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CHARACTERIZATION ASSESSMENT OF COLUMBITE ORE FOR NIOBIUM PENTOXIDE VALUE ADDITION FOR ORTHOPEDIC APPLICATION

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ABSTRACT

The demand for stable source for niobium pentoxide has market prospects for orthopedic implants, especially if economically sourced and processed. Columbite ore was extracted and sampled from four pits in Kuru mine site in Jos South Local Government Area, Nigeria. The columbite was processed using a three-disc rapid magnetic separator, and characterized using ED-XRFS, XRD, and SEM/EDS. The magnetic samples were mainly iron with impurities, while the ferromagnetic samples were columbite with niobium pentoxide, and the non-magnetic samples were more of silica. The crude was found to contain 32.1 % ZrO_2, 3.42 % ThO_2, 12.31 % Nb_2O_5, 6.65 % Fe_2O_3, 52.19 % SiO_2, and 1.61 % TiO_2, along with other trace compounds. The 45 A current application yielded the highest ferromagnetic product with a high grade of Niobium pentoxide, with a recovery rate of 67.67 %. The sample morphology showed coarse interlocking within crystal aggregates in the ore matrix. The elements present were Ti, Al, Mn, O, B, Ta, and Nb. The processed concentrate sample contained 1.8414 % SiO_2, 13.9485 % TiO_2, 20.9295 % Fe_2O_3, 46.5767 % Nb_2O_5, 7.9113 % Ta_2O_5, and other constituent compounds in trace form. The processed tailings sample contained 12.1418 % MgO, 4.3061 % Al_2O_3, 1.3150 % SO_3, 40.8441 % SiO_2, 4.8829 % CaO, 6.4475 % Cr_2O_3, 25.9409 % Fe_2O_3, and 1.0330 % NiO.

Keywords: niobium pentoxide, columbite, magnetic separation, orthopedic, Kuru mines.

INTRODUCTION

The demand for advanced biomaterials in the medical field has prompted extensive research into materials that offer superior performance, biocompatibility, and longevity. Among these, niobium pentoxide (Nb_2O_5) is emerging as a material of considerable interest for orthopedic applications [1]. This interest is primarily driven by Nb_2O_5's remarkable properties, which include excellent biocompatibility, corrosion resistance, and exceptional mechanical strength [2, 3]. These attributes make Nb_2O_5 an ideal candidate for implants and other medical devices that require long-term reliability and compatibility with human tissues. As orthopedic treatments often involve the insertion of foreign materials into the body, the choice of material is crucial to ensure that implants not only perform their intended functions but also do so without eliciting adverse biological responses [4, 5]. Niobium pentoxide's unique ability to form a stable and inert oxide layer on its surface further enhances its biocompatibility, reducing the likelihood of immune reactions and promoting the successful integration of implants with bone and other tissues. This stability, combined with its mechanical properties, positions Nb_2O_5 as a superior material for the next generation of orthopedic implants, capable of enduring the mechanical stresses and biological environments encountered in the human body [6].

Orthopedic implants and devices are critical in improving the quality of life for individuals suffering from musculoskeletal disorders [7]. The materials used in these implants must not only withstand the mechanical stresses of daily activities, such as walking, running, and lifting but also be biocompatible to avoid adverse reactions in the body [8, 9]. Niobium pentoxide meets these stringent requirements, making it a valuable material in orthopedics. Its mechanical strength ensures that implants can endure the repetitive and often substantial forces exerted on them without deforming or failing [10]. Furthermore, its ability to form a stable oxide layer enhances its biocompatibility and promotes bone growth and integration, crucial factors for the success of orthopedic implants. The interaction between the implant and the surrounding bone tissue is vital for the stability and longevity of the implant. Niobium pentoxide's surface properties support the attachment and proliferation of bone cells, facilitating the integration process and ultimately leading to more successful and durable implant outcomes [11].

