Physicochem. Probl. Miner. Process., 59(3), 2023, 170297

http://www.journalssystem.com/ppmp

Physicochemical Problems of Mineral Processing

ISSN 1643-1049

Grindability characterization and work index determination of alluvial ferro-columbite deposits for efficient mineral processing

Nnaemeka Stanislaus Nzeh [1], Abraham A. Adeleke [1,2], Patricia Abimbola Popoola [1]

[1] Chemical, Metallurgical & Materials Engineering Dept, Tshwane University of Technology, Pretoria, Private Bag X680, Pretoria West 0183, Republic of South Africa

[2] Materials Science & Engineering Dept, Obafemi Awolowo University, Ile-Ife 220002, Nigeria

Corresponding author: nstannzeh@gmail.com (N.S. Nzeh)

Abstract: This study emphasizes on the physicochemical and grindability characteristics and work index of an alluvial formed silica dominated ferro-columbite mineral from Rayfield-Jos minefields in Plateau state, Nigeria. Investigations were also carried out in order to determine the mineralogy of the mineral deposits and most essentially the actual energy consumed during comminution and milling of the mineral so as to achieve the liberation size prior to high efficient mineral processing or beneficiation and the extraction of value metals. The distribution of the mineral particles as well as their sizes was determined, with a mineral liberation size fraction range essentially established as -150+90 μm particle sizes. Mass percentage of each size fraction obtained from PSD analysis conducted before and after comminution was also determined, obtaining 80% passing for both the mineral feeds and comminuted products. Berry and Bruce modified Bond's work index was therefore obtained, and was determined to be within the range of 2.0414 to 2.5667 kWh/ton. Hence, the energy consumed or required to comminute or grind the Fe-columbite mineral to 80% passing is expected to fall within the range of 0.3613 to 0.4543 kWh. Thus, it could be said that a low milling work index as well as moderately low energy is required for comminution and this can be attributed to the mineralogy, mineral source and alluvial formation of the mineral reserve. Therefore, the grindability/PSD result of the mineral sample indicates that its mineralogy is considered a class of moderately soft mineral type in terms of texture with easy grindability.

Keywords: alluvial, columbite, mineral, comminution, milling, work index, energy, grindability

1. Introduction

Characterization, being an essential part of mineral processing and beneficiation is referred to the general or broad procedure whereby the structure and entire responses of materials or particles in the minerals are determined (Yerima and Abdulrahman, 2015; Alabi et al. 2015; Nzeh et al. 2023a). It is very essential for materials or primary/secondary minerals to undergo characterization. This is done in order to establish a relationship between the material structures, properties, responses or performance as well as the processing techniques involved; by the description of the material characteristics or features relating to its physicochemical structure and composition that is significant to the investigations of the material's properties, processing and applications. When exploring and exploiting minerals, it is necessary to critically consider the inherent mineral properties and the major compositions; as well as the behaviour or response during processing, beneficiation and extraction (Abraham et al., 2012; Gbadamosi et al., 2021). Moreover, these mineral characteristics or properties will also determine the economical aspect of exploiting the minerals commercially from the minefield (Gbadamosi et al., 2021). Apart from elemental/chemical composition, microstructural or morphological, minerals can also be characterized in terms of their responses to comminution.

Preceding other mineral ore beneficiation processes, comminution is described as an important aspect of mineral processing (Lynch, 2015; Alabi and Awogbamila, 2020). It is comprised of the sequence of crushing and grinding. This is the reduction process of solid minerals, whereby crushing involves

DOI: 10.37190/ppmp/170297

the reduction in particle size fraction of the run-off-mine mineral ore particles to a size fraction where grinding is subsequently carried out until mineral liberation or production of separate value and gangue particles (Magdalimovic, 1989; Wills and Napier-Munn, 2006; Adeoti et al., 2019). However, comminution, especially grinding/milling process is regarded as the highest energy consuming procedure during mineral processing and as such, proper investigations on the required energy consumed during comminution have become imperative over the decades (Napier-Munn et al., 1996; Norazirah et al., 2016; Adeoti et al., 2019; Adetula et al., 2019; Alabi and Awogbamila, 2020). According to Higgins (1998) and Adetula et al. (2019), mineral (particle) size reduction is somewhat expensive. More so, the required energy and the cost per ton of mineral that pass through comminution generally increases, beginning from blasting (with about 0.43 kWh/t) to crushing (with 3.24 kWh/t) and to grinding (with 10 kWh/t) (Higgins, 1998; Adetula et al., 2019). In essence, the overall power/energy consumption and draw of power/energy in a comminution system during milling process are essentially key factors (Siddall et al., 1996; Adetula et al., 2019). However, milling is reported to be more or less inefficient, despite it being the highest energy consuming and most expensive procedure (Adetula et al., 2019). As of a result of this, it is of great importance to optimize the usage of the available power/energy in the comminution system. In this regard, and with respect to the limited available energy resources, developing and designing comminution equipment of high level of mechanical reliability and efficiency is imperative, thus, making energy efficiency of high priority (Norazirah et al., 2016; Adetula et al., 2019; Alabi and Awogbamila, 2020). In addition, the design of the comminution system or circuit during metallurgical or mineral processing plant design of the mines is based on the abrasive index and grindability processes employed on the mineral samples as well as the expected processed mineral tonnage (Levin, 1992; Adetula et al., 2019).

Moreover, in recent decades, the problem of insufficient or the conservation of power and energy resources are being encountered in several parts of the world. It has been stated by Napier-Munn et al. (1996) and Adeoti et al. (2019), that 30 to 50% of total energy consumption by mineral ore metallurgical plants during the processing of minerals or ore deposits is related to comminution. This however may be extended up to 70% during the comminution of hard ores (Napier-Munn et al., 1996; Adeoti et al., 2019). It was also observed that almost 1.5% of the annual electrical energy produced in USA is utilized in material comminution processes by the mineral processing industries (Whittles et al., 2003; Adeoti et al., 2019). This may essentially be attributed to the mineral grindability, which is the measure or degree of ease or resistance of a material towards comminution. Meanwhile, this grindability resistance is the comminution parameter known as the milling work index (Magdalimovic, 1989; Yaro, 1996; Wills and Napier-Munn, 2006; Alabi et al., 2012, Alabi et al., 2015, Adeoti et al., 2019; Adetula et al., 2019; Bwala et al., 2021). Table 1 enlists some mineral ores with known work indexes.

Over the decades, the work index determination on certain heavy minerals, such as the Nb and Ta minerals were not completely investigated. It is expressed numerically as the kilowatt hour per short ton of mineral (kWh/t) which is required to reduce the mineral from an infinite particle size to 80% passing 100 μm (Magdalimovic, 1989; Wills and Napier-Munn, 2006; Alabi et al., 2012; Alabi et al., 2015; Adeoti et al., 2019; Bwala et al., 2021). In essence, it is described as a measure of the resistance of a mineral to comminution (Todorovic et al., 2017). However, work index is expressed theoretically as an essential parameter in mineral processing, characterization and process design (Adeoti et al., 2019); which determines the rate of energy consumption during mineral mechanical activation, comminution or breakdown of run-off-mines to the appropriate mineral liberation sizes (economic optimal particle size fraction) in order to prevent under/over-grinding as well as selecting suitable comminution equipment. When there is under-grinding, there is insufficient liberation of the mineral which limits recovery of the value element during separation/purification stages. On the other end, an over-grinding increase the consumption of energy during grinding which in turn is not cost effective, and may as well tend to decrease recovery of the final products. This explains comminution and thus, work index as a key and vital elements for efficient mineral processing and beneficiation process routes (Onemine, 2010; Alabi and Awogbamila, 2020).

Apart from under/over-grinding, there are other demerits of comminution which affects subsequent beneficiation and processing of the mineral. Reasonable quantities of material are lost to the tailings, which is considered a wastage in terms of material, time, energy, effort and resources (Riantie et al.,

2013; Alabi and Awogbamila, 2020). However, the major shortcoming encountered during comminution is explained that most of the energy input during crushing or grinding is absorbed by the milling machines, converting the energy into heat and noise while just a little fraction of the energy is made available for the ore reduction (Alabi and Awogbamila, 2020). According to Wills and Finch (2015), less than 1% of the total input energy is made available for the actual mineral particle size reduction, and the bulk of the input energy is utilized in producing heat and noise (Wills and Finch, 2015; Alabi and Awogbamila, 2020). Further, plastic materials would consume energy when changing shape; however, they tend to retain this shape without the creation of a significant new surface (Alabi et al., 2015; Lynch, 2015; Norazirah et al., 2016; Alabi and Awogbamila, 2020).

