Comminution Behavior and Magnetism Study of Vanadium Ore from the South African Northern Limb of the Bushveld Igneous Complex

Antoine F Mulaba- Bafubiandi[1,2], and Malefo Moroka[1]

***Abstract*—**The observed energy needs promoted by the conversion and usages of electric vehicles called for an extensive exploitation and beneficiation of vanadium bearing minerals as vanadium redox battery are developed. This paper discusses the mineralogical composition of the vanadium bearing minerals contained in the ferrotitanovanadium ores from the Langa reserves (South Africa) as analyzed with XRD, XRF and SEM and the Mossbauer spectroscopy. The characterization and magnetism study helped to decide on the downstream usage of magnetic separation as concentration means after the optimized comminution assisted by Microwave pre-processing was conducted. It is observed from the vanadium recovery versus grind experiment that the 80% passing a lower screen size of 75µm at higher milling time of 25 minutes and 80% passing the higher screen size of 1000µm at lower milling time of 5minutes.And for milling time of 10 minutes, the recovery of vanadium bearing minerals is 33% . Results also show that the increase in recovery from 22% to 81% as milling time increases from 5 minutes to 25 minutes. And for 10 minutes milling time, 80% passing particle screen size of 212µm. From recovery versus grind graph of feed particle size less than 2mm, It is noticed that P80 curve shows the 80% passing a lower screen size of 3×10^-30µm at higher milling time of 25 minutes and 80% passes the higher screen size of 212µm at lower milling time of 5minutes. And for milling time of 10 minutes it shows that recovery of vanadium bearing minerals is 77%. The recovery curve shows that the increase in recovery from 57%% to 94% as milling time increases from 5 minutes to 25 minutes. And for 10 minutes milling time, 80% passing particle screen size of 75µm. It is observed that the energy consumption varies with milling time and feed particle size. From the results presented, total energy consumed increases as milling time increases and feed size particles increases. This can be seen from the particles size of greater than 2mm have higher energy consumption of 0,0032Kw/h and particle size that are less than 2mm shows energy consumption of 0,029 at time increasing

***Keywords*—** Langa reserves (South Africa milling curve, vanadium bearing minerals,), Vanadium redox battery**.**

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I. Introduction

Milling is a critical industrial operation found in mineral, metallurgical, power generation, and chemical sectors, and it consumes a significant amount of energy, contributing substantially to overall plant operational expenses. The main objective of milling in the context of Platinum Group Minerals (PGMs) is to reduce the size of materials, primarily to release the valuable constituents that are locked within the surrounding gangue material. Once the material has been reduced to a size that allows for adequate liberation of these valuable components, subsequent processes such as magnetic separator can effectively separate these valuable elements from the ore. The size reduction procedures, particularly the final grinding stage that occurs just before the concentration process, represent the primary step in preparing the feed for the concentration process.

Moreover, choice of concentration process and techniques plays a crucial role in liberating ferrovanadium ore. Optimal methods need to be selected to address the specific challenges presented by the ore's composition and characteristics. In this paper will be determining the mineralogical composition of the Vanadium bearing minerals contained in the ferrotitanovanadium ores as analyzed with XRD, XRF and SEM and the FTIR. The characterization and magnetism study helped to decide on the downstream usage of magnetic separation as concentration means after the optimized comminution process specifically milling.

In order to optimize the milling stage, requirements of the magnetic separation in terms of particle size have to be fully understood first, since either over-grinding or low liberation leads to the reduction in the amount of vanadium bearing minerals required recovered. This would be done by running experiments by varying the parameters of milling process.

