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(Research Article)

Desliming process of kuru columbite (Plateau state) using scrubbing method towards niobium pentoxide recovery

Alabi OO *, Gbadamosi YE and Akinpelumi T

Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, P.M.B. 704, Akure Ondo State, Nigeria.

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Abstract

The presence of niobium in nature is associated with pegmatite in the form of columbite which necessitates its recovery process from the crude. Kuru Columbite Ore was sourced from ten different pits properly mixed to obtain homogenization and 20 kg was sampled out using the random sampling method, it was ground using the Denver milling machine and prepared towards characterization followed by desliming process by repeated scrubbing technique. Chemical characterization of the crude sample was carried out using Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRF). Mineralogical analysis was carried out using X-ray Diffractometer (XRD) and Scanning Electron Microscope equipped with Energy Dispersive Spectrometer (SEM-EDS). 100 g of the crude sample using the cone and quartering method was sampled out for the particle size analysis and 1000 g each of sieve fractions –710+500 µm, –500+355 µm, –355+250 µm, –250+180 µm, and –180+125 µm was used for the scrubbing process. ED-XRF analysis revealed that the crude sample contains 11.4% Nb_2O_5, 39% SiO_2, 6.56% Fe_2O_3, 0.34% TiO_2 and other compound in trace forms. Mineralogical analysis of the Kuru columbite revealed the present of niobium oxide, iron tantalum, with other associated minerals. SEM images revealed that the ore contains coarse grains of niobium pentoxide which are disperse within the matrix of the ore, this implies that it requires less energy to liberate niobium pentoxide within the matrix of its ore and are evenly dispersed. The EDS of these microstructures revealed the presence of O, Mg, Al, Si, and Fe. The particle size analysis revealed the liberation size to be -250+180 µm and the chemical analysis of the Kuru Columbite ore of the concentration test using scrubbing process revealed that the economic liberation (-250+180 µm) have the highest assay of 48.6% Nb_2O_5. Kuru Columbite Ore has successfully being upgraded from 11.4% to 48.6% via repeated scrubbing process.

Keywords: Desliming; Kuru Columbite; Scrubbing; Niobium Pentoxide; Recovery

1 Introduction

Ores are composed of varieties of minerals, among which the mineral of interest lies and it is necessary to beneficiate the mineral of interest from the ore, because most minerals are finely disseminated and ultimately associated with gangue, they must be ultimately unlocked or liberated before separation can be undertaken (Wills and Napier-Mum, 2006; Onyedika *et al*., 2012). The chemical and mineralogical characterization an ore is of a high importance in order to establish the relationship between structures of materials, the processing techniques of materials and the properties of materials by describing the features of the composition and structure of material which is significant to the study of the properties, processing methods and application. In addition, the characteristics of minerals also often determine the economical aspect of commercial exploitation of the mine site because it varies from one deposit to the other (Barum, 2014; Gbadamosi *et al*., 2021; Gitari *et al*., 2017). Naturally occurring deposits of minerals hardly meet the criteria

* Corresponding author: Alabi OO
Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, P.M.B. 704, Akure Ondo State, Nigeria.

necessary for industrial use, in which desliming procedure performed in mineral processing helps to increase separation efficiency, which require a minimum investment for mixing equipment. The scrubbing process helps in mineral separation, from deleterious materials by agitation with water and then thoroughly flushed away from the desired product, it occurs through material on material attrition created by the violent agitation and a product of high quality is obtained, that is a silt free mineral product that meets standard specifications. Efficiently removing the deleterious materials increases the value of the final product (Nagaraj, 2018).

Niobium pentoxide (Nb_2O_5) is the inorganic compound that is colourless insoluble solid that is fairly unreactive, it is the pioneer to all materials made of niobium, the supreme application being alloys, but other specialized applications include capacitors, lithium niobate, and optical glasses. Niobium Pentoxide (Nb_2O_5) is used principally as an additive to improve the strength and corrosion resistance of steel and due to its refractive nature; it has gained application in jet engine components manufacture, rocket assemblies, and heat resisting and combustion equipment. Also, Niobium is used in lamp filaments, production of jewelry and it is capable of being colored by an electrolytic process (Alabi *et al*., 2015; Alabi *et al*., 2016; Alvesa and Coutinho, 2015).

