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Liberation Characteristics of Muro Iron Ore for Efficient Beneficiation Process

Olushola Bamidele Nenuwa[1]*, Oladunni Oyelola Alabi[2], Christopher Olatunde Ikubuwaje[3]

[1,3]Department of Mineral & Petroleum Resources Engineering, Federal Polytechnic, P.M.B. 5351, Ado-Ekiti, Ekiti State, Nigeria
[2]Department of Metallurgical & Materials Engineering, Federal University of Technology, P.M.B. 704, Akure, Ondo State, Nigeria

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***Corresponding author:** Olushola Bamidele Nenuwa
Department of Mineral & Petroleum Resources Engineering, Federal Polytechnic, P.M.B. 5351, Ado-Ekiti, Ekiti State, Nigeria

Abstract

The liberation size of Muro iron ore was determined by obtaining iron ore samples from Toto Local Government Area, Nasarawa State. The collected samples were subjected to crushing and grinding to reduce their size. The elemental composition and mineralogical characteristics of the ground sample were then examined via SEM-EDS analysis. Particle size analysis of the homogenised iron ore sample was conducted, and the sieve fractions obtained were chemically analysed with the X-ray Fluorescence Spectrometer (XRF). The SEM-EDS analysis revealed that iron (Fe) and silicon (Si) were the most predominant elemental constituents with atomic percentages of 38.7% and 51.2%, respectively. The iron-bearing grains are most abundant at grain sizes less than 100µm. The mesh of grind (D80 value) of the iron ore was found to be 276 µm, the actual liberation size of the ore is -1180 + 850 µm, having the highest recovery of iron (Fe) at 44.85%. The 50% intersection, which indicates the economic liberation size of the ore is at -75 + 53 µm. Whenever Muro iron ore is ground in preparation for the concentration process, the economic liberation size of -75 + 53 µm should be adopted to prevent energy wastage through over-grinding and poor recovery due to under-grinding.

Keywords: Comminution, Liberation size, Mineral processing, Muro iron ore, Nasarawa state, Particle size analysis.

1. INTRODUCTION

The first and most important step in mineral processing is liberation, or the release of valuable minerals from the gangue and the second step is concentration, which is the separation of the valuable minerals from the gangue (Adeleke, 2023). Mineral concentration will be ineffective if liberation is not well accomplished. Liberation is the freeing or detachment of dissimilar mineral grains (Subba Rao, 2011). The liberation of the valuable minerals from the gangue is accomplished by comminution or size reduction, which involves crushing and grinding. The target of comminution is to liberate mineral particles at the coarsest possible particle size, which will ensure that energy is conserved and over-grinding, which can make the subsequent separation process difficult or expensive, is avoided. Under-grinding or too little liberation is also not desirable, as it can lead to losses. Therefore, good liberation is a prerequisite to a successful mineral processing operation; without it, the economic separation process will be impossible (Wills & Finch, 2016).

Mineral liberation is the extent to which the particles are made of discrete mineral grains. A fully liberated particle is composed of only one mineral, while composite particles have mixtures of mineral grains, and a locked particle has no surface exposure to the selected mineral. The various processes that the ore is being prepared for will require different degrees of liberation to be effective (Dunne *et al.,* 2019). The degree of liberation is the percentage of the mineral that exists as free particles in the broken ore to the total mineral content in locked and free particles. Liberation is easier if there are weak boundaries between mineral and gangue particles, but most times, the bond between mineral and gangue is strong, thereby causing random breakage and production of a significant amount of middlings (Wills & Atkinson, 1993). An economic degree of liberation is attained by grinding ores to an optimum grind size, which is determined by laboratory and pilot-scale test work. Grinding is the most energy-intensive operation in mineral processing, and it accounts for up to 50% of a concentrator's energy consumption (Fuerstenau & Han, 2009; Radziszewski, 2013). Although under-grinding is

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undesirable during size reduction, over-grinding will also produce very fine and difficult-to-treat “slime” particles, which may be lost in the tailings or even discarded before the concentration process. Grinding is then a compromise between producing high-grade concentrates, operating costs, and losses of fine minerals.

