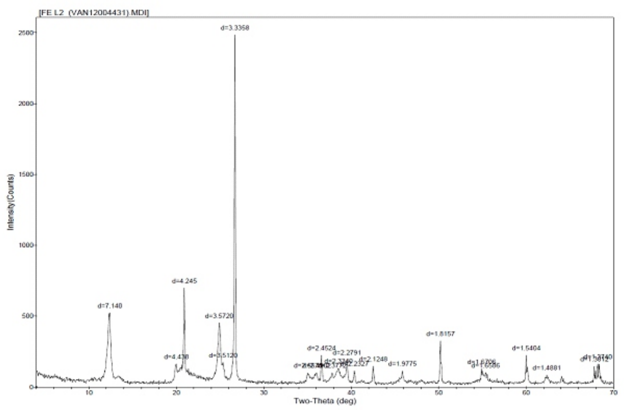


Fig.4: X-ray diffraction pattern for sample FEL2

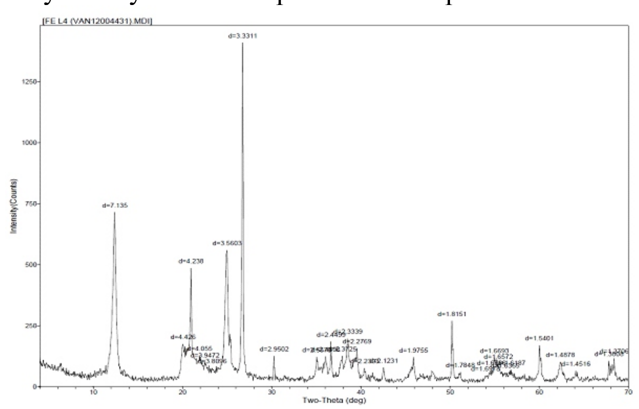


Fig.6: X-ray diffraction pattern for sample FEL4

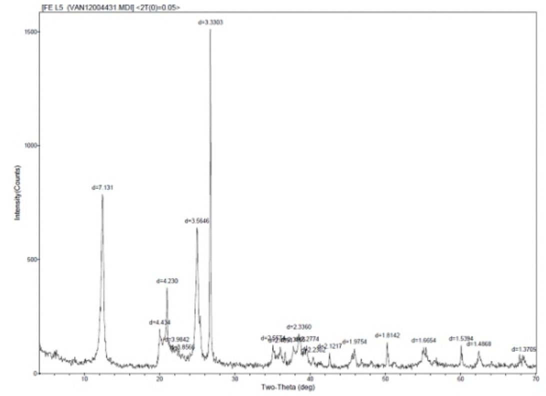


Fig.7: X-ray diffraction pattern for sample FEL5

The nature of the kaolinite in the rock samples is suggestive of a transformational and neoformational origin with acid tropical environment where the degree of leaching is probably intensive. The d-spacing and the plane axis values of the kaolinite also indicated Dioctahedral, Trioctahedral and Pinacoid crystal shape with range of forms {001},{060}, {133},{134}and {002}. Similarly the quartz d-spacing and plane axis values suggested Orthorhombic and Trigonal crystal shape with crystal forms {101}, {100}, {112}, {211}, {110}and {102}.
The figures 8 to 12 indicates the various pattern showing the associated mineral composition in the subsequent claystone samples.