About 34 minerals deposits have been identified in Nigeria and one of such is Niobium bearing ore. Large deposits of Niobium ore are known to occur in Nasarawa, Gombe, Plateau and Kogi States as well as the Federal Capital Territory. The deposits are both alluvial and primary in the numerous pegmatite bodies that infest these areas. The study area (Kuru) is a district in Jos South Local Government Area of Plateau State North Central Nigeria, falls within the coordinates of latitude 9º49'59.98"N longitude 8º50'59.99"E measured using Global Positioning System (GPS). The study area (Kuru) is made up of settlements like; Science School Kuru, Kuru Karama and Kuru Babba. Kuru is an old mining town boarded by some other mining villages such as Bisichi in the Northeast and Bukuru- Rayfield to the north. It is essentially known for the mining of tin and columbite with other associate minerals such as tantalum and kaolin (Abere *et al*., 2017; Alabi *et al*., 2015; Mallo, 2000).

The enormous application enhance the reason for this research since there is steady and increasing demand of Niobium pentoxide and the recovery will promote the development of the ore in the form of revenue generation to the economy.

2 Material and methods

2.1 Materials

The material employed for the research work was the crude sample of Kuru Columbite, Plateau State, Nigeria. The procedure adopted for the research work includes: sample collection and preparation, which was followed by the determination of the physical and chemical characteristics and desliming process by scrubbing.

2.1.1. Sample Collection

Kuru Columbite samples was collected from ten (10) different pits of the Kuru mining site, it was homogenized using the hand held shovel on a flat cemented clean surface. 20 kg were weighed using cone and quartering sampling technique. 20 kg of the as received crude was sampled out for the research work.

2.1.2. Sample Preparation

20 kg of crude sample collected was crushed using the Fritsch Pulveristte Laboratory Jaw Crusher (Model LF6797AC, Size: D-12) and the crushed product was then ground in a Denver Laboratory Milling Machine (Ball mill) for the chemical and mineralogical characterization analysis.

2.2 Method

2.2.1. Chemical Characterization of Crude Columbite ore using the Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRFS)

Energy Dispersive X-ray Fluorescence Spectrometer, model: PANanalytical Minipal 7 was used to chemically characterize the crude sample. 20 g of the sample was pulverized to pass through a 200-250 mesh sieve, properly mixed with a cellulose flakes binder in the ratio of 5 g to 1 g cellulose flakes binder and pelletized at a pressure of 10-15 tons/inch^2 in a pelletizing machine. The pelletizing sample was stored in a desiccator for analysis. The ED-XRFS machine was switched on and allowed to warm up for 2 hours. Finally, appropriate program of various elements of interest were employ to analyze the crude sample to detect the presence or absence of elements. Result of the analysis was reported in percentage (%) for minor and major concentrations of elements.

2.2.2. Mineralogical Characterization of Crude Columbite ore using the X-ray Dffractometer Analyzer (XRD)

Qualitative and Quantitative determination of the nature of the phases and the amount of the phases present in the sample was determined by a PANanalytical Empyrean diffractometer with PIXcel detector. The material was prepared for XRD analysis using a back loading preparation method. The phases were identified using X'Pert High score plus software. The relative phase amount in % weight was evaluated using the Rietveld method.

2.2.3. Mineralogical Characterization of Crude Columbite ore using Scanning Electron Microscope Equipped with Dispersion Spectrum Analysis (SEM-EDS)

Morphological and qualitative analysis of the bulk ore was performed using SEM-EDS. The SEM provided information on the physical properties of the mineral, while EDS provided information on their chemistry. Scanning Electron Microscope studies for mineral analysis of representative sample was conducted in two stages using SEM model JEOP 840. Sample for analysis was cut, mount in embedded epoxy resin, and finally polished to obtain a mirror-like surface. The polished surface was finally carbon coated before analysis in order to make the mineral's surface conductive. The EDS detector attached to the SEM carried out the qualitative chemical analysis of the sample.

2.2.4. Particle Size Analysis of the Crude Kuru Columbite Ore

100 g of the prepared sample (crushed and ground) was fed into the uppermost sieve of the set of sieves arranged in conformity with √2 series ranging from 1000 to -63um (Alabi *et al*., 2017). Then, attached to the bottom sieve is a tight fitting pan to receive the final undersize, also a lid was placed on top of the uppermost sieve to prevent the escape of the sample. The set of sieves was carefully placed on an Automated Endecott test sieve shaker model EFL2mk11 (5471) and agitated for 30 minutes. During agitation, undersize particles descend through successive sieves until they are retained on a sieve having aperture size slightly smaller than their diameter (Alabi *et al.,* 2016). The set of sieves was separated and the retained on each sieve was weighed.

