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Beneficiation and Characterization of Wase Galena Deposit Using Gravity and Froth Flotation Techniques

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Abstract

This research investigated the beneficiation of lead in Wase lead ore deposit with the aid of gravity and froth flotation techniques with a view to determine the chemical composition and the mineral content of the crude lead ore; determine the liberation size of the valuable mineral; and to investigate the best technique to produce lead as charge into the blast furnace. The crude ore was crushed and ground to obtain head sample for chemical and mineralogical analyses using Energy Dispersive X-Ray Fluorescence Spectrometer (ED-XRFS) and X-Ray Diffraction (XRD) respectively; and for particle size analysis. The particle size was subjected to air floating machine, Humphrey spiral concentrator, Wilfley shaking table using six kilograms (6kg) as charge and one kilogram (1kg) as charge for froth flotation at the liberation size. The ED-XRFS analysis of the crude ore shows that it contains 68.50% PbO and a lead metal of 66.42% Pb with other associated minerals such as 19.50% Fe_2O_3, 3.77% ZnO, and 2.08% SiO_2 while others are in traces. The mineralogical analysis reveals that the mineral composition of the ore contains 13.38% galena, 6.53% hematite, 3.43%sphalerite, 1.94% azurite and other minerals by weight. The particle size analysis indicates that the liberation size of the ore is at -0.1+0.2 mm. It was discovered that the Wilfley shaking table technique meet the required standard of assay and recovery for the production of lead as charge into the blast furnace and hence, the most suitable technique for the beneficiation of this ore.

Keywords

Ore, Blast Furnace, Liberation Size, Gravity, Mineral, Beneficiation, Assay, Wilfley

1. Introduction

Nigeria has a wealth of mineral resources distributed all over the Country which can be explored for possible exploitation to broaden the Country's economic base. Lead is part of the solid minerals which can contribute greatly towards achieving the laudable objectives of boosting and diversifying of the Nigerian economy when exploited. It is one of the softest and heaviest of the common metals. It is a main-group element represented by symbol Pb and has an atomic number of 82. It is bluish gray in color with a bright metallic luster when newly cut or melted. It takes on a silvery gray patina when exposed to atmosphere but changes to dark grey or black on exposure to industrial atmospheres. Lead has a specific gravity of 11.34 in cast form and 11.35 to 11.37 after rolling or extruding. It melts at 327.4°C and boils at about 1740°C. Its ultimate tensile strength is roughly 2000 pounds per square inch (136 kg/cm^2) and has a Brinell hardness of approximately 4.5. Lead does not conductor heat and electricity. Lead resists corrosion due to a thin protective coating that forms on its surface. Lead forms alloys with several metals such as tin, antimony and bismuth [1].

Lead is hardly found in native state in nature but it is associated with other elements to form a series of minerals such has galena (PbS); cerussite ($PbCO_3$) and anglesite ($PbSO_4$) but the principal ore of lead is galena which is about 86.6% Pb. Lead is however, occur in ore with zinc, silver, and most abundantly copper, and it is extracted together with these metals [1]. Lead was used during the Roman Empire for water pipes, glazes on prehistoric ceramics, cosmetic kohl and used by ancient Egyptians to darken their eyelids [2]. Some modern day uses of lead include burial vault liners, glassware, pewter, stabilizers for PVC, crystal glass, and ceramic glazes, used as pigments. Lead is also used in containers and pipes in order to prevent rust and corrosion; and most steel bridges are painted with lead-based paints [2]. Lead/zinc occurrence and distribution is dominant in the Northern part of Nigeria due to the peculiar geology of the area. This occurrence is based on the saline water intrusion in the sedimentary basins or fractured/shear zones in crystalline rocks while the mineralization is mostly associated with quantity of copper and silver, present in lodes filing the fractures within the sedimentary rocks in Benue Trough in Abakaliki area of Ebonyi as well as in crystalline basement rocks. Lead/zinc can be found in Nigeria viz: Nasarawa (Akwanga, Eggon and Wamba), Taraba, Plateau, Abia, Bauchi, Zamfara, Abuja and Ebonyi [2]. Several researchers have worked extensively on different beneficiation techniques of beneficiating the World lead ore deposit. In India, Mandre and Shara investigated the dissolution behavior of galena and sphalerite and their preferential leaching from one another. They discovered that the dissolution of individual minerals depends on their nature of association with other sulphides [3]. Jianhua *et al* investigated the electronic structures of natural galena having ten types of impurities with the aid of the density-functional theory and concluded that the Sb and Mn impurities might result to the over-oxidation of galena, and hence not suitable for the froth flotation of galena [4]. Emin investigated the effects of hydrodynamic conditions on true flotation and entrainment of complex sulphides ores such as lead, zinc and copper sulphides ores using a fractional factorial experimental design and determined the contributions of true flotation and entrainment in flotation of a complex sulphide ore [5]. However, in Iran, Dehghan*et al* studied and optimized the zinc extraction from a low-grade sphalerite concentrate using ferric sulphate and sulfuric acid media as a potentially promising process for zinc production from low grade zinc resources [6]. Ajayi employed the technique of froth floatation to beneficiate lead-zinc ore of Abakaliki [7]. *Abere et al* worked on the Enrichment of Zinc Concentration of Ishagu Lead-Zinc Ore Deposit using gravity and froth flotation Beneficiation Techniques and concluded that the air floating gravity separation technique is the most efficient route to add value to the zinc concentrate of the ore [8]. Detailed process mineralogy was conducted by Mei *et al* on a non-sulphide lead-zinc ore from the province of Sichuan, thus providing basic data and a theoretical foundation for developing techniques to utilize this resource. They discovered that both lead and zinc levels in the ore exceeded the minimum industrial grade for lead-zinc oxide ores with relatively high amounts of silver and iron and then concluded that physical separation alone could not achieve high recovery of lead and zinc from this ore [9]. However, this work seeks to beneficiate Wase lead ore deposit with gravity and froth flotation techniques and investigate the best technique to produce lead as charge into the blast furnace.