The grindability characterization and work index determination as well as the energy consumed during comminution or required to comminute certain critical minerals is key and has been de-emphasized in recent time. Columbite is one of such minerals to require comprehensive characterization as a result of its complex mineral composition and structure. Comminution and hence work index is a key process factor in the processing and upgrade of columbite for the recovery of Nb and/or Ta; and this area has been neglected in the Nigerian mining and solid minerals sector, especially on primary and secondary mineral sources such as columbite-tantalite ore deposits. In this regard, the study is aimed at investigating the grindability and also determining the work index of alluvial columbite mineral in order to determine the actual energy consumed during comminution and milling to the liberation size prior to beneficiation and metal extraction process. This is therefore significant in columbite mineral liberation and design/development of effective mineral processing/beneficiation routes for overall efficiency of recovery, separation and extraction of value metals such as Nb and Ta. This will also act as a deciphering factor to be considered when choosing and selecting suitable comminution equipment and design system**.**

1.1. Theoretical considerations of comminution and milling work index

There are a lot of established comminution and milling theories. It has been established theoretically that the milling work index is a grindability analysis used to determine the work index of fine mineral feed samples applying a closed cycle ball mill to mechanically activate or liberate the mineral (Todorovic et al., 2017; Alabi et al., 2015). The liberation of minerals involves the specific particle size fraction called the liberation size which essentially has to be reached by the mineral particles in order to achieve significant separate mineral particles of value mineral or gangues. However, these mineral particles in their liberation size can possibly be separated from the minerals as concentrates and tailings having an acceptable efficiency by the commercial process unit (Alabi et al., 2015). According to Adeoti et al. (2019), Alabi et al. (2015), the textural relationship between mineral particles in the mineral ore matrix and the relation to selection of process requires the liberation size determination.

Also, work and energy are related quantities possessing same units, which one can refer to as two sides of the same coin. Energy can simply be defined as the capacity to do work (which in this case, is the milling process). Thus, the energy required or consumed during the comminution of a coarse mineral is directly proportional to the new surface area of the finer mineral particles produced (Adeoti et al., 2019; Alabi et al., 2015). A milling work index is therefore represented as a comminution value or parameter of minerals usually known to determine the characteristics of minerals, which is utilized in the designing of industrial comminution plants (Todorovic et al., 2017; Lvov and Chitalov, 2019). Therefore, generally and theoretically established, work index, as a comminution parameter is the rate or level of a material's resistance to crushing and grinding. Several techniques of predicting (rod or ball) milling energy requirements for accurate measure of mineral's grindability were developed by Bond (Wills and Napier-Munn, 2006; Gupta and Yan, 2006; Bwala et al., 2021).

1.2. Bond ball milling and work index

The Bond ball milling work index is regarded a grindability analysis used to determine the Bond work index of mineral samples, applying a closed cycle ball mill to mechanically activate the mineral particles. A Bond work index is represented as a value or parameter of minerals usually known to determine the characteristics of the minerals, which is utilized in the designing of industrial comminution plants. This parameter is the most widely used over the decades in determining the measure of resistance of

minerals to comminution (Todorovic et al., 2017). The Bond work index is usually determined by carrying out a stimulated dry grinding process in closed circuit in a Bond ball mill to obtain a circulating load of 250%. The procedure is performed on mineral samples usually in weights between 10 to 15 kg. Series of grinding cycles is conducted and at the end of each grinding cycle; the ground mineral samples from the Bond ball mill undergoes screening on a control sieve. The undersize mineral sample particles are removed and another grinding cycle begins at the sieve oversize with a fresh mineral sample added. The ball mill revolution number is calculated for all grinding cycles with the use of the previous cycle data to obtain the 250% circulating load. This is repeated for about 7 to 10 grinding cycles until a constant is formed in the last three (3) milling cycles by the control sieve undersize produced per ball mill revolution. The bond work index is then be calculated with the mathematical formula in Equation 1 and a ball milling particles distribution curve is obtained (Todorovic et al., 2017; Lvov and Chitalov, 2019). A standard Bond work index test however has been reported to require constant screening of undersized mineral particles in a closed system circuit. This technique has been adopted by several researchers in determining Bond work index of different minerals (Levin, 1992; Denis, 2003; Doll and Baratt, 2011; Bwala et al., 2021).

$$BW_i = 1.1\frac{44.5}{Pc^{0.22}G^{0.82}=(\frac{10}{\sqrt{P_{80}}}-\frac{10}{\sqrt{F_{80}}})} \quad (1)$$

where BW_i = Bond work index (in kWh/t), P_c = Test sieve mesh size (in µm), G = Weight of test sieve fresh undersize per mill revolution (in g/rev), P_{80} = Opening of sieve size passing 80% of last cycle test undersize product (in µm), F_{80} = Sieve mesh size passing 80% of feed before grinding (in µm).

However, several other feasible comminution theories and techniques in determining work index have been developed. Amongst lots of other comminution theories such as Bonds, Kicks, Ritingers which were considered not satisfactory (Wills and Finch, 2015; Alabi and Awogbamila, 2020); this study focuses on work index determination, adopting modified Bond's method established by Berry and Bruce in 1966 (Adeoti et al., 2019; Berry and Bruce, 1966).

1.3. Berry and Bruce milling and work index

Berry and Bruce work index as the study case, is known as a modified, comparative method of Bond's work index grindability equation (Todorovic et al., 2017; Lvov and Chitalov, 2019; Bwala et al., 2021). Its procedure or calculations involves the utilization of a reference material (mineral sample) with a known grindability and work index. This method has been established very feasible, reliable and efficient over the years by several authors. The data/information obtained from the work index test calculations may hence be used to obtain the amount of power or energy required for comminution as well as the grinding efficiency (Alabi et al., 2012). This mineral feature has been reported useful in designing the processing of minerals by various researchers. Thus, determination of this characteristic, explaining the required energy to comminute a ton of mineral from a particular feed size to specific product size for primary ore deposits as well as secondary sources is imperative (Adeoti et al., 2019). Table 1 displays the work index of different minerals.

Table 1. Average work indexes of various minerals (Wills and Napier-Munn, 2006; Thomas et al., 2014; Alabi et al., 2015; Adetula et al., 2019; Bwala et al., 2021)

Mineral ore	Work index	Mineral ore	Work index
Barite	4.28 – 6.24	Graphite	1.75 – 45.03
Bauxite	2.38 – 9.45	Gold ore	3.00 – 42.00
Coal	1.63 – 11.37	Limestone	2.69 – 14.00
Columbite	3.94 – 10.81	Molybdenium	11.60 – 14.10
Copper ore	12.70 – 14.00	Silica sand	2.65 – 16.46
Dolomite	2.82 – 11.27	Silver ore	13.00 – 22.00
Emery	3.48 – 58.18	Tantalite	3.60 – 11.90
Fluorspar	2.98 – 9.76	Titanium ore	4.23 – 11.88
Granite	2.68 – 15.13	Quartzite	2.71 – 17.40

2. Experimental

2.1. Preparation of research material/mineral samples

The research material used for this study is an alluvial (silica dominated) columbite mineral of low-grade sourced in the middle belt region of Nigeria from the Rayfield minefields at the localities of Rayfield, Jos South, Plateau state. Nigeria is however located between longitude of 3° and 15°E and latitude of 4° and 14°N (Yerima and Abdulrahman, 2015). Rayfield, as case study area is about 15 km away from Jos main city, capital of Plateau state, Nigeria (Ayeni et al., 2012a; Ayeni et al., 2012b). The study area, Rayfield-Jos lies in Plateau state between longitude of 8° 54 43E and latitude of 9° 50 1N with a bearing of 105.4°ESE (Mindat, 2021); surrounded by Bukuru, Vom and Lere localities. A true representative sample of the columbite mineral was collected randomly from different dumps or deposits in the mine field with a total land mass of approximately 50 hectares; from a soil depth of about 2 to 3m deep (1.5 × 1.5m) which were deposited in an alluvial form through alluvial veins influenced by flowing water. Thus, it is imperative to also note that the research mineral samples collected or procured, initially originated from the remnant deposits or tailings dump of a heavily exploited cassiterite mineral reserve from a somewhat abandoned mine site in the Rayfield-Jos minefields. This mine site was heavily exploited by the colonial miners decades ago, and has since then been left unexploited and untouched for many decades, and hence, has been deposited over the years by alluvial formation and several deposition of different heavy mineral particles. This has however been reported elsewhere (Nzeh et al., 2023a). Thus about 10 kg of columbite sample was obtained from the different dumps/deposits at the Rayfield-Jos mine site using the grab sampling, coning and quartering techniques. Fig. 1 and 2 shows Rayfield-Jos mine field and samples of the alluvial columbite mineral, respectively.

