

**American Chemical Science Journal**  
3(3): 287-293, 2013

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## **XRD Characterization of Sand Deposit in River Niger (South Eastern Nigeria)**

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### **Authors' contributions**

*This work was carried out in collaboration of all authors. Author DCB collected and performed all analyses, characterization, and wrote the first draft of the work. Author OF managed the literature searches. Author EUE designed the study, managed the analyses of the work, interpreted the results, corrected, and prepared the final manuscript. All authors read and approved the final manuscript.*

**Research Article**

**Received 12<sup>th</sup> February 2013**  
**Accepted 18<sup>th</sup> March 2013**  
**Published 1<sup>st</sup> June 2013**

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### **ABSTRACT**

In this study the sand deposits from River Niger in Anambra State, South Eastern Nigeria, were characterized for its potential utilization as industrial raw materials for ceramics and enamel wares. Physical, chemical and mineralogical characteristics of the sand sample (A) were determined. X-ray diffraction (XRD) was used in the mineralogical characterization. Results obtained were analyzed using Bragg-Wolf equation and International Centre for Diffraction Data software. The results show that the sample contain the phyllosilicate minerals of mica group and identified as shirozulite ( $\text{KMn}_3[\text{Si}_3\text{Al}]\text{O}_{10}[\text{OH}]_2$ ) a new manganese dominant of monoclinic arrangement. The physico-chemical analysis of the deposits corroborates the XRD results. The results concluded that the samples could be utilized as industrial raw materials for ceramic and enamel wares.

**Keywords:** *Characterization; XRD; Shirozulite; crystals; clay; mineralogy; analysis.*

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## DEFINITIONS, ACRONYMS, ABBREVIATIONS

*XRD – X-ray Diffraction.*

### 1. INTRODUCTION

Quartz is the most common mineral on the face of the Earth. It is found in nearly every geological environment and is at least a component of almost every rock type. It frequently is the primary mineral, >98%. It is also the most varied in terms of varieties, colors and forms. [1]. This variety comes about because of the abundance and widespread distribution of quartz. A collector could easily have hundreds of quartz specimens and not have two that are the same due to the many broad categories. Multiple combinations of the various forms of the quartz could produce hundreds of unique possibilities. Quartz also has associated minerals of numerous and varied forms which includes: amazonite a variety of microcline, tourmalines especially elbaite, wolframite, pyrite, rutile, zeolites, fluorite, calcite, gold, muscovite, topaz, beryl, hematite and spodumene [2]. Most of these minerals are of mica group. Shirozulite is a new Mn-dominant trioctahedral mica. The mineral occurs in tephroite-rhodochrosite ores in contact with a Ba-bearing K-feldspar vein. Mica has been known as a typical insulator and is used as filler for cosmetics, paints, machinable-ceramics, etc. The component of mica is  $\text{SiO}_2$  (40-50 wt %) [3]. Very strongly bonded  $\text{SiO}_4$  tetrahedral range as a sheet of hexagonal net between the sandwich-type plates called “tablets” are pulling up and between these tablets exist alkaline metals or alkaline earth metals which are weakly bonded with 12 oxygen atoms on the upper and lower plates [3]. Clay product derived from the weathering and hydrothermal reactions of rocks is a versatile industrial mineral that has amazing variety of uses and application as in petroleum cracking, cosmetic base, paper chalk, ceramics porcelain, dinner works, architectural tiles, pharmaceutical industries, etc [4,5]. The growing interest and importance of surface properties in engineering applications is obvious. Evidence is the relationship between coating properties and surface function performance. Among various coating systems for industrial and engineering applications, glass-ceramic coatings have advantage of chemical inertness, high temperature stability and superior mechanical properties such as abrasion and impact as compared to non oxide coatings in use such as paints, metal, polymer, rubber. Besides imparting required functional properties such as heat, abrasion and corrosion resistance to suit particular end use requirements, the glass ceramic coatings in general also provide good adherence, defect free surfaces and refractoriness. Conventional enamel coatings and glass ceramic coatings are still imported into Nigeria till date, where as the resources needed for their production are abundant, largely underdeveloped and are exported in raw form [6]. Increased and continuous dependence on these imported products may have serious implications on the local market. The development of local capabilities is therefore imperative in view of the need to build solid foundation for diversification of Nigeria economy. In this study the sand deposits from River Niger in Anambra State, South Eastern Nigeria was explored and characterized for its potential utilization as industrial raw materials for ceramics and enamel wares.

