ELEMENTAL AND MICROSTRUCTURAL CHARACTERIZATIONS OF IRON CONCRETION

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Abstract

A sample of Iron concreation is taken from the Moenyin Township, Kachin State. The elemental and characterization of this iron concretion are examined by utilizing XRD (X-ray diffraction) and EDXRF (Energy Dispersive X-ray Flurorescence) which are very useful for qualitative and quantitative analysis. The microstructural of iron concretion was studied by SEM(scanning electron microscopy). This study shows the different minerals in the sample and provide valuable information.

Keywords - Iron Concretion, XRD, EDXRF,SEM

Introduction

A concretion is hard, compact mass of matter formed by the precipitation of mineral cement within the spaces between particles and is found in sedimentary rock or soil. A iron concretion is the most abundant sedimentary rock. It is a rock composed mainly of clay size mineral grains and is also called a mud rock. It is a sedimentary rock that is usually from the compaction of at least 50 % silt and clay size particles. It can also contain organic material, iron oxide and heavy mineral grains. It is made up of very thin layers and has thin beds of either sandstone or limestone. The quantity of iron concretion depends on their thickness and extent, depth and pressure, fluid saturations, permeability and among other factors. Iron concretion can contain a wide range of minerals, although only the clay minerals and non clay mineral were found by X-ray diffraction. The type of clay found in iron concretion is a function of rock type and climate. A special variety of iron concretion is an iron rich material. A iron concretion was taken from Moenyin Township, Kachin State. Depositional environment was once thought to exert a considerable influence on clay mineralogy but it is now known that alteration of the clay framework does not occur, although there is a change in the exchangeable cation population. Rock type also plays its part in clay mineralogy. Illites probably derive from weathering of preexisting illites and chlorites from preexisting chlorites, thus certain rock type generate particular maineralogies. Black organics iron concretion are found in the earth. They can serve as a source rock for many oil and gas deposits. From the tiny particles of organic matter that were deposited with mud to form iron concretion. Iron concretion which are deposited in oxygen-rich environment often contain tiny particles of iron oxide or iron hydroxide minerals such as hematite, goethite or limonite. The presence of hematite can produce red iron concretion while the presence of limonite or goethite can produce yellow or brown iron concretion. High ratios of Fe^{+3}/ Fe^{+2} are associated with red colors and low with yellow or brown. Because the Fe^{+3}/ Fe^{+2} ratio is controlled by the oxidation state which in turn is controlled by the amount of organic matter in sediments, all color in iron concretion is ultimately controlled by the amount of organic matter present. The elemental analysis is characterized by using EDXRF (Energy Dispersive X-ray Fluorescence). The structural information is obtained by using the X-ray diffraction in the

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laboratory of UY. X-ray diffraction is widely used for the phase identification of a crystalline material and can provide valuable information. The objective of this experiment is to identify the different materials and grains size present in iron concretion.

Experiment

The range of particle size in the iron concretion is 16.12mm. The sample was made grounded to obtain powder by using pastel and motor. Experimental arrangement of sample preparation are shown in Figure 1(a-e). The elemental and structural characteristics are studied by X-ray diffraction (XRD) and energy dispersive X-ray fluorescence (EDXRF). Iron concretion was prepared by pressing into a pellet at 1 ton of dynamic pressure. The powder sample was put in the sample holder of the x-ray diffractometer and a test was conducted on it. The EDXRF spectrometer used was a tube excited system with molybdenum as anode, and silver in the secondary target arrangement. Figure 2 show as the x-ray diffractometers with interlocks and shielding. The beam function only with the door closed.

(a)

(b)

Figure 1(a-b) Photographs of iron concretion

(c)

Figure 1(c) Photograph the diameter measurement of iron concretion

Figure 1(d) Sample preparation preparationpreations

Figure 1(e) Photograph of the top-view of as-prepared powder of iron concretion

Figure 2 photograph of X-ray diffractometers

Results and Discussion

X-Ray Diffraction (XRD) Analysis

X-ray diffraction is used to determine crystallinity of material. XRD uses the total X-ray scattering both the crystalline and amorphous phases to determine the crystallinity. The analysis of iron concretion powder was determined by using a X-ray Diffractomerter (Rigaku Miniflux 600). XRD was performed using monochromatic CuKα radiation (λ=1.54056Å¬) operated at 40kV (tube voltage) and 40mA (tube current). Sample was scanned from 10° to 70° in diffraction angle 2θ with a step-size of 0.02°. The X-ray diffractograms of iron concretion is shown in figure 3. The upper side of XRD profile was represented the observed profile while the lower side indicated the standard or reference JCPDS (Joint Committee on Powder Diffraction Standard) library file. On the XRD patterns, five peaks were clearly observed. They were (110), (120), (111), (221) and (151) respectively.