Despite the clear advantages of using niobium pentoxide in orthopedic treatments, many low- and middle-income countries (LMICs) face significant challenges due to the scarcity and prohibitive cost of this material [12]. The high cost of importing niobium pentoxide often places it out of reach for healthcare systems in these regions, limiting the availability of advanced orthopedic treatments. This scarcity not only hinders the quality of care that can be provided but also exacerbates health disparities, as patients in wealthier countries have access to more advanced and effective treatments [13]. The lack of local sources and processing capabilities for niobium pentoxide compounds is the problem. Without the infrastructure to produce high-purity Nb_2O_5 domestically, LMICs are dependent on external suppliers, making them vulnerable to market fluctuations and supply chain disruptions. Consequently, there is an urgent need to develop local sources and processing methods to make niobium pentoxide more accessible and affordable in these regions. By establishing local production capabilities, LMICs could significantly reduce costs, improve the availability of high-quality orthopedic implants, and enhance overall healthcare outcomes [14]. Moreover, local production would reduce dependency on international markets, ensuring a more stable and consistent supply of niobium pentoxide.

Located in Jos South Local Government Area, Plateau State, North Central Nigeria, the Kuru Mines are renowned for their rich deposits of columbite, an ore that contains significant quantities of niobium and tantalum. Positioned at latitude 9º49′59.98″N and longitude 8º50′59.99″E, these mines have historically been a major source of niobium, contributing to the global supply of this essential element [15, 16]. Despite this, the full potential of Kuru Mines in producing high-purity niobium pentoxide for advanced applications has not been fully tapped [17, 18]. This study aims to explore and realize this potential, focusing on the sourcing and processing of columbite from the Kuru Mines to produce niobium pentoxide for orthopedic applications. By leveraging the rich columbite resources available at the Kuru Mines, there is an opportunity to develop a sustainable and economically viable supply chain that can meet the growing demand for niobium pentoxide in the medical field. This initiative could not only transform the local mining industry but also position Nigeria as a key player in the global market for advanced biomaterials [19].

This study has processed and characterized Nb_2O_5 derived from columbite obtained from the Kuru Mines. This characterization not only validates the feasibility of local Nb_2O_5 production but also establishes a foundation for further research and development in the field of orthopedic biomaterials.

EXPERIMENTAL

Ore sourcing

The raw material, columbite ore, was extracted from four distinct pits at the Kuru mine site in Kuru town, Jos South Local Government Area, Plateau State, Nigeria. To ensure homogeneity, the ore from these pits was thoroughly mixed. The required crude ore sample was then obtained using the grab sampling method. This was followed by a random sampling method to achieve a representative subsample, ultimately reducing the quantity to the desired 50 kg for further processing and analysis.

Experimental procedure

Characterization of columbite ore

Samples of the columbite ore were ground into fine powders of 125 μm and dried in an oven at 105°C for one hour. The dried powders were then mixed with a binder and pelletized under a pressure of 10 - 15 tons/inch^2 using a pelletizing machine. The pelletized samples were stored in a desiccator until analysis. The analysis was performed using a Minipal 4 Spectro Xepos Energy Dispersive X-Ray Fluorescence Spectrometer. The samples were subjected to a 90 - min scan, with 30 min allocated for major elements and 60 min for minor elements. The spectrometer software analyzed all elements in the periodic table from sodium (Na) to uranium (U), reporting only those elements detected above the limits. The results were expressed as oxides and reported in percentage (%) composition.

The mineralogical analysis was conducted using a PANalytical EMPYREAN diffractometer with Co Kα radiation to identify the phases present in the materials. The samples were scanned over a 2θ range of 10° to 90° to detect and assess the crystalline phases. For the morphological and elemental analysis, a VEGA3 TESCAN scanning electron microscope equipped with an EDS analyser was employed. The SEM analysis was conducted at an accelerating voltage of 20 kV, which allowed for a detailed examination of the powder morphology and elemental composition. Additionally, Fourier Transform Infrared (FTIR) spectroscopy was used to analyse the functional groups within the processed powder, with spectra collected over the range of 600 - 4000 cm^{-1}.

Extraction process of niobium pentoxide from columbite

The collected columbite ore sample from the Kuru mine site was first sieved using a 2000 μm sieve to separate debris from the ore. Subsequently, five subsamples, each weighing 5 kg, were randomly selected from the homogenized 50 kg batch. These subsamples were then subjected to a crushing and grinding process to achieve a particle liberation size of 355 μm. Each 5 kg sample was processed sequentially through a three-disc rapid magnetic separator. The separation process was conducted at varying current intensities of 40 Amps, 45 Amps, 50 Amps, 55 Amps, and 60 Amps for each batch. This variation allowed for the differentiation and collection of distinct mineral products based on their magnetic properties.

