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Full Length Research Paper

Instrumental analysis of Arrinrasho clay for characterization

***Edah A.O[1] , Kolawole J.A[2], Solomon A.O[3], Shamle N[1] and Awode A.U[1]**

[1]Department of Chemistry University of Jos, Jos Nigeria.
[2]Department of Pharmaceutical Chemistry University of Jos, Jos Nigeria.
[3]Department of Geology and Minning University of Jos, Jos Nigeria.

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Instrumental characterizations of Arrinrasho clay were performed by X-ray techniques of XRF, XRD and FCC15. XRF gave the major chemical composition of the clay samples as alumina and silica. XRD analysis revealed the structures of the minerals in the clay, confirming the presence of Parahopeite, Nacrite, Kaolinite, Halloysite Chrysotile, talc, reibeckite, Antigorite, Grunerite and Cristobalite in the clay samples. The FCC15 gamma scout radiation meter gave the ionizing and background radiations ranges as 2.30 – 3.60 mS/yr and 1.01 – 1.56 mS/yr respectively.

Keywords: Instrumental Characterizations, XRF, XRD, FCC15.

INTRODUCTION

The complex nature of clay makes its study and findings an ever fresh area of interest especially to the world of science. Clay and its minerals have played major roles in anthropogenic activities (Sidhu and Gosh, 1996). The low cost of clay and its relative abundance in nature, high sorptive /electric charge properties, plus ion exchange ability and compatibility with several materials, gives it a wide range of application (Barbel and Kurnianwan 2003) and (Costanzo, 2001). Clay materials present layer and sheet orientations. The several possible structural presentation of the elements in clays results in different classes of clay, such as Kaolinite, Serpentine, pylophyllite (talc), smectite (Bentonite/montmorillonite, saponite), sepiolite and vermiculite (Shichi and Takaji 2000). The particle size, surface area and high charge density of clay material are some of the properties that make for the adsorption capacity of clays (Alkan *et al* 2004). XRF technique may be used to determine the concentration of major metallic elements such as Na, Si, K, Fe, Cu, Zn, minerals Zn, Fe, Mg, Ca, toxic elements such as As, Pb and Cd, contaminants in the form of Ag and Hg and many materials used as fillers, pigments and additives.(Uribe, 2000). Though, Silica, alumina and water are the basic components of clay. Iron alkalies and alkali earth metals may be present in good measures (Stevens and Anderson, 1996). Clay is structured at an atomic, molecular and macro level, and these structures interact to produce the variations in observed behavior (Velde, 1995). The atomic lattice of clay minerals present two unique structural units of octahedral and tetrahedral conformations. The octahedral conformation, this involves oxygen and hydroxyl groups that are linked to Aluminum, Iron and Magnesium atoms at equidistance from six oxygen or hydroxyl species and the tetrahedral Silica conformation, where the units are oriented into hexagonal network, which is repeated continuously, forming a sheet of silica. The different ratios of the basic components of clay and the several possible combinations of the orientations of layers and sheets in conjunction with the particular metal present, determines the mineral type of clay (Holtz and Kovacs, 1981). In view of this natural architectural design of clays, it is imperative to always characterizes them before putting it to use. There is no report in the literature to our knowledge that the huge clay deposit at Arrinrasho has been characterized. This work is therefore set to use

*Corresponding author E-mail: edahalex2005@gmail.com
Tel: +2348065585338

standard methods to characterize Arrinrasho clay deposit.

MATERIALS AND METHOD

Major instruments and equipment were, X-ray Fluorescence Spectrometer, (Panalytic, PW4030), X-ray diffractometer model 2000 (Shimadzu …), FCC15 (Gamma scout radiation meter).