The crystallite size (G) was determined by Debye-Scherrer formula,

$$G = \frac{\beta\lambda}{B\cos\theta_B}$$

where, G = crystallite size (nm)

β = Scherrer constant ≈ 0.899

λ = wavelength of X-ray diffraction (1.54056Å)

B = FWHM (full width at half maximum peak)

θ_B = Bragg angle (deg)

The FWHM and crystallite size of iron concretion powder were shown in Table 1. The average crystallite size of iron concretion powder was in the range of 37.89 nm. The XRD measurement showed that all peaks of iron concretion were consisted with that iron concretion standard (JCPDS) file having a Al-substituted goethite.

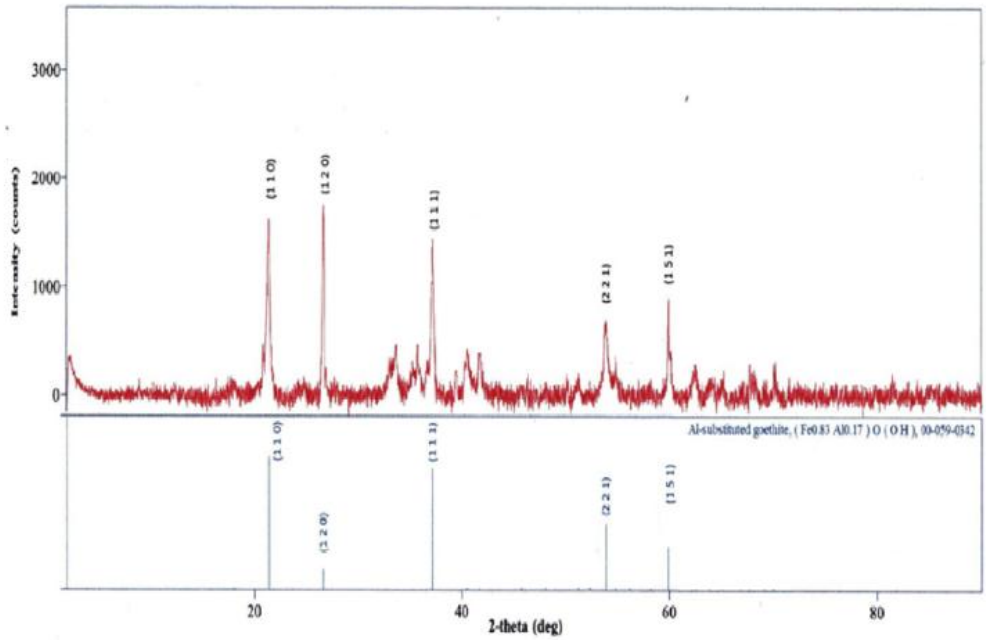

Figure 3 XRD pattern of iron concretion

Table 1 Crystallite size of iron concretion

No:	Peaks(hkl)	FWHM(deg)	Crystallite size (nm)
1	(110)	0.1647	49.03
2	(120)	0.1839	44.34
3	(111)	0.2194	31.16
4	(221)	0.2695	33.03
5	(151)	0.2870	31.91
		Average crystallite size	= 37.89 nm

EDXRF Analysis

The samples were collected from Moenyin Township, Kachin State. The samples were analyzed using EDXRF spectrometer at Universities' Research (URC) in University of Yangon. The spectrometer was operated at its optimum conditions. The spectrometer was operated at its optimum conditions. The detection unit is a Canberra Si (Li) detector with a resolution of 170eV at 5.9 keV. X-ray spectra were acquired on a PC-based MCA, Canberra S100. The sample was then irradiated for 3000s. The X-ray tube was operated at its optimized condition of 40 kV and 10mA. The EDXRF spectrum of the iron concretion was then recorded. It is shown in Figure 4.The concentration of elements contained in iron concretion sample, are listed in Table 2.

