



Chemical and Mineralogical Characterization of Isanlu-Isin Columbite for Effective Beneficiation Towards Niobium Pentoxide Recovery

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Abstract: The characterization of Isanlu-Isin columbite was conducted to obtain data suitable for its beneficiation to metallurgical grade. Samples from various locations within the mining site underwent homogenization, crushing, and grinding for characterization. Chemical analysis was performed using Energy Dispersive X-ray Fluorescence Spectroscopy (ED-XRF), while mineralogical analysis employed a Scanning Electron Microscope with Energy Dispersive Spectroscopy (SEM-EDS), X-ray Diffraction (XRD), and petrological examination. ED-XRF results revealed that the crude samples contain 10.070% Nb₂O₅, along with trace amounts of other minerals (gangue). Mineralogical characterization confirmed the presence of columbite, quartz, and tantalite. SEM imagery showed a uniformly distributed coarse grain structure, while EDS analysis identified Mn, Nb, Si, and Fe in their oxide forms. Silicon and niobium were found to be the predominant elements in the matrix. This characterization confirms that the ore contains niobium pentoxide, the primary mineral of interest. The data obtained serves as a foundation for effective pre-processing strategies to recover niobium pentoxide in the extractive metallurgy industry.

Keywords: Niobium Pentoxide, Columbite, Characterization, Liberation size, Homogenization

1. Introduction

Columbite otherwise known as niobium ore, contain niobium pentoxide used when smelted to produce niobium (Nb) metal. Niobium (Nb) uses have led to significant technological and industrial advancements across various industries such as steel, metallurgy, nuclear energy, electronics, aviation, and communication [1, 2]. Niobium is consequently a critical metal with substantial strategic value and considerable demand [3]. Niobium is a crucial alloying element for steel, and Nigeria has the world's largest reserves of solid minerals, including coal, iron ore, quartz, zinc, uranium, lead, feldspar, limestone, cassiterite, columbite, titanite, rutile, and uranium [4, 5]. Columbite deposits in Nigeria are found in various regions, including the Plateau, Kwara, Kaduna, Bauchi, Kano, Kebbi, Gombe, Abuja, Nassarawa, and River States [6].

1.1 Niobium Pentoxide (Nb₂O₅)

Niobium pentoxide (Nb₂O₅) is a colorless, insoluble, and non-reactive precursor to niobium-based materials, including alloys, optical glasses, capacitors,

and steel for corrosion resistance, and is used in various industries [7-10]. Niobium, when added to steel in small amounts, significantly enhances its strength, mechanical properties, and corrosion resistance, making it useful in various industries like construction and heavy machinery [11]. Columbite, a superconductive niobium mineral, is utilized in industrial processes to produce valuable metals, weldable steel, and tantalite capacitors, increasing alloy strength globally [12]. High manufacturing and processing costs, coupled with unique properties and applications, have led to a high demand for industrial metals, resulting in increased costs [13, 8]. Niobium pentoxide, a highly studied ores for the production of niobium metal, is crucial for its diverse applications, including manufacturing consumer goods like super alloys and hearing aids. Niobium and tantalum are in high demand due to their role in modern industrial materials and high-tech consumer goods, making columbite a promising mineral for effective exploitation [8]. It is the source of the metals niobium and tantalum, which are crucial strategic elements used in high-tech applications, such as the primary strategic materials used in the aerospace sector [14].



1.2 Characterization of the Mineral

Characterization is essential for both primary and secondary minerals [8]. Giving an explanation of the material's composition and physicochemical structure [15, 16]. The analysis of an ore characteristics, processing, and applications requires a thorough understanding of its basic properties, compositions, reactions, and techniques during extraction, processing, and beneficiation. [16-18]. Understanding the chemical and mineralogical characteristics of an ore is crucial for determining its structure, processing techniques, and attributes, as it explains its composition and impacts the economics of commercial mining activities [16, 18]. Before further processes for the beneficiation of an ore, comminution is an essential step in the mineral processing, which involves the crushing and grinding [19-20]. However, the process of reducing the size of a solid minerals begins with crushing, which reduces the ore particles from runoff mines to a size fraction. Grinding is then carried out until the mineral is liberated, enabling the separation of mineral of interest from the impurities. [20, 21].

