

Studies On the Characteristics of Some Imiegba Clays

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Abstract

The microstructural attributes and the elemental composition of two clays from imiegbe community in Edo state were studied using scanning Electron Microscopy (SEM) and Electron Dispersive system (EDS). The clay samples; Inare clay sample I, had the following elemental composition: carbon, oxygen, aluminum, silicon, potassium, titanium, and Iron. Inare clay sample II had the following elemental composition: carbon, oxygen, aluminum, silicon, and Iron. All elements being present at varying weight percent contributions. Inare clay I and II exhibit characteristics reminiscent of Illite clays, characterized by their 2:1 structural configuration and prevalent presence of silicon, aluminum and iron. These elements were captured graphically. The element profiles and weight composition were captured via the K shell of the atoms of the elemental constituents. Inare clay I had potassium and titanium which were absent in Inare clay sample II. A blend of the two clays could provide a mix that will expand the utility of the composites beyond that of the individual clays.

Introduction

Clay, an ancient and integral material in human history, holds significance ranging from prebiotic biomolecule synthesis to modern daily use. It serves as a vital component in ceramics like porcelain, bricks, and tiles, and finds utility in various products such as plastics, paints, paper, rubber, and cosmetics. Furthermore, clay's eco-friendly attributes render it suitable for depollution purposes. Nonetheless, defining "clay" remains intricate due to its varying attributes across disciplines. The joint nomenclature committees (JNCs) of the Association Internationale pour l'Etude des Argiles (AIPEA) and the Clay Minerals Society (CMS) proposed that 'clay' denotes a naturally occurring material predominantly comprising fine-grained minerals, typically demonstrating plasticity with water content and solidifying upon drying or firing. Specifically, clay features layered structures of fine-grained minerals, characterized by plasticity at suitable moisture levels and subsequent permanent solidification (Njoka et al., 2015).

Clay classification pertains to (1) a natural material with plastic properties, (2) particles smaller than two micrometers, and (3) fine mineral fragments primarily composed of hydrous-layer silicates of aluminum, occasionally containing magnesium and iron. The scope of clay minerals, in this context, mainly covers hydrous-layer silicates and closely related short-range ordered aluminosilicates occurring in very fine-grade forms. While clay mineral types include various minerals within the particle size range mentioned, this definition narrows down to hydrous-layer silicates. The term 'clay' is applied to materials resulting from weathering, hydrothermal processes,

or sediment deposition. It is also used to describe the finest fraction in sediment or soil classifications. Distinguishing between clay size and clay minerals is pivotal as identical-sized clay samples can exhibit diverse behaviors based on the prevalent clay minerals. Variations in clay minerals lead to distinct physical, chemical, and mechanical traits, dependent on mineral formation. For instance, montmorillonite swells significantly in the presence of water due to water uptake between its unit layers, while kaolins remain relatively unaffected.

Clay mineral formation in soil involves gradual processes influenced by the weathering environment, mainly dictated by climate and landscape drainage. These clay minerals constitute the fundamental building blocks of clay raw materials. Their crystal structure usually involves sheets arranged in structural layers, formed from tetrahedral and octahedral units. The arrangement creates a hexagonal network within each sheet. These structural elements combine to form various layers, such as 1:1 or 2:1 layer, leading to different crystalline clay mineral groups including Kaolinite, Smectite, Vermiculite, Illite, and Chlorites.

Kaolinite group which includes clay minerals like kaolinite, hallosite, nacrite and dickite, is a 1:1 type clay mineral. It is composed of one layer of silica and one layer of alumina, which is formed under acidic conditions through advanced weathering processes or hydrothermal changes of feldspars and other aluminosilicates (Miranda-Trevino & Coles, 2003) The chemical formula of kaolinite is $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ (39% Al_2O_3 , 46.5% SiO_2 and 14.0% H_2O) and its structure possesses strong binding forces between the layers which resists expansion when wetted (Trckova et al., 2004)