Fig. 1. Rayfield-Jos mine field

(**i**) (**ii**)

Fig. 2. Rayfield-Jos alluvial columbite mineral (**i**) As-received (**ii**) Washed and dried sample

2.2. Methodology

2.2.1. Materials

Other than the test mineral ore (t), Berry and Bruce modified Bond's method however requires the usage of a reference mineral (r) with a grindability/work index which is known. The test mineral sample (Rayfield-Jos columbite deposits), of which the work index is to be determined, was collected essentially from abandoned tailing dumps in Rayfield-Jos minefields, deposited in somewhat fine alluvial form and of a low-grade nature (with high degree of silica composition), and hence, to a certain degree, often referred to as tailings by some researchers (Ayeni et al., 1999, 2012a, 2012b). More so, the need for a pre-comminution or size reduction may be overlooked as the size fraction of the columbite sample is of

acceptable feed size. The reference material were ore samples of Gyel-Bukuru columbite mineral, obtained from Jos, Plateau state in Nigeria; which have been reported to have a known work index value of 2.72 kWh/ton and 3.42 kWh/ton (Alabi et al., 2015). Comminution was however carried out on the both columbite samples in order to determine the liberation particle size. The equipment used for comminution was a laboratory ball mill and a laboratory pulverizer 'Polymix PX-MFC 90D'. Both test and reference feed (F) samples were comminuted for a particular time (T) using a laboratory ball mill and their products (P) were weighed on a weighing beam balance and then subsequently subjected to PSD tests.

2.2.2. Mineralogical analysis

The mineralogy of the columbite test sample was characterized using X-ray diffraction (XRD) patterns. A quantity of the columbite particles were prepared and homogenized according to the standard panalytical backloading system to provide random distribution. A computer controlled PANalytical X'pert Pro Powder diffractometer was utilized to analyse the positioned mineral samples at room temperature, 30 to 40 kV power and at a current of 30 to 40 mA. This diffractometer was θ-θ conFig.d with X'celerator detector and variable divergence. Fixed receiving slits with Fe filtered Co-Kα radiation (γ = 1.789 Å) and a monochromatized Cu- Kα radiation source (γ = 1.5406 Å) at a scanning angle range of $2\theta = 10^{o}$ to 90^{o} and a scan rate of $0.02^{o}.s^{-1}$ was applied. Phases and crystal sizes present in the columbite particles thus was identified and the different peaks matched by an X'pert Highscore Plus software.

2.2.3. Morphological and microstructural analysis

I. Scanning electron microscope (SEM) and Energy Dispersive Spectrum (EDS)

A high performance field emission-scanning electron microscopy (FE-SEM) JSM-7600F, Joel Japan was used to examine the surface morphology and dispersion of homogenized columbite mineral test sample using secondary electron imaging. An energy dispersive x-ray spectrometer detector, Oxford X-Max with an INCA X-Stream pulse analyzing software and back scattered electron (BSE) imaging detector equipped to the FE-SEM gave the elemental composition and surface topography of the columbite samples. The test samples was coated with conductive carbon with the aid of a carbon tape/vacuum coater and positioned for analysis. The INCA analyzer was set at an acquisition time of 70 secs and 2 secs process time. The FE-SEM analysis was conducted applying a spectrum range of about 1 to 20 keV and an accelerated voltage of about 20 to 30 kV incident electron energy beam for both fractured and smooth surfaces, respectively. The obtained SEM micrograph was subjected to 'Image J' software for further and clearer photomicrograph interpretation (indicating the micropores embedded in the columbite test mineral deposit).

II. Optical microscope (OM)

An optical microscopy was conducted using an optical microscope (OM) Olympus BX 51 TRF on the columbite test sample. This was used to produce and examine the sample photomicrographs. The process adopted a combination of light and lenses to magnify the material image at different magnifications including 5x, 10x, 15x and 20x. The objective lens is made up of two operational features which includes magnifications (ranging from 5x to 100x) and the numerical aperture (ranging from 0.14 to 0.7) corresponding to the focal lengths of 40 mm to 2 mm respectively.

2.2.4. Chemical analysis

X-ray fluorescence (XRF) chemical composition analysis was conducted on both test and reference samples (as seen in Tables 2 and 3, respectively). The mineral samples were prepared as boric acid powder briquettes and then ARL Perform'X Sequential XRF as well as the Uniquant software was used to analyze both the test and reference mineral samples, respectively. This software was however set to detect all elements ranging from Na to U in the periodic table and report the elements above the limits of detection. Values were normalized, as there was no LOI in determining the crystal water and changes in oxidation state.

2.2.5. Determining work index using Berry and Bruce modified Bond method

The test and reference mineral ores of same mass were ground for same period of time (T) using a laboratory ball milling machine (Bwala *et al*., 2021). The input power (P) into the mill is constant and the input energy (E) remains the same for both test and reference mineral ores. Arithmetically, the modified Bond's work index equation is expressed in Equations 2 to 6 (Bwala *et al*., 2021):

$$W = P \times T; \text{ and } E = P \times T \tag{2}$$

where Work (W) = Energy (E); P = Power; T = Time. Equation 2 can thus be modified as:

$$W = 10 \times Wi \left(\frac{1}{\sqrt{P_{80}}} - \frac{1}{\sqrt{F_{80}}}\right) \tag{3}$$

Therefore, Equation 3 can be rewritten as:

$$W_t = W_r = Wi_t \left(\frac{10}{\sqrt{P_{t80}}} - \frac{10}{\sqrt{F_{t80}}}\right) \tag{4}$$

$$W_r = W_t = Wi_r \left(\frac{10}{\sqrt{P_{r80}}} - \frac{10}{\sqrt{F_{r80}}}\right) \tag{5}$$

Thus, Equation 4 and 5 can also be expressed as:

$$Wi_t = Wi_r \left(\frac{10}{\sqrt{P_{r80}}} - \frac{10}{\sqrt{F_{r80}}}\right) \Big/ \left(\frac{10}{\sqrt{P_{t80}}} - \frac{10}{\sqrt{F_{t80}}}\right) \tag{6}$$

where W = Work input, W_t = Work input for test mineral, Wi_t = Work index for test mineral, Wi = Work index, W_r = Work input for reference mineral, Wi_r = Work index for reference mineral, F_{r80} = 80% of feed for reference mineral, F_{t80} = 80% of feed for test mineral, P_{t80} = 80% of products for test mineral, P_{r80} = 80% of products for reference mineral.

2.2.6. Procedures involved in determining work index using Berry and Bruce method

The procedures adopted in determining the Berry and Bruce modified Bond's work index of the alluvial columbite mineral is represented in the following steps:

(a) 200 to 300g of both test and reference mineral samples were comminuted for about 60 mins in a laboratory ball milling machine.
(b) Hitherto, PSD analysis for both samples was determined using an automatic vibratory sieve shaker (King Test VB 200/300), with a number of different sieves sieving into several particle size fractions (+500–38 μm) for about 30 mins, before being fed into the ball mill for comminution. After comminution, both samples were also taken for PSD analysis, following same sieving procedure.
(c) Each particle size fractions obtained from PSD analysis before and after comminution for both tests and reference mineral samples were weighed on a sensitive mass beam balance and weights recorded and denoted as 'feed' and 'products' accordingly.
(d) The Berry and Bruce modified Bond's work index was therefore determined using the values obtained from the PSD analyses and calculations following the mathematically expression presented in Equation 6. Hitherto, the particle size passing 80% in the feeds and products for both test and reference mineral samples were calculated using 'Gaudin Schumann' formula in Equations 7 to 9 (Bwala *et al*., 2021).

$$P(X) = 100 \left[{}^{X}/_{K}\right]^{\alpha} \tag{7}$$

$$\text{where } \alpha = \frac{[\log P(X_2) - P(X_1)]}{\log (X_2) - (X_1)} \tag{8}$$

$$Size_2 = \frac{(\%\, passing\ size_2)^2}{(\%\, passing\ size_1)^2} \times size_1 \tag{9}$$

3. Results and discussion

3.1. Mineralogy and phase determination

The analytical test for identifying and quantifying the mineral phases was carried out as seen in Fig. 3. The XRD analysis conducted produced scattered X-ray peaks indicating crystalline materials. Each mineral in the ore produced a specific X-ray fingerprint of different intensities against a scattering angle.

This is regarded as the characteristic of the columbite mineral crystalline atomic structure as well as the presence of quartz and zirconia. The intensity of the various peaks indicated different compositions of different minerals ranging from ferro-columbite and/or columbite-tantalite (coltan), zirconia to quartz. The mineral sample showed high intensity peaks of quartz compared to the other mineral compositions. This indicates that the columbite mineral's major impurity is silica. Cassiterite and pseudo-rutile minerals are also present in somewhat lower quantities. However, it also showed the presence of ferro-columbite and zirconia in almost similar increased proportions. More so, the mineralogy and phase determination of the standard reference sample is reported elsewhere (Alabi et al., 2015, 2016).