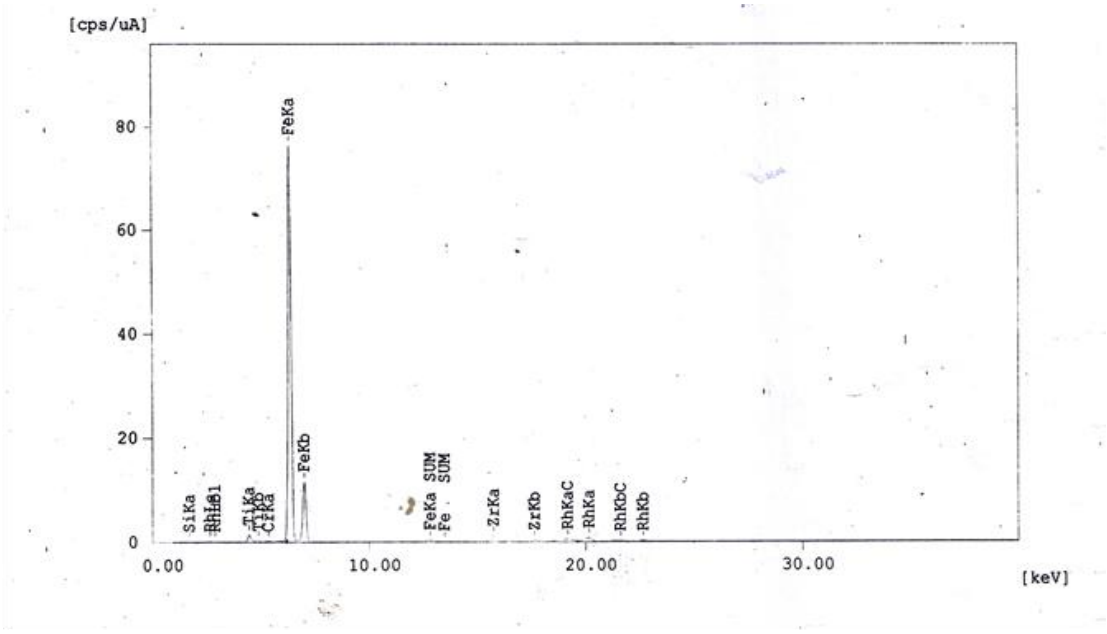

Figure 4 The EDXRF spectrum of iron concretion

Table 2 The elemental composition in iron concretions

Atomic No(Z)	Element	Concentration(%)
26	Fe	85.694
14	Si	11.017
81	Ti	2.473
40	Zr	0.706
24	Cr	0.110

Scanning Electron Microscopy (SEM) Analysis

The Scanning Electron Microscopy (SEM) is a type of electron microscope capable of producing high-resolution image of a surface iron concretion powder were obtained by calcination of the sample. Figure 5 showed the SEM analysis of iron concretion powder exhibit grained microstructure with small crystalline size. The grain size were calculated by using well known bar code system with Image J software. The average grain size of the sample powder was found to be about 1.145 μm. From the image, it was clearly found that the little amount of pores and grain growth pattern. This figure indicated that most of the grain size was regular structure and a few number of larggrain size was found. The sample powder appear with little agglomeration in light contrast with background particles. According to SEM, analysis, the SEM image was successfully implemented the smooth in morphology, grain size and particle quality of iron concretion powders.

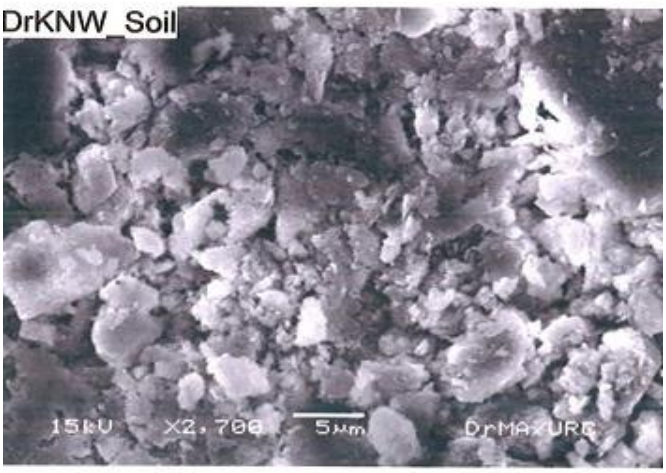

Figure 5 The surface morphology of iron concretion

Conclusions

Iron concretion can contain large amounts of various minerals depending on its burial history and sedimentation. Elemental and structural characterizations of iron- concretion were characterized by XRD and EDXRF measurements Experimental results concluded as follows. XRD results show that the sample is Al-substituted goethite structure, their different crystalline structures make them distinct minerals. The mineral enthusiast chemical formula was Fe Al O(OH). EDXRF results show that the elemental compositions in iron concretion are Fe, Si, Ti, Zr, Cr are found in this sample. There were more concentration of iron in this sample. According to the SEM image the grain size was regular structure and a few number of large grain size was found. Goethite is a common weathering product of iron-bearing minerals. Iron concretion resists earthquake. So, it can be used for construction such as the wall, the ceiling and the building block. The research can be used as a fertilizer.

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