II. THEORETICAL BACKGROUND

A. Vanadium ore classification

Vanadium ore deposits are classified into several types, including vanadiferous (titano) magnetite deposits, sandstone-hosted (u)-vanadium deposits, calcrete-hosted (u)-vanadium deposits, vanadate deposits, and deposits associated with crude oil, coal, and shales. The primary vanadium ore source is vanadiferous (titano) magnetite deposits, which are characterized by rutile, ilmenite, and titanomagnetite hosts. Sandstone-hosted (SSV) deposits are characterized by calcrete-hosted uranium and vanadium ores, with the Colorado Plateau being the top producer. Vanadate deposits, formed when lead, copper, and zinc sulfide orebodies underwent supergene alteration, are more prevalent in arid climates. Vanadium is also associated with deposits of crude oil, coal, and shale, with concentrations in asphaltene ranging from 1,200 to 5,000 ppm. Vanadium deposits linked to graphite can be found in metamorphic terranes, bauxites, laterites, supergene iron ores, laterite, phosphate, and bauxite ore .

B. Vanadium reserves in the world

Global vanadium resources were projected to be 63 million metric tons in 2012, with 14 million metric tons of reserves. The primary source of vanadium is vanadiferous titanomagnetite (VTM) deposits, which are magmatic accumulations of ilmenite and magnetite [. Vanadium-titanium magnetite is primarily found in Russia, South Africa, China, the US, Canada, Norway, Finland, Sweden, India, Australia, and New Zealand. China, Russia, New Zealand, Austria, and formerly South Africa and Finland use vanadium slag for extraction.

C. Vanadium reserves in Africa

Vanadium deposits and occurrences in Eastern Africa include the Kabanga-Musongati alignment in Burundi, Ethiopia, Kenya, Madagascar, and Madagascar. Burundi has nine main stratigraphic units with Fe-Ti-V oxide deposits, while Ethiopia has three types of iron deposits with potential vanadium content. Kenya's Mozambique Belt contains gneiss with scapolite, vanadian zoisite, vanadian diopside, vanadian sphene, and vanadian magnetite, as well as green vanadium grossular porphyroblasts. Madagascar's Green Giant and Molo deposits in the Tulear Region have significant vanadium-graphite concentrations ..

In Malawi, sandstone-hosted uranium mineralizations are found in Karoo sediments, while in Mozambique, the Montepuez Graphite Project contains graphite-bearing mica schist and gneiss with variable amounts of vanadium. In Somalia, surficial uranium-(vanadium) deposits are found in the valley-fill calcrete type. Tanzania's Mozambique Belt contains Neoproterozoic high-grade metasedimentary suites, while Uganda's Bukusu alkaline complex contains magnetite ore with low levels of vanadium. Western Africa's Angola is rich in iron ore resources, with the Kassala and Kitungo deposits being the most significant 4].

D. Vanadium Reserves in South Africa

South Africa's vanadiferous (titano) magnetite (VTM) deposits are the world's largest vanadium source, consisting of magmatic accumulations of (titano)magnetite and ilmenite. The Bushveld Complex, located in the North-West Limpopo and Mpumalanga Provinces, is known for housing the largest chromite deposits and associated platinum group metals. Mining operations are significant in these deposits .

E. Vanadium application

Vanadium, a versatile transition metal with high strength, corrosion resistance, and the ability to form alloys with other metals, has numerous applications across various industries.

Steel Alloys:

Vanadium-containing steels are utilized in construction, automotive others for their enhanced strength and toughness, making them suitable for building structures, bridges, and aircraft components. [1].

Energy Storage:

Vanadium is a key component in vanadium redox flow batteries (VRFBs), a large-scale energy storage technology utilizing renewable energy sources like wind and solar power. [2].

C1123008Vanadium is used as a deoxidizer and desulfurizer in the production of certain ferrous and non-ferrous alloys. It can help improve the quality and properties of metals during the smelting process [1].