### 2. METHODOLOGY

Sand sample A was collected from banks of River Niger located in Anambra State, South Eastern Nigeria. Physical, chemical and mineralogical characterizations were determined. The chemical composition was done after acid digestion using Buch Scientific 210 VGP Atomic Absorption Spectrometer. Loss of Ignition (LOI) was done by gravimetric method

while pH determination was carried using Jenway pH meter. Colours were compared with the standards. The mineralogical characterization was done using monochromatic x-ray (MD 10 mini diffractometer, version 2) with Beta filter CuK $\alpha$  radiation of wavelength ( $\lambda$ ) of 1.5406Å and automatic slit. A set of  $2\theta$  angle ranging from 15° – 75° was used. This was done at the Engineering Materials Development Institute (EMDI), Akure, Ondo State of Nigeria. In the X-ray absorption analysis, a crystalline powdered specimen was exposed to a beam of X-rays of suitable energy. Diffraction occurs, the angles of deviation and relative intensities of the deviated beams were measured. The structural properties were determined from the measured beams as crystal systems, crystal structure, inter planar distance and lattice constant.

### 3. RESULTS AND DISCUSSION

#### 3.1 Physical Characterization

The physical characterization of the sand sample were shown in Table 1

**Table 1. Physical characterization of sand sample A**

Particle size distribution (PSD) [7]	A
Clay	50%
Sand	30%
Silt	20%
Colour	Red gray
Specific gravity	0.894
Density	0.89g/cm <sup>3</sup>

From the results shown in Table 1, the particle size distribution gave the highest percentage for clay followed by sand and the least percentage for silt. This is an indication that they will be of immersed geological, industrial and agricultural importance [8,9].

#### 3.2 Chemical Composition

The result of the chemical composition is shown in Table 2. The result shows high silica and alumina contents of ratio 2:1. The presence of oxides of alkali and alkaline earth metals were observed with CaO, TiO<sub>2</sub>, NaO and MnO in very low concentration in the samples studied. This shows that the samples fall into clays recommended for refractory work [10]. For good refractory characteristics, clay should have a percentage composition of Al<sub>2</sub>O<sub>3</sub> between 30% and 50% with a limited amount of Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub> and CaO [11]. In Table 2 is shown the chemical composition of sample A.

Table 2. Chemical composition of sample A

Component	A wt (%)
SiO <sub>2</sub>	59.30
Al <sub>2</sub> O <sub>3</sub>	27.14
Fe <sub>2</sub> O <sub>3</sub>	Nil
TiO <sub>2</sub>	0.03
MgO	2.23
MnO	0.17
CaO	0.07
K <sub>2</sub> O	3.29
Na <sub>2</sub> O	0.05
*LOI	12.75
pH	5

\*LOI: Loss on Ignition

The oxides of sodium, potassium and magnesium are the main fluxing and ion-exchange materials in clays. Thus, the vitrification and ion exchangeable materials of these samples are expected to be low. This is however an added advantage for their application in ceramics and enamel industry since a high level of CaO can cause undesirable expansion and subsequent cracking in structures [12,11].

### 3.3 X-ray Diffraction Investigation

The results of the X-ray diffraction (XRD) reveals that the sample comprised of different types of minerals with quartz, phyllosilicates of mica group and feldspar being predominant in the samples and minor amounts of illites. In Figs. 1a and 1b is shown the XRD spectra and interpretation of sample A.