Some researchers have reported on the mineralogical and chemical composition of various columbite mine site, exception the Isanlu-Isin deposit, which has not been investigated nor reported on. In addition, there is an increase in demand for niobium metal due to Nigeria's increased demand and consumption of steel. The chemical and mineralogical characterization of ores varies from deposit to deposit, and it is essential to understand the specific chemical and mineralogical composition of any deposit for effective processing methods [7] reported on the Gyel columbite deposit, and [16] reported on the Ray-field deposits in Jos. In order to determine the elemental composition, mineral phases present in the ore matrix, and the ore morphological conditions, respectively, this study focused on the characterization Isanlu-Isin columbite using energy dispersive x-ray fluorescence spectrometer (ED-XRFS), a petrological analyzer, an X-ray diffractometer (XRD), and a scanning electron microscope (SEM) with an energy dispersive spectrometer (EDX). The information produced will aid in providing sufficient understanding of the ore exploitation as a new resource of heavy minerals in Nigeria. Additionally, the beneficiation of niobium pentoxide mineral from columbite can be significantly influenced by liberation characteristics and associations, grain size distribution, constituents, and phase composition, among other factors. Consequently, understanding the chemical and

mineralogical compositions of ores helps improve their recovery rates during the value-adding process [5].

2. Research Method

2.1 Research Materials

The materials used for this research work include about seven kilogram (1.0 kg) of crude sample of Isanlu-Isin columbite.

2.2 Sample collection

Isanlu-Isin columbite samples were sourced from different pits on the mine site. In the mine field with point coordinate 8° 14' 12"N, 7° 16' 04"E, which covered an area of roughly 50 hectares, a true representative sample of columbite was randomly selected from various deposits. The soil was deposited in an alluvial form through alluvial veins influenced by flowing water, and the soil depth was between 5-to-6-meter depths the area cut (1.5 × 1.5 meters).

2.3 Sample preparation

A sample of one kilogram (1.0 kg) of crude was taken from the seven kilograms (7.0 kg) of received crude and homogenized using the cone and quartering sampling method, followed by the random sampling method. This allowed for the selection of the minimum representatives for characterization.

2.4 Chemical Characterization of the Isanlu-Isin Columbite

A Xenometric Genius 'IF' model energy dispersive X-ray fluorescence spectrometer was used to chemically analyze the crude material. The material was ground up to fit through a 200–250 mesh screen using 20.0 g at a pressure of 10–15 tons/inch², mixed with a cellulose flakes binder in a 5:1 ratio (5.0 g sample(s) to 1.0 g cellulose flakes binder), and then pelletized in a pelletizing equipment. The pelletizing sample or samples for examination were stored in a desiccator. Two hours were spent allowing the EDXRF apparatus to warm up once it was turned on. Lastly, suitable programming of different elements of interest were used to examine the crude sample or samples in order to determine if they were present or absent. A percentage (%) or parts per million (ppm) were used to report the analysis's findings for minor and major elements [7].



2.5 Mineralogical Characterization of Crude Columbite

The crude columbite sample of Isanlu-isin was mineralogically characterized using an X-ray diffractometer (XRD), a scanning electron microscope with energy dispersive spectrometer (SEM-EDS), and a petrological analyzer.

2.5.1 X-ray Diffractometer (XRD) Analysis of the Isanlu-isin Columbite Sample

The Rigaku Miniflex model diffractometer, variable divergence, and receiving slits with Fe-filtered Co-K α radiation were used to analyze the material that was ready for XRD examination. X'Pert Highscore Plus software was used to identify the stages. 0.040° was the position of the receiving slit. Using a 2 θ scale, the counting range was 5 to 70°. It took 1.5 seconds to count. An Anton Paar HTK 16 heating chamber with a Pt heating strip was used to acquire the temperature-scanned XRD data. The outcome included graphic representations of the qualitative data. The Rietveld method was used to estimate the relative phase amounts (weight percentage) [18].