Characterisation

X-ray Fluorescence, X-ray Diffraction, and Scanning Electron Microscopy are powerful analytical techniques used to analyse ores and minerals, providing valuable information on composition, structure, and morphology.:

X-ray Fluorescence (XRF):

X-ray Fluorescence is a non-destructive analytical technique widely used in the mining and metallurgy industries to determine the elemental composition of ores. It operates on the principle that when a sample is irradiated with high-energy X-rays, it emits characteristic X-ray fluorescence signals unique to its constituent elements. Several studies have explored the application of XRF in ore analysis. (Seiler S, et al) investigated the use of energy-dispersive XRF to analyse, including iron, copper, and gold ores. The study highlighted the accuracy and rapidity of XRF in quantifying elemental compositions. [3] focused on the study the distribution and quantification of trace biological elements in different environmental matrices using wavelength-dispersive XRF. The research emphasized the importance of calibration and sample preparation in obtaining accurate results. [4] discussed the advancements in portable and handheld XRF instruments, making in-situ analysis of ores more accessible and convenient for mining and exploration.

X-ray Diffraction (XRD):

X-ray Diffraction is a technique used to identify the mineralogical composition and crystalline structure of ores. XRD provides valuable information about the minerals present in an ore sample, their relative abundance, and their crystallographic characteristics: [5] presented a comprehensive review of XRD applications in ore characterization. The study discussed the identification of mineral phases and the quantification of mineralogical composition. [6].explored the use of XRD in analysing the mineralogical composition of cassiterite-bearing skarn ore to determine the efficiency of magnetic separation. The research demonstrated the utility of XRD in monitoring ore processing operations. Moreover, [7] has discussed the combination of XRD with other techniques, such as XRF and SEM, to provide a more holistic understanding of ore samples, enabling comprehensive characterization.

Scanning Electron Microscopy (SEM):

SEM is a microscopy technique that provides high-resolution images and allows for the examination of the morphology and microstructure of ore samples at the microscopic level. SEM is often used in conjunction with other analytical methods for a complete ore analysis:
[8] discussed the use of SEM for investigating the texture, grain size, and mineral associations in various ore samples. The study emphasized the importance of sample preparation and imaging parameters for accurate results. According to , the application of SEM-EDS (Energy Dispersive X-ray Spectroscopy) for the analysis of ore samples, can successfully determine both morphological and elemental information. The research highlighted the versatility of SEM-EDS in ore characterization. [9] focused on the integration of SEM with other techniques like XRD and XRF for a comprehensive understanding of clayey ceramics containing manganese ore tailings, allowing researchers to link mineralogical and chemical information with microstructural data.

Rod milling.

Rod milling is a form of comminution, which involves the mechanical reduction of material size9 [10]. In rod milling, cylindrical rods are used as grinding media within a rotating drum. The rods are generally made of steel and range in length from several inches to several feet. As the drum rotates, the rods tumble and crush the material, reducing its size through a combination of impact and attrition.

Equipment

The primary equipment used in rod milling is the rod mill, a rotating cylindrical mill with an internal lining, where the grinding action takes place. The mill is usually driven by a motor and can operate in wet or dry conditions. It may have a discharge mechanism to control the flow of milled material [11].

Rod milling is a widely used process in various industries, including ore grinding, aggregate production, chemical processing, and raw material preparation. It is crucial to consider factors such as rod charge, mill speed, feed size, liner design, and product size control when milling. The L/D ratio, the ratio of rod length to mill diameter, plays a significant role in determining grinding efficiency [12]. Mill speed is also crucial, as it impacts the grinding action and the material being milled. Feed size also influences the product size and overall efficiency. Liner design, the internal lining of the mill, protects the shell and affects the grinding action. The choice between wet and dry grinding depends on the material's nature and specific process requirements. Overall, rod milling is a versatile and efficient method for various applications.

Efficiency and Energy Consumption:
Rod milling is known for its efficiency in achieving fine particle sizes [13]. However, the energy consumption can be significant. Optimizing operational parameters, such as rod charge and mill speed, is crucial to achieve high efficiency and minimize energy consumption.
Rod milling calculations involve various parameters and equations to determine key aspects of the rod milling process, including critical speed, power consumption, and product size distribution [10]. Below, I'll outline the calculations commonly used in rod milling:

1. Critical Speed Calculation:

The critical speed of a rod mill is the rotational speed at which the centrifugal force on the rods equals the gravitational force. It is expressed in revolutions per minute (RPM). The formula for calculating the critical speed of a rod mill is [13]:

Equation 1 Bond work index

$$\omega = \frac{48{,}3}{\sqrt{R-r}}$$

Where:
ω the critical speed (in RPM), R is the radius of the mill (in meters) and r is the radius of the rods
2. The Bond rod mill work index (RWI) is calculated using the following formula [15]:

Equation 2 Bond work Index

$$E = BWI\left(\frac{10}{F80} - \frac{10}{P80}\right.$$

Where:
E the energy required to grind the sample per mass (in kWh/t), RWI is the Bond rod mill work index (in kWh/t), P80 is the product size in micrometres (µm) at which 80% of the material passes in the product distribution and F80 is the feed size in micrometers (µm) at which 80% of the material passes in the feed distribution.
3. Mill Load Calculation:
The mill load, expressed as a percentage, represents the proportion of the mill's volume occupied by grinding rods and the material being milled (Deniz V,20130. It can be calculated using the formula:

$$Mill\ load(\%) = \frac{(Media\ volume + Material\ volume)}{Mill\ volume} \times 100$$

Where:
Rod Volume is the volume of grinding rods, Material Volume is the volume of the ore or feed material Mill Volume is the total internal volume of the rod mill
The equation for material volume can be calculated use below equation (Deniz V,2013):

$$Material\ volume = \frac{Material\ volume}{Material\ density}$$

Therefore, J can be calculated from the formula below (Deniz V,2013):

$$J = \frac{Rods\ mass(bulk\ density)}{Mill\ volume} \times \frac{1}{0,6}$$

Also, to calculate the f_c use the formula below (Deniz V., 2013)

$$f_c = \frac{Material\ mass(bulk\ density)}{Mill\ volume}$$

The following calculation is then for calculating U amount of material to be fed into the milling vessel [16]

Equation 3 Mill filing calculations

$$U = \frac{f_c}{0,4J}$$

Where:

F_c is the Material volume based on mill volume and J is the fraction volume occupied by media bed

Then finally to calculate number of rods to be used equation below:

$$n = \frac{J}{volume\ per\ rods}$$

Product Size Distribution Calculation:

The product size distribution is determined experimentally by conducting particle size analysis tests. Various methods, such as sieve analysis or laser diffraction, are used to calculate the particle size distribution of the final product after milling. These calculations are essential for optimizing operational parameters, ensuring energy efficiency, and achieving the desired product quality.

Below is the formula of square root equation used to calculate descending order of screen size for preparation of polymer screens [17]:

$$Y = \frac{x}{\sqrt{2}}$$

Where: Y is screen following the upper screen in descending order and X is the bigger screen.

III. RESEARCH METHODOLOGY

A positivism research philosophy was utilized on this research project as there was need of data collection which is qualifiable for the establishment of data which were necessary for XRF, XRD, FTIR and PSD experiments data of vanadium ore.

A deductive approach was the path of this research project, in which previous assumptions were examined on the PSD and magnetism study on vanadium ore. This approach will have a significance in the understating of the topic statement and the research questions and research problem.

The research project was developed under the quantitative and qualitative approach which evolves under controlled laboratory experiments and collecting quantitative data, where different types of information such, composition, morphology, microstructure, grain size form analytical technique and also graphs, numerical values, sizes of milling (pulverizing, crushing and particle sizes) were analysed and be delivered as data. Statistical data analytical was used to analyse the data of the experiment, of which will later deviate into quantitative data.

The research strategy that was utilized is an experimental research strategy, as this particular project based on experiment analysis. Data will be collection and analysis with fixed of variables. For example, the product size distribution is determined experimentally by conducting particle size analysis tests. Moreover, for milling few grinding experiments have been ran for comparison

The time horizon was cross sectional, because the ore sample was initially crushed and pulverized for characterization, which was used to determine the optimal parameters of milling process. And data was collected not after a long period of time compared to longitudinal time horizon. The experiment and data collection of the study of particle size distribution on vanadium ore.