The separation yielded three distinct product streams: magnetic, ferromagnetic, and non-magnetic fractions. These fractions were carefully collected, weighed, and prepared for subsequent chemical characterization. The chemical analysis was performed using an energy-dispersive X-ray fluorescence spectrometer (ED-XRFS) to determine the elemental composition of each fraction. The magnetic fraction predominantly consisted of iron (Fe), with minor impurities detected. The ferromagnetic fraction was found to be rich in columbite, containing a high grade of niobium pentoxide (Nb_2O_5). The non-magnetic fraction primarily comprised silica (SiO_2). This detailed characterization provided essential insights into the mineralogical composition and the efficiency of the separation process, facilitating further processing and refinement of niobium pentoxide for orthopedic applications.

Characterization of the extracted niobium pentoxide

The chemical and mineralogical characterization of the extracted niobium pentoxide was achieved through a combination of Energy-Dispersive X-ray fluorescence Spectrometry (ED-XRFS), X-Ray Diffraction (XRD), and Scanning Electron Microscopy with Energy Dispersive Spectrometry (SEM/EDS) analysis. These techniques provided comprehensive insights into the chemical composition, mineral phases, and morphology of the materials.

RESULTS AND DISCUSSION

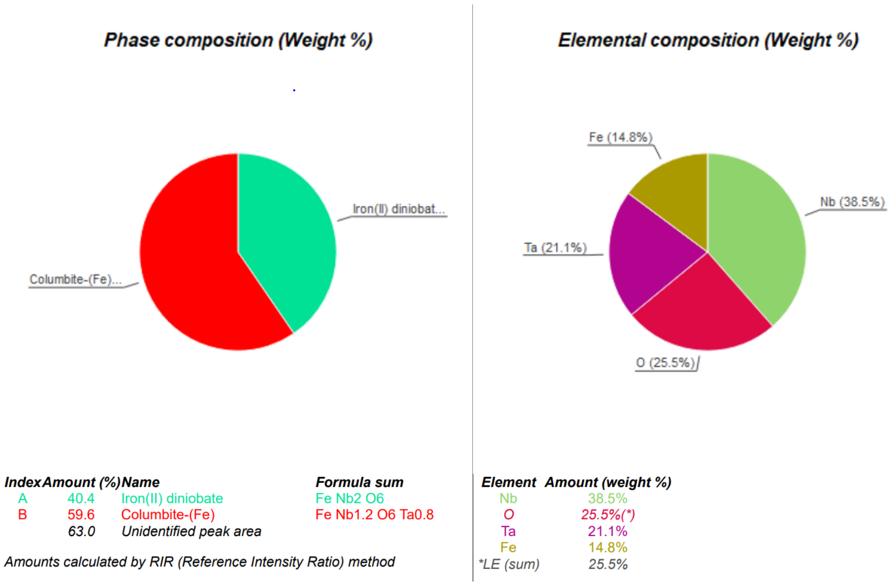
Characterization and processing of crude sample

Table 1 shows the chemical composition of the raw columbite ore using an energy-dispersive X-ray fluorescence spectrometer (ED-XRFS). The analysis revealed that the crude contains 32.1 % $ZrO_{2,}$ 3.42 % ThO_2, 12.31 % Nb_2O_5, 6.65 % Fe_2O_3, 52.19 % SiO_2, 1.61 % TiO_2, and other compound in trace form, thus confirming the presence of niobium in its oxide in the ore sample. The study indicates that the ore's low niobium content necessitates processing to be utilized directly. Hence the need to process using the three-disc rapid magnetic separator at a current variation.

The mineral distribution of the raw columbite ore was achieved in Fig. 1. The phase composition revealed

Table. 1. Chemical analysis of crude sample of columbite ore.

Composition	SiO_2	Fe_2O_3	ZrO_2	Nb_2O_5	TiO_2	ThO_2	Ta_2O_5	HfO_2	PbO
Content, %	52.19	6.65	32.1	12.31	1.61	3.42	1.50	1.76	0.14



Index	Amount (%)	Name	Formula sum
A	40.4	Iron(II) diniobate	Fe Nb2 O6
B	59.6	Columbite-(Fe)	Fe Nb1.2 O6 Ta0.8
	63.0	Unidentified peak area	

Amounts calculated by RIR (Reference Intensity Ratio) method

Element	Amount (weight %)
Nb	38.5%
O	25.5%(*)
Ta	21.1%
Fe	14.8%
*LE (sum)	25.5%

Fig. 1. Mineral distribution of the raw columbite ore.

the presence of 40.4 % Iron (ii) diniobate ($FeNb_2O_6$), 59.6 % Columbite ($FeNb_{1.2}O_6Ta_{0.8}$), and 63 % unidentified peak area. The analysis reveals the presence of niobium pentoxide (a mineral of interest) from the columbite ore. The elemental composition shows the presence of Fe, Ta, O, Nb, and LE (sum) with weight percentages of 14.8, 21.1, 25.5, 38.5, and 25.5 respectively. The result of the XRD analysis of the raw columbite ore sample is presented in Fig. 2. The background and peak position were identified, and a search match was performed based on the peak position. Niobium pentoxide, Calc. (exp. peaks), Background, Iron (ii) diniobate, and Columbite were the identified peaks. The mineral phases present in the ore are compared favourably with the literature and the focus of mining the ore is on niobium pentoxide for orthopedic application [20].

The result obtained in Table 2 shows that 45 A current application gave the highest ferromagnetic product with a high grade of niobium pentoxide of about 64.231 % Nb_2O_5 with a recovery of 67.67 %. Hence, the whole process was repeated using a sieve 355 µm product at 45A current application to obtain the needed quantity and quality of niobium pentoxide needed for orthopedic medical application. Also, the findings show a direct relationship between assay Nb_2O_5 ferromagnetic and recovery as against the standard rule (i.e. the inverse relationship between assay grade and recovery) which established the optimization process of the separation procedure.

Characterization of processed sample

Sample morphology of the peak processed columbite ore was revealed in Fig. 3 at an image view of 100 magnification of 500 µm. The sample morphology shows the coarse interlocking nature of the mineral within the crystal aggregates in the ore matrix. The minerals are separated by coarse grain boundaries which facilitate the highest value of ferromagnetic product with a high grade of Niobium pentoxide.

Fig. 4 presents the energy dispersion spectroscopy (EDS) peaks for various elements of the processed columbite at 100 µm in the SEM micrograph magnification. The elements present were Ti, Al, Mn, O, B, Ta, and Nb with the highest peak value.

Table 3 shows the chemical composition of the processed concentrate sample using an energy-dispersive X-ray fluorescence spectrometer (ED-XRFS). This reveals that the processed concentrate sample contains 1.8414 % SiO_2, 13.9485 % TiO_2, 20.9295 % Fe_2O_3, 46.5767 % Nb_2O_5, 7.9113 % Ta_2O_5, and other constituent compounds in trace form, thus confirms the effective

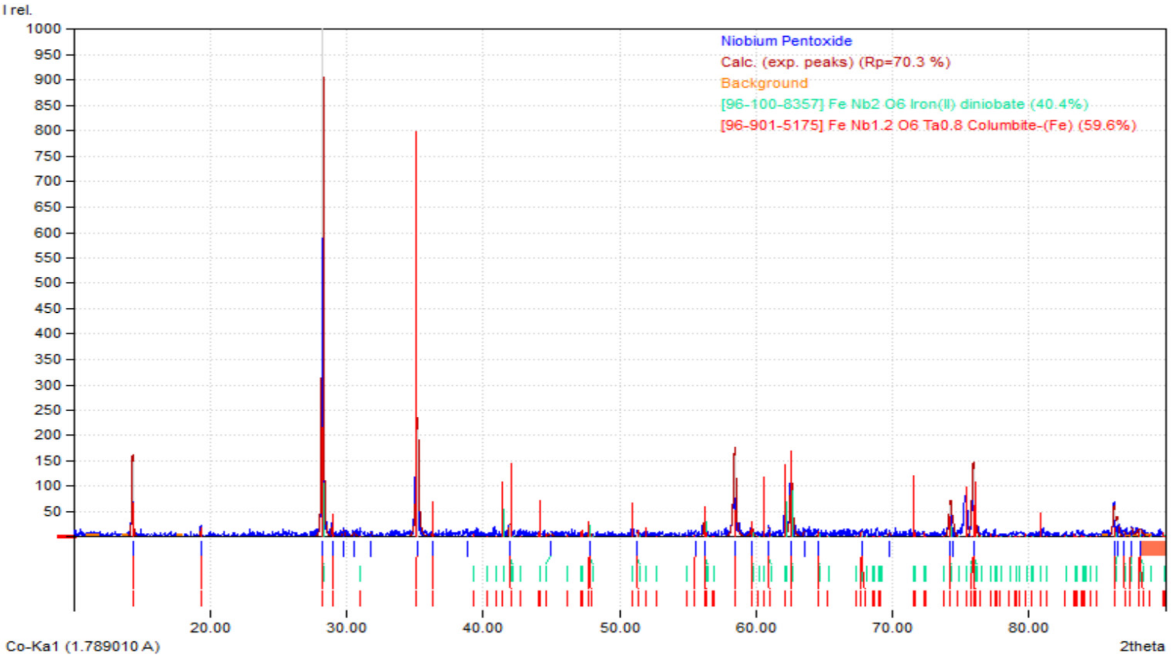


Fig. 2. XRD of raw columbite ore.

Table. 2. Total weight of product obtained after magnetic separator process at different amperes.

Current, A	Wt. charged, kg	Assay Nb_2O_5 charge, %	Wt. magnetic, kg	Wt. ferromagnetic, kg	Wt. non-magnetic, kg	Assay Nb_2O_5 ferromagnetic, %	Recovery, %
40	5.00	12.31	1.59	1.023	2.387	43.120	71.67
45	5.00	12.31	1.42	0.345	3.235	64.231	67.67
50	5.00	12.31	1.69	0.743	0.947	51.360	64.50
55	5.00	12.31	1.76	0.810	2.567	49.170	64.47
60	5.00	12.31	2.31	1.069	1.621	34.910	60.63

recovery process of Niobium pentoxide with the grade percentage of 46.5767.

Table 4 shows the chemical composition of processed tailings sample using Energy Dispersive X-ray Fluorescent Spectrometer (ED-XRFS). This reveals that the processed tailings sample contains 12.1418 % MgO, 4.3061 % Al_2O_3, 1.3150 % SO_3, 40.8441 % SiO_2, 4.8829 % CaO, 6.4475 % Cr_2O_3, 25.9409 % Fe_2O_3, 1.0330 % NiO and other constituent compounds in trace form. The percentage composition of SiO_2 (40.8441 %) is high compared to the processed concentrate (1.8414 % SiO_2), the result affirms the effective recovery process of Niobium pentoxide from columbite.

The phases present in the peak processed sample are presented in Fig. 5. The XRD analysis result of the sample in Fig. 5 shows the peaks are indexed as grossular ($Si_{24}Al_{16}Ca_{24}O_{96}$), pyrochlore ($Nb_{16}Ca_{16}O_{56}$), silicon dioxide ($SiO_2$), and $Fe_4Nb_8O_{24}$. The $Si_{24}Al_{16}Ca_{24}O_{96}$ phase was observed at a wide range of 2θ values between 35.4° and 75.6°. The $Nb_{16}Ca_{16}O_{56}$ phase was observed at a wide range of 2θ values between 35.4° and 76.3°. The SiO_2 phase was observed at a wide range of 2θ values between 35.4° and 74.4°. The $Fe_4Nb_8O_{24}$ phase was observed at a wide range of 2θ values between 14.4° and 85.5°. The crystallographic data of the detected phases present in the sample 0934 is shown in Table 5.

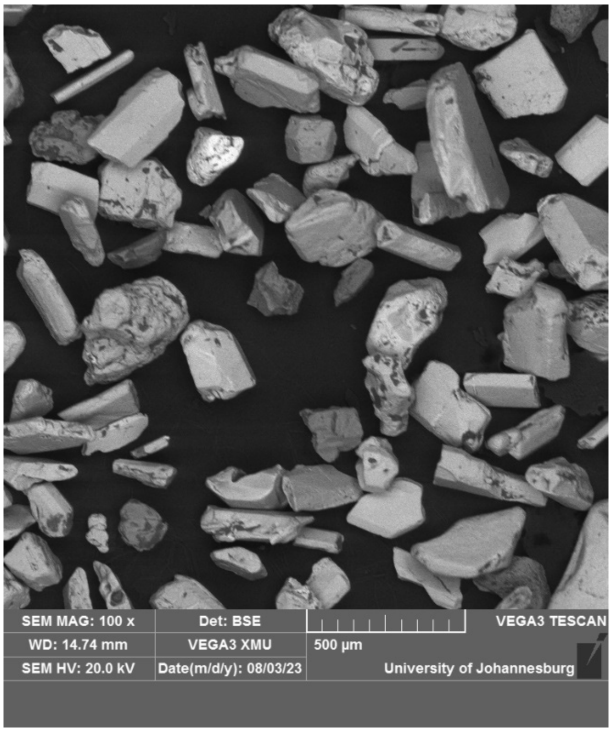


Fig. 3. Sample morphology of the peak processed columbite ore.

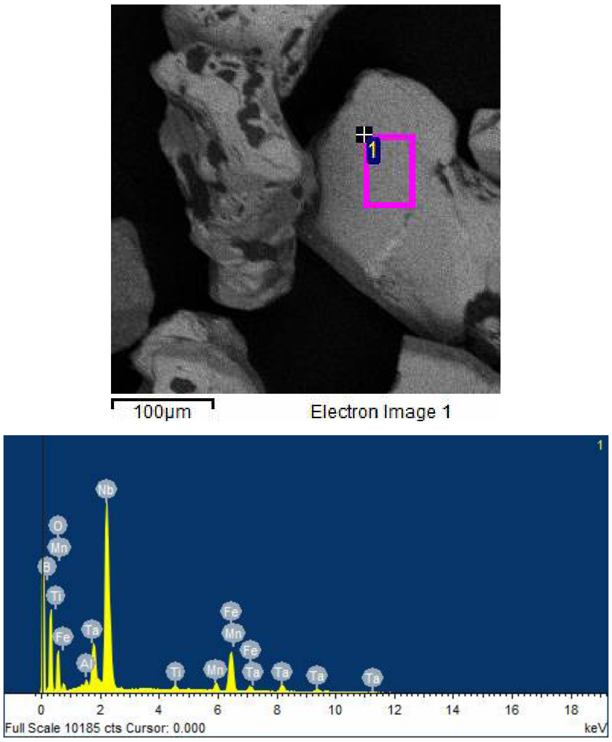


Fig. 4. SEM-EDS spot analysis of the peak processed columbite ore.

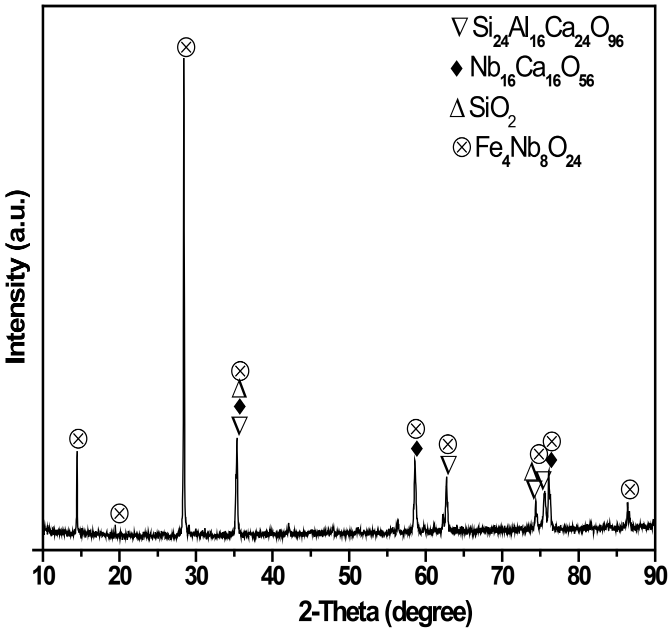


Fig. 5. XRD pattern of the peak processed columbite.

Table 3. ED-XRFS analysis of the peak processed columbite ore.

Component	Result, mass %
MgO	0.0422
Al_2O_3	0.7552
SiO_2	1.8414
P_2O_5	0.3639
SO_3	0.0000
K_2O	0.1369
CaO	0.2938
TiO_2	13.9485
Cr_2O_3	0.0594
MnO	2.0919
Fe_2O_3	20.9295
Y_2O_3	0.3866
ZrO_2	3.1402
Nb_2O_5	46.5767
SnO_2	0.8565
Dy_2O_3	0.0577
Yb_2O_3	0.1062
HfO_2	0.1938
Ta_2O_5	7.9113
ReO_2	0.2399
ThO_2	0.0588
U_3O_8	0.0097

Table 4. ED-XRFS analysis of the tailings of the columbite ore.

Component	Result, mass %
Na_2O	0.0917
MgO	12.1418
Al_2O_3	4.3061
SiO_2	40.8481
P_2O_5	0.0216
SO_3	1.3150
Cl	0.1867
K_2O	0.1472
CaO	4.8829
TiO_2	0.5627
V_2O_5	0.1173
Cr_2O_3	6.4475
MnO	0.3231
Fe_2O_3	25.9409
CO_2O_3	0.0619
NiO	1.0330
CuO	1.0740
ZnO	0.4368
SrO	0.0156
PbO	0.0460

Table 5. Crystallographic data of phases present in the peak processed sample.

Alloy	Phase	Space group(no.)	*a*,Å	*b*,Å	*c*, Å	Cell vol., 10^6 pm^3	Reference
Peak processed Sample	$Si_{24}Al_{16}Ca_{24}O_{96}$	Ia-3d (230)	11.8450	11.8450	11.8450	1661.90	96 - 900 - 0237
	$Nb_{16}Ca_{16}O_{56}$	Fd-3m (227)	10.3400	10.3400	10.3400	1105.51	96 - 101 - 1129
	SiO_2	P42/mnm (136)	4.1640	4.1640	4.1640	46.19	96 - 154 - 4735
	$Fe_4Nb_8O_{24}$	Pbcn (60)	14.2660	5.7330	5.0500	413.02	96 - 100 - 8357

CONCLUSIONS

The sourcing of columbite ore from Kuru Mines and processing for niobium pentoxide value addition for orthopedics applications have been considered. On account of the results obtained the following conclusions are drawn.

- The analysis revealed that the crude contains 32.1 % ZrO_2, 3.42 % ThO_2, 12.31 % Nb_2O_5, 6.65 % Fe_2O_3, 52.19 % SiO_2, 1.61 % TiO_2, and other compound in trace form;
- The phase composition and XRD analysis revealed the presence of 40.4 % Iron (ii) diniobate ($FeNb_2O_6$), 59.6 % Columbite ($FeNb_{1.2}O_6Ta_{0.8}$), and 63 % unidentified peak area;
- The recovery process shows that 45A current

application gave the highest ferromagnetic product with a high grade of Niobium pentoxide of about 64.231 % Nb_2O_5 with a recovery of 67.67 %;

- The sample morphology shows the coarse interlocking nature of the mineral within the crystal aggregates in the ore matrix;
- The elements present were Ti, Al, Mn, O, B, Ta, and Nb with the highest peak value;
- This reveals that the processed concentrate sample contains 1.8414 % $SiO_{2,}$ 13.9485 % TiO_2, 20.9295 % Fe_2O_3, 46.5767 % Nb_2O_5, 7.9113 % Ta_2O_5 and other constituent compounds in trace form;
- This reveals that the processed tailings sample contains 12.1418 % MgO, 4.3061 % Al_2O_3, 1.3150 % SO_3, 40.8441 % $SiO_{2,}$ 4.8829 % CaO, 6.4475 % Cr_2O_3, 25.9409 % Fe_2O_3, 1.0330 % NiO and other constituent compounds in trace form; and
- The XRD result of the processed sample revealed the peaks are indexed as grossular ($Si_{24}Al_{16}Ca_{24}O_{96}$), pyrochlore ($Nb_{16}Ca_{16}O_{56}$), silicon dioxide ($SiO_2$), and $Fe_4Nb_8O_{24}$.

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