2. Materials and Methods

The raw material used in this research is lead ore obtained from Wase, Plateau State, Nigeria. The chemicals used include lime, water, potassium ethyl xanthate (99% SIGMA), methyl iso-butyl carbonol (99% MERCK) while the equipment used include shovel, digital pH meter (metrohm model 780), syringe, sieve shaker and set of sieves, Denver jaw crusher, Denver cone crusher, ball mill, Pan analytical Minipal Energy Dispersive X-Ray Fluorescence Spectrometer (ED-XRFS), Leco optical microscope, X-Ray Diffractometer (XRD), Kip Kelly air floating machine, Humphrey Spiral concentrator (B124TA) and Denver Laboratory Floatation Cell. The ore sample was taken from coordinates $9^{\circ}14'0''$ Longitude (East) and $10^{\circ}34'0''$ Latitude (North) of Wase. The sampling was carried out at the site using random and shoveling methods. 100 kg of sample was obtained from the overburden at a depth of 1.7 meters. The quantity of the ore obtained was further sampled at National Metallurgical Development Centre (NMDC), Jos, using cone and quartering method to obtain head sample for mineralogical and chemical analyses of the crude, and for particle size analysis.

2.1. Comminution

Comminution is a process by which particle size of ore is progressively reduced until the clean particles of mineral are liberated or unlocked from associated gangue for onward separation by such methods as are available. Comminution in the mineral processing plant takes place as a sequence of crushing and grinding processes. Crushing is the first mechanical stage in the process of comminution in which the main objective is the liberation of the valuable minerals from the gangue. The Crushing of the ore was accomplished by compression of the ore against rigid surfaces, or by impact against surfaces in a rigidly constrained motion path. Crushing is usually a dry process, and is performed in several stages, with small reduction ratios ranging from three to six in each stage. The reduction ratio of crushing stage can be defined as the ratio of maximum particle size entering to maximum particle size leaving the crusher. The crushing was scheduled into two major stages, the primary and secondary crushing schedule according to [10]. The primary crushing schedule was carried out by the use of primary crusher such as Denver Jaw crusher. The crusher was used to reduce the size of the lumps of run-of-mines at the mineral processing laboratory of NMDC Jos. This schedule was carried out on secondary crusher, such as Denver cone crusher, to further reduce the size of the ore to particle size less than 2 mm. The feed of the secondary crusher was the product from the primary crusher. Grinding is the last stage of comminution and is usually performed wet to provide a slurry feed to the concentration process, although dry grinding has limited applications. In this stage the particles were reduced in size by a combination of impact and abrasion. It was carried out in a rotating cylindrical steel vessel known as ball mill which was made up of a charge of loose crushing bodies, the grinding medium, which was free to move inside the mill, thus comminuting the ore. The grinding medium was steel balls.