The cation exchange capacity (CEC) of kaolinite is less than that of montmorillonite due to its low surface area and low isomorphous substitution that result from its high molecular stability and this contributes to its low plasticity, cohesion, shrinkage and swelling. However, the material can adsorb small molecular substances such as lecithin, quinolone, paraquat, diaquat polyacrylonitrile, some proteins, bacteria and viruses. Industrial uses of Kaoline includes; manufacture of paper, paint, rubber, ceramic, plastic and pharmaceutical products, catalyst for petroleum cracking and auto exhaust emission catalyst control devices, cosmetics base and pigments (Olaremu, 2015)

Smectite, which includes montmorillonite, beidellite, nantronite, saponite and hectorite, are 2:1 layer clay mineral formed from the weathering of soils, rocks (mainly bentonite) or volcanic ash and belongs to a group of hydroxyl alumino-silicate. The variation of physical and chemical properties of bentonites within and between deposits is caused by differences in the degree of chemical substitution within the smectite structure, the nature of the exchangeable cations present, type and the amount of impurities present (Erdoğan, 2015). Minerals associated with smectites include quartz, cristobalite, feldspars, zeolites, calcite, volcanic glass and other clay minerals such as kaolinite. The groups of smectite clays are distinguished by differences in the chemical composition pertaining substitutions of Al_3^+ or Fe_3^+ for Si_4^+ in the tetrahedral cation sites and Fe_2^+ , Mg_2^+ or Mn_2^+ for Al_2^+ in the octahedral cation sites. Smectites have very thin layers and small particle sizes which contributes to high surface area and hence a high degree of absorbency of many materials such as oil, water and other chemicals (Abdou et al., 2013). Additionally, smectites have higher cation exchange capacities, swelling and shrinkage properties than other clays. The variable net negative charge on smectites structural layers attracts water molecules into the

interlayer area thus causing expansion, and the amount of swelling is related to the type of interlayer cation present. For example, the sodium rich smectite clays expand more than those containing calcium ions. Smectites are valuable minerals for industrial applications due to their high cation exchange capacities, high surface area, surface reactivity, adsorptive capacity and catalytic activity (Akpomie, 2017). This group of clays has found applications in bonding foundry sands, drilling fluids, iron ore pelletizing, agriculture (as carrier material for pesticides, fertilizers and for coating seeds), paper making, paints, pharmaceuticals, cosmetics, plastics, adhesives, decolorization and ceramics (Miranda-Trevino & Coles, 2003). The material is also used as clarifying agents for oils and fats, chemical barriers, liquid barriers and catalysts (Abubakar, 2014)

Vermiculite is a hydrated magnesium aluminium-iron silicate which possesses 2:1 type of clay minerals (Tang, 2012). It has a layer charge of 0.9-0.6 per formula unit, and contains hydrated exchangeable cations primarily Ca, and Mg in the interlayer (Schulze, 2005). The high charge per formula unit gives vermiculite a high cation exchange capacity and causes this clay type to have a high affinity of weakly hydrated cations such as K^+ , NH_4^+ and Cs^+ . Upon rapid heating at a temperature of 900°C or higher, the water in raw flakes vermiculite flashes into steam and the flakes expand into accordion-like particles, a phenomenon known as exfoliation (Marcos, 2020). The expanded or exfoliated material is low in density, chemically inert and adsorbent has excellent thermal and acoustic insulation properties, is fire resistant and odourless. The common applications of exfoliated vermiculite include making of friction light weight aggregates, thermal insulator, brake linings, various construction products, animal feeds and in horticulture (Heckroodtt, 1991)

Incorporation of vermiculite in fertilizers makes them more efficient in releasing nutrients and hence making the fertilizers more economical to the consumers. The expansion of vermiculite on heating generates sufficient internal pressure which can be utilized to break hard rock during tunneling work. Vermiculite ores contain variable amounts of other minerals such as feldspars, pyroxenes, amphiboles, carbonates and quartz that are formed along with vermiculite in the rock and occur as major components, as well as minor components such as phosphates, iron oxides, titanium oxides and zircon (Marcos et al., 2020).