Iron ore is the primary raw material from which metallic iron is extracted to make steel (Lu, 2022). Muro iron ore is one of the many iron ore deposits found in Nigeria, which is still under investigation (Anike *et al.,* 1993; Obaje, 2009). Intensive field and laboratory studies are expected to be carried out on the deposit. Muro Banded Iron-Formation (BIF) is associated with the Proterozoic metasedimentary “schist belts” in northern Nigeria. The BIF and associated metasediments form the Muro Hills stretching for 13 km in a NE-SW direction. Two facies of the BIF, namely, oxide and carbonate facies, have been recognised, with the former predominating and the latter being poorly developed and rather sporadic (Adekoya *et al.,* 2012). Previous studies conducted on the characterisation of Muro iron ore reported that the total iron (Fe) content is 25 – 35% and the silica (SiO_2) content is in the range of 54%. The estimated reserve of the deposit is around 10.6 million tonnes (Ibrahim & Biliaminu, 2010; Nenuwa et al., 2022). The average work index of Muro iron ore was calculated as 26.13kWh/t, and the energy requirement for grinding the ore was found to be 3.62kWh/t (Nenuwa *et al.,* 2021).

The liberation size of an ore is a very important parameter in mineral processing design as it gives the processor clear information about the particle size the grinding operation should target. The liberation sizes of Itakpe iron, Arufu lead ore, Madaka manganese ore, and Akiri copper ore have been previously investigated (Ettu *et al.,* 2014; Ogundeji *et al.,* 2018; Ola-Omole & Nheta, 2020; Usaini *et al.,* 2014). Hence, this study aims to determine the liberation size of Muro iron ore, which will set the stage for an effective and efficient concentration of the iron ore. The outcome of this study will provide the economic liberation size at which more of the iron concentrates are freed and can be ultimately recovered from the gangue.

2. MATERIALS AND METHOD

2.1Accessibility to the site and sample collection

The crude iron ore samples used for this study were sourced from Muro, a town in Toto Local Government Area, Nasarawa State, Nigeria. Fig. I reveals an outcrop of the Muro iron ore deposit, and Fig. II is a geological location map of Nasarawa state showing the location of Muro. The sample location was accessed from Abuja - Abaji - Toto - Gadabuke Expressway. The site is about 8 km from Gadabuke junction, and it is accessible through narrow, untarred roads and pathways. The iron ore deposit is located behind Cheku Primary School, Muro community. 70 kg samples of the iron ore were collected with the aid of a sledge hammer and sample bags from four (4) different points at an interval of 100 meters apart within the deposit. The Global Positioning System (GPS) was used to measure the exact locations of the points from where the samples were collected. Table I presents the GPS readings showing the coordinates and elevation of the points.