2.2. Chemical Composition of Crude Lead Sample

The chemical analysis was carried out with the aid of ED-XRFS at NMDC Jos. The samples were thoroughly mixed to homogenize and then sampled using riffles sampler. The resulting sample was taking for the chemical analysis.

2.3. Grain size Analysis

The samples for the grain size analysis were prepared and molded through the Bakelite powder in a mounting press at 300°C The prepared samples were crushed and ground using series of grits paper on a calibrated Leco optical microscope. This shown the grain size of the Lead ore sample at varying shapes and colours measured at a class interval of 100. The midpoint of class interval (X) and frequency (F) as seen in equation 1 were used to determine the variance (S^2) which was otherwise used to evaluate the standard deviation as indicated in equation 2 which gave the required grain size of the lead ore and also its liberation size.

$$\text{Variance } (S^2) = \sum X^2F - \sum XF^2/\sum F \tag{1}$$

$\sum F - 1$

$$\text{Standard Deviation} = \sqrt{S^2} \tag{2}$$

2.4. Sieve Size Analysis

Sieve analysis was carried out to evaluate the particle size distribution of the ore in each sieve size fraction and to determine the liberation size of the ore. Cone and quartering sampling techniques were used to sample 5 kg of the lead ore where 150 g was obtained and sieved on an Automated Endecott test sieve shaking machine for 30 minutes. Sieve aperture sizes ranging from 11.2 to 0.065 mm were used. Each sieve size fraction obtained was weighed, recorded and analyzed with ED-XRFS.

2.5. Gravity Separation

The ore was subjected to the following gravity techniques such as air floating concentration; Humphrey Spiral Concentration; and Wilfley shaking table at the liberation size.

2.6. Air floating Beneficiation Technique

6.0kg of the ore at a liberation size of -0.1+0.2 mm was weighed and charged to the Kip Kelly Air floating machine at a feed rate of 50kg per hour with an inlet air opening at 2cm at a deck slope of tilted at an angle of 1800 [11]. The concentrate and tailing were weighed and analyzed with ED-XRFS.

2.7. Humphrey Spiral Concentration Technique

6.0kg of the ore at the require liberation size of -0.1+0.2 mm was added to 25 liters of water and mixed in order to form slurry [12]. The slurry was charged into Humphrey Spiral Concentrator at a feed rate of 50 liters per hour and its valve was set at 2cm wide. The concentrate and tailing were collected in a bucket and allowed to settle for 12 hours and then decanted, sun dried, weighed and analyzed with ED-XRFS machine for chemical composition.

2.8. Wilfley Shaking Table Technique

6.0kg of the ore at the required liberation size of -0.1+0.2 mm was charged into the wilfley shaking table at the Mineral Beneficiation Pilot Plant of NMDC, Jos after forming slurry and added to water at ratio of 1:5 with deck tilted at 1800 at feed rate of 50 liters per hour while the deck's speed was set at 250 revolution per minute (RPM) [13]. The resulting products were weighed and analyzed with ED-XRFS for chemical composition analysis.

2.9. Froth Flotation

The froth flotation was carried out in a standard 1 litre Denver Laboratory Flotation Cell at the Mineral Processing Laboratory of NMDC Jos using -0.10 mm + 0.20 mm size of the ore sample. 300 g of the dry sample was added to 700ml of water in the flotation cell. The flotation cell was started and the mixture was agitated by the cell's impeller for two minutes. The pH of the pulp was taken using the pH meter. 0.215 g/l and 0.250 g/l of lime were added to the pulp before the pH could be stable at 7.5 and 8.0 respectively where Zn/Fe are least hydrophobic while Pb/Cu are most hydrophobic (great pH to recover Pb). 2 g/l of potassium ethyl xanthate was added to the pulp at pH 7.5 using syringe. The pulp was then agitated and conditioned for 10 minutes. 0.25 g/l of methyl iso-butyl carbonol was added with the aid of syringe as frother and the cell's air inlet was opened, which led to formation of bubbles/foams. The impeller was allowed to agitate the pulp for 5 minutes with formation of more bubbles/foams. The froth was scraped into a plastic collector and allowed to settle before it was decanted and the solid was therefore sun dried.

3. Results

The results obtained in this work are presented in tables 1 to 7 respectively; chemical analysis of crude lead ore, analysis of metal content in the ore, mineralogical composition of the ore, the grain size of the ore analyzed through the statistical approach, fractional sieve size analyses, the chemical composition of the processed ore using the gravity and froth flotation techniques, and recovery of lead in the ore.