2.5.2 Scanning Electron Microscope with Energy Dispersion Spectrum Analysis (SEM-EDS) of the Isanlu-isin columbite sample

Using the Phenom Pro 'X' model, morphological and qualitative analyses of the bulk ore were carried out. While the EDS offered information on the mineral's chemistry, the SEM offered information on its physical characteristics. Research on minerals using a scanning electron microscope SEM model JEOP 840 was used in two stages of analysis of representative samples. After

being sliced and mounted in embedded epoxy resin, the samples for analysis were polished to a mirror-like surface. To make the mineral's surface suitable for investigation, the polished surface was ultimately carbon coated. A qualitative chemical analysis of the material was performed using an EDS detector that was connected to the SEM [5].

2.5.3 Petrological Analysis of the Isanlu-isin Columbite Sample

From the Isanlu-isin columbite sample's alluvial, a chip of roughly 1/8 of an inch by 1 inch was cut. Its surfaces were then trimmed, and the chip was placed on a grinding machine to smooth up its surface. To identify the various minerals and ascertain the microstructure of the ore, the samples were placed on a slide and examined with a Leica Petrological Microscope at an adequate magnification and image display. For the characterisation study, the remaining samples were appropriately combined to achieve homogenization using the cone and quartering sampling technique.

3. Results

Result of the characterization of Isanlu-Isin columbite are presented thus;

The pie chart in Figure 3 is the result obtained from x-ray diffractometry analysis of Isanlu-isin columbite showing a summary distribution of the minerals in the ore matrix in their different phase and composition. The pie chart shows quartz at 63% wt. columbite at 16% wt. muscovite at 21% wt. and tantalite at 0.1% wt.

Table 1. Chemical Composition of Isanlu-isin Columbite using Energy Dispersive X-Ray Spectrometer (ED-XRFS)

Comp.	SiO ₂	Fe ₂ O ₃	NiO	Nb ₂ O ₅	WO ₃	CaO	Al ₂ O ₃	Ta ₂ O ₅	ZrO ₂	SnO ₂	ThO ₂
Conc. (%)	57.381	5.732	0.017	10.070	0.090	0.277	8.044	1.257	8.470	0.872	BDL

Table 2. Chemical Composition of Igbara Oke Riverside Silica using Energy Dispersive X-Ray Spectrometer (ED-XRFS)

Component	SiO ₂	Fe ₂ O ₃	NiO	Nb ₂ O ₅	WO ₃	CaO	Al ₂ O ₃	Ta ₂ O ₅	ZrO ₂	SnO ₂
Conc. (%)	84.655	1.501	0.003	0.013	0.003	0.752	5.790	0.053	0.087	0.000