Figure I: An outcrop of the Muro iron ore deposit

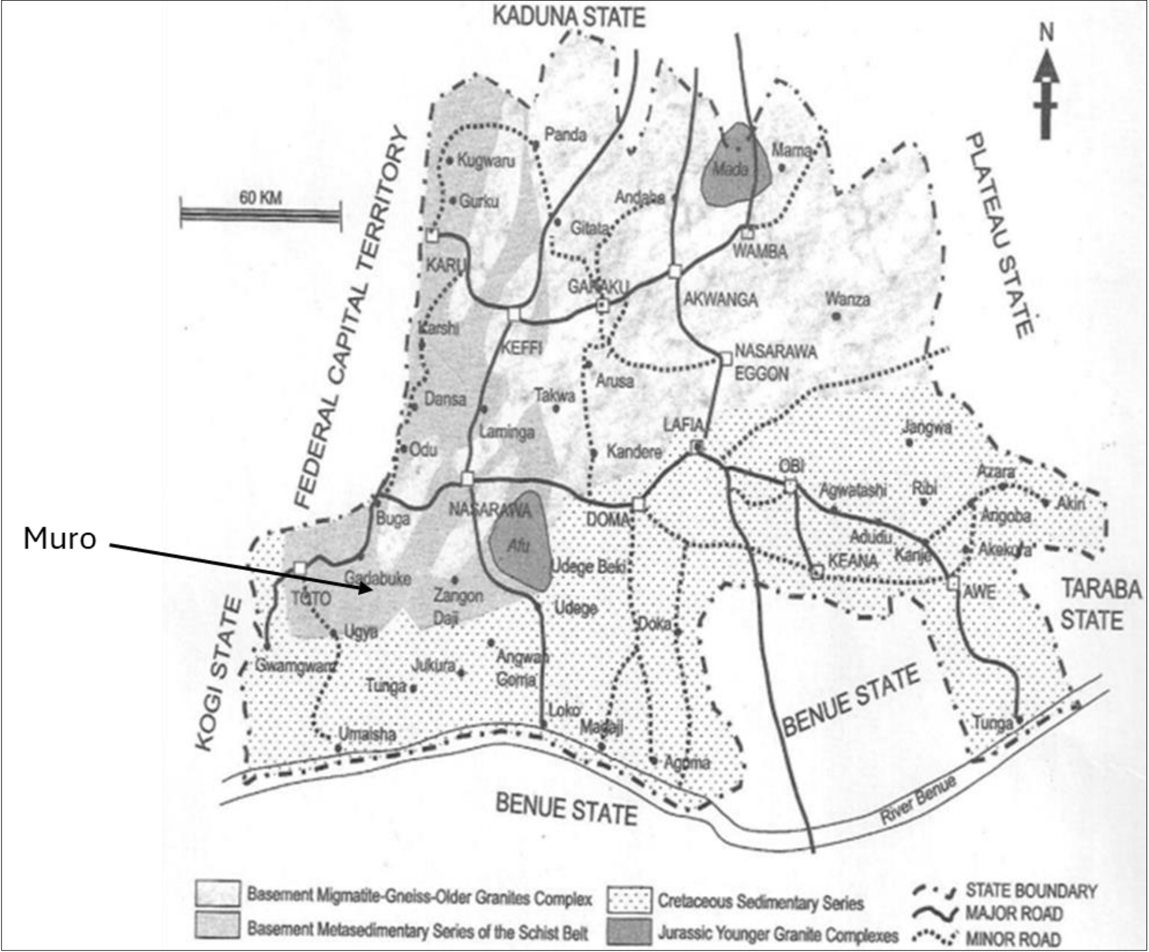

Figure II: Geological location map of Nasarawa state showing Muro, the study area (Usaini *et al.,* 2014)

Table I: GPS readings of the locations from where the iron ore samples were collected

Locations	Northing (N)	Easting (E)	Elevation (m)
Point 1	08° 20.151’	007° 14.055’	234.08
Point 2	08° 20.146’	007° 14.056’	238.65
Point 3	08° 20.136’	007° 14.057’	241.40
Point 4	08° 20.170’	007° 14.025’	226.77
Source: Fieldwork			

2.2 Sample preparation

Sample preparation involved crushing and grinding the four different samples collected. The iron ore samples of about 30cm in size were reduced with a sledge hammer to smaller sizes that could be accepted by the jaw crusher. The samples were then crushed using a laboratory jaw crusher and pulverised with the ball mill. The crushed iron ore samples 1, 2, 3 and 4, weighing 500g respectively, were charged separately into the ball mill and ground for 20 minutes. The product from the ball mill was sieved using a sieve size of 1180µm to obtain uniform sizes. 100g each of samples 1, 2, 3 and 4 from the pulverised samples were thoroughly mixed to produce the homogenised sample.

2.3 Particle size analysis and liberation size determination

The particle size distribution and the liberation size of the homogenised iron ore sample were determined by fractional sieve analysis, which was carried out using Endecolts mechanical sieve shaker (Fig. III). An array of sieve sets ranging from 1180 µm to 53 µm was arranged using a sieve scale in a stack with the coarsest sieve on the top and the finest at the bottom. A tight-fitting pan was placed below the bottom sieve to receive the final undersize, and a lid was placed on top of the coarsest sieve to prevent the escape of the sample. The arranged sieves were placed on the sieve shaker, which vibrates the materials vertically. 100 g of the homogenised crude iron ore was weighed and charged into the upper sieve (1180 µm) and agitated for 15 minutes, causing the undersize mineral particles to fall through successive sieves until they were retained on a sieve having an aperture smaller than the diameter of the particles. After this operation, the size fraction retained on each sieve was collected, weighed, and recorded. Thereafter, the size fractions were finally packed in small sample bags and properly labelled according to their sieve sizes. These packed samples were chemically analysed with the aid of an Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRF). The sieve size fraction that contains the highest percentage assay of iron (Fe) content was acknowledged as the actual liberation size of the iron ore, while the 50% intersection indicated the economic liberation size.

Figure III: Mechanical sieve shaker

2.4 Mineralogical Characterisation

The morphological and qualitative analyses of the homogenised Muro iron ore sample were performed using a JEOL-JSM-7600 Scanning Electron Microscope equipped with Energy Dispersive Spectroscopy (SEM-EDS) operated at 15kV. The back-scattered electron (BSE) image revealed the spatial distribution of mineral phases and compositional contrast, while the EDS analysis provides information on the elemental composition. For the SEM, a small amount of powder sample was taken and placed on the sample holder, which was then positioned inside the machine. A vacuum was built in the sample chamber, and the sample was irradiated to generate emission from which micrographs of the sample were produced. During the process, the EDS analysis was also carried out on the sample to establish the spectra of the major elements.