Table 1. *ED-XRF Analysis of crude lead ore.*

Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Cr_2O_3	MnO	Fe_2O_3	NiO	CuO	ZnO	La_2O	Ag_2O	PbO
0.65	2.08	0.06	0.43	0.02	0.09	1.02	19.50	0.04	0.64	3.77	0.04	0.02	68.50

Table 2. *Analysis of metal content in the ore.*

Oxide	%Composition	Element	%Composition
PbO	68.50	Pb	66.42
ZnO	3.77	Zn	3.03
Fe_2O_3	19.50	Fe	15.28
CuO	0.64	Cu	0.51

Table 3. *Mineralogical composition of the ore.*

Major Minerals	Minor Minerals	Gangues
Galena (PbS)	Sphalerite (ZnS)	Kaolinite [$Al_2Si_2O_5(OH)_4$]
Hematite (Fe_2O_3)	Azurite (CuS)	Quartz (SiO_2)
	Rutile (TiO_2)	Muscovite [KAl3SiO10(OH)2]

Table 4. *Statistical table of grain size analysis.*

Class Interval	X	F	FX	X^2F
001-100	50.5	233	11766.5	594208.25
101-200	150.5	256	38528	5798464
201-300	250.5	204	51102	12801051
301-400	350.5	310	108655	38083577.5
401-500	450.5	190	85595	38560547.5
501-600	550.5	298	164049	90308974.5
601-700	650.5	200 ΣF = 1691	130100ΣFX = 5897955	84630050Σ X^2F = 270776872.8

From Equation 1, the variance (S^2) is given by:

$S^2 = 270776872.8 – 589795.5^2 /1691$

1691 - 1

$S^2 = 38500.01578$

Hence, from Equation 2;

Standard Deviation = $\sqrt{(38500.01578)^2}$= 196.21

Table 5. *Fractional sieve size analyses.*

Sieve sizes (mm)	Weight (g)	Weight (%)	Cumulative Weight (%) Retained	Cumulative Weight (%) Passing	% Pb
+11.2	7.03	4.89	4.89	95.11	37.80
-11.2+9.5	6.28	4.37	9.26	90.74	36.53
-9.5+2.0	10.92	7.59	16.85	83.15	38.61
-2.0+1.4	11.55	8.03	24.88	75.12	39.40
-1.4+1.0	12.89	8.96	33.84	66.16	31.34
-1.0+0.7	13.32	9.26	43.10	56.90	34.55
-0.7+0.5	13.01	9.05	52.15	47.85	37.91
-0.5+0.35	14.13	9.83	61.98	38.02	29.54
-0.35+0.10	14.05	9.77	71.75	28.25	30.08
-0.10+0.20	13.53	9.41	81.16	18.84	43.50
-0.20+0.09	12.01	8.35	89.51	10.49	35.28
-0.09+0.065	11.46	7.97	98.78	1.22	40.21
-0.065	3.60	2.50	99.98	0.02	38.65

Table 6. *ED-XRF Analysis of processed lead ore using Air float, Spiral Wilfley and Froth floatation techniques.*

% composition		Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Cr_2O_3	MnO	Fe_2O_3	NiO	CuO	ZnO	Pb
Air Float	Conc	0.62	0.34	0.01	1.20	0.02	0.08	0.82	18.58	0.03	0.60	3.73	70.20
	Tailings	2.74	65.43	0.84	2.81	1.08	0.91	1.85	3.05	0.01	0.01	2.61	10.01
Spiral	Conc	0.05	10.54	1.03	0.17	1.21	0.06	1.05	19.06	0.04	0.58	3.66	56.71
	Tailings	2.42	60.47	3.21	2.34	1.87	1.89	2.15	10.28	0.01	0.02	2.11	12.32
Wilfley	Conc	1.12	2.08	1.22	1.82	0.01	0.11	0.64	20.05	0.21	0.65	3.02	68.40
	Tailings	2.53	67.25	2.10	3.76	0.80	0.94	0.93	6.51	0.08	0.12	0.31	14.20
Froth Flotation	Conc	0.04	13.05	1.02	1.89	3.44	0.09	0.35	19.91	0.10	0.68	4.05	60.50
	Tailings	1.05	61.31	2.42	3.08	2.51	1.13	1.03	2.58	0.62	1.32	2.12	17.29