Some impurities like asbestiform amphibole minerals found in vermiculite have toxicity impact on human health as they lead to development of diseases such as malignant mesothelioma, asbestosis or lung cancer; hence, characterization of clays is important in order to identify such impurities.

Illite clay mineral group is also called clay micas. Mica is a group of phyllosilicate minerals with crystalline structure that can be split or delaminated into thin sheets that are platy, flexible, clean, elastic, transparent to opaque, resilient, reflective, refractive, dielectric, chemically inert, insulating, light weight and hydrophilic (Tang et al., 2012). Studies have shown that there are over 30 members of mica group, but six forms that are found in nature and commonly used in microscopy and other analytical applications consist of muscovite, biotite, phlogopite, lepidolite, fuchsite and zinnwaldite. Three members (illite group) which includes illite, glauconite and muscovite are referred to as clay minerals because they exhibit characteristic properties of clay, with illite mineral being the most common. Illite is formed from weathering of potassium and

aluminum rich rocks like muscovite and feldspar under alkaline conditions. Illite group is a 2:1 layer silicate clay mineral which is non expansive because the space between the crystals of individual clay particles is filled by poorly hydrated potassium cations or calcium and magnesium ions which hinder water molecules from entering the clay structure. The cation exchange capacity of Illite ranges between 20-40meq per 100g. (Gray M et al., 2013.) The colour of the minerals ranges from grey white, silvery white to greenish grey. The illites find application in structural clay industry and in agro minerals due to high potassium content (Njoka et al., 2015; Van, 2002). Mica clay ores contain a variety of impurities which includes quartz, feldspar, kaolin and pyroxene. Presence of these minerals in mica ores will impact upon the industrial value of these deposits and the processing complexity thus reducing or increasing its value depending on the applications. (Tang et al., 2012)

Chlorites are hydrous aluminosilicates that are arranged in a 2:1 structure with an interlayer (Wiewiora, 1990). They incorporate primarily Mg, Al and Fe cations and to a less extent Cr, Ni, Mn, V, Cu and Li cations in the octahedral sheet within the 2:1 layer and in the interlayer hydroxide sheet. They also exhibit a large substitution of Si by Al cations in the tetrahedral sheet (Iannucelli et al., 2016). The colour of chlorites varies from white to almost black or brown with a tint of green where these optical properties are coupled to the chemical composition of chlorite. Knowledge of the chemical composition of chlorite is important in the study of phase relationships in low and middle grade metamorphic rock (Rakhila et al., 2018).

Considering the diversity of clay mineral groups in nature, the initial mineralogical and chemical examination of clay ores can be used to indicate the suitability of the material for different applications. The aim of this study is to characterize two clays (Inare I and Inare II) from Imiegba community, Edo state using Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). Results from this study will open ways for further research on the beneficiation of the clays.

MATERIALS AND METHODS

Study area

Imiegba is situated in Etsako East Local Government Area of Edo State, Nigeria, its geographical coordinates are 7° 12' 0" North, 6° 27' 0" East and its original name (with diacritics) is Imiegba.

Sampling Methods

The procedure for collecting and preparing clay samples was executed following the protocol outlined in the study by Njoka et al. (2015). The acquisition of samples was done using sampling cups.