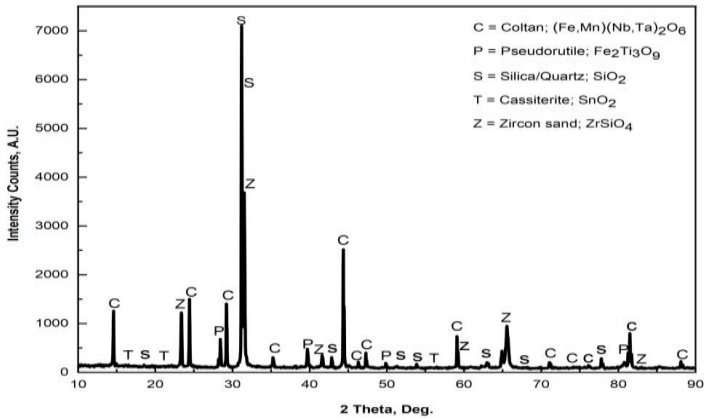


Fig. 3. Mineral phases of the Rayfield-Jos columbite test sample

3.2. Morphology and microstructure

Fig. 4 shows the microstructural and photomicrographical representation of the alluvial columbite from Rayfield-Jos using a scanning electron and optical microscope respectively. The SEM-EDS was carried out in order to investigate the mineral's surface morphological characteristics as well as determining the elemental composition of the columbite mineral. The examined SEM microstructure in Fig. 4i indicated significant amount of aggregate mineral particulates in particle sizes ranging mainly between 150 up to 500 μm, and of shapes varying from sub-hedral, sub-angular, spherical to sub-angular, as well as significant micropores found within the particles (in Fig. 4ii). Thus, in line with literature (Agulyansky, 2004; Ryan, 2018) and also confirmed by the chemical composition analysis carried out, it is evident that the major constituents are suspected to be columbite, silica and zircon sand, respectively. The OM photomicrographs in Fig. 4iii indicate the presence of silica, zirconia and columbite particles embedded in each other. This confirms results of the SEM test. In addition, the peaks from the semi-quantitative EDS analysis shown in Fig. 5 explained the presence of various elements, ranging from Nb, O, Si, Sn, Zr, Fe, Al, Ta, and Mn, with significant compositions at different spectra. Thus, from the EDS analysis in Fig. 5, showing two different spectra, it is however evident that the major elemental composition associated and dominating the columbite mineral includes Nb, Fe, as well as Si, which explains the mineral to be a silica-based alluvial ferro-columbite (Fe-columbite), and thus corresponds to the mineral phases, such as: columbite-tantalite (coltan), silica/quartz, zircon sand as well as pseudo-rutile, obtained from the ED-XRF analysis conducted. However, the morphology and microstructure of the standard reference sample can be found elsewhere (Alabi et al., 2015, 2016).

3.3. Chemical composition

For the objective of characterization, efficient mineral processing and subsequent metal extraction, the associated elements with Nb contained in a columbite-tantanlite (coltan) mineral deposit are to be considered, as they are regarded as the major factors contributing to the physicochemical properties/characteristics of the mineral sample (Adetunji et al., 2005; Habinshuti et al., 2021). Hence, proper characterization analyses is thus imperative, and therefore required to determine these associated elemental composition in the columbite or tantalite mineral sample brings about the feasible selection of suitable, efficient and appropriate processing/beneficiation methods. The chemical composition results are shown in Table 2 and 3.

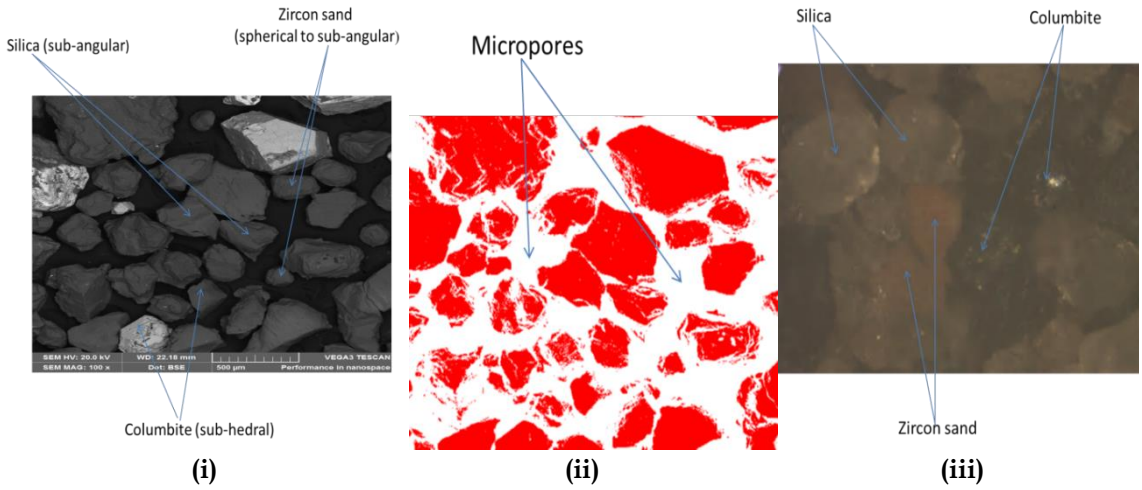


Fig. 4. Microstructural view of the Rayfield-Jos columbite test samples

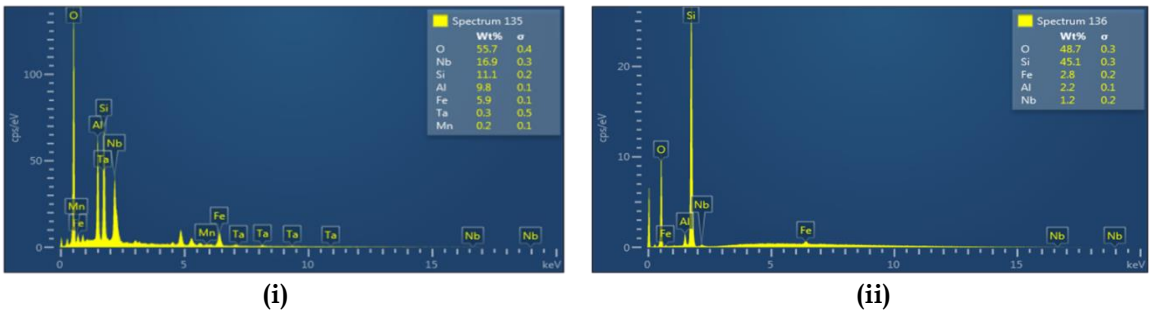


Fig. 5. EDS spectra of the Rayfield-Jos columbite test samples

Table 2. ED-XRF analysis of head sample of the test mineral from Rayfield-Jos

Elements	SiO_2	ZrO_2	Nb_2O_5	Ta_2O_5	Fe_2O_3	TiO_2	Al_2O_3	SnO_2	Others
Composition (wt %)	69.60	9.88	6.60	0.70	4.58	2.39	1.90	0.99	3.36

Others = oxides of Mn, Hf, P, Ce, Ca, Mg, Cr, W, Y, K and U

Table 3. ED-XRF analysis of head sample of the reference mineral from Gyel-Bukuru

Elements	SiO_2	ZrO_2	Nb_2O_5	Ta_2O_5	Fe_2O_3	TiO_2	Al_2O_3	SnO_2	MnO	Others
Composition (wt %)	27.00	14.70	6.71	0.79	23.10	22.20	4.20	6.47	1.01	*

*Others = trace amounts of oxides of Hf, V, Zn, Ca, W, Y, Ag, Rb, Pb, U and Th

From the ED-XRF chemical composition analysis of the test sample showing elements in oxide forms (as in Table 2), it can be seen that the mineral sample from Rayfield-Jos is a promising mineral deposit for the extraction of Nb. However, the mineral deposit is of low-grade, as it has been reported elsewhere that the Nb/Ta deposits with Nb_2O_5 and/or Ta_2O_5 composition of < 25% are termed as low-grade deposits (Adetunji et al., 2005; Alabi et al., 2016; Nzeh et al., 2022a, 2022b, 2023b). The test sample is mainly predominated by silicon dioxide (SiO_2), as well as zirconium dioxide (ZrO_2), niobium penta-oxide (Nb_2O_5), iron III oxide (Fe_2O_3), titanium dioxide (TiO_2), and oxides of aluminum (Al_2O_3) and tin (SnO_2) as major constituents with percentage compositions of 69.60%, 9.88%, 6.60%, 4.58%, 2.39%, 1.90% and 0.99% respectively. However, Ta_2O_5, HfO_2, P_2O_5, CeO_5, MnO, CaO, MgO, Cr_2O_3, WO_3, Y_2O_3, and K_2O were in somewhat minute compositions. The radioactive material (U_3O_5) was detected in trace amounts. More so, LOI was not determined during this analysis. In addition, the reference mineral sample was seen to possess almost similar chemical composition with the test mineral sample and thus, suitable to be used as a standard reference material.