The data collected will primary data which was derived from the conducted experiment. For example, particle size distribution, milling curve, elemental composition and energy consumption curve

Data collection of the research project was done with an experiment that is controlled in a laboratory. Also results from previous literature review on the PSD study of vanadium ore using from was collected as data to optimized with own inputs and to compare the new data done on the experiment and its realism.

TABLE I. MATERIALS AND EQUIPMENT USED

Equipment and material	Functions
Magnetism detector	Analysis of magnetism of ore
Rod mill	To grind the ore
Sieves for screening	To screen particle size
Crusher (jaw crusher and cone crusher)	comminution of the ore
Water	For wet milling
X-ray Fluorescence (XRF)	Elemental composition of sample
X-ray Diffraction (XRD)	Mineral composition of sample
Jones Spinning riffle	Sampling
Scanning Electron Microscope (SEM)	used to examine the surface morphology
Drying oven	To dry resulted cake from pressure filter
Pressure filter	Separating water from sample

1. *Experimental set up*

Initially the ore sample of 4.8kg was crushed to particles size ranges between 5mm and 0,075mm. Therefore, it was sampled into four samples of 1200g, one sample of 1200g was sampled again into 6 samples of 200g. This samples where then screened of which resulted into two samples of different size particle. From each sample of different size particle were samples 3 sample of 200g, resulting in 6 sample of which we ground using Rods milling. Moreover, 3 samples each of 206 were obtained and characterized using XRF, XRD AND SEM, of which their result were used to optimized mill parameters. After milling for different milling time 25min, 10min and 5, 2 set of the three sample of particle size less than 2mm and other of particle size greater than 2mm. The milled product was screen 10 minutes each and particle size distribution was determined and there for milling curve were constructed form PSD.

After experiments, the data was collected and analysed including XRF, XRD, SEM, milling experiments.

IV. RESULTS AND DISCUSSION

XRF RESULTS

TABLE II. ELEMENTAL ANALYSIS OF THE AS RECEIVED MATERIAL

Component	Unit	Result	Judgement
MgO	mass%	0.0296	
Al2O3	mass%	1.9574	
SiO2	mass%	2.3142	
P2O5	mass%	0.0138	
SO3	mass%	0.2667	
Cl	mass%	0.0336	
K2O	mass%	0.0123	
CaO	mass%	0.0433	
TiO2	mass%	8.3604	
V2O5	mass%	1.7155	
Cr2O3	mass%	0.0894	
MnO	mass%	0.1838	
Fe2O3	mass%	84.7291	
NiO	mass%	0.1717	
CuO	mass%	0.0311	
ZnO	mass%	0.0269	
PbO	mass%	0.0213	

The XRF result, table 2, show elemental composition by metal oxide mass % with the abundance of Fe 84,7291%, Ti 8,3604%, Si 2,3142%, Al 1,9574% and v 1,7155%.