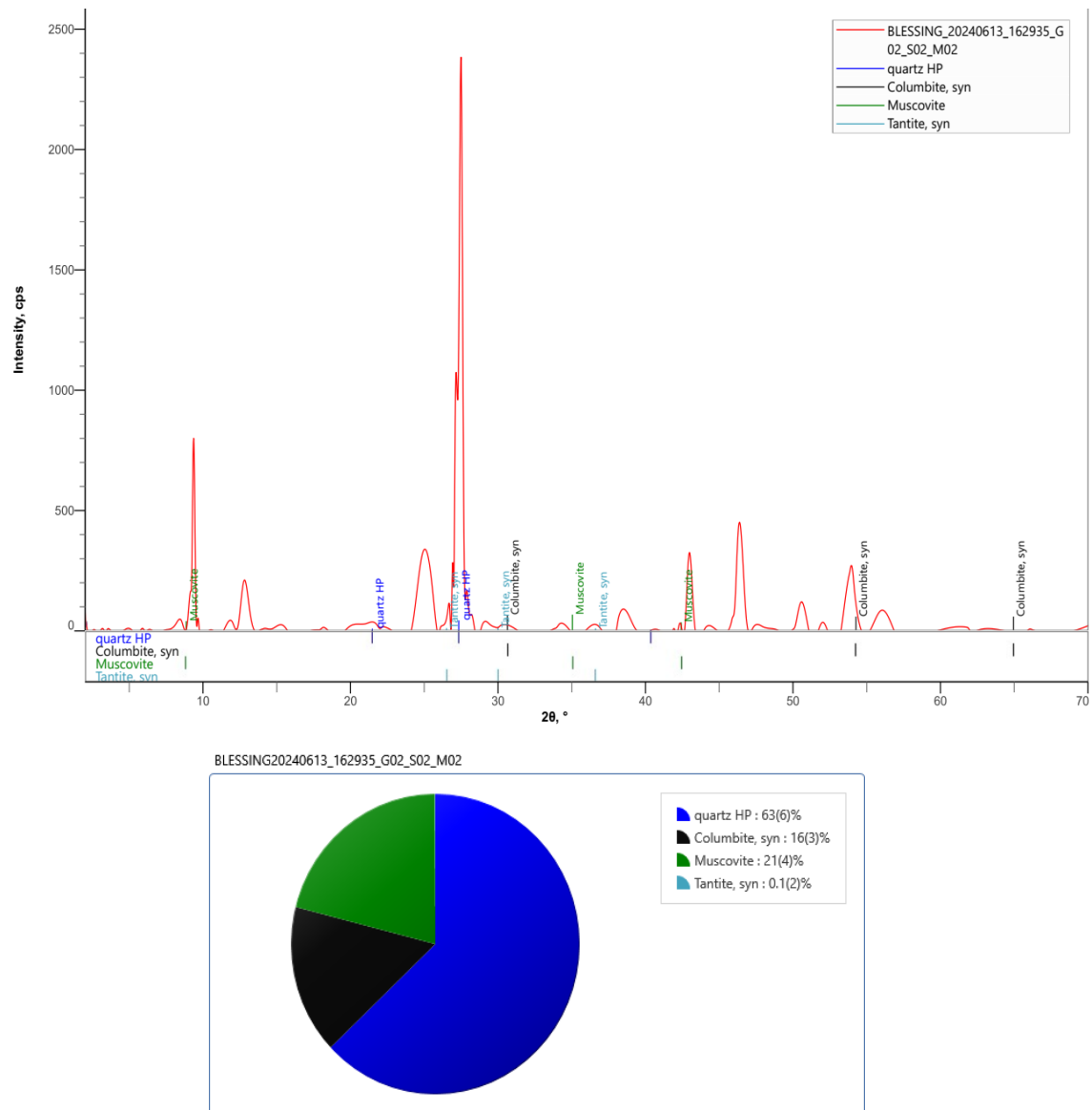


Figure 1. Quantitative X-ray diffractometry analysis of the Columbite ore

Table 3. EDS Result of Isanlu-isin Columbite

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	57.95	59.39
26	Fe	Iron	8.25	4.69
13	Al	Aluminum	10.87	14.09
39	Zr	Zirconium	4.52	3.49
19	K	Potassium	3.24	2.26
20	Ca	Calcium	0.87	0.13
16	S	Sulfur	2.00	1.65
41	Nb	Niobium	8.62	7.48
12	Mg	Magnesium	1.70	1.07
22	Ti	Titanium	0.44	0.54
15	P	Phosphorus	0.56	0.44
25	Mn	Manganese	0.27	0.39
11	Na	Sodium	0.62	0.37
24	Cr	Chromium	0.25	0.34
23	V	Vanadium	0.21	0.27

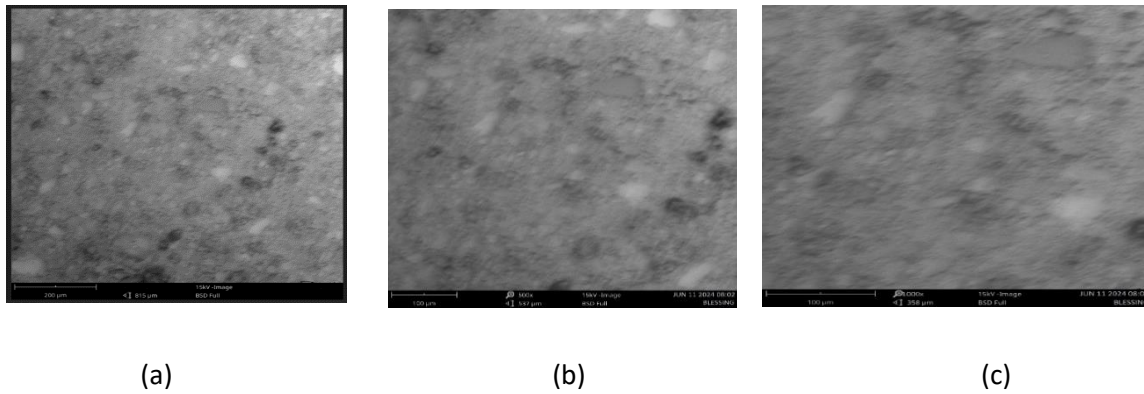


Figure 2(a,b,c). The Micrograph of Scanning Electron Microscope of Isanlu-Isin Columbite at 1, 500, and 1000 magnifications.