Sample and Sampling

A hundred samples of clay were excavated from open pits at Arrinrasho in Barkin Ladi, Plateau central Nigeria. The sampling points were located by GPS corresponding to latitude (09° 51' 56.4˝ to 09° 28̋ 55.5˝), longitude (008° 51'05.6˝ to 08° 51'11.4˝) and an elevation between (4305 to 4590)ft above sea level. The clay was reduced to powder. The pulverized composite samples of clay were collected in pre-labeled sampling poly bags for analyses. The composite samples were labeled 102, 104, P104, and EX110

Sample Preparation

The pellet for the Energy Dispersive X-ray Fluorescence spectrometer were prepared by weighing 0.005g of the powder sample, into an agate mortar and properly mixed with three drops of binder (consisting of polyvinylchloride dissolved in Toluene). The mixed clay and binder were scraped into a mold with a punch and die. This was compressed into pellet with a force of 20 tones in a hydraulic press. The clay samples were properly pulverized to powder for in readiness for XRD as reviewed by Bish and Post in 2004. Sample Preparation: To ensure homogeneity, each of the samples was prepared using the Moore and Reynolds procedure. The analytical method adopted was as found in JCPD Data Book, 1974.

Determination of Physical Parameters

Standard methods were used for the physical parameters as listed below. The color, moisture content, particle size, pH, tap density, and conductivity of the samples were determined using tintometer, calibrated oven, standard sieves and shaker, pH-meter and conductivity meter respectively. All instruments were calibrated before use.

Quantitative Determination of Elements by XRF

A modular system Energy Dispersive X-ray Fluorescence Spectrometer, (Panalytic, PW4030), was employed in the elemental analysis. The pellets prepared above were placed in ten sample holders, while the remaining two sample holders housed the standards. Each set of samples were placed in the sample chamber of the spectrophotometer. A voltage of 30kv and a current of 1mA were applied to generate the X-ray needed to irradiate the samples for a preset period of 10 minutes. The spectrum from the sample were analyzed and concentration of the elements determined by a modular system with mini Pal Analytic soft ware.

Calibration

The calibration factors for the EDXRF were sourced from The International Atomic Energy Agency (IAEA). The standard samples were irradiated simultaneously with the samples under investigation.

X-Ray Diffraction (XRD) Determination

The XRD patterns of the Arrinrasho clay samples were determined by employing the Shimadzu X-ray diffractometer model 2000. The sample holder had provision to vibrate and rotate the sample from several orientations (Sarrazin et al., 2004).

Determination of Radiation Properties

Measurements were done with FCC15 (Gamma scout radiation meter). The batteries were guaranteed by the manufacturers. (The instrument is accompanied with the following features •Measures alpha, beta, gamma and x-rays •Reliable LND712 Geiger - Müller detector •Large easy-to-read 4-digit LCD display •Ultra long life 10 year built-in battery •Two Kilobytes on board memory •Integrated serial data transfer port •Compact, ergonomic hand held design • High impact Novodur® housing •Wide temperature range (-40 ~ +75℃))

RESULTS

The following observations and results were obtained on the Arrinrasho Clay Samples (ACS)

DISCUSSION

The result displayed in Table 1, shows the physical properties of the Arrinrasho pulverized clay samples (ACS).The color ranged from white to slightly off white, its texture is smooth, displaying a free flowing pattern on discharge. The particle size passed 30µm mesh, indicating a fine nature. These are comparable to the acceptable properties of clays characterized by United States Geological Agency (Foley, 1999).

Table 2, shows the XRF elemental analysis of Arrinrasho clay. This reveals that it mainly contains Silica, Alumina and Iron Oxides, and minor components of Copper, Nickel, Zinc, Chromium, Titanium, Calcium, and Manganese Oxides.

