



Characterisation of Mica from Bukkuyum Area of Zamfara State, Nigeria

Abubakar G. Salisu*¹, Hadiza A. Isa¹, Hamisu Adamu¹ and Zalihatu M. Idris²

¹Department of Applied Science, Kaduna Polytechnic, Kaduna, Nigeria.

²Department of Science Laboratory Technology, Abdu Gusau Polytechnic,
Zamfara State, Nigeria.

*Corresponding author: abasal012@gmail.com

<https://www.tetfundkadpolycoex.org.ng>

ARTICLE INFORMATION

Article history:

Received 13 Jun., 2023

Revised 26 Sep., 2023

Accepted 20 Oct., 2023

Available online 20 Oct., 2023

Keywords:

Evaluation

Mica

XRD

XRF

SEM

Abstract

The term mica is a generic term that applies to a group of aluminosilicate minerals that possess a sheet-like structure. The most common commercial micas are Muscovite is a potassium-based mica, usually colorless to pale green, others include Phlogopite, Lepidolite, Zinnwaldite, Biotite and Roscoelite. Muscovite mica enjoys the most diverse market applications. Muscovite and phlogopite possess unique characteristics and are highly valued because of their physical, chemical, electrical, thermal, and mechanical properties. These properties include high dielectric strength, low coefficients of expansion, cold and heat resistance, and high tensile strength, making these minerals excellent raw materials for electrical insulators, reinforcing materials, and high-temperature applications. As with all industrial minerals, several factors determine the test program implemented in any evaluation project. The material to be tested could be from new, unexploited deposit or from existing. This paper is aimed at evaluating Mica from Bukkuyum local government area of Zamfara state of Nigeria, using Empyrean diffractometer DY 674 (2010) for XRD phase analysis of the powdered sample. The peaks generated matched those of a muscovite in ICDD PDF 2 (2010) database. Minipal 4 energy dispersive X-ray Fluorescence (XRF) provided the elemental analysis in their oxides with 48.7% SiO₂, 33.3% Al₂O₃, 10.2% K₂O, 1.04% Fe₂O₃ as major composition with loss on ignition (LOI) of 0.79% and SEM provided the photomicrograph picture with the fiber histogram giving the particle size statistics of 959.92nm, 4.22µm and 11.23µm, with the objective of giving adequate information for any of its numerous industrial applications.

1.0 Introduction

The term mica is derived from the Latin micare, meaning to shine or glitter. Today, it is a generic term that applies to a group of aluminosilicate minerals that possess a sheet-like structure. Mica is a mineral that has a rich history of industrial applications, over the centuries, mica uses have evolved from decorative ornaments, mirrors, stove windows, and shock-resistant materials in early

military applications to its more high-tech applications today in construction materials, paper, plastics, paints, electronics, and others (John and James, 2006; Bailey, 1984),.

The most common commercial micas are Muscovite: a potassium-based mica, usually colorless to pale green. Phlogopite: a magnesium-based mica that is yellow to dark brown. Lepidolite: a lithium-based mica with a purplish hue. Other micas with less commercial value include Zinnwaldite: a lithium/iron-based mica that is gray to brown; Biotite: a magnesium/iron-based black mica; and Roscoelite: vanadium/potassium/ magnesium mica that can be green or brown, Muscovite mica enjoys the most diverse market applications, followed by phlogopite and veimiculite (Harben, 2002). Muscovite and phlogopite possess unique characteristics and are highly valued because of their physical, chemical, electrical, thermal, and mechanical properties. These properties include high dielectric strength, low coefficients of expansion, cold and heat resistance, and high tensile strength, making these minerals excellent raw materials for electrical insulators, reinforcing materials, and high-temperature applications (Hendrick, 2002).

Elemental substitution takes place within the various micas quite readily. The mica group includes the more common and most widely used mica species such as muscovite, paragonite. and phlogopite. Other micas in this group are biotite and lepidolite. Muscovite and paragonite (both are commonly referred to as muscovite) enjoy the most industrial uses and subsequent exploitation. Muscovite is the most common of all the micas, having a chemical formula $KAl_2(OH)_2(AlSi_3O_{10})$ with a theoretical composition of 11.8% K_2O , 45.2% SiO_2 , 38.5% Al_2O_3 and 4.5% H_2O . Paragonite is similar in composition, with the potassium replaced by sodium (Sinkankas, 1964; Skillen, 1992).