Instruments used and procedures

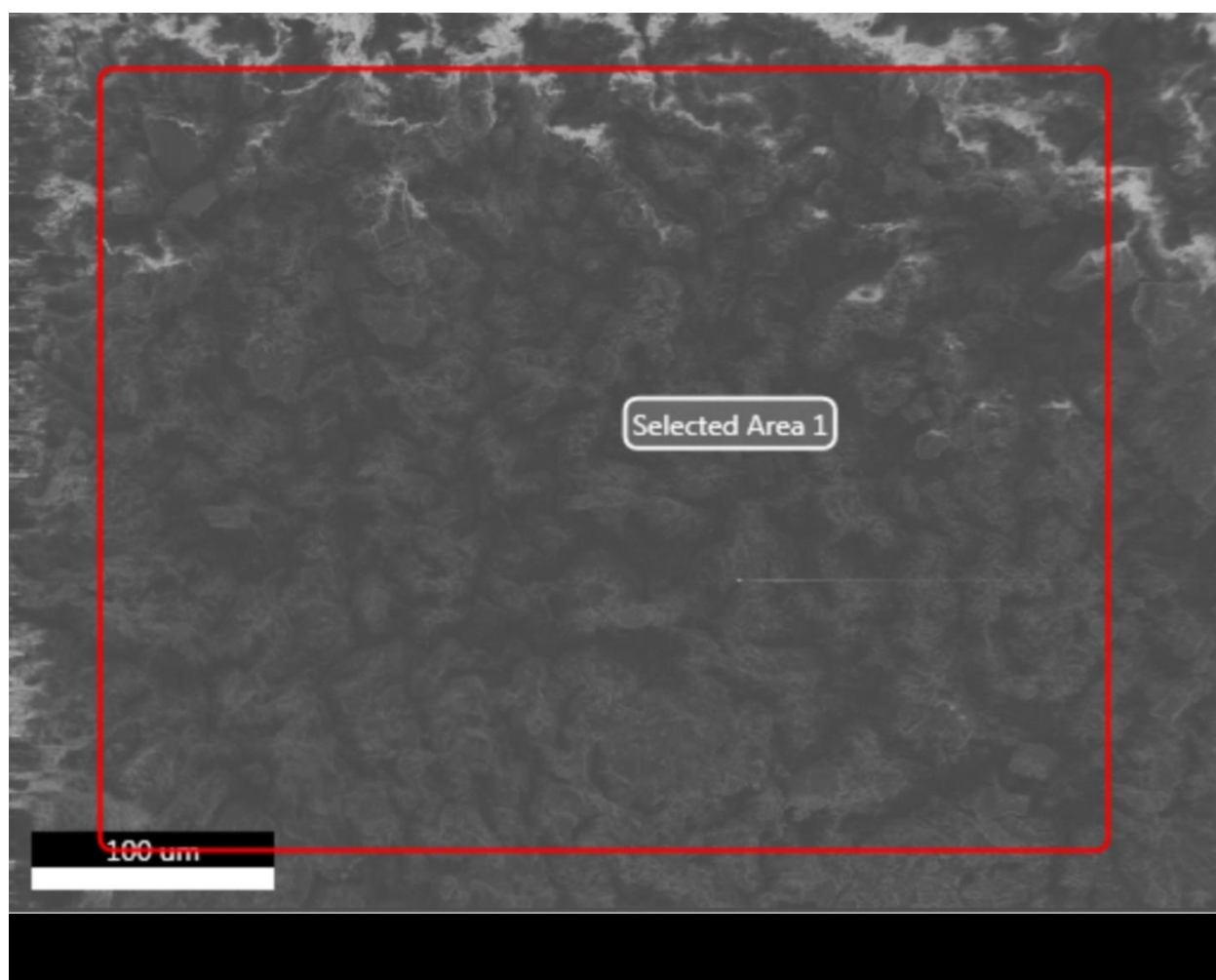
For the analysis, the Scanning Electron Microscope (EVO|LS10 MODEL) and the Electron Dispersive System (EVO|LS10 MODEL) instruments were utilized as outlined below:

The individual samples, namely Inare I and Inare II clay, were weighed, and subsequently, 0.1g of each sample was separately placed onto distinct sample stubs using aluminum tape for fixation.

These sample stubs were then introduced into the SEM. The instrument was initiated to scan the samples for a duration of 90 seconds. During this process, an electron beam was emitted, which interacted with the samples, causing electron scattering. A concave lens within the sample chamber focused and consolidated the scattered electrons. This interaction was then detected by a series of detectors, which generated images of the samples, along with providing information about the elemental composition and spectrum percentages.