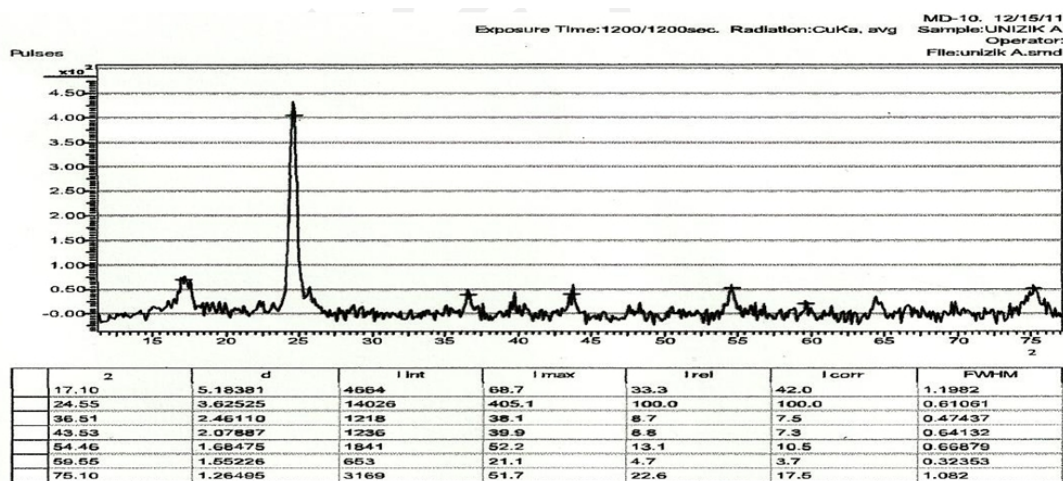
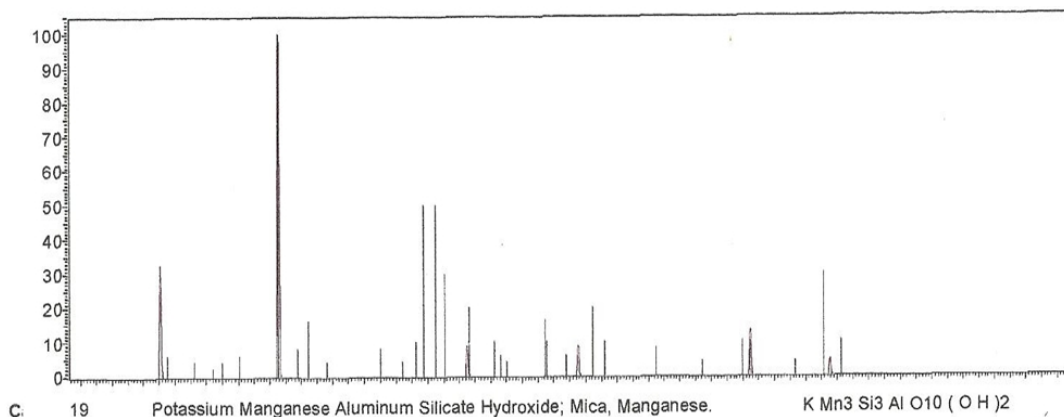


Fig. 1a. X-ray diffraction of sample A on CuKα; in the 2θ region, glancing angle 15°–75°

Sample A identified as Shirozulite of formula  $K(Mn^{2+}, Mg)_3(AlSi_3O_{10})(OH)_2$  in Fig. 1b.



**Fig. 1b. XRD interpretation of sample A**

In Table 3 is shown structural parameters of sample A

**Table 3. Structural parameters of sample A**

Empirical formula	$\text{KMn}_3\text{Si}_3\text{AlO}_{10}(\text{OH})_2$
Wavelength (Å)	1.5405
Crystal system	Monoclinic
Crystal size ( $\text{mm}^3$ )	0.113
Space group	C2-m
a (Å)	5.41(7)
b (Å)	9.37(1)
c (Å)	10.23(9)
$\beta$ ( $^\circ$ )	99 (7)
Volume, V ( $\text{Å}^3$ )	512.19
Z	2
$2\theta$ range ( $^\circ$ )	15 -75

The basal (001) d spacing of the Braag's Wolf equation ( $n\lambda = 2d\sin \theta$ ) of the sample gave a monoclinic prismatic crystal of class space group C2/m which was sharp at 5.18381Å and collapsed at 1.26495Å during the heating processes at the corresponding glancing angles of analysis. The chemical composition shown in Table 2 compared well with the mineralogical composition. The description and crystal structure by the report corresponds with the results presented in Table 3. For instance the reported lattice constant a = 5.41Å, cleavage of (001) perfect and red gray colour in Table 3 compared well with a value of 5.3791Å, cleavage of (001) perfect and dark reddish brown colour obtained by an earlier report [13]. Shirozulite has found application in micro porous materials because of the presence of suitable amounts of micro-crystalites uniformly distributed in a glassy face [6]. The identified mineral, Shirozulite, is new, a manganese dominant trioctahedral mica discovered in 2004 in Japan [14]. In most third world countries like Nigeria for instance non-black fillers including clays have been largely imported where as clay deposits are in great abundance in nearly every local government area of South East of Nigeria [14]. This study has implied that this local raw material should be used in place of the imported ones.

#### 4. CONCLUSION

In this study sand deposits from River Niger in Anambra State, South Eastern Nigeria have been characterized for its possible utilization in ceramics and enamel industries. The results showed that the sample is rich in quartz which is the major component of mica and feldspar. The level of its alkali and alkaline earth metal oxides is an advantage to the desired application in ceramics and enamel industries.

#### ACKNOWLEDGEMENTS

The assistance of the XRD department of the Engineering Materials Development Institute (EMDI), Akure, Ondo State of Nigeria is hereby acknowledged.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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