2.2.5. Desliming Process by Scrubbing of the Kuru Columbite Ore

5 kg of the crude sample was divided into five (5) portions, each portion was crushed using Fritsch Pulveristte Laboratory Jaw Crusher (Model LF6797AC, Size: D-12), to reduce the size to less than 1.0 mm, this was further ground using a Denver Laboratory Milling Machine (Ball mill) at the Mineral Processing Laboratory of the Metallurgical and Materials Department, Federal University of Technology, Akure (FUTA). The ground samples each was sieved to 100% passing sieve fractions of –710+500 µm, –500+355 µm, –355+250 µm, –250+180 µm, –180+125 µm.

Using 1000 g of –710+500 µm sieve size fraction, highly concentrated pulp was prepared by adding 10 litres water to 1000 g of the crude sample, and scrubbing tests was performed in a vessel five different times for about 25 min with the stirring rate of 1500 rpm. 20 g of Sodium Silicate was pre-dissolved in 5 mls of water and then added to the scrubbing suspension at room temperature. After scrubbing, the pulp was stirred and dispersed for 5 min with extra 25 litres of water. The float product was collected into a container and labeled as "T" while the sink product was labeled as "C". The resulting products (C and T) were allowed to settle down for 24 hours then the water was decanted. The resulting pulp products (C and T) were filtered using a 750 diameter filter paper and the cake from the filtering process was dry in a carbolite oven model OV95C at a temperature of 110º C for 12 hours until there is almost moisture free 99.9 % dried sample. Then, the resulting products (C and T) were weighed, sampled out and analyzed for the determination of their chemical composition. The procedure was repeated for the other sieve size fractions.

3 Results

Table 1 Chemical Characterization of Crude Columbite ore using the Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRFS)

Compositions	**SiO_2**	**Fe_2O_3**	**ZrO_3**	**Nb_2O_5**	**TiO_2**	**ThO_2**	**K_2O**	**HfO_2**	**PbO**
Content (%)	39	6.56	30.55	11.4	0.34	2.47	3.66	1.55	0.058

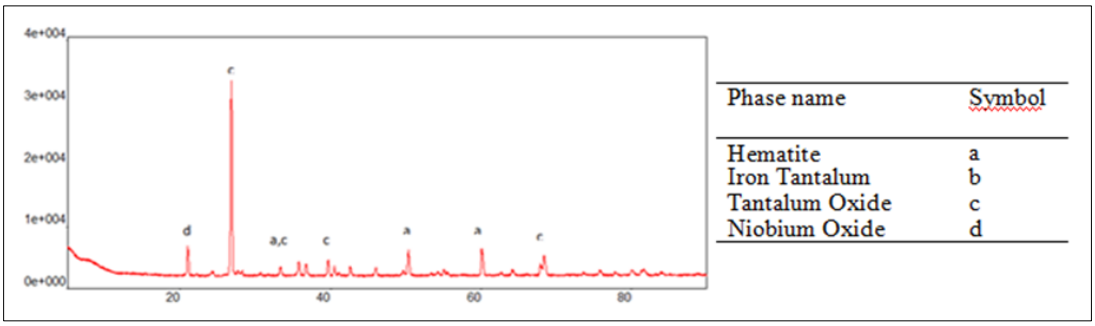

Figure 1 Mineralogical Characterization of Crude Columbite ore using the X-ray Dffractometer Analyzer (XRD)