RESULTS AND DISCUSSION

Inare I



Electron Scan Micrograph of Inare I

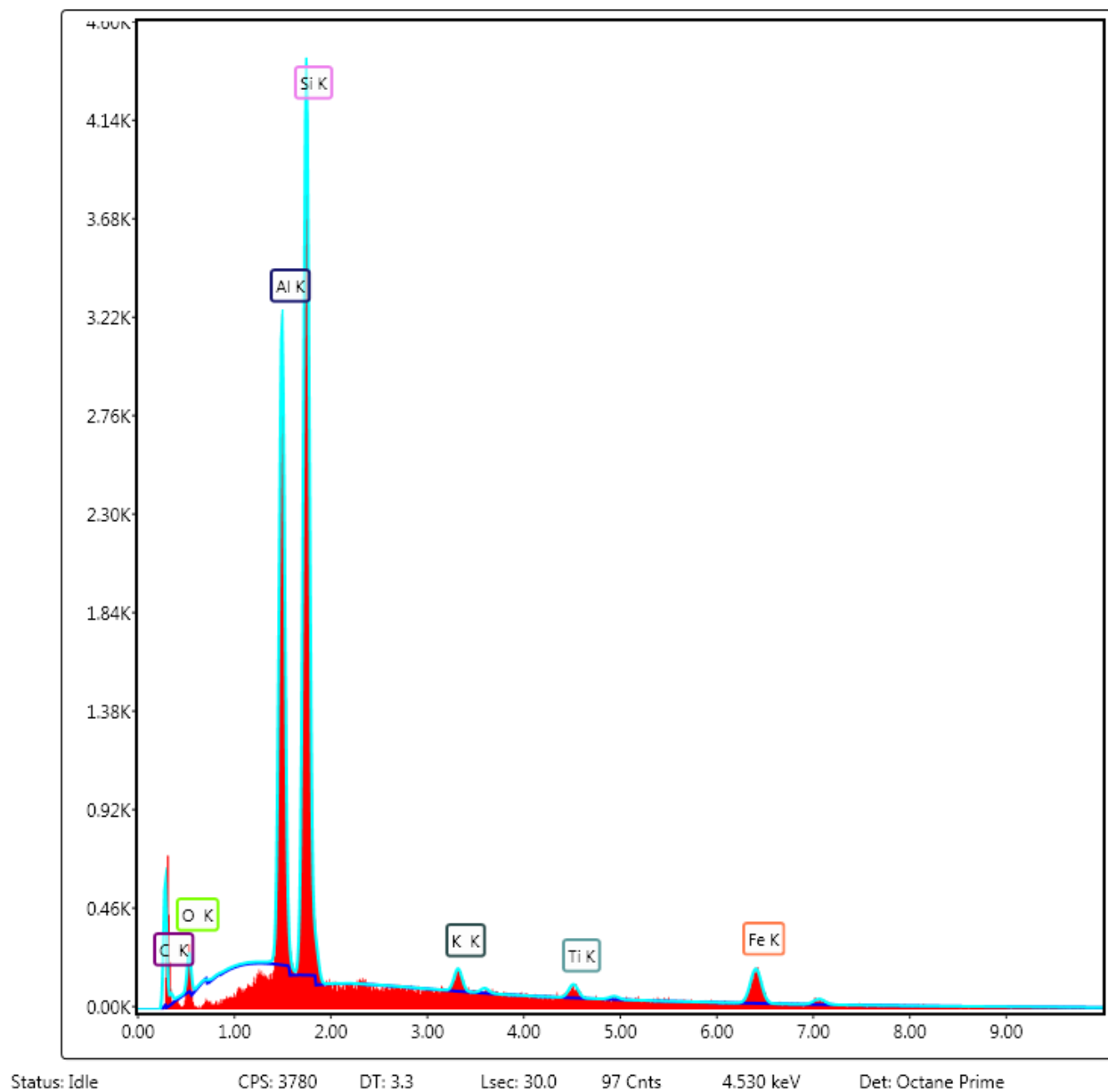


Table 1

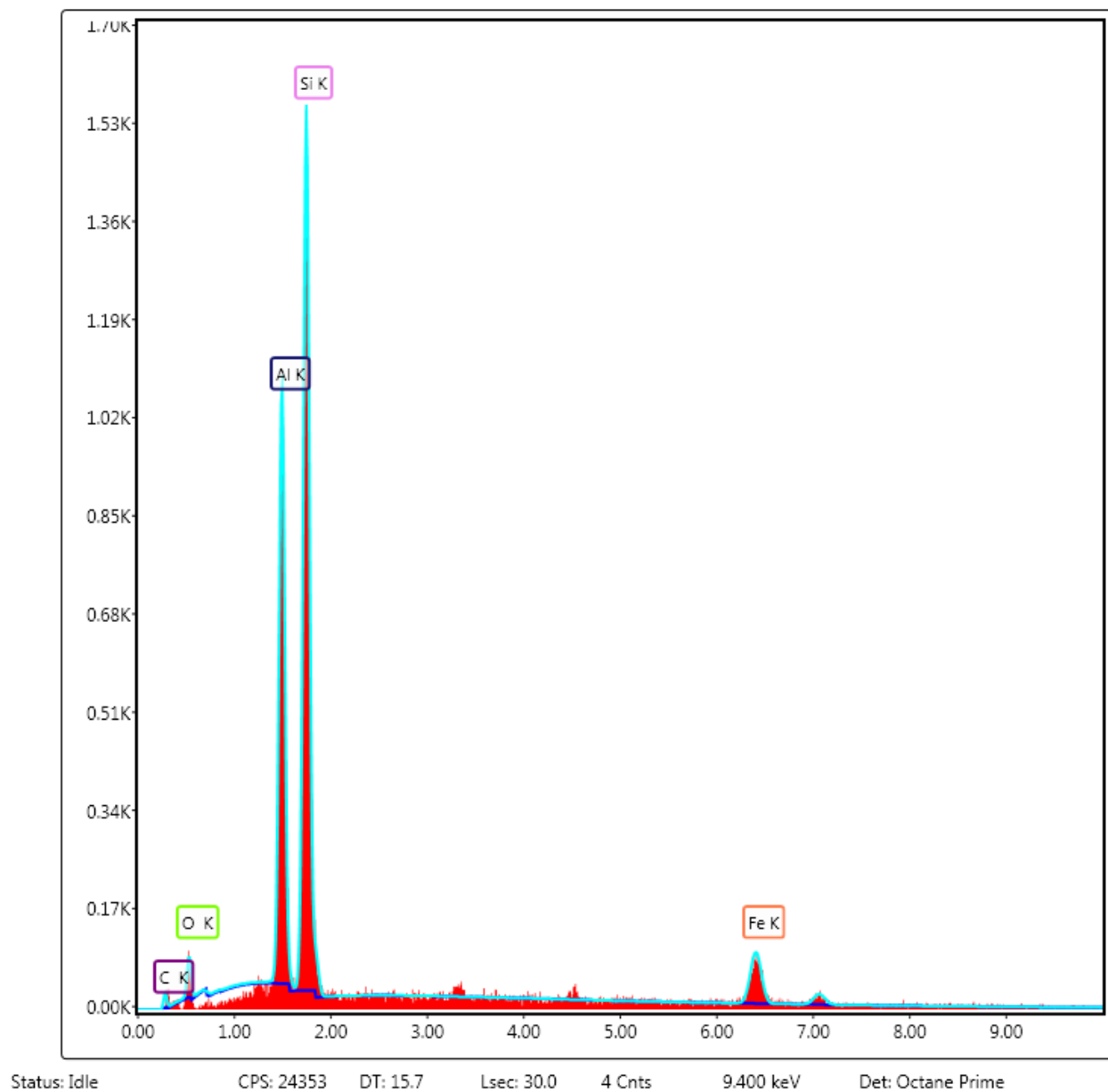
Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	R	A	F
C K	38.02	59.8	12.27	82.13	0.0652	1.109	0.9492	0.1546	1
O K	4.23	4.99	15.44	29.75	0.0098	1.0571	0.9708	0.2182	1
Al K	16.7	11.69	3.91	720.17	0.1327	0.934	1.0107	0.84	1.0126
Si K	27.89	18.76	4.06	1077.16	0.211	0.9534	1.0171	0.7906	1.0036
K K	1.52	0.73	18.82	35.66	0.0128	0.8794	1.043	0.9371	1.0257
Ti K	1.55	0.61	22.25	24.99	0.0132	0.8087	1.0534	0.9839	1.0648
Fe K	10.09	3.41	8.54	74.88	0.0846	0.7892	1.0577	1.002	1.0595

