Sedimentation Analysis of Some Sulphide Ore Concentrates and Comparative Studies on their Leach-Ability in Butanoic Acid Solution

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Abstract: Sedimentation analysis of galena and sphalerite concentrates has been carried out to determine the average grain sizes. Comparative studies on the leach-ability of these ore concentrates in butanoic acid solution were also done. Results of the sedimentation analysis indicate that the average grain sizes of Ishiagu galena and sphalerite are approximately 100 and 150 μm respectively. This implies that galena is finer than sphalerite. It was found that increase in the mass-input of galena and sphalerite, initial and final pH of the leaching solution decreases the concentration of dissolved metal as a result of minimized reductive tendency of H^+ under this condition. The concentrations of lead and zinc dissolved per unit mass of galena and sphalerite concentrates leached in butanoic acid solution was evaluated as 10.575 and 5.077 mg/Kg/g respectively. This implies that the concentration of dissolved lead in butanoic acid is approximately twice that of zinc. This also implies that the reaction between galena and butanoic acid is twice that between sphalerite and the same acid (experimental process and conditions being the same). [New York Science Journal 2010;3(5):100-105]. (ISSN 1554 – 0200).

Keywords: Sedimentation analysis, sulphide ore concentrates, leaching, butanoic acid.

1. Introduction

It has been reported (Reed, 1988) that processing systems with particle distribution of particle sizes and shape are produced by proper selection and blending of raw materials with different initial characteristics and by subsequent crushing, grinding, dispersion, classification and granulation. Studies have been carried out by Mc Geary (1961) on the packing of coarse (0.37mm) monosize spherical particles using axial vibration. During the experiment, packing was observed to be mostly in an orthorhombic arrangement and packing density of 62.5%. The report concluded that packing density of nearly monosized spherical particles of silica and alumina of colloidal size packed by filter pressing deflocculated slurry is about 60-65%. The accuracy of particle size data has been reported (Barsoum, 1979) to depend somewhat on the sample preparation, the particle shape and technique used for the analysis. Barsoum (1979) reported that techniques in current use are microscopy, sieving, sedimentation, electrical sensing, and laser diffraction and light intensity fluctuation. Sedimentation analysis has been reported (David, 1979) to be more precise and sensitive to the size distribution of clay suspension than electrical sensing technique. Previous research (Singer and Singer, 1963) revealed that in electrical sensing techniques, the resistance of an electrolyte current path through a narrow orifice between two electrode increases when ceramic particle pass through the orifice. The resistance pulse for a stream of dispersed particle passing through the orifice are converted into the voltage pulse, amplified, scaled and counted electronically. In sedimentation analysis using hydrometer method