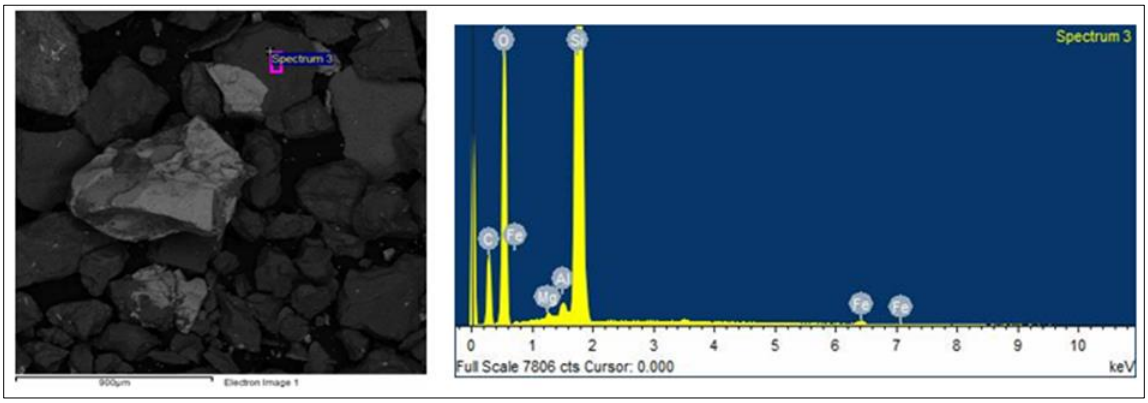

Figure 2 Mineralogical Characterization of Crude Columbite ore using Scanning Electron Microscope Equipped with Dispersion Spectrum Analysis (SEM-EDS)

Table 2 Elemental Composition of Crude Kuru Columbite ore using Scanning Electron Microscope Equipped with Dispersion Spectrum Analysis (SEM-EDS)

Element	Weight %	Atomic %
O K	41.66	70.10
Mg K	0.35	0.38
Al K	1.56	1.56
Si K	1.57	1.57
Fe K	54.86	26.45
Total	100	

Table 3 Fractional Sieve Size Analysis Result of Kuru Columbite Ore

Sieve range (µm)	Nominal aperture (µm)	Weight retained (g)	% Weight retained	% Cumulative Weight retained	% Cumulative Weight passing
+1000	1000	-	-	-	-
-1000+710	710	0.40	0.41	0.41	99.59
-710+500	500	0.60	0.61	1.02	98.98

-500+355	355	5.50	5.61	6.63	93.37
-355+250	250	12.00	12.24	18.87	81.13
-250+180	180	59.50	60.71	79.58	20.42
-180+125	90	5.80	5.92	85.50	14.50
-125+90	90	3.70	3.78	89.28	10.72
-90+63	63	7.7	7.86	97.14	2.86
-63	-63	2.8	2.85	100.00	0.00

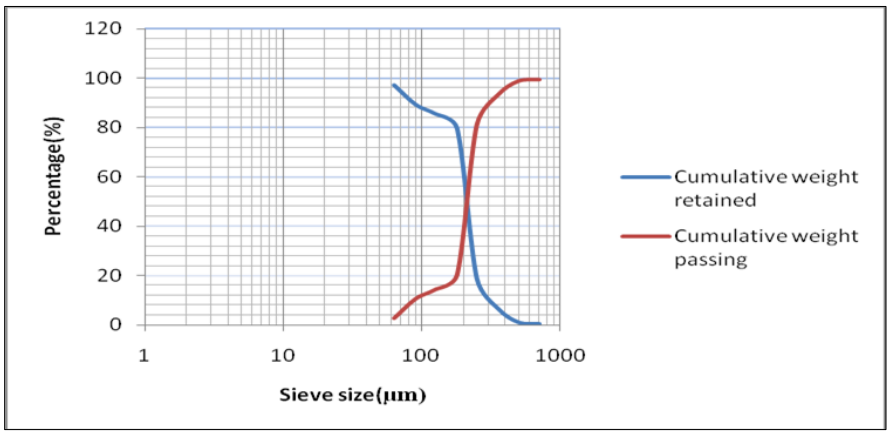

Figure 3 log-log of % cumulative weight retained and passing against sieve sizes of the fractional sieve analysis of Kuru Columbite ore

Table 4 Yield Results of Desliming Process by Scrubbing

Sieve Size Range (µm)	Charge (g)	Concentrate (g)	Tailings (g)
-500 + 355	1000	502.3	149.8
-355+ 250	1000	484.4	280.7
-250 + 180	1000	450.9	203.3
-180 + 125	1000	411.3	280.7
-125 + 90	1000	405.8	247.9

Table 5 Chemical Analysis of the Concentrate Processed Samples of Kuru Columbite ore

Sieve Size Range (µm)	SiO_2	Fe_2O_3	ZrO_2	Nb_2O_5	TiO_2	ThO_2	Ta_20_5	HfO_2	PbO
-500+355	14.0	11.3	43.0	20.2	1.3	1.1	1.4	2.3	0.1
-355+250	15.0	16.3	BDL	37.5	1.5	3.5	1.7	4.7	0.1
-250+180	14.0	19.3	BDL	48.6	2.8	3.0	2.5	4.5	0.1
-180+125	10.0	9.9	42.8	26.7	1.5	0.9	1.3	2.0	0.1
-125+90	8.0	15.7	37.3	23.6	2.5	1.2	2.2	2.2	0.1