The quantity of Silica and Alumina lie between (49.01 –

Table 1. Observation and Result of physical parameters

Parameter	Observation/Result	Parameter	Observation/Result
Color	White to Off white	Moisture content	2.78 ± 0.23
pH	6.5 - 7.5	Radioactivity Level	(3.2 - 3.6)mS/yr
Particle size	30µm	Tap density	53.0 ± 0.25g/cm^3
Conductivity	357 ± 20µS/cm	Texture	Smooth/free flowing

Table 2. The XRF elemental analysis of Arrinrasho clay showing the % of compounds present

Samples	SiO_2	Al_2O_3	CaO	MnO	TiO_2	Fe_2O_3	Cr_2O_3	ZnO	NiO	CuO
102	49.01	34.20	0.27	0.01	0.01	3.96	0.14	0.21	0.09	0.10
104	61.50	33.40	0.61	0.01	0.01	2.78	0.05	0.07	0.02	-
P104	58.01	36.40	0.29	-	-	3.28	0.07	0.08	0.03	0.04
EX110	62.60	34.20	0.28	-	-	1.58	0.07	0.10	0.02	-

Table 3. The XRD Mineral analysis of Arrinrasho clay showing the number and major minerals present

Composite Sample	NMI	MMP
102	9	Parahopeite,Nacrite, Kaolinite, Anthop
104	8	Halloysite, Chrysotile, talc, Riebeckite, Antigorite
P104	7	Grunerite, Cristobalite, Riebeckite, Antigorite.
EX110	4	Halloysite,Kaolinite, Antigorite, Anthop

NMI: Number of mineral identified. MMP: Major Mineral Present

Table 4. Showing Ionizing Radiation properties of Arrinrasho clay

Composite Sample	Ionizing Radiation mS/yr	Background Radiation mS/yr
102	2.30	1.01
104	3.10	1.56
P104	3.60	1.53
EX110	2.63	1.01

62.60%) and (33.40 – 36.40%) respectively. The Iron and Zinc Oxides were (1.58 – 3.69%) (0.07 – 0.21%) respectively. The Iron content being lower than 4.00% coupled with TiO_2 at 0.01% gave the Arrinrasho clay sample a brilliant white to slightly off-white powdery appearance when pulverized. Table 3, gives the summary of the XRD analysis of the ACS, the minerals confirmed include Parahopeite, Nacrite, Kaolinite, Anthop (composite 1),Halloysite, Chrysotile, talc, Riebeckite, Antigorite(composite2),Grunerite, Cristobalite, Riebeckite, Antigorite (composite 3) and Halloysite,Kaolinite, Antigorite, Anthop (composite 4). These minerals have been reported in standard instrumental characterization of clays employing XRF and XRD techniques by (Preeti and Singh, 2007). This analytical technique probes the crystal lattice structure of the pulverized samples. An x-ray is beamed into the samples, the distance between the waves as they exit the sample is due to the diffraction of the waves by the basal plane. This d-spacing refers to the spacing between planes of the crystal lattice. In the occurrence of several lattice structures or spacings between platelets, there will be multiple peaks on the graph generated (Ryan, 2007).

The results in Table 4, displays the summary of the FCC15 gamma scout radiation meter. This device is sensitive and reliable. The ionizing radiations in mS/yr were 2.30, 3.10, 3.60, and 2.63 for the composites samples 1, 2, 3 and 4 respectively. These values of ionizing radiations are within the limits of 5.00 mS/yr of human exposure per year as set by USEPA, 2007.

CONCLUSION

The physical, chemical and instrumental (XRF, XRD and FCC15) analysis confirmed that the Arrinrasho clay samples had Alumina, Silica as the major constituents and Iron, Calcium and Zinc Oxides as the minor

constituents, while Manganese, Titanium, Chromium, Nickel and Copper Oxides were comparatively at the trace levels.

The X-ray diffraction, suggests, Parahopeite, Nacrite, Kaolinite, Anthop, Halloysite, Chrysotile, Talc, Reibeckite, Antigorite, Grunerite and Cristobalite as the major phases. The FCC15 gamma scout radiation meter gave a maximum ionizing radiation of 3.60 mS/yr from the clay samples.

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