In furtherance, it was also observed from the results that the ED-XRF analysis gave the percentage amount of Fe_2O_3 and MnO in the test sample to be 4.58% and 0.23%; Nb_2O_5 and Ta_2O_5, 6.60% and 0.70%, respectively, indicating the somewhat higher presence of Fe and Nb compared to Mn and Ta. Thus, with respect to the general chemical formula/composition of columbite mineral ore, $[(Fe,Mn)(Nb,Ta)_2O_6]$, and with silica dominating more in regards to the mineral content of the test sample, this simply confirms that the alluvial columbite test sample is more of a silica-based Fe-columbite mineral. It therefore explains the possibility of a relatively high magnetic field. Hence, the presence of Fe_2O_3 and other ferro-magnetic and strong paramagnetic responses such as TiO_2 and NiO compounds. Weak paramagnetic responses such as MnO, Nb_2O_5 and Ta_2O_5 were also present indicating significant difference in magnetism and the utilization of such material response differences for separation (Nete et al., 2012; Nete, 2009; Ayeni et al., 1999; Kimmel and Kitchell, 2003). The sample chemical composition analysis also showed the difference in density and specific gravity of the particles present in the columbite deposit. Hence, significant presence of heavy materials such as stannic oxide (SnO_2), haematite (Fe_2O_3), titania (TiO_2), zirconia (ZrO_2) and Nb_2O_5 amidst lighter materials such as silica (SiO_2) and aluminum (Al_2O_3), etc. were indicated, which however may be utilized further in separating each other (Ryan, 2018; Nete, 2009; Liu et al., 2016; Deblonde et al., 2016; Cook, 2000; Everistus, 2010; Ogbonna et al., 1999).

In addition therefore, the knowledge of the difference in their electrical conductive/electrostatic responses can also be utilized (Nete, 2009; Deblonde et al., 2016; Everistus, 2010; Ayeni et al., 1999; Kimmel and Kitchell, 2003). Thus, the material particles with varying density and specific gravity, magnetic, electrical and conductive responses therefore explains the possibility of exploiting their characteristics, upgrading and processing the columbite adopting certain physical separation techniques such as gravity, magnetic and electrostatic separations respectively for subsequent extraction and recovery of Nb. More so, the radioactive property (uranium) detected in very trace quantities was negligible. This was however less than the critical value (< 0.5 wt.%). This therefore is a key factor by which its absence enables better handling, transportation and reduces the process complexity and cost during Nb extraction/recovery (Zhang et al., 2017).

3.4. Particle size distribution analysis

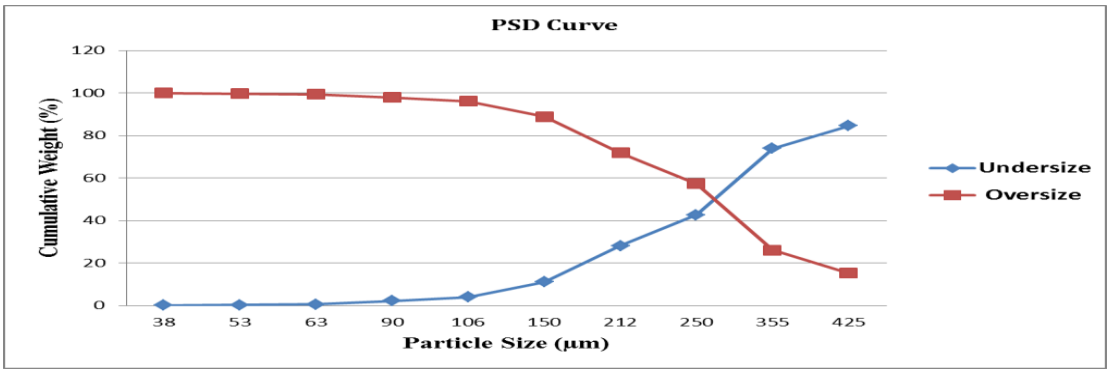
More essentially, the results of the PSD analyses of the test and reference before (feeds) and after (products) comminution in the ball mill are presented in the section. Tables 4 to 7, and Fig. 6 to 7 represent the PSD values and curves for the feed to ball mill and products from ball mill of the test and reference samples, respectively. However, -150+90 μm was essentially established as the mineral liberation size fraction range of the test sample.

Table 4. PSD analysis of test sample feed to ball mill

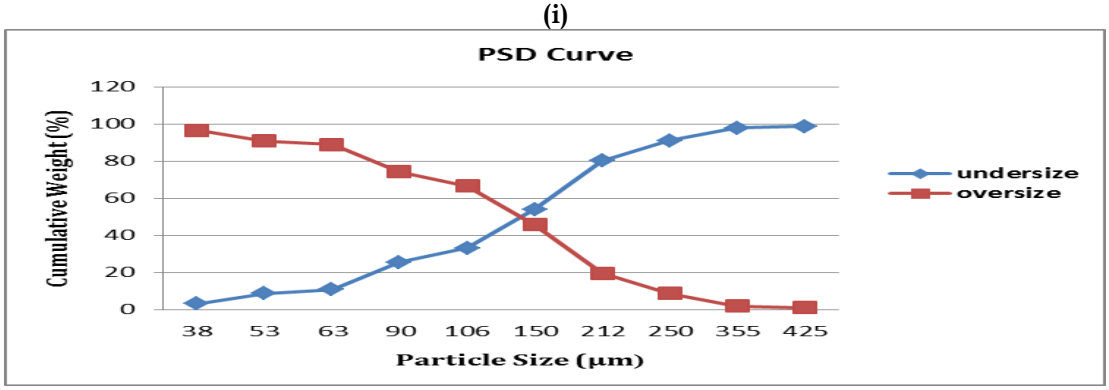
Particle size (μm)	Wt (g)	Wt (%)	Normal aperture (μm)	Cum. under-size (%)	Cum. over-size (%)
+425	45.6	15.38	425	84.61	15.38
-425+355	31.6	10.66	355	73.95	26.04
-355+250	92.8	31.31	250	42.64	57.35
-250+212	43.0	14.51	212	28.13	71.86
-212+150	50.6	17.07	150	11.06	88.93
-150+106	21.2	7.15	106	3.91	96.08
-106+90	5.2	1.75	90	2.16	97.83
-90+63	4.8	1.62	63	0.54	99.45
-63+53	0.8	0.27	53	0.27	99.72
-53+38	0.6	0.2	38	0.07	99.92
-38	0.2	0.07	-	-	-
Total	**296.4**	**99.99**			

Table 5. PSD analysis of test sample product from ball mill

Particle size (μm)	Wt (g)	Wt (%)	Normal aperture (μm)	Cum. under-size (%)	Cum. over-size (%)
+425	2.4	1.22	425	98.78	1.22
-425+355	1.6	0.81	355	97.97	2.03
-355+250	13.3	6.77	250	91.20	8.80
-250+212	21.3	10.83	212	80.37	19.63
-212+150	51.6	26.25	150	54.12	45.88
-150+106	40.8	20.75	106	33.37	66.63
-106+90	15.1	7.68	90	25.69	74.31
-90+63	28.9	14.70	63	10.99	89.01
-63+53	4.0	2.03	53	8.96	91.04
-53+38	11.0	5.60	38	3.36	96.64
-38	6.6	3.36	-	-	-
Total	**196.6**	**100.00**			



(i)



(ii)

Fig. 6. PSD curve of test sample **(i)** feed to ball mill **(ii)** product from ball mill

Therefore, using the mathematical expression by 'Gaudin Schumann' in Equation 9 and the PSD values from Tables 4 and 5, respectively, in order to determine the test sample 80% passing for feeds (F_{t80}) and products (P_{t80}):

$$F_{t80} = \left(\frac{80\%}{84.61\%}\right)^2 \times 425\ \mu m = \left(\frac{0.8}{0.8461}\right)^2 \times 425\ \mu m$$

$$\text{Thus, } F_{t80} = 0.8940 \times 425 = 379.95\ \mu m \text{ at } 80\% \text{ passing}$$

$$P_{t80} = \left(\frac{80\%}{80.37\%}\right)^2 \times 212\ \mu m = \left(\frac{0.8}{0.8037}\right)^2 \times 212\ \mu m$$

$$\text{Thus, } P_{t80} = 0.9908 \times 212 = 210.05\ \mu m \text{ at } 80\% \text{ passing}$$

Similarly, using the same Gaudin Schumann's expression and values from Tables 6 and 7 in order to obtain the 80% passing for feeds (F_{r80}) and products (P_{r80}) of the reference sample (Gyel-Bukuru columbite mineral) with known work index (Wi_r) of 2.72 kWh/ton and 3.42 kWh/ton (Alabi et al., 2015):

Table 6. PSD analysis of reference sample feed to ball mill

Particle size (μm)	Wt (g)	Wt (%)	Normal aperture (μm)	Cum. under-size (%)	Cum. over-size (%)
+425	49.0	16.39	425	83.61	16.39
-425+355	25.4	8.50	355	75.11	24.89
-355+250	57.4	19.20	250	55.91	44.09
-250+212	30.4	10.17	212	45.74	54.26
-212+150	64.0	21.41	150	24.33	75.67
-150+106	48.0	16.06	106	8.27	91.73
-106+90	13.3	4.45	90	3.82	96.18
-90+63	9.8	3.28	63	0.54	99.46
-63+53	0.9	0.30	53	0.24	99.76
-53+38	0.5	0.17	38	0.07	99.93
-38	0.2	0.07	-	-	-
Total	**298.9**	**100.00**			

