Mineralogical Study of Gypsum Occurrence from the Mutwe Plain, Chad Basin, North East Nigeria, using the X-ray Diffraction Techniques

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Abstract

The mineralogical study of seventeen samples and its hosting upper Cretaceous Fika Shale in the Mutwe plain, Chad basin has been studied using the X-Ray Diffraction (XRD) techniques. The predominant minerals observed occur either as impurities within the gypsum or in association with hosting shale in the diffractogram. From this study, it has been found that most of the samples contain the original rock-forming clay minerals. The Gypsum disseminates as specks and nodules with shale and the presence of the mineral glauconite in the sediments indicates deposition in a shallow marine environment.

Keywords: Fika shale; Chad basin; X-Ray diffraction (XRD); Gypsum and Mineralogical association

Introduction

Various reports and publications provide detail information about the mineralization’s [1-5]. The present work tends to study the mineralogy of the Gypsum occurrences from the Mutwe plain Chad basin using the X-Ray Diffraction (XRD) techniques. The x-rays are electromagnetic radiation of wavelength of about one angstrom (1Å), which is about the same size as the atom. They occur in the portion of the electromagnetic spectrum between gamma rays and ultra violet rays that could be used in identification of minerals present in a powdered sample.

The x-ray diffraction involves passing an incident x-ray of known wavelength from a source at a known angle (θ) to the surface of a solid which it penetrates to some depths. As it passes through the solid, it is reflected from successive planes of atomic arrangement of the solid following established optical laws as illustrated below in figure 1. The path difference between two successive waves is 2λ=2d sin θ where λ is the wavelength of the incident x-ray and d’ is the distance that separates successive atomic planes of the solid.

For constructive interference between these waves the path difference must be an integral number of wavelengths.

\[ n \lambda = 2 \lambda \text{ or } 2x \]

This leads to the Bragg’s equation:

\[ n \lambda = 2d \sin \theta \]

\[ \theta = \frac{1}{2} \sin ^{-1} \left( \frac{n \lambda}{d} \right) \]

\[ n \lambda = 2d \sin \theta \]

\[ \theta = \frac{n \lambda}{2d} \]

\[ d = \frac{n \lambda}{2 \sin \theta} \]

\[ \theta = \sin ^{-1} \left( \frac{n \lambda}{2d} \right) \]

Figure 1: Reflection of x-rays from two planes of atoms in a solid. Note ‘x’ in this diagram is wavelength.

The present study tends to concentrate on the Gypseous sediments of the Mutwe plain, Chad basin, which is about the mineralization’s [1-5]. The present work tends to study the mineralogy of the Gypsum occurrences from the Mutwe plain Chad basin using the X-Ray Diffraction (XRD) techniques. The predominant minerals observed occur either as impurities within the gypsum or in association with hosting shale in the diffractogram. From this study, it has been found that most of the samples contain the original rock-forming clay minerals. The Gypsum disseminates as specks and nodules with shale and the presence of the mineral glauconite in the sediments indicates deposition in a shallow marine environment.

X-ray diffraction has been in use in two main areas for the fingerprint characterization of crystalline materials and the determination of their structure. Each crystalline solid has its unique characteristic x-ray powder pattern which may be used as a ‘fingerprint’ for its identification [6,7].

The geology setting

The Nigerian sector of the Chad Basin, known locally as the Bornu Basin, is one of Nigeria’s inland basins occupying the northeastern part of the country. It represents about one-tenth of the total area extent of the Chad Basin, which is a regional large structural depression common to five countries, namely, Cameroon Central African Republic, Niger, Chad, and Nigeria. The Bornu Basin falls between latitudes 11°N and 14°N and longitudes 9°E and14°E, covering Borno State and parts of Yobe and Jigawa States of Nigeria [8]. The Chad Basin belongs to the African Phanerzoic sedimentary basins whose origin is related to the dynamic process of plate divergence. The basin belongs to a series of Cretaceous and later rift basins in Central and West Africa whose origin is related to the opening of the South Atlantic [9]. The Precambrian Basement Complex constitutes the bedrock on which sediments ranging in age from Paleozoic to the Quaternary have been deposited. Furon [10] suggested that the Chad Basin was a tectonic cross point between a NE-SW trending “Tibesti-Cameroon Trough” and a NW–SE trending “Air-Chad Trough” in which over 3,600 m of sediments have been deposited. The Chad Basin in the Nigerian sector shows a depositional sequence from top to bottom: Quaternary, Early Pliocene, Continental Terminal, Gongle Sandstone, Fika Shale, and Gongla, Yolde and Bima Sandstones Formations [11]. Sedimentary sequences were deposited from the Paleozoic to Recent, accompanied by a number of stratigraphic gaps. Sediments are mainly continental, sparsely fossiliferous, poorly sorted, and medium- to coarse-grained, feldspathic sandstones called the Bima Sandstone. A transitional
calcareous deposit—Gongilaf ormation—that accompanied the onset of marine incursions into the basin overlies the Bima Sandstones. These are overlain by graptolitic shale [12]. The oldest rocks in the Chad basin belong to Bima Sandstone and the youngest to the Chad Formation as shown in the stratigraphic column of the study area (Figure 2). The geology of the study area is shown on figure 3.