3. RESULTS AND DISCUSSION

3.1 Particle size distribution

The particle size distribution of the homogenised Muro iron ore sample and the percentage iron (Fe) content for each sieve size are presented in Table II.

Table II: Results of sieve analysis of the homogenised Muro iron ore sample

Sieve size range (µm)	Weight retained (g)	% Weight retained	Nominal aperture Size	Cumulative % weight retained	Cumulative % weight passing	% Fe present (Grade)
+1180	1.28	1.32	1180	1.32	98.68	38.12
-1180+850	0.07	0.07	850	1.39	98.61	44.85
-850+425	8.89	9.18	425	10.57	89.43	43.66
-425+300	5.96	6.15	300	16.72	83.28	34.00
-300+125	12.76	13.17	125	29.89	70.11	30.60
-125+75	9.09	9.38	75	39.27	60.73	32.01
-75+53	12.27	12.67	53	**51.94**	**48.06**	29.44
-53+pan	46.56	48.06	Pan	100.00	0.00	39.08

It was observed from the sieve test results presented in Table II that the cumulative percentage weight passing following the order of sieve arrangement from the coarsest to the finest was obtained as 98.7%, 98.6%, 89.4%, 83.3%, 70.1%, 60.7%, 48.1% and 0%. The mesh of grind or D80 value is the optimum particle size resulting from a specific grinding operation. It is also referred to as the particle size for which 80% of the material passes through the sieve or is finer than the sieve's diameter. The mesh of grind is the liberation mesh decided on as correct for the commercial treatment of the material. The mesh of grind (D80 value) for Muro iron ore is calculated based on results obtained from Table II and the Gaudian Schumann expression as follows:

If size1 = 300 μm is equivalent to 83.28% passing size

Then size2, which is equivalent to 80% passing size, is given as:

$$\text{Size2} = \left(\frac{\frac{80}{100}}{\frac{83.28}{100}}\right)^2 \times 300$$

$$= \left(\frac{0.8}{0.8328}\right)^2 \times 300$$

The mesh of grind (D80 value) is equivalent to 276 µm at 80% passing

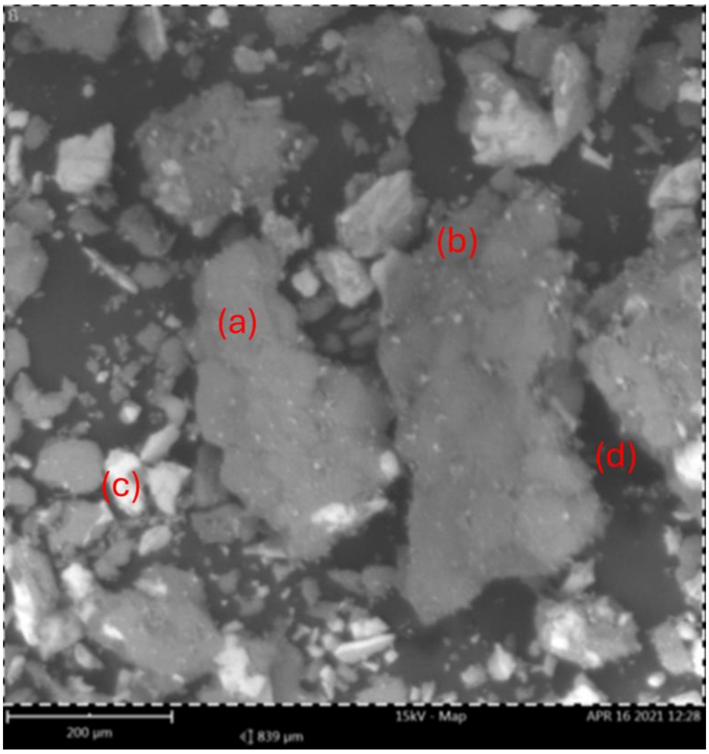

Figure IV: SEM microstructure in the back-scattered electron (BSE) mode of homogenised Muro iron ore sample (a) Iron-bearing mineral (b) Silica (c) Gangue mineral (d) Pore

Table III: EDS result showing elemental composition of the homogenised Muro iron ore sample