Table 6 Chemical Analysis of the Tailings Processed Samples of Kuru Columbite ore

Sieve Size Range	SiO_2	Fe_2O_3	ZrO_2	Nb_2O_5	TiO_2	ThO_2	Ta_20_5	HfO_2	PbO
-500+355	26.0	22.01	27.1	12.1	1.55	2.43	1.04	1.24	0.15
-355+250	27.0	20.48	33.6	15.9	1.37	2.91	1.25	1.71	0.12
-250+180	23.0	24.36	BDL	16.6	1.63	3.73	1.49	1.73	0.14
-180+125	22.0	23.16	32.1	17.6	1.61	3.42	1.50	1.76	0.14
-125+90	21.0	22.26	BDL	14.4	1.45	3.46	1.39	1.71	0.14

BDL = Below Detectable Level

4 Discussion

Table 1 shows the chemical composition of crude kuru columbite ore using Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRFS). The analysis revealed that the crude contains 11.4 % Nb_2O_5, 6.65 % Fe_2O_3, 39 % SiO_2, 0.34 % TiO_2, and other compound in trace form, thus confirming the presence of niobium in the crude.

Mineralogical Characterization of Crude Columbite ore using the X-ray Dffractometer Analyzer (XRD) reveals the mineral phases present in ore sample. Figure 1 shows the XRD pattern of the crude sample, the peak of the various mineral compounds present and their respective phase amounts. The mineral present in the ore are as follows: Hematite, iron Titanium, Tantalum oxide, niobium pentoxide. The diffractogram revealed that tantalum oxide and niobium oxide are predominant when compared with other minerals in the presence matrix.

Figure 2 presents the scanning electron microscopy (SEM) micrograph of the crude sample at 900 microns while Table 2 shows the detailed EDS analysis data of mineral present in weight percentage in the crude sample. 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 the easier their liberation (Alabi *et al*., 2015). The mineral phases present within the ore matrix were revealed using EDS result. The locations of the analyzed zones are EDS peaks of the determined elemental composition with their relative amounts in Table 2. The analysis of the crude revealed the presence of O, Mg, Al, Si, Fe.

Table 3 Figure 3 shows the particle size analysis of the crude and the log-log plot of % cumulative weight retained and passing against sieves sizes respectively. Table 3 shows the percentage weight retained of each sieve fractions. From the graph, the two curves obtained are mirror images of each other and they intercept at -250 + 180 µm. Thus the economic liberation size of the mineral is -250 + 180 µm.

Table 4 – 6 reveals the yield and the chemical characterization of the processed samples of the scrubbing process. Table 4 shows that the yield of the concentrate after repeated scrubbing process is higher that the yield result of the tailings. Table 5 – 6 reveals the chemical composition of the processed sample from the scrubbing process and -250+180 µm sieve size fraction of Nb_2O_5 has the highest percentage composition compare to other sieve size fraction which indicate that beneficiation of Nb_2O_5 took place.

5 Conclusion

The de- sliming process of kuru columbite (Plateau state) using scrubbing method towards niobium pentoxide recovery has been successfully carried out and the following conclusion were drawn:

- the chemical characterization of the crude Kuru Columbite Ore via ED-XRFS revealed the presence of the niobium bearing mineral with the compound composition of 11.4% Nb_2O_5, and some other compound in trace form as gangue in the ore.
- the mineralogical characterization XRD revealed that it contains hematite, niobium oxide, and quartz. SEM images revealed interlocking nature of some mineral within the crystal aggregates of the crude and the analysis of region within the ore matrix revealed the presence of O, Al, Fe, Si, and Mg; such that iron, silicon, and oxygen are the major elemental constituents of the matrix.
- the particle size analysis revealed it's liberation size to be -250+180 µm; and

- the chemical analysis of the Kuru Columbite ore of the concentration test using scrubbing process revealed that the economic liberation (-250+180 μm) has the highest assay (grade) of 48.6% Nb_2O_5.

It is therefore, scientifically proven that niobium pentoxide quality in Kuru columbite can be improved upon to Metallurgical grade by dis – sliming process using sodium silicate as the dis-sliming agent at asieve size of -250 + 180 μm.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest.

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