Table 7. PSD analysis of reference sample product from ball mill

Particle size (μm)	Wt (g)	Wt (%)	Normal aperture (μm)	Cum. under-size (%)	Cum. over-size (%)
+425	10.7	5.57	425	94.44	5.57
-425+355	6.5	3.38	355	91.06	8.95
-355+250	22.8	11.87	250	79.19	20.82
-250+212	16.6	8.64	212	70.55	29.46
-212+150	39.0	20.30	150	50.25	49.76
-150+106	37.8	19.68	106	30.57	69.44
-106+90	16.8	8.75	90	21.82	78.19
-90+63	21.2	11.04	63	10.78	89.23
-63+53	2.2	1.15	53	9.63	90.38
-53+38	9.8	5.10	38	4.53	95.48
-38	8.7	4.53	-	-	-
Total	**192.1**	**100.01**			

$$F_{r80} = \left(\frac{80\%}{83.61\%}\right)^2 \times 425\ \mu m = \left(\frac{0.8}{0.8361}\right)^2 \times 425\ \mu m$$

Thus, $F_{r80} = 0.9155 \times 425 = 389.09\ \mu m$ at 80% passing

$$P_{r80} = \left(\frac{80\%}{79.19\%}\right)^2 \times 250\ \mu m = \left(\frac{0.8}{0.7919}\right)^2 \times 250\ \mu m$$

Thus, $P_{r80} = 1.0206 \times 250 = 255.15\ \mu m$ at 80% passing

Therefore, recalling and rewriting Equation 6, the work index of the alluvial columbite mineral (test sample), can be determined following the mathematical calculations in Equation 10:

$$Wi_t = Wi_r \left(\frac{10\sqrt{P_{r80}} - 10\sqrt{F_{r80}}}{10\sqrt{P_{t80}} - 10\sqrt{F_{t80}}}\right) \quad (10)$$

where work index of the reference mineral (Wi_r) = 2.72 kWh/ton, 80% of feed for test mineral (F_{t80}) = 379.95 μm, 80% of products for test mineral (P_{t80}) = 210.05 μm, 80% of feed for reference mineral (F_{r80}) = 389.09 μm, 80% of products for reference mineral (P_{r80}) = 255.15 μm.

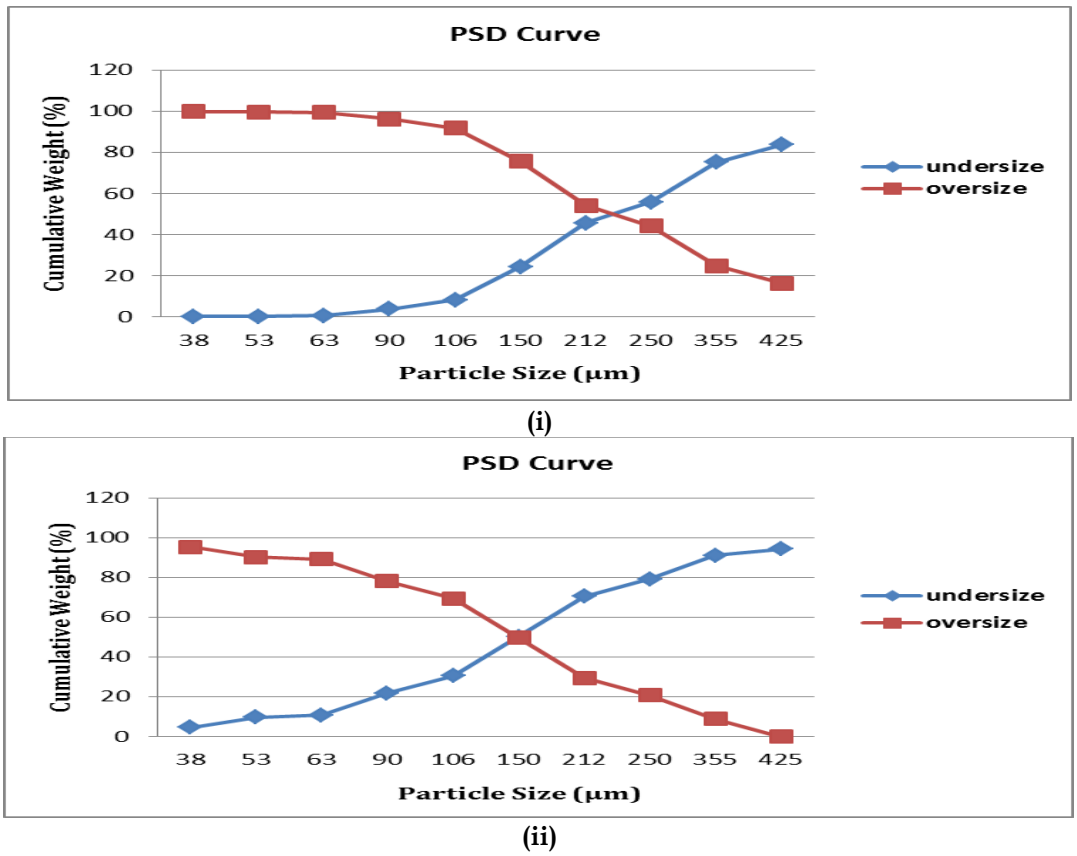


Fig. 7. PSD curve of reference sample (i) feed to ball mill (ii) product from ball mill

$$Wi_t = 2.72\left(\frac{10\sqrt{255.15}-10\sqrt{389.09}}{10\sqrt{210.05}-10\sqrt{379.95}}\right) = 2.0414 \text{ kWh/ton}$$

Also, using the reference mineral work index (Wi_r) value of 3.42 kWh/ton; while applying the same parameters and values to calculate the test mineral work index (Wi_t).

$$Wi_t = 3.42\left(\frac{10\sqrt{255.15}-10\sqrt{389.09}}{10\sqrt{210.05}-10\sqrt{379.95}}\right) = 2.5667 \text{ kWh/ton}$$

Thus, the milling work index (Wi_t) of the alluvial columbite mineral sample falls within the range of 2.0414 to 2.5667 kWh/ton and a mean Wi_t of 2.3041 kWh/ton was calculated. Therefore, recalling Equation 3, and with a known milling work index, the energy required to comminute or grind the columbite mineral to 80% passing can be determined (within the range of 0.3613 to 0.4543 kWh) by substituting the known value parameters into the Equation 3.

$$W = 10 \times 2.0414\left(\frac{1}{\sqrt{210.05}}-\frac{1}{\sqrt{379.95}}\right) = 0.3613 \text{ kWh}$$

$$W = 10 \times 2.5667\left(\frac{1}{\sqrt{210.05}}-\frac{1}{\sqrt{379.95}}\right) = 0.4543 \text{ kWh}$$

Therefore, the values in Tables 4 to 7 as well as the Figs. 6 to 7 displays the various results of the PSD analyses conducted on both the test (Rayfield-Jos alluvial columbite deposit) and reference (Gyel-Bukuru columbite ore) mineral samples. As a result of the presence of little rocky particles in the feed of the alluvial columbite deposit (test sample), a cumulative of 15.38% oversize mineral particles was retained on the coarsest sieve of 425 µm size. More so, about 84.61% of the sample undersized particles passed through the 425 µm sieve cumulatively. Similarly, 355 µm sieve retained sample particles of about 10.66% with undersize particles of about 73.95% passing. Also, 31.31 % was retained on the 250 µm sieve with about 42.64% passing of undersized particles. In the same way, 150, 90 and 53 µm sieves retained oversized sample particles of 17.07, 1.75 and 0.27% respectively; having about 11.06, 2.16 and 0.27% undersize particles passing accordingly. It was observed further that 0.2% particles of the columbite mineral sample were retained on the 38 µm sieve which was considered the finest sieve.

However, about 0.07% finer particles passed through the sieve and were received on the pan. This is presented in Table 4 and graphically in the PSD curve in Fig. 6i.

In contrast as regards to the under-size and over-size particles, the products realized after the milling process of the test columbite mineral sample had just about 1.22% cumulative mineral particles retained on the 425 μm sieve with about 98.78% cumulative particles passing through the sieve. About 2.03% of the mineral sample particles were also retained on the 355 μm sieve with 97.97% particles passing through it. 91.20% of the mineral particles however passed through the 250 μm sieve while 8.80% particles were retained. More so, 150, 90 and 53 μm sieves retained oversized sample particles of 45.88, 74.31 and 91.04%, accordingly; having about 54.12, 25.69 and 8.96% undersize particles passing, respectively. It was also observed that 96.64% particles of the columbite mineral sample were retained on the 38 μm sieve, considered as the finest sieve. Thus, about 3.36% finer particles passed through this very sieve and were retained on the pan. This is however illustrated in Table 5 and graphically in the PSD curve in Fig. 6ii.