Element Name	Element Symbol	Atomic Conc. (%)	Weight Conc. (%)
Iron	Fe	38.71	52.60
Silicon	Si	51.15	34.95
Silver	Ag	0.77	2.03
Niobium	Nb	0.67	1.51
Yttrium	Y	0.68	1.48
Calcium	Ca	1.19	1.16
Cobalt	Co	0.68	0.98
Aluminium	Al	1.40	0.92
Potassium	K	0.89	0.84
Manganese	Mn	0.58	0.77
Chlorine	Cl	0.73	0.63
Vanadium	V	0.41	0.51
Sulfur	S	0.51	0.39
Sodium	Na	0.60	0.33
Magnesium	Mg	0.43	0.25
Chromium	Cr	0.20	0.25
Titanium	Ti	0.20	0.24
Phosphorus	P	0.21	0.16

3.2 Results of Mineralogical Characterisation via SEM-EDS

Figure IV is the SEM image in the back-scattered electron (BSE) mode of the homogenised Muro iron ore sample with a field of view (FOV) of 839µm. Table III is a presentation of the elemental composition of the sample as revealed by the EDS. Figure V shows the EDS spectra obtained for the iron ore sample. The results revealed that the iron ore microstructure consists of various mineral crystals of different shapes, sizes and colour separated by grain boundaries. The regions of high average atomic number appear brighter relative to regions of low atomic number. This means that the heavier the element, the lighter the colour. The greyish portions, as shown in Figure IV(a), are the iron-bearing minerals, while the darker portions in Figure IV(b) are the silica. The whitish grains scattered within the matrix, as displayed in Figure IV(c), are other gangue minerals associated with the iron ore, while the black regions are pores, as shown in Figure IV(d). The mineral crystals in the iron ore matrix differ in size, shape, and orientation. The grain sizes of the iron-bearing mineral, as revealed by the SEM micrograph, ranged between <100µm and 400µm, but the most abundant grains are less than 100µm. It can also be observed from the results that the minerals are separated by a large grain boundary, which is an indication that the minerals are loose and can be easily liberated from each other during comminution with minimal energy. The EDS results for the iron ore in Table III and Figure V revealed the various elements present in the Muro iron ore, including their atomic and weight concentrations in percentage. From the result, iron (Fe) and silicon (Si) were the most abundant elemental constituents, with atomic percentages of 38.7% and 51.2%, respectively (Si had the highest peak). The atomic concentrations of other elements in the iron ore are minor. The weight concentrations of iron and silicon are 52.60% and 34.95%, respectively.

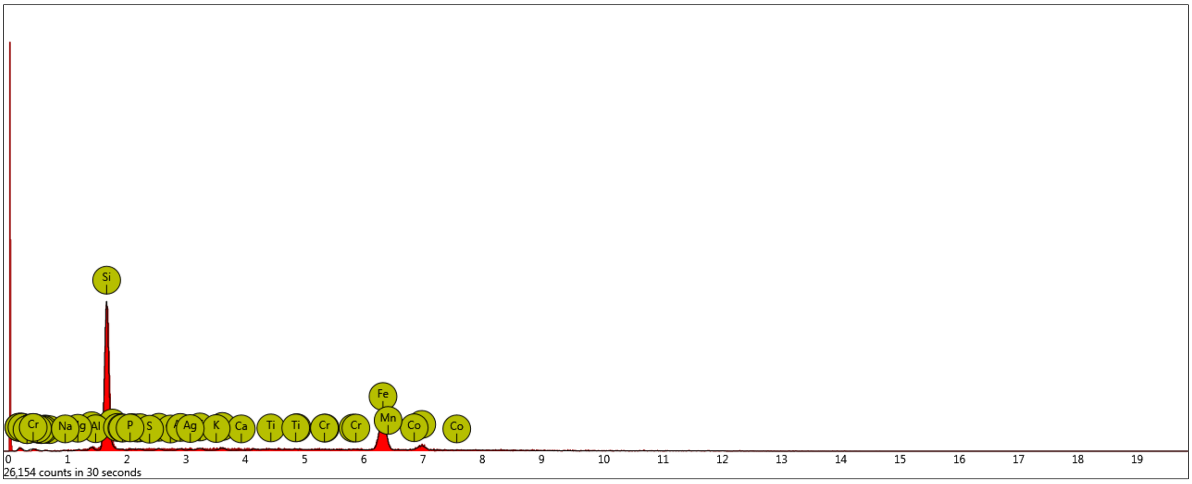

Figure V: EDS pattern of the homogenised Muro iron ore sample

Table IV: Chemical composition (weight percentage) of sieve fractions of Muro iron ore as determined by XRF analysis *(Composition of Fe was obtained by oxide-to-element conversion)