XRD results

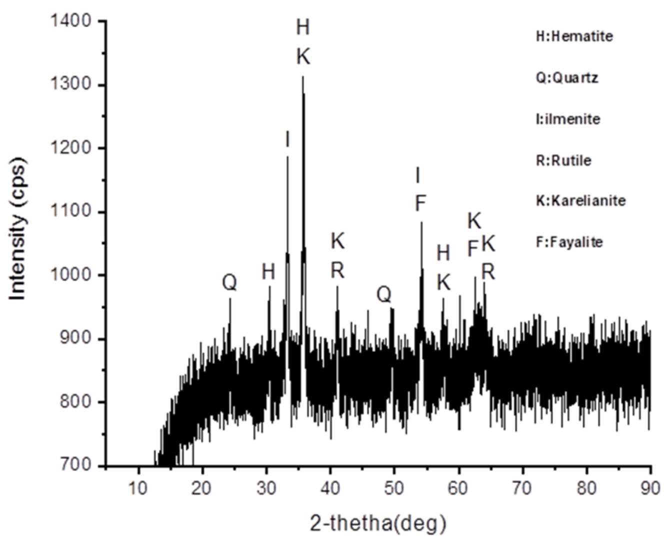
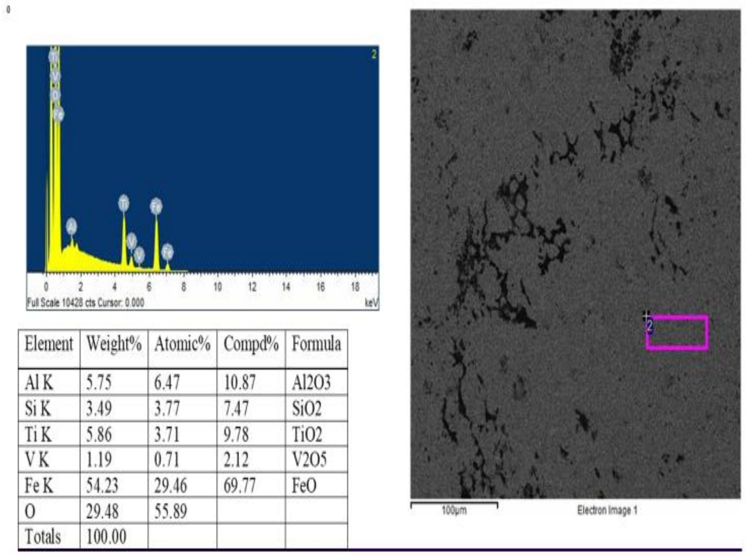


Fig. 1. XRD results of the as received sample.

The XRD shown high peaks of hematite following by ilmenite, karelianite, fayalite and then quartz. This show that the ore mostly compost of iron bearing minerals and that vanadium bearing minerals are less. It is shown that the width varies from peak to peak.

SEM results



Element	Weight%	Atomic%	Compd%	Formula
Al K	5.75	6.47	10.87	Al2O3
Si K	3.49	3.77	7.47	SiO2
Ti K	5.86	3.71	9.78	TiO2
V K	1.19	0.71	2.12	V2O5
Fe K	54.23	29.46	69.77	FeO
O	29.48	55.89		
Totals	100.00			

Fig 2. SEM micrograph and EDS of seleted points ontheas received sample

The SEM-EDX analyses were used to determine the morphological structure and the percentage composition of each element in the ferrovanadium ore. The SEM-EDX graphical images of the ferrovanadium ore showed microcrystalline, intergrown grain crystal-like particles, which were uniform and clustered. The elemental composition of these microcrystalline particles was composed of mainly V, O, Al, Si, Ti and Fe with the percentage weight content in the order of V(1.19wt%) <Si(3.49wt%) <Al(5.75wt%) <Ti(5.86wt%) <O(29wt%) <Fe(54.23wt%). Moreover, the analysis shown grain size that ranges between 100µm and 70µm. Below is the results of recovery and grind for feed particle size greater than 2mm.

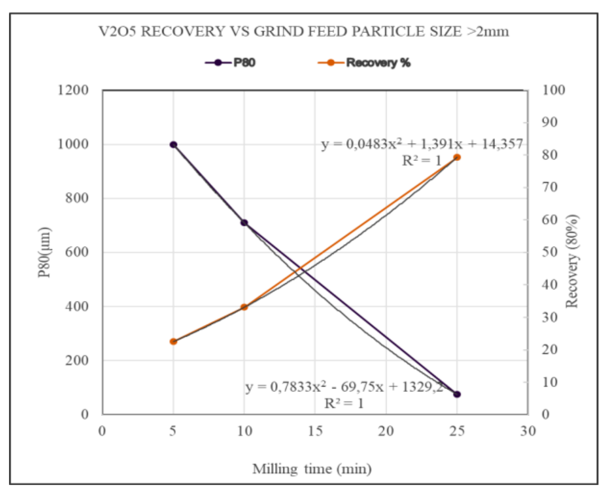


Fig.3. Recovery of V_2O_5 in function of grind feed particle size bigger than 2mm

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It Is discovered from the V recovery vs Grind graph that the 80% passing a lower screen size of 75µm at higher milling time of 25 minutes and 80% passes the higher screen size of 1000µm at lower milling time of 5minutes. And for milling time of 10 minutes it shows that recovery of vanadium bearing minerals is 33%. From the recovery curve it is show that the increase in recovery from 22% to 81% as milling time increases from 5 minutes to 25 minutes. And for 10 minutes milling time, 80% passing particle screen size of 212µm. Below is the results of recovery and grind for feed particle size less than 2mm.