Methodology

The mineralogical analysis of the seventeen samples were done in the laboratory of the National Steel and Raw materials Exploration Agency (NSRMEA), it entailed washing, crushing, and pulverization to a powdery form where the particle sizes are in the order of one hundred microns. Machines used in the size reduction of samples include, the jaw crusher, the laboratory disc mill, vibrating cup mill, and the pulverizer. Each of the pulverized powdered samples was put into an XRD sample holder which is disc like in shape, made of steel material. The disc has in its center a hollow of about 1.2 cm diameters. The hollow is filled and compacted to the point where all voids are filled up. The sample surface was then polished and flattened before insertion into the diffractometer for mineralogical analysis. To avoid the contamination of sample containers, they were cleaned up with acetone after each analysis before further usage. Incident lights varying in angle from 5° to 70° were then passed through the samples in the diffractometer and minerals present in the samples displayed in the form of their characteristic peaks at the different angles of incidence. The corresponding inter planer distances at the various peak angles for the minerals (d-values) were also displayed on the monitor of the computer coupled to the diffractometer.

Discussion

Mineralogy

Minerals in the various samples were identified by comparing the peak values displayed on the diffractograms (Figures 3-19) some of the minerals are discussed below:

- Apatite: ChlorApatite Ca5Cl (PO₄)₃, Fluorapatite: Ca₅(PO₄)₃F
  Chloro phosphate and fluoro phosphate of calcium
  Apatite occurs as a primary constituent of igneous rocks, but only in accessory amounts. It is also present in small quantities in metamorphic rocks, especially in crystalline limestone. In the study area this mineral was identified in gypsum samples from the Mura, Manawachi area and the Kwayaya deposit. The mineral occurs also in shale and clay samples 2738, 2752, 2757 and 2760 from Manawachi (Figures 5, 12, 13 and 15 respectively).

- Biotite: K(Mg,Fe)₃(AlSi₃)O₁₀(OH,F)₂
  This is a silicate of magnesium, iron, aluminum and potassium with hydroxyl and fluorine. Occur as original constituent of igneous rocks of all kinds—granites, diorites, gabbros, etc. It occurs abundantly as a mineral of metamorphic origin in biotite gneisses, biotite schists, and in contact altered clayey rocks. This mineral occurs in several shale samples and as an impurity in a number of gypsum samples in the entire study area. Samples 2727, 2744, and 2757 in figures 4, 9 and 13 respectively must have been derived from the weathering of...
Chamosite: $\text{Fe}_3\text{Al}_2\text{Si}_2\text{O}_{15}\text{Aq}$

The hydrated iron silicate chamosite is an important constituent of sedimentary iron ores. Occurs in gypsum sample 2749 in the Mura deposit (Figure 10) and also in one Kabul hill clay sample 2758 near Kwayaya (Figure 14). Indicates that the sediments were derived from metamorphic rock constituents of the Basement Complex.

Chloritoid: $(\text{Fe}^{2+}\text{Mg, Mn})(\text{Fe}^{3+},\text{Al})\text{Al}_3\text{O}_2(\text{SiO}_4)(\text{OH})_4$

Dark green to greenish black. Occurs only in metamorphic rocks of sedimentary origin, especially those of argillaceous composition. Occur only in one shale sample at the Kwayaya deposit (Figure 17). Indicates that the sediments were derived from metamorphic rock constituents of the Basement Complex.

Clinozoisite: $\text{Ca}_2\text{Al}_3(\text{SiO}_4)_3\text{OH}$, Epidote-zoisite: $\text{Ca}_2(\text{Al,Fe})_3(\text{SiO}_4)_3\text{OH}$

This is a silicate of calcium and aluminum commonly a secondary mineral in igneous rocks, where it is produced by the alteration of ferromagnesian minerals and calcium. Plagioclase and also as a constituent of impure calcerous rocks. Occur here as impurity in gypsum sample 2740 from the Mura deposit (Figure 6), and gypsum sample 2760 from Kwayaya (Figure 15).

Glauberite: $\text{Na}_2\text{Ca}(\text{SO}_4)_2$

This is an anhydrous sulphate of sodium and calcium. Generally known to occur as a saline residue associated with other sodium minerals of this type as is the case at Sass fort in Germany. This mineral was observed in both shale and gypsum samples throughout the area. It was identified in shale samples 2727, 2744, 2767 (Figures 4, 9 and 19) and as an impurity in samples 2741 and 2752 of gypsum (Figures 7 and 12).
Glauconite: K(Fe³⁺Al)(SiAl)₄O₁₀(OH)₂

It is essentially a hydrous silicate of iron and potassium, though aluminum, magnesium, and calcium are often present. In form, it is amorphous, granular or earthy, and in colour, olive-green-yellowish, grayish and blackish green. Its presence in sediment indicates shallow water, marine origin for the deposit. It occurs mainly in the shale's-samples 2748, 2751, and 2763 from Mura and Kwayaya (Figures 11-17).