As with all industrial minerals, several factors determine the test program implemented in any evaluation project. The material to be tested could be from new, unexploited deposit or from existing. This paper is aimed at evaluating Mica from Bukkuyum local government area of Zamfara state of Nigeria, using Empyrean diffractometer DY 674 (2010) for XRD phase analysis of the powdered sample. The peaks generated match those of a muscovite in ICDD PDF 2 (2010) database. Minipal 4 energy dispersive X-ray Fluorescence (XRF) provided the elemental analysis in their oxides, and SEM provided the photomicrograph picture with the fiber histogram giving the statistics of particle size, with the objective of giving adequate information for any of its numerous industrial applications.

2.0 Experimental Procedure

2.1 Materials and Methods

The sample of Mica sheets was collected from Bukkuyum local government area, Zamfara state of Nigeria. Using, Empyrean diffractometer DY 674 (2010) for (XRD) phase composition analysis, Minipal 4 energy dispersive X-ray Fluorescence for (XRF) elemental analysis and PhenomProX Scanning Element Microscope (SEM) for micrograph morphological picture of the Mica sample.

2.2 XRD Analysis of the Sample

The sample was finely ground, homogenized, and sieved to pass through the 75 microns. A representative portion of the powdered sample was then prepared using the sample preparation block and compressed in the flat sample holder to create a flat, smooth surface that was later

mounted on the sample stage in the XRD cabinet.

The samples were analyzed using the reflection-transmission spinner stage using the Theta-Theta settings. Two-Theta starting position is 0.00483 and ends at 75,000 with a two-theta step of 0.026 at 3.57 seconds per step. Tube current was 40mA and the tension was 45VA. Fixed Divergent Slit size of 1° is used and the goniometer radius was 240mm. The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. A peak intensity occurs when the mineral contains lattice planes with d-spacings appropriate to diffract X-rays at that value of θ , although each peak consists of two separate reflections ($K_{\alpha 1}$ and $K_{\alpha 2}$), at small values of 2θ the peak locations overlap with $K_{\alpha 2}$ appearing as a hump on the side of $K_{\alpha 1}$, Greater separation occurs at higher values of θ . Typically these combined peaks are treated as one. The 2λ position of the diffraction peak is typically measured as the center of the peak at 80% peak height.

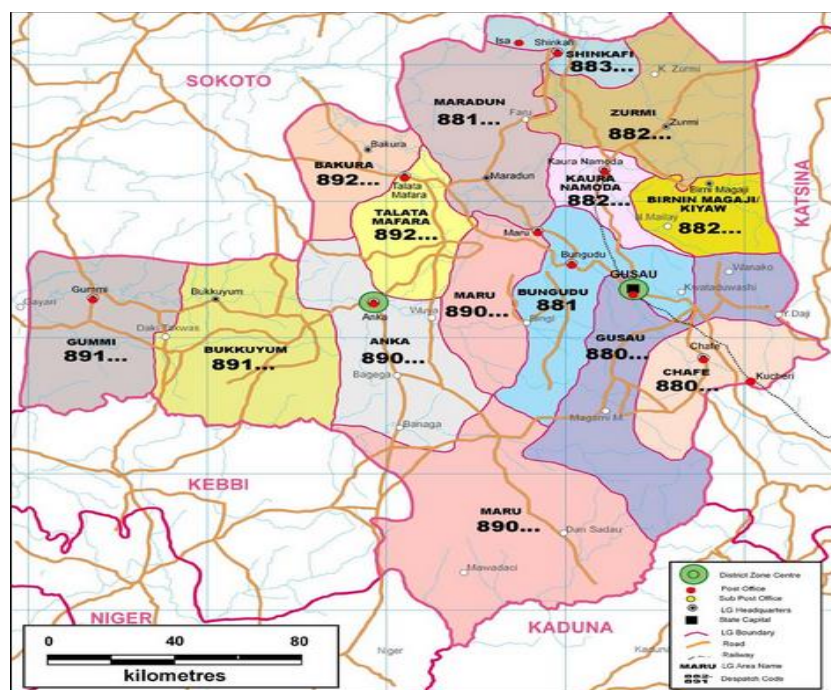


Figure 1: Map of Zamfara state showing fourteen local government areas, with Bukkkuyum local government area where the barite found second from left.