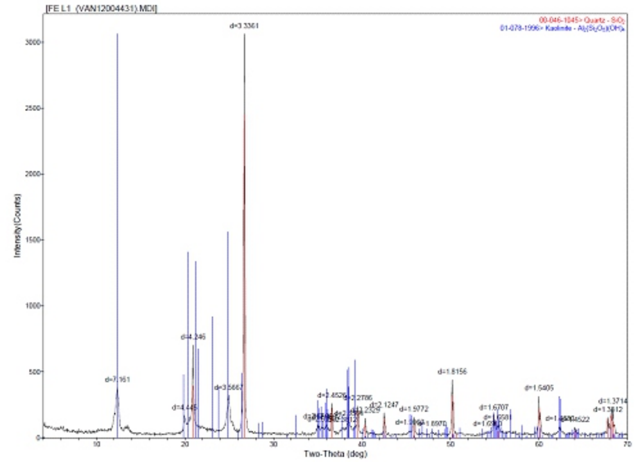


Fig.8: Pattern showing the associated mineral composition in sample FEL1

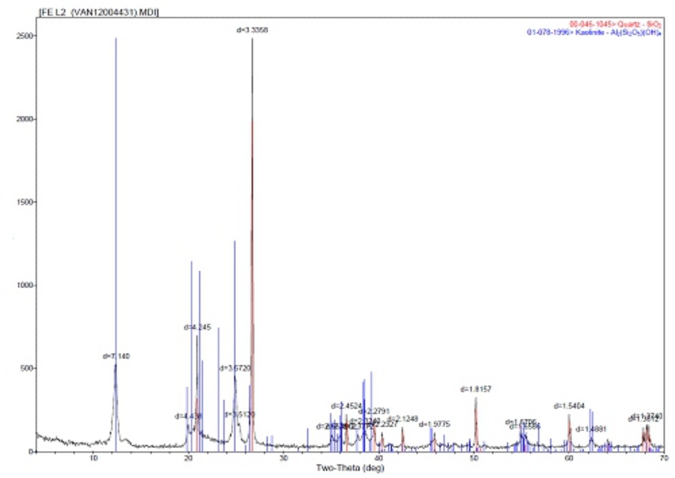


Fig.9: Pattern showing the associated mineral composition in sample FEL2

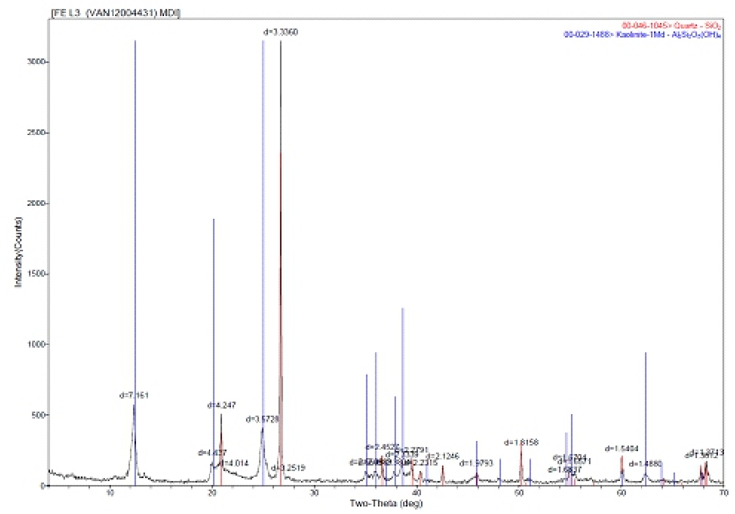


Fig.10: Pattern showing the associated mineral composition in sample FEL3

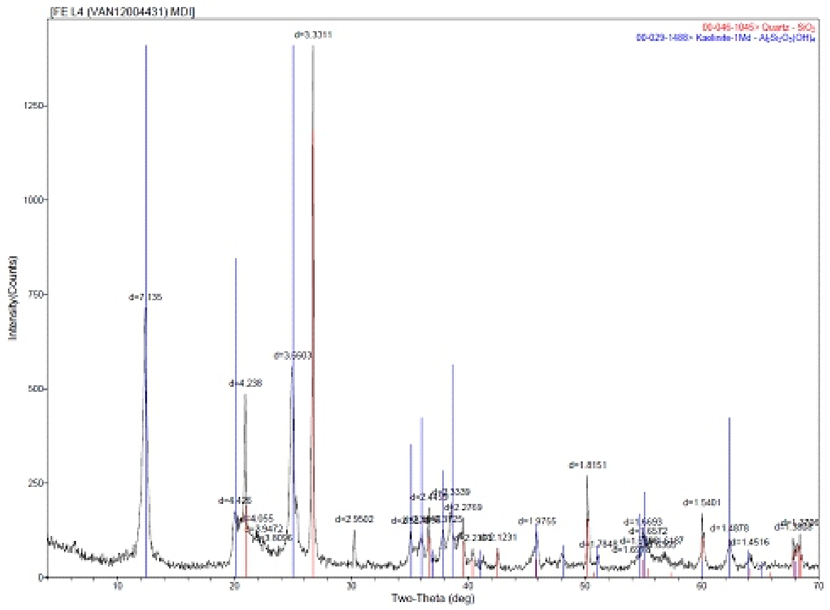


Fig.11: Pattern showing the associated mineral composition in sample FEL4

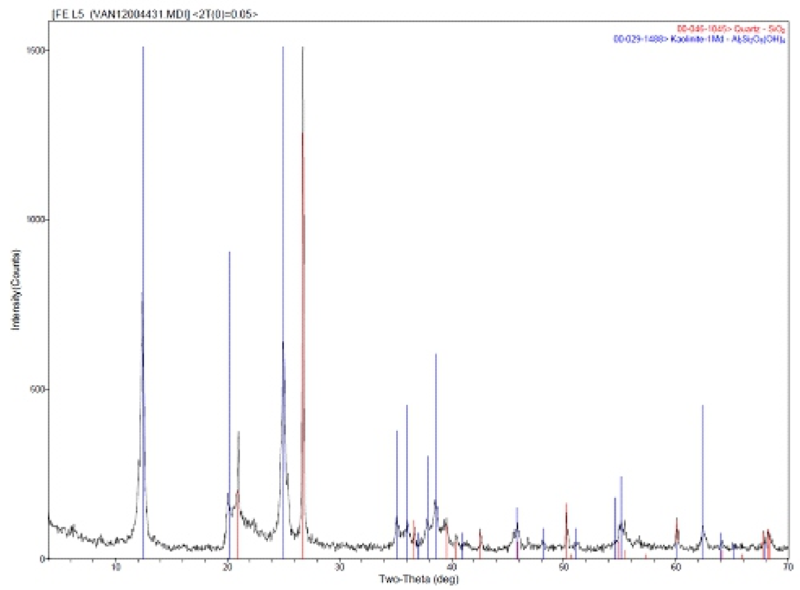


Fig.12: Pattern showing the associated mineral composition in sample FEL5

CONCLUSION

The x-ray diffraction pattern of the samples indicated peaks suggestive of significant, minor to moderate abundance of Quartz and Kaolinite.

The Kaolinite is probably sourced from an acid tropical environment where leaching is intensive typical of a low latitude region particularly off major rivers draining regions of tropical weathering. The plane axis (hkl) values of kaolinite as estimated from the d-spacing value and determinant function suggest crystal forms which range from {001}, {060}, {002}, {133} and {134}of dioctahedral, trioctahedral and pinacoid (basal) crystallography shapes. Similarly, the quartz are orthorhombic and trigonal in shape.

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