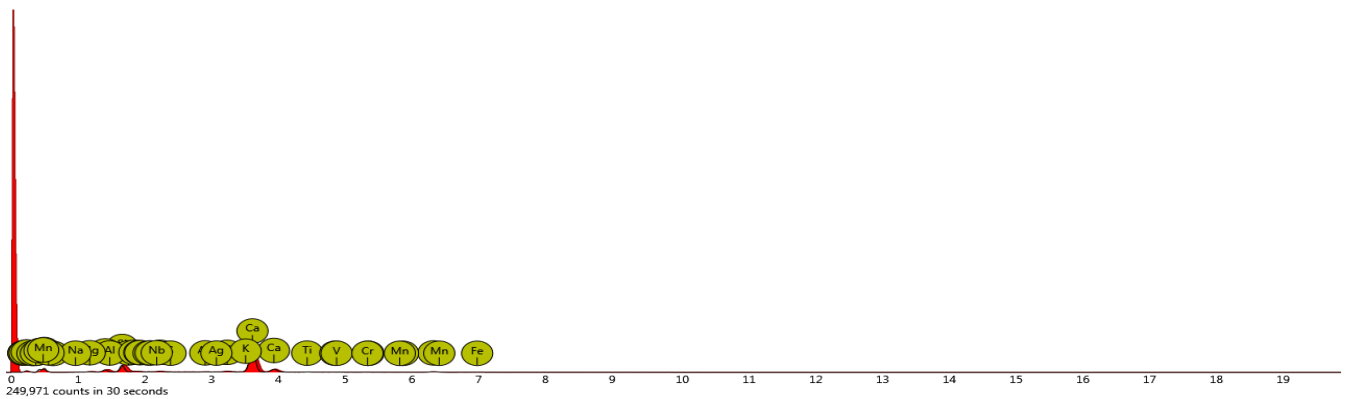


Figure 3. Scanning Electron Microscope (SEM) of Columbite samples of Isanlu-Isin at 1, 500, and 1000 magnifications, and the Ore elemental composition at 815, 537 and 358 um