Glaucophane: Na₂(MgFe)₂(Al,Fe³⁺)₂Si₈O₂₂(OH)₄

Silicate of sodium, magnesium, iron and aluminum Occur in metamorphic rocks such as glaucophaneschists, produced by the metamorphism of soda-rich igneous rocks and as a constituent of metamorphic rocks of sedimentary origin. Such rocks are within the Basement complex from which the sediments in the basin have been derived. It was found as an impurity in one gypsum sample 2749 at Mura (Figure 10).

Goethite: Fe₂O₃H₂O

Hydrous iron oxide with 62.9% iron. Brownish black, yellowish or reddish. Distinguished from limonite by being crystalline. Commonly occur in association with limonite, and hematite. Goethite is the chief constituent of limonite. Both result from the alteration of other iron minerals; from a highly ferruginous rock. Limonite is commonly yellowish brown. It is found as a constituent of the clays and the Kabul hill iron stones near Manawachi (Figure 13).

Ilmenite: FeTiO₄

Iron Titanium oxide. Specimens vary in composition, especially in the ratio of titanium to iron. It occurs as an accessory constituent in the more basic igneous rocks especially in gabbro and norites. Many important deposits of ilmenite are of detrital character chiefly beach sands in places like Australia and Senegal. Founding gypsum samples 2740 and 2748 at Mura (Figures 6 and 11).

Magnesite: MgCO₃

Occur sometimes in certain bedded deposits and interpreted as saline deposits. Occurs in one shale sample 2757 from Manawachi (Figure 13).

Muscovite: KAl₂(AlSi₃)O₁₀(OH,F)₂

Occurs as an original constituent of acid igneous rocks, such as granites and pegmatites. Workable deposits of Muscovite occur in large plates in pegmatites. Muscovite is a common constituent of metamorphic rocks-gneisses and mica schist’s etc. Muscovite is a very common constituent of detrital sedimentary rocks-micaceous sandstones, clays, etc. It is next to quartz in abundance in both the gypsum and the hosting shale’s. It was found in four of the shale
samples 2727, 2748, 2761, and 2767 from Mura and Kwayaya (Figure 3, 10, 15 and 18 respectively). It was also present in four gypsum samples 2740, 2741, 2749, and 2760 (Figures 6, 7, 10 and 15 respectively).

Orthoclase: $\text{KAlSi}_3\text{O}_8$

Orthoclase occurs as an essential constituent of the more acid igneous rocks such as granites, seynites, felsite. It occurs also as grains in feldspathic sandstones or arkoses, these resulting from the incomplete alteration and sorting of weathered granitic rocks. Occur in gypsum sample 2769 from Kwayaya (Figure 20).

Quartz: $\text{SiO}_2$

Present in virtually every sample analyzed, in the form of silica.

Sillimanite: $\text{Al}_2\text{SiO}_3$

Aluminum silicate. Occurs in the inner zone of hornfels. Resulting from the contact metamorphism of argillaceous rocks and in high grade of regionally metamorphosed rocks. It was found in Manawachi gypsum sample 2760 (Figure 15) and in one clay sample 2758 (Figure 14).

Strontionite: $\text{SrCO}_3$

Strontium carbonate with a small proportion of calcium carbonate usually present. Occurs in veins traversing cretaceous marls and limestones. Found in clay sample 2758 near Kwayaya (Figure 14).

**Environment of formation and mineralogical association**

Gypsum is generally known to be an evaporite and according to [13-15]. The mineralogical analysis carried out on the gypsum samples; indicate that the predominant mineralogical impurities in the samples are quartz, muscovite, biotite, glauberite and glauconite. Other less common impurities include goethite, carnalite, chlorite, chamosite,
and apatite. The minerals identified in the sediments include those derived from the igneous and metamorphic rocks characteristic of rocks of the Nigerian Basement Complex as well as those from the older sedimentary rocks. Minerals like apatite, muscovite, biotite, quartz and chlorite were evidently derived from a granitic terrain, while minerals like actinolite, glauconphane, silimanite and staurolite are likely to have been derived from a terrain of predominantly metamorphic rocks. The presence of the mineral glauconite in the sediments indicates deposition in a shallow marine environment. The presence of the minerals glauberite, and carnalite, in association with gypsum make the deposits comparable and similar in some degree to other evaporite successions in other parts of the world like Stassfort in Germany, the Saline valley in California, U.S.A, and the Karabogaz Gulf on the eastern side of the Caspian Sea.

Conclusion

The mineralogical analysis of the samples and its hosting Upper Cretaceous Fika Shale reveal predominant minerals occur either as impurities within the gypsum or in association with hosting shale in the diffract gram, these were similar across the study area and were consistent with general geology of the area.

References