Conc. (Wt.%)	**Pan**	**-75 +53µm**	**-125 +75µm**	**-300 +125µm**	**-425 +300µm**	**-850 +425µm**	**-1180 +850µm**	**+1180µm**
SiO_2	43.1	57.2	53.4	55.3	50.2	36.2	32.2	44.8
CaO	0.12	0.079	0.23	0.14	0.30	0.54	2.71	0.10
Cr_2O_3	0.12	0.10	0.11	0.10	0.11	0.13	0.12	0.12
Fe_2O_3	55.93	42.14	45.81	43.79	48.66	62.48	64.19	54.56
CuO	0.053	0.064	0.085	0.063	0.059	0.048	0.067	0.050
ZnO	0.02	-	0.041	-	-	-	0.03	-
Br	0.059	-	0,038	0.033	0.041	0.068	0.069	-
Rb_2O	0.061	0.042	0.044	0.041	0.047	0.073	0.067	0.066
RuO_2	0.25	0.38	-	0.042	0.48	0.38	0.29	0.32
La_2O_3	0.03	0.02	0.07	0.04	0.03	0.02	0.05	0.02
Re_2O_7	0.02	-	0.04	-	-	-	0.07	-
PbO	0.24	-	-	-	-	-	-	-
K_2O	-	-	0.12	-	0.089	0.045	0.051	0.051
Y_2O_3	-	0.02	-	0.02	-	-	-	-
*Fe	39.08	29.44	32.01	30.60	34.00	43.66	**44.85**	38.12

3.3 Chemical composition of sieve fractions and liberation size

Table IV presents the result of the chemical analysis of all the sieve fractions of the homogenised Muro iron ore sample. The percentage iron (Fe) content for all the sieve fractions was obtained by oxide-to-element conversion, and they are shown in Table IV. It was found that particle size of 1180 µm contained 38.12% Fe, 850 µm contained 44.85% Fe, 425 µm contained 43.66% Fe, 300 µm contained 34% Fe, 125 µm contained 30.6% Fe, 75 µm contained 32.01% Fe, 53 µm contained 29.44% Fe, particle size less than 53µm (pan) contained 39.08% Fe. Significant liberation of the iron-bearing mineral was achieved at a particle size of -1180 + 850µm, which had the highest percentage of iron (Fe) of 44.85% compared to the other sieve fractions. This is taken as the actual liberation size of Muro iron ore. From Table II, the 50% intersection, which is acknowledged as the economic liberation size, is -75 + 53 µm. The economic liberation of minerals has always been useful in mineral processing plants because it saves time, energy and resources. The economic liberation size implies that more of the valuable minerals are liberated and can be recovered at this particle size. This result is in agreement with the SEM-EDS analysis result, which reveals that the iron-bearing grains with sizes less than 100µm are the most abundant in the SEM matrix. The result is also consistent with a previous study, which recommended grinding to -74µm to guarantee complete recovery of the iron value (Okafor *et al.,* 1998). If the sample is concentrated at a particle size that is less than the economic liberation size, it can result in slime formation, in which the valuable minerals are lost to the tailings. However, if larger particles are engaged in the concentration process, the recovery will be low because valuable minerals are still locked up with the gangue minerals.

4. CONCLUSIONS

Mineralogical characterisation of Muro iron ore via SEM-EDS and particle size analysis to determine the liberation size was carried out. Iron (Fe) and silicon (Si) were the most abundant elemental constituents, with atomic percentages of 38.71% and 51.15%, respectively. Particle size analysis showed that the Muro iron ore had a mesh size (D80 value) of 276 µm at 80% passing. The actual liberation size of Muro iron ore is -1180 + 850 µm, with the highest iron (Fe) recovery at 44.85%, while the 50% intersection indicates the economic liberation size is -75 + 53 µm. During the grinding of Muro iron ore, the economic liberation size of -75 + 53 µm should be adopted to prevent over-grinding, which could lead to energy wastage and under-grinding that would cause inefficient liberation of the valuable portion from the gangue and low recovery during the subsequent concentration process. Muro iron ore is another potential iron ore deposit that can be explored and exploited for use in the iron and steel industries. Further research should be conducted on Muro iron ore to establish an additional database that could be used in the development of a suitable process route for the beneficiation of iron ore. The government should also pay more attention to the development of the Muro iron ore deposit and other similar deposits within the country.

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