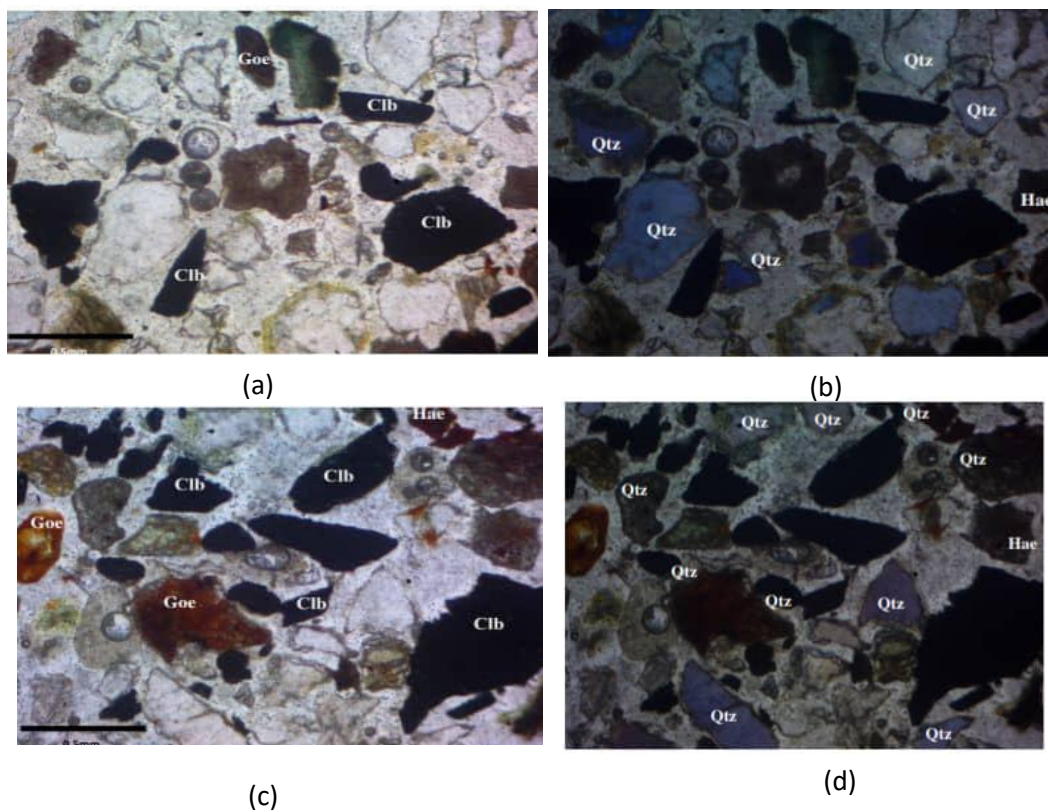


Figure 5. Micrograph image of Isanlu-Isin columbite using petrological microscope

This shows that the most predominant phase in the matrix of the ore is quartz and shows the highest peak, also having the relative figure of merit as follows; quartz (SiO_2 , 3.446), Columbite (FeNb_2O_6 , 3.138), Muscovite [$\text{H}_2\text{KAl}_3(\text{SiO}_3)_3$, 3.480], Tantalite (Ta_2O_5 , 3.315). However, the characterization affirms that quartz is the major gangue which needs to be separated from the ore. Hence, need for processing of the columbite to appraise the niobium oxide present in the ore for use in metallurgical application.

3.1 Results of the mineralogical characterization analysis by scanning electron microscope equipped with energy dispersive spectrum

The results of the mineralogical characterization analysis by scanning electron microscope equipped with energy dispersive spectrum is presented in Table 1, Figure 2 (a,b,c) and the spectrum in Figure 3

Key Words:

Qtz = quartz, Hae = haematite, Goe = goethite; Clb = Columbite (12% to 17%)

4. Discussion

The chemical characterization results are presented in Table 1, and it shows that the crude columbite; contains the presence of niobium pentoxide (Nb_2O_5) at an assay of 10.070% in the crude sample, with other mineral constituents in the crude sample, and their composition.

Table 1 shows the chemical composition of Isanlu-Isin columbite using Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRFS). The analysis revealed that the crude contains 10.070 % Nb_2O_5 , and 5.732 % Fe_2O_3 , 57.381 % SiO_2 , 0.684 % TiO_2 , while other minerals in their compound exists in trace form with some radioactive compound such as ThO_2 , among others below detectable level, thus confirming the presence of low-grade niobium oxide in (10.070%) in the crude ore sample. This assay shows that Niobium pentoxide is of low grade in the ore and it is not economical unless beneficiated using processing technique to make it more economical by value addition to 50 – 60% Nb_2O_5 , which is regarded as suitable for the pyrometallurgical extraction of Niobium metal. The chemical analysis of Igbara-Oke Riverside Silica is as presented in Table 2; this reveals a quartz with high silica (SiO_2) content of 84.655% SiO_2 . This confirms the mineral as silica due to high presence of silica-oxide, while niobium oxide occurred in a very low

percentage at 0.013% SiO_2 suggests that the riverside silica contains little mineral of economic value, which shows a good relationship as to niobium. there is also presence of Fe_2O_3 at 1.501%, Al_2O_3 at 5.790%, and Ta_2O_5 at 0.053%, all ore deposit in trace forms, which a niobium ore over burden should have as standard. Mineralogical characterization of the crude Isanlu-Isin columbite using X- ray diffractometer (XRD) was shown on Figure 2. The diffractometer shows mineral present at different phases within the ore matrix. It shows the mineral present in the ore as form of tantalite, quartz, columbite, and muscovite. The diffractogram revealed that tantalum, and niobium in the form of columbite are predominant when compared with other minerals in the ore matrix.

The pie chart in Figure 2 shows the quantitative X-ray diffractometry analysis of the Columbite ore revealing the mineral phases present with the composition within the ore matrix, having quartz as the major mineral present which needs to be separated from the columbite; the mineral of interest. And since we have more of quartz as the major gangue in the ore, it will be easier to employ gravity separation method to produce niobium pentoxide; the mineral of interest. Scanning electron microscope with energy dispersive spectrum (SEM) the mineral particle distributed within the matrix of an ore on a magnification of x200, x500 and x1000 respectively. Which shows that the particles of the mineral present are in large, grains and not closely bonded within the matrix of the columbite, hence reasons for fairly low work index and energy application to the tune of all respectively to affect the crushing and grinding process. (Comminution process) for minerals liberation from the ore.

Table 3, and Figure 3 - 4 reveals the scanning electron microscope (SEM) micrograph of the crude sample which was analyzed using a 15kV mode and a BSD Full detector, at a magnification of 90 with 815, 537, and 358 microns. Followed by results of EDS analysis results for the mineral present in the crude sample as a weight percentage: Silicon has atomic and weight concentrations of 57.95 and 59.39, respectively. The Niobium has an atomic concentrate of 8.62 and weight concentrate of 7.48. iron, aluminium and potassium as an atomic concentrate of 8.25, 10.87, and 3.24 respectively. Heavy mineral such as iron, silicon and niobium were revealed. So also, potassium, calcium, titanium, manganese, sodium, chromium and vanadium as minor minerals present in smaller quantity. The SEM micrograph revealed the interlocking nature of minerals within the crystal aggregates in the

ore matrix. From this, it can be observed that the minerals are coarsely packed as such easy liberation via comminution is facilitated; because the more coarsely packed the minerals, it become easy the liberation of the ore [7]. The EDS result was used to identify the mineral phases that were present in the ore matrix. The EDS peaks of the identified elemental composition are where the examined zones are located. The analysis of the crude revealed the presence of Nb, Mn, Ca, Si, Fe while, Nb, Ta, are below detectable level (BDE). However, further point analysis carried out revealed that silicon, Aluminum and niobium are the predominant elemental constituents of minerals in the ore matrix, the result obtained further compliment the characteristics carried out on the crude sample using ED-XRF and XRD and also reveals that Isanlu-isin columbite contains niobium the mineral of interest which can be beneficiated for niobium pentoxide production, in association with other minerals, which serves as impurities to the minerals (Nb_2O_5) of interest and it needs to be processed for its elimination to upgrade the niobium pentoxide present. Figure 5 (a,b) shows the Petrological micrograph of crude Isanlu-Isin columbite. This confirms the presence of quartz (Qtz), Hematite (Hae) and Geothite (Goe) within the ore matrix with Columbite (Clb) between 12% to 15% formation in this ore. The petrology results shown in Figure 5(c,d) a composition of columbite was seen to be between 12% to 15% and this confirms the 10.070% assay of niobium present in the ore as revealed in the XRF result.

5. Conclusion

On premise of the experimental results obtained alongside the course of these research and their discussion, the following conclusion were drawn;

- i. The crude columbite chemical analysis using ED-XRF showed that it is a mineral that contains low grade niobium pentoxide, with a compound composition of 10.070 percent Nb_2O_5 .
- ii. The crude columbite mineralogical characterization showed that it comprises of tantalite, muscovite, quartz, and columbite. However, the imagery showed that some minerals in the crude ore crystal aggregates interlocked, and a study of the ore matrix showed that Nb, Al, Fe, Si, and Mn were present; as a result, iron, silicon, and oxygen as the elements of the matrix.

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Author Contribution Statement

Omowunmi B. Akinnodia: Concetualization, Methodology, Investigation, Data Collection, Formal Analysis, Writing Original Draft. Oladunni O. Alabi: Formal Analysis, Writing Review & Editing. Fatai O. Aramide: Formal Analysis, Writing Review & Editing. Yemisi E. Gbadamosi: Formal Analysis, Writing Review & Editing. All the authors read and approved the final version of the manuscript.

Does this article screened for similarity?

Yes

Conflict of interest

The Author's declares that there is no conflict of interest anywhere.

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