Table 7. *Recovery of lead in the ore.*

		Weight of charged	Weight of product (%)	% Pb	Recovery
Air float	Conc.	6	2.64	70.20	61.04
	Tailings		3.36	10.01	19.53
Spiral	Conc.	6	1.80	56.71	55.84
	Tailings		4.20	12.32	21.61
Wilfley	Conc.	6	2.48	68.40	69.20
	Tailings		3.52	14.20	28.55
Froth flotation	Conc.	1	0.29	60.50	85.34
	Tailings		0.71	17.29	33.42

4. Discussion

The XRF analysis of the crude lead ore presented in Table 1 indicates that the crude lead ore contains 68.50% PbO with other gangue minerals of composition 19.50% Fe_2O_3, 3.77% Zn, 2.08% SiO_2, 1.02% MnO and 0.64% CuO while others occur in minute quantity. The gangue minerals require beneficiation to be isolated from the mineral of interest. According to [14]; the ore meets the requirement of 1 – 5%Pb for exploration of a mine field. It can be observed from Table 2 that lead (Pb) has a percentage composition of 66.42%Pb metal in the ore. However, it can be observed from Table 3 that the major minerals present in the ore are galena and hematite while sphalerite, azurite and rutile are the minor minerals. The major gangue minerals present in the ore are quartz, kaolinite, and muscovite. Hence, Table 3 confirmed the result obtained in Table 1. From Table 4, the particle size of lead was measured using statistical analysis as shown in Equations 1 and 2 to be approximately 0.20 mm (200 µm) whereas in equation 2, the standard deviation is 196.21 (approximately 200 µm) i.e, the grain size that will liberate the valuable mineral is between 101 – 200 µm (Table 4) which implies that the average grain size needed to achieve liberation of the valuable mineral is about 200 µm which falls within the interval of the liberation size for the beneficiation of lead ore [15].

Table 5 presents the results of the sieve size analysis where it can be deduced that at -0.10+0.20 mm (101-200 µm), the cumulative weight percent retained and the cumulative weight percent passing are respectively 81.16 and 18.84% with an assay of 43.50%Pb. Also, at -0.09+0.065 mm; the cumulative weight percent retained and the cumulative weight percent passing are respectively 98.78 and 1.22% giving an assay of 40.21%Pb. It can be deduced from table 5 that beneficiation could be effective at -0.10+0.20 mm and -0.09+0.065 mm when compared to the other particle sizes. However, the gravity separation technique will be the most effective at particle size -0.10+0.20 mm due to the coarseness of the particle size at the liberation size [16] but froth flotation technique will be preferable at -0.09+0.065 mm because the particle size is finer [17].

From Table 6, it can be observed that the assays of the concentrate and tailings for Pb are 70.20% and 10.01% for air float respectively while the corresponding recovery are 61.04 and 19.53 (Table 7). Also, the assays of the concentrate and tailings for Pb in spiral method are 56.71 and 12.32% respectively with the corresponding recovery of 55.84 and 21.61 as indicated in Tables 6 and 7. In Wilfley shaking table, the assays of the concentrate and tailings for Pb are 68.40 and 14.20% respectively with the corresponding recovery of 69.20 and 28.55% (Tables 6 and 7).

The froth flotation has an assay of concentrate and tailings of 60.60 and 17.29%; and recovery 62.08 and 33.42% for Pb respectively. From Tables 6 and 7, the air float method has the higher grade of 70.20% Pb but with a low recovery of 61.04% when compared to the Wilfley shaking Table with a grade and recovery of 68.40 and 69.20%Pb respectively. The spiral and froth flotation beneficiation techniques do not meet the standard assay/grade required as charged into the blast furnace and hence, not suitable for the concentration of this ore. However, the air float and Wilfley shaking techniques meet the standard assay of 65%Pb required as charge into the blast furnace for lead production [1]. It is more preferable to employ the Wilfley shaking table with an assay of 68.40%Pb and recovery 69.20%Pb for the beneficiation process because its assay and recovery meet the 65% Pb standard requirement as charge into the blast furnace when compared to the air float techniques which has low recovery and this is in accordance with [17].

5. Conclusion

The ore was proven to be a complex ore where the major minerals are galena and hematite while sphalerite, azurite and rutile are the minor minerals. The average grain size requires to liberate the valuable mineral is between 0.1+0.2 mm (100-200 µm). Also, the Wilfley shaking table is the most suitable technique for the beneficiation of Wase lead ore deposit because the assay and recovery are within the standard requirement as charge into the blast furnace but if a high assay of 70.20% Pb with a low recovery of 60.04% Pb required; then the air float technique should be considered as well.

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