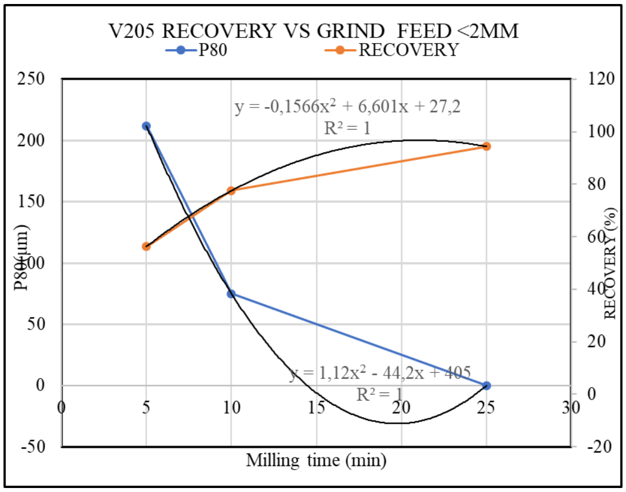

Fig. 4. Vanadium recovery versus milling time

From recovery vs Grind graph of feed particle size less than 2mm, It Is discovered that P80 curve shows the 80%passing a lower screen size of 3×10^-30µm at higher milling time of 25 minutes and 80% passes the higher screen size of 212µm at lower milling time of 5minutes. And for milling time of 10 minutes it shows that recovery of vanadium bearing minerals is 77%. From the recovery curve it is show that the increase in recovery from 57%% to 94% as milling time increases from 5 minutes to 25 minutes. And for 10 minutes milling time, 80% passing particle screen size of 75µm.

Below is the graph showing energy consumption against milling time.

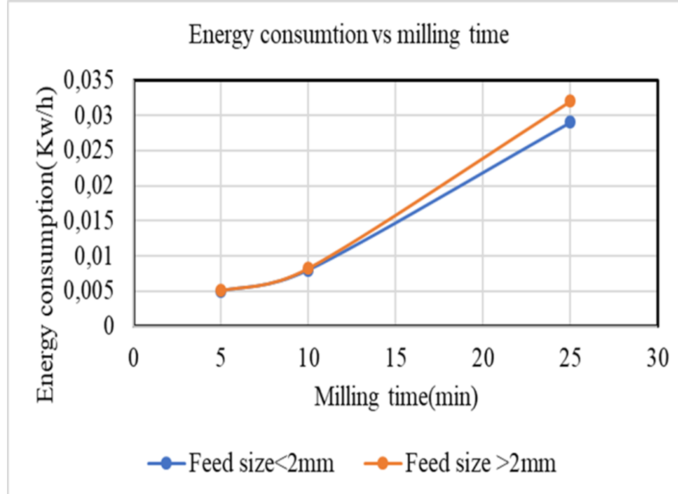

Fig 5. Energy consumption with milling time

The graph shows the energy consumption varies with milling time and feed particle size. From the results presented, total energy consumed increases as milling time increases and feed size particles increases. This can be seen from the particles size of greater than 2mm have higher energy consumed of 0,0032Kw/h and particle size that are less than 2mm shows energy consumption of 0,029 at time increasing. It can be observed from both graph that energy increase as milling time increases.

V. Conclusion

The vanadium bearing ore from the Langa farm (Limpopo) was characterised using XRD, XRF and SEM. The paper elaborates more on the comminution behaviour of the feed. The energy consumption was discussed. The longer the milling time the higher the energy consumption. Optimal liberation size fraction was studied.

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