The PSD tables and curves of both test and reference mineral samples in Tables 4 to 7 and Figs. 6 to 7, showed a normal pattern of mineral particle distribution which indicated that the mineral particle size fractions is in line with comminution theories. The results in Tables 4 and 5 indicated that the alluvial columbite mineral particulate can be defined generally within a variety of particle size fraction from 38 to 500 μm (-500+38 μm). Also, the PSD graphical representation or curve in Fig. 6 shows the cumulative weight % undersize and oversize of the columbite mineral particles plotted against the sieve sizes. Thus the results indicated the 80% mineral particles passing sieve size fractions of both feed and products of the test samples were 380 μm and 210 μm, respectively, which are in the sieve range of -425+355 and -212+150 μm sieve sizes. This explained that the test sample, alluvial columbite mineral deposits are relatively of moderately fine or fairly coarse mineral particles in size. A mesh of grind of 250 μm sieve size fraction was determined for the test sample as a liberation size was observed to be 150 μm sieve size fraction after the milling process. However, obtained from the PSD values and curve in Tables 6 and 7 and Fig. 7, 390 μm and 255 μm particle sizes were determined as the 80% mineral particles passing sieve size fractions for the feed and products of the reference mineral samples, respectively, which are in the sieve range of -425+355 and -355+250 μm sieve sizes.

The work index value of the alluvial Fe-columbite mineral deposits (test sample) was calculated to fall within the range of 2.0414 to 2.5667 kWh/ton. Hence, the energy consumed or required to comminute or grind the Fe-columbite mineral to 80% passing is expected to be within the range of 0.3613 to 0.4543 kWh. The work index results obtained in this study corroborates and corresponds with the results reported by Alabi et al. (2015). In comparison with the obtained results and data collated on the columbite mineral used as the standard reference material/mineral, the milling work index as well as the energy required in the comminution of the sample Fe-columbite deposit is somewhat low. This can however be attributed to the mineralogy of the mineral source and alluvial formation of the mineral reserve. Finally, the grindability results and PSD curve of the alluvial Fe-columbite mineral indicates that it can be considered a class of medium soft mineral type in terms of texture. Thus, it is imperative to note that the grindability or comminution characteristics and hence work index value varies with the particle size of the test mineral samples; since the grindability/comminution character and work index of most primary and secondary minerals is dependent on the average particle sizes of the minerals or ore deposits, which also has been reported by several researchers (Adetula et al., 2019; Alabi et al., 2015; Yaro, 1996; Nzeh et al., 2023c).

4. Conclusions

The grindability characterization and work index of an alluvial columbite mineral sourced and collected randomly from different mineral deposits at a mine-field in the localities of Rayfield, Jos South in Plateau state, Nigeria were investigated. The resulting data realized from this study investigation establish that the (alluvial) columbite sample is a silica-based ferro-columbite mineral deposit. Hence, the mineral deposit was efficiently characterized by its response to comminution. PSD curves were also generated and were used as an essential tool in determining the liberation size of the test mineral to fall within -150+90 μm particle sizes. Further, a range of work index values between 2.0414 to 2.5667 kWh/ton with a minimum mean value of 2.3041 kWh/ton was determined using the modified Bond's

method by Berry and Bruce. Thus, the minimum energy consumed or required to comminute or grind the alluvial Fe-columbite mineral deposit (test sample) is estimated to be between 0.3613 to 0.4543 kWh. This implies that the cost of energy consumed during the comminution process can therefore be estimated. Thus, the cost estimation of the energy consumed is determined by the product of a unit cost of energy by the total energy consumed during the process. It can then be concluded that a minimum average amount of 0.4078 kWh energy is required to grind one tonne of the columbite test sample from 80% passing 380 μm sieve size to a particle size allowing 80% passing 210 μm sieve size. This parameter is however relevant in the exploration, exploitation and characterization of mineral deposits, and may be utilized in the design system set up, suitable equipment selection for ore comminution and mineral processing, as well as a panacea in the development of an economic but effective beneficiation process route of Rayfield-Jos columbite mineral ore for efficient recovery and extraction of Nb and Ta metals.

Acknowledgements

The authors would like to acknowledge the Mineral Processing and Extractive Metallurgy Laboratory of the Chemical, Metallurgical and Materials Engineering Department in the Faculty of Engineering and the Built Environment, Tshwane University of Technology, Pretoria, South Africa. The authors appreciate Engr. Dr. Oladunni Oyelola Alabi and Engr. Prof Suleiman Bolaji Hassan of the Nigerian Metallurgical Society (NMS), for their assistance in the procurement of both test mineral and reference mineral samples utilized in this work. Special appreciation also goes to Dr. Swithin Nnabuchi Nzeh (GP United Kingdom; MBBS, MRCGP, Dip. Paed, RCPathME) as well as Ms. Theresa Chirenda, Engr. Annikie Ditshego Mashao, Engr. Godson Nnadozie Nzenwata, Mrs. Cecilia Nnenna Nzeh and Ms. Esther Sochikaima Stan-Nzeh for their very vital contributions towards the actualization and success of this study.

References

ABRAHAM, J. B., ANDREY, V. K., JOSEPH, K. B. , PÄR, J. G. (2012), *Characterization of Chemical Composition and Microstructure of Natural Iron Ore from Muko Deposits*, ISRN Materials Science. 2012:9. DOI: 10.5402/2012/174803.

ADEOTI, M. O., DAHUNSI, O. A., AWOPETU, O. O., ARAMIDE, F. O., ALABI, O. O., JOHNSON, O. T. , ADULKAREEM, A. S. (2019), *Determination of work Index of graphite from Saman-Burkono (Nigeria) using modified Bond's method*, Nigerian Journal of Technology, Vol. 38, No. 3, pp. 609-613.

ADETULA, Y. V., OZAH, B., ALABI, O. O., AJAYI, J. A. , AKOJA, A. (2019), *Determination of Work Index for Iperindo Lode Gold Deposit at Ilesha Goldfield Osun State, Nigeria Using Modified Bond Index*, American Journal of Materials Synthesis and Processing, Vol. 4, No. 1, pp. 37-42.

ADETUNJI, A. R., SIYANBOLA, W. O., FUNTUA, I. I., OLUSUNLE, S. O. O., AFONJA, A. A. , ADEWOYE, O. O. (2005), *Assessment of beneficiation routes of tantalite ores from key locations in Nigeria*, Journal of Minerals and Materials Characterization and Engineering, Vol. 4, No. 2, pp. 85-93.

ALABI, O. O, YARO, S. A., DUNGKA, G. T., ASUKE, F. , DAUDA, E. T. (2015), *Determination of Work Index of Gyel-Bukuru Columbite Ore in Plateau State*, Nigeria, Journal of Minerals and Materials Characterization and Engineering, Vol. 3, pp. 194-203.

ALABI, O. O., YARO, S. A., DUNGKA, G. T., ASUKE, F. , HASSAN, B. (2016), *Comparative beneficiation study gyel columbite ore using double stage (magnetic-to-magnetic and magnetic-to-gravity) separation techniques*, Journal of Minerals and Materials Characterization and Engineering 4, pp. 181-193.

ALABI, O. O. AND AWOGBAMILA, O. W. (2020), *Determination of grindability characteristics of Duguri (Nigeria) galena towards effective beneficiation process*, Science and Technology, 6(22), pp. 83-92.

ALABI, O. O., SHEHU Y. A. , BINTA, H. (2012), *Determination of Bond Index of Wasagu Manganese Ore in Kebbi State, Nigeria*, International Journal of Scientific and Engineering Research, Vol. 3, No. 10, pp 1-8.

AYENI, F. A., ALABI, O. O., OLA, S. O. (1999), *Beneficiation of Tailings*, Proceedings of the 16th Nigerian Metallurgical Society Annual Conference, Abuja, 3-5 November 1999, pp. 67-75.

AYENI, F. A., IBITOYE, S. A., ADELEKE, A. A. (2012a), *Development of a wet magnetic-gravity processing route to recover columbite from Jos minefield tailings dump, Nigeria*, Journal of Mining and Metallurgy, Vol. 48 A (1), pp. 63-72.

AYENI, F. A., MADUGU, I. A., SUKOP, P., IBITOYE, S. A., ADELEKE, A. A. , ABDULWAHAB, M. (2012b), *Secondary Recovery of Columbite from Tailing Dump in Nigerian Jos Mines Field*, Journal of Minerals and Materials Characterization and Engineering, Vol. 11, pp. 587-595.

BERRY, T. F. , BRUCE, R. W. (1966), *A Simple Method of Determining the Grindability of Ores*, Canadian Mining Journal, Vol.8, No. 7, pp 63-65.

BWALA, D. M., ABDULFATTAH, F., ALABI, O. O. , ADEWUYI, B. O. (2021), *Determination of work index of Filin Kokowa gold deposit in Toro Local Government, Bauchi State Nigeria*, Nigerian Journal of Technology, Vol. 40, No. 3, pp. 387-392.

COOK, N. J. (2000), *Mineral characterization of industrial mineral deposits at the geological survey of Norway: a short introduction*, NGU-BULL 436, pp. 189-192.

DENIZ, V. (2003*). Relationships between bond's grindability (gbg) and breakage parameters of grinding kinetic on limestone*, In Proceedings of 18th International Mining Congress and Exhibition of Turkey-IMCET, pp. 451-456.

DOLL, A. , BARRATT, D. (2011), "Grinding: Why so many tests?", In 43rd Annual Meeting of the, Canadian Mineral Processors, Ottawa, Ontario, Canada.