Results are presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table, and x-y plot. Intensity (I) is reported as peak height intensity. That is intensity above background, or as integrated intensity, the area under the peak. The relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak (*relative intensity* = $I/I_1 \times 100$).

The d-spacing of each peak is then obtained by solution of the Bragg equation for the appropriate value of λ . Once all d-spacing have been determined, automated search/match routines compared the unknown to those of known materials. Because each mineral has a unique set of d-spacing, matching these d-spacings provides an identification of the unknown sample. A systematic procedure was used by ordering the d-spacings in terms of their intensity beginning with the most

intense peak. Files of d-spacings for hundreds of thousands of inorganic compounds are liable from the International Centre for Diffraction Data as the Powder Diffraction File (PDF). The peaks obtained from the analyses were matched with the minerals from ICDD database which was attached to the software of the machine.

2.3 XRF Analysis of the Sample

Solid sample pellet of 10g was placed in the special “dekat” plastic cup sample holders. The sample was then loaded on the sample changer and the position was noted. Beads used for the major oxide analysis which are SiO₂, Al₂O₃, Fe₂O₃, MgO, TiO, MgO, CaO, K₂O and P₂O₅ expressed in weight percent were precast. 1.0g of pulverized sample was weighed and mixed exactly with 5times of X-Ray flux (66.0% lithium Tetraborate, 34.0% Lithium Metaborate). This weighed mixture was mixed properly in a Platinum crucible and ignited in a pre-set furnace at 1150°C for 10 minutes. Immediately the weighed mixture in the crucible was placed on the crucible holder, the preparation process commenced as the start button was pressed on. It was heated, mixed and automatically cast into a dish (mold). During this process a releasing agent was added (ammonium iodide tablet). Upon completion of cooling the cold glass bead was unloaded ready for analysis. The glass bead was labeled and slotted into the Minipal 4 energy dispersive XRF for major oxide analysis. For XRF Trace elemental analysis, the pellets were prepared by weighing 1.0g of oven dried pulverized sample and 5.0g flux (cellulose powder) added as a binder. This is mixed thoroughly with glass rod in a bowl. The well mixed was then compressed by applying a pressure of 1500kgm using manual compressor. The steps carried out for measurements were; Loading the sample, and opening the measure window for the required application i.e.:

- Selected measure application from the menu bar
- clicked on the required application to select it
- Clicked on the "sample identity" file to insert the 'text cursor.
- Typed a name for the sample to be measured.
- clicked on the position of the sample in the "select changer position" to measure

2.4 SEM of the Sample

The Solid samples pellet was prepared for scanning electron microscopy by crushing and grinding, to acquire such images on the morphology and surface features of the samples at micrometer scales. The SEM has an electron optical column in which a finely focused high intensity electron beam was scanned over a specified area of the specimen small particles mounted on a stub, the impact of the electron beam on the surface of a solid sample causes various types of radiation signals that were recorded by detector above the specimen.

3.0 Results and Discussion

3.1 XRD Phase analysis

From Figure 2 show the XRD peaks of the crude Mica powder, which indicates peaks corresponding to muscovite pattern with reference code 01-070-1869 on ICSD card.

3.2 XRF Elemental analysis

From table 1 the elemental composition mica from bukkuyum local government area of Zamfara state, Nigeria are presented in their oxides.