From the data presented in the above table, it is evident that the composition of Inare I includes carbon, oxygen, aluminum, silicon, potassium, titanium, and iron. Carbon is observed to have the highest weight percentage. This significant weight percentage can be attributed to the organic matter present in the Inare I sample, which likely originates from the remnants of plants or animals. The table also illustrates the weight percentages of silicon, oxygen, and aluminum. According to sources like Stephen A. Nelson (2014), in the context of trioctahedral smectite, each oxygen (O) and hydroxide (OH) ion is encompassed by three divalent ions like Fe^{2+} and Mg^{2+} , a characteristic that aligns with the presence of such ions in Inare I, as indicated in the table above. Consequently, the notable levels of carbon and silicon, coupled with the presence of oxygen, aluminum, and iron, suggest that the clay sample may belong to either the kaolinite or smectite clay mineral classification.

Inare II



Electron Scan Micrograph of Inare II



Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	R	A	F
C K	8.6	19.15	29.21	3.34	0.0108	1.1803	0.9195	0.1064	1
O K	4.33	7.24	16.71	11.45	0.0153	1.1269	0.9427	0.3133	1
Al K	23.43	23.23	5.1	247.47	0.1858	0.9979	0.9865	0.7844	1.0128
Si K	42.02	40.02	5.26	385	0.3072	1.019	0.9937	0.7153	1.0032
Fe K	21.62	10.36	8.9	41.45	0.1908	0.8468	1.0451	0.9966	1.0457

Table 2

The data presented in the table above reveals the elemental composition of Inare II, encompassing carbon, oxygen, aluminum, silicon, and iron. Analyzing Figure 2, it becomes evident that silicon exhibits the highest peak, trailed by aluminum and iron, succeeded by carbon and oxygen.

Illite is characterized by the chemical formula $(K, H_3O)(Al, Mg, Fe)_2(Si, Al)_4O_{10}\{(OH)_2(H_2O)\}$. The presence of silicon, aluminum, iron, and potassium in Table 2 aligns with the attributes of an Illite microstructure, known for its 2:1 arrangement of silica tetrahedrons, alumina octahedrons, and silica tetrahedrons.

Consequently, the substantial weight percentages of silicon, aluminum, and iron strongly suggest that Inare II belongs to the Illite clay group, characterized by a 2:1 structural configuration. This microstructural makeup supports the classification of Inare II within the Illite clay category.

CONCLUSION

The analysis of Imiegba clay samples, Inare I and Inare II, provides insights into their elemental composition and microstructural attributes. The presence of carbon in Inare I suggests organic matter influence, while significant levels of silicon, aluminum, oxygen, and iron in both samples hint at possible affiliations with kaolinite or smectite clay mineral classification. Notably, Inare II's dominance of silicon, aluminum, and iron supports its classification as an Illite clay, known for its 2:1 structural configuration. This study enhances understanding of these clays' characteristics and their potential applications.