GBADAMOSI, Y. E., ALABI, O. O. , BORODE, J. O. (2021), *Evaluating the potentials of liberation size determination in Anka (Zamfara state, Nigeria) manganese ore and its comminution tendency using bond index technique*, Journal of Materials Science Research and Reviews, Vol. 8(1), pp. 7-18.

DEBLONDE, G. J. P., WEIGEL, V., BELLIER, Q., HOUDARD, R., DELVALLÉE, F., BÉLAIR, S. , BELTRAMI, D. (2016), *Selective recovery of niobium and tantalum from low-grade concentrates using a simple and fluoride-free process*, Separation and Purification Technology, 162, pp. 180-187.

GUPTA, A. , YAN, D. (2006), *Mineral Processing Design and Operation- An Introduction*, Amsterdam: Elsevier.

EVERISTUS, N. (2010), *Niobium Ore Mining and Processing in Nigeria: The Opportunities*, Nigeria Business Place, Abuja. pp. 1-3.

HABINSHUTI, J. B., MUNGANYINKA, J. P., ADETUNJI, A. R., MISHRA, B., OFORI-SARPONG, G., KOMADJA, G. C., TANVAR, H., MUKIZA, J. , ONWUALU, A. P. (2021), *Mineralogical and physical studies of low-grade tantalum-tin ores from selected areas of Rwanda*, Results in Engineering, Vol. 11, pp. 1-11.

HIGGINS, M. (1998), *Jksimblast – Blast Simulation and Management*, Presented At Blasting Analysis International Eighth HighTech Seminar, Nashville, Tennessee, pp. 1-9.

KIMMEL, J. L , KITCHELL, R. W. (2003*), Oxygen reduced niobium oxides*, United States Patent, US6576099B2, pp. 1-30.

LEVIN, J. (1992), *Indicators of grindability and grinding efficiency*, Journal of South Africa Institute Minerals and Metallurgy, Vol. 92, No. 10, pp. 283-290.

LIU, M., YOU, Z., PENG, Z., LI, X. , LI, G. (2016), *Enrichment of rare earth and niobium from a REE-Nb-Fe associated ore via reductive roasting followed by magnetic separation*, The Minerals, Metals & Materials Society, pp. 1-10.

LVOV, V. V. , CHITALOV, L. S. (2019), *Comparison of the different ways of the ball bond work index determining*, International Journal of Mechanical Engineering and Technology, Vol. 10(3), pp. 1180-1194.

LYNCH, A. J. (2015), *Comminution handbook*, Carton, Victoria, Australia Institute of Mining and Metallurgy, Vol. 6: pp. 43-49.

MAGDALIMOVIC, N. M. (1989), *Calculation for Energy required for grinding in ball mill*, International Journal of Mineral Processing, Vol 25, No. 2, pp 41-43.

MINDAT. (2021), *Rayfield, Jos south, Plateau state, Nigeria*, [Online], Available from: https://www.mindat.org/feature-2324561.html. [Accessed on: 25th August, 2021].

NAPIER-MUNN, T. J., MORELL, S., MORRISON, R. D , KOJOVIC, T. (1996), *Mineral comminution Cirtcuit, their operation and optimizatio*", JKMRC Monograph Series in Mining and Mineral Processing 2, University of Queensland Australia. 2, 143-154.

NETE, M. (2009), *Dissolution and analytical characterization of tantalite ore, niobium metal and other niobium compounds*, [Published Dissertation]. University of the Free State, Bloemfontein, pp. 1-22.

NETE, M., PURCELL, W., SNYDERS, E., NEL, J. T. , BEUKES, G. (2012), *Characterization and alternative dissolution of tantalite mineral samples from Mozambique*, The Journal of the Southern African Institute of Mining and Metallurgy, Vol. 112, pp. 1079-1086.

NNAEMEKA S. NZEH, PATRICIA POPOOLA, SAMSON ADEOSUN, ABRAHAM ADELEKE. (2023a). *Characterization of Rayfield-Jos Columbite Deposit for Efficient Beneficiation and Recovery of Niobium and Tantalum*.

The Minerals, Metals & Materials Society 2023: Characterization of Minerals, Metals & Materials 2023, The Minerals, Metals & Materials Series. pp. 191-206. https://doi.org/10.1007/978-3-031-22576-5_18.

NNAEMEKA STANISLAUS NZEH, MAITE MOKGALAKA, NTHABISENG MAILA, PATRICIA POPOOLA, DANIEL OKANIGBE, ABRAHAM ADELEKE , SAMSON ADEOSUN. (2023b). *Pyrometallurgical approach in the recovery of Niobium and Tantalum. Pyrometallurgy – New Perspectives, Intechopen*, pp. 1-23.

NORAZIAH, C., AB. AZIZ, A. , GRATIM, H. (2016), *An Ambient Agent Model for Analyzing Managers' Performance During Stress*, In: 2016 International Conference on Applied Science and Technology (ICAST 2016), Kedah, Malaysia, 11-13.

NZEH, N. S., ADEOSUN, S., POPOOLA, A. P., ADELEKE, A. , OKANIGBE, D. (2022a), *Process applications and challenges in mineral beneficiation and recovery of niobium from ore deposits–A review*, Mineral Processing and Extractive Metallurgy Review, Vol. 43, No. 7, pp. 833-864.

NZEH, N. S., POPOOLA, A. P. I., ADELEKE, A. A. , ADEOSUN, S. O. (2022b), *Factors and challenges in the recovery of niobium and tantalum from mineral deposits, recommendations for future development–A review*, Materials Today: Proceedings, Elsevier, Vol. 65, pp. 2184-2191.

NZEH, N. S., POPOOLA, A. P. I., OKANIGBE, D., ADEOSUN, S. O., ADELEKE, A. A. 2023c. *Work index evaluation of Rayfield-Jos columbite mineral for effective processing and recovery of niobium and tantalum*. Materials Today: Proceedings, Elsevier, pp. 1-8. DOI: https://doi.org/10.1016/j.matpr.2023.08.059.

OGBONNA, A. I., NWAKAUDU, S. M. , ONYEMOBI, O. O. (1999), *Strategic Mineral Deposits in Nigeria (The Neglected Case of Niobotantalites)*, Proceedings of 16th Nigerian Metallurgical Society Annual Conference, Abuja, October 1999. pp. 67-75.

ONEMINE (2010), *Summary and determination of Bond Work Index using an Ordinary Laboratory Batel Ball Mill*, http://www.onemine.org/serch//summary.emf. On 14/04/2021.

RIANTIE, A., WAHYUNI, N., NORA, I. (2013), *PemiscihTimbal (Pb) Dalam Galena Dengan Metode Flotasi Manggunakan Deterjen*, Positron, 3(1):01 -05.

RYAN. (2018), *Facts about niobium: Production, properties, applications, and availability*, ADMAT Refractory Metals, [Online], Available from: https://www.admatinc.com/facts-about-niobium-production-properties-applications-and-availability. [Accessed on: 27th June, 2020].

SIDDALL, B., HENDERSON, G. , PUTLAND, B. (1996), *Factors Influencing Sizing Of SAG Mills From Drill Core Samples*, Proc. Conf. International AG And SAG Grinding Technology, UBC, Vancouver, Canada, Vol. 2, pp. 463.

THOMAS, D., ASUKE, F. , YARO, S. (2014), *Determination of Some Conceptual Mineral Processing Parameters of Soba-Wanka Pyrochlore-Col Tan Mineral Ore Deposit*, in Nigeria Engineering Conference, pp. 32-41.

TODOROVIC, D., TRUMIC, M., ANDRIC, L., MILOSEVIC, V. , TRUMIC, M. (2017), *A quick method for bond work index approximate value determination*, Physicochemical Problems of Mineral Processing, Vol. 53(1), pp. 321-332.

WHITTLES, D. N., KINGMAN, S. W. , REDDISH, D. J. (2003), *Application of numerical modelling for prediction of the influence of power density on microwave-assisted breakage*, International Journal of Mineral Processing, Vol. 68, pp 71-91.

WILLS, B. A. , FINCH J. (2015), *Will's Mineral Processing Technology*, 8th Edition, Butterworth-Heinemann, 8,. 212-234.

WILLS, B. A. , NAPIER-MUNN, T. J. (2006), *Will's mineral processing technology - An introduction to the practical aspects of ore treatment and mineral recovery*, Elsevier Science and Technology Books, 7th Edition.

YARO S. A. (1996), "Grindability Test For Ririwai Lead-Zinc Complex Ore", Journal of Nigeria Mining.

YERIMA, M. L. , ABDULRAHMAN, A. S. (2015), *Physiochemical Analysis of Chanchaga Ore, North Central Nigeria*, Journal of Applied Sciences 15 (7), pp. 1020-1025.

ZHANG, Y., DU, M., LIU, B., SU, Z., LI, G. , JIANG, T. (2017), *Separation and recovery of iron and manganese from high-iron manganese oxide ores by reduction roasting and magnetic separation technique*, Separation Science and Technology, Vol. 52, No. 7, pp. 1321-1332.