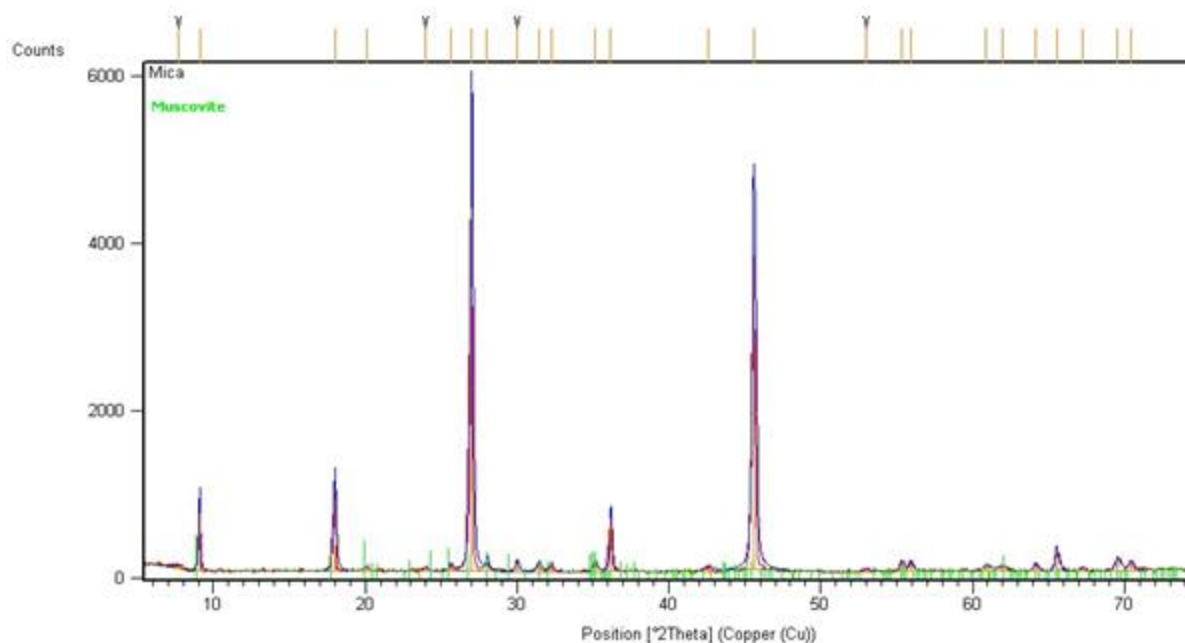


Figure 2: X-ray diffraction of crude Mica from Bukkuyum local government, Zamfara State, Nigeria.

Table 1: X-Ray Fluorescence (XRF) Elemental Analysis Result of crude Mica From Bukkuyum Local Government Area, of Zamfara State, Nigeria

OXIDES	PERCENTAGE PRESENCE
SiO ₂	48.7
Al ₂ O ₃	33.3
K ₂ O	15.2
Na ₂ O	0.67
CaO	0.01
MgO	0.04
TiO ₂	0.17
Fe ₂ O ₃	1.04
MnO	0.02
BaO	0.03
LOI	0.79

3.3 SEM Morphological Analysis

From the figure 3 and figure 4 it shows the surface, the fracture pattern of the sheet and range of fiber particle size respectively.

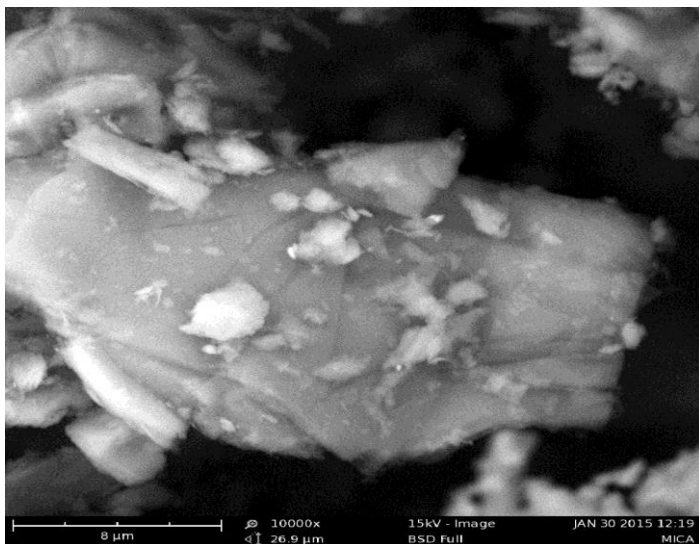


Figure 3: Scan Electron Photomicrograph showing the fracture surface of fine crude Mica that is representative of the Mica deposit in Bukkuyum local government area of Zamfara State, Nigeria.

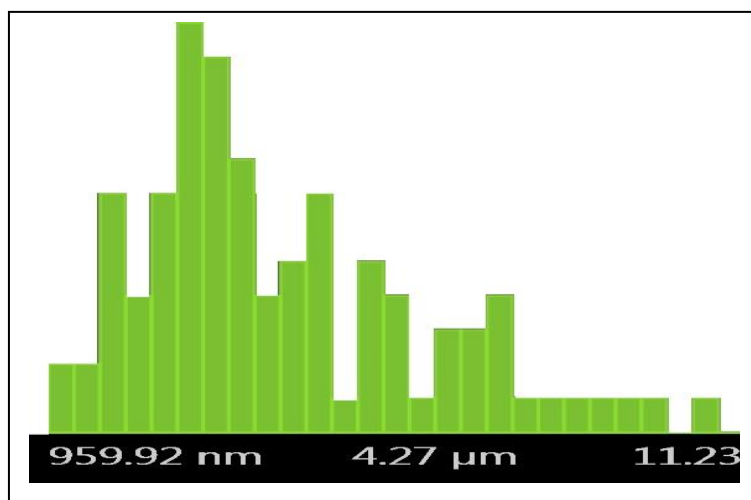


Figure 4: The fiber histogram showing the particle size distribution of the crude crushed Mica that is representative of the Mica deposit in Bukkuyum local government area of Zamfara State, Nigeria.

4.0 Conclusion

From the evaluation results and analysis, the following conclusions are deduced.

1. From XRD analysis confirms the muscovite phase
2. The XRF provided the elemental composition in their oxides SiO₂ of 48.7, Al₂O₃ of 33.3, k₂O of 15.2, Na₂O of 0.67, CaO of 0.01, MgO of 0.04, TiO₂ of 0.17, Fe₂O₃ of 1.04, MnO of 0.02, BaO of 0.03 and LOI of 0.79

3. The SEM shows the morphology along with statistics of fiber particle size of 959.92 nm, 4.27µm and 11.23µm
4. Finally, it can be concluded that the Mica from bukkuyum local government area of Zamfara state of Nigeria is a muscovite with chemical formula $K_{0.77}Al_{1.93}(Al_{0.5}Si_{3.5})O_{10}(OH)_2$ from the XRD result and contains impurities of Fe_2O_3 of 1.04%, and CaO of 0.01%, MgO of 0.16%, TiO_2 of 0.17% with LOI of 0.79% from the XRF result. The morphology results 959.92nm, 4.27µm and 11.23µm. This result can be improved on by numerous beneficiation processes depending on the area of industrial or engineering application.

Acknowledgements

Authors would like to thank (NGRL), Kaduna for their kind help of XRD and XRF analysis, and also Department of Chemical Engineering, ABU, Zaria for the SEM morphology analysis.

References

- Bailey, S.W 1984. Classification and Structures of the Micas. Pages 1-12 in reviews in mineralogy. Volume 13. Washington, DC: mineralogical society of American.
- Harben, P.W 2002, Mica, pages 220-227 in Industrial Minerals Handbook 4th edition, survey. UK; Industrial Mineral Information.
- Hendrick, J.B 2002, Mica. Pages 51.1-51.5 in Mineral Yearbook Washington DC, US, Geological Survey
- John W.S and James T. T Jr.2006 mica pages 637-652 in 7th Edition Industrial Minerals & Rocks. Commodities, Markets and Uses, edited by Jessica E.K, Nikhil C.T, James M.B and Stanley T.K Published by SMME Inc. USA.
- Sinkankas, J 1964. Phyllosilicates, pages 480-486 in Mineralogy for Amateurs, New York; Van Nostrand Reinhold.
- Skillen A. 1992, Mica, grounds for optimism. Industrial Minerals 302-25