References

- Abdou, M. I., Al-sabagh, A. M., & Dardir, M. M. (2013). Evaluation of Egyptian bentonite and nano-bentonite as drilling mud. *Egyptian Journal of Petroleum*, 22(1), 53–59.
<https://doi.org/10.1016/j.ejpe.2012.07.002>
- Akpomie, K. (2017). *Industrial Potentials of Adiabo clay in Calabar municipal of Cross River State South South Nigeria*. <https://www.researchgate.net/publication/308680627>
- Erdoğan, Y. (2015). Physicochemical Properties of Handere Clays and Their Use as a Building Material. *Journal of Chemistry*, 2015, 1–6. <https://doi.org/10.1155/2015/374245>
- Gray M, Neerdael B, & Degnan P (2013). (n.d.). *Characterization of Swelling Clays as Components of the Engineered Barrier System for Geological Repositories Results of an IAEA Coordinated Research Project 2002-2007*.
- Grim, R. E., & Kodama, . Hideomi. (2022). Clay Mineral. In *Britannica*.
<https://www.britannica.com/science/clay-mineral>
- Heckroodt, R. O. (1991). Clay and clay materials in South Africa*. In *J. S. At., Inst. Min. Metal* (Vol. 91, Issue 10).
- Iannuccelli, V., Maretti, E., Sacchetti, F., Romagnoli, M., Bellini, A., Truzzi, E., Miselli, P., & Leo, E. (2016). Characterization of Natural Clays from Italian Deposits with Focus on Elemental Composition and Exchange Estimated by EDX Analysis: Potential Pharmaceutical and Cosmetic Uses. *Clays and Clay Minerals*, 64(6), 719–731.
<https://doi.org/10.1346/CCMN.2016.064038>
- Marcos, C., Adawy, A., & Rodríguez, I. (2020). Relationship between Textural Parameters of Lamellar Products Obtained by Acid Activation of Pure and Commercial Vermiculites and Their Iron and Water Content. *Minerals*, 10(8), 661. <https://doi.org/10.3390/min10080661>
- Miranda-Trevino, J. C., & Coles, C. A. (2003). Kaolinite properties, structure and influence of metal retention on pH. *Applied Clay Science*, 23(1–4), 133–139.
[https://doi.org/10.1016/S0169-1317\(03\)00095-4](https://doi.org/10.1016/S0169-1317(03)00095-4)
- Murray, H. H. (1991). Overview — clay mineral applications. *Applied Clay Science*, 5(5–6), 379–395. [https://doi.org/10.1016/0169-1317\(91\)90014-Z](https://doi.org/10.1016/0169-1317(91)90014-Z)
- Njoka, E. N., Ombaka, O., Gichumbi, J. M., Kibaara, D. I., & Nderi, O. M. (2015). Characterization of clays from Tharaka-Nithi County in Kenya for industrial and agricultural applications. *African Journal of Environmental Science and Technology*, 9(3), 228–243. <https://doi.org/10.5897/AJEST2014.1816>
- Olaremu, A. G. (2015). Physico-Chemical Characterization of Akoko Mined Kaolin Clay. *Journal of Minerals and Materials Characterization and Engineering*, 03(05), 353–361.
<https://doi.org/10.4236/jmmce.2015.35038>

- Rakhila, Y., Ezzahi, A., Elmchaouri, A., & Mestari, A. (2018). Synthesis and Characterization of a Red Clay Based New Composite Ceramic Material. *Advances in Materials Physics and Chemistry*, 08(07), 295–310. <https://doi.org/10.4236/ampc.2018.87020>
- Tang, Q., Wang, F., Tang, M., Liang, J., & Ren, C. (2012). Study on Pore Distribution and Formation Rule of Sepiolite Mineral Nanomaterials. *Journal of Nanomaterials*, 2012, 1–6. <https://doi.org/10.1155/2012/382603>
- Trckova, M., Matlova, L., Dvorska, L., & Pavlik, I. (2004). Kaolin, bentonite, and zeolites as feed supplements for animals: health advantages and risks. *Veterinárni Medicína*, 49(10), 389–399. <https://doi.org/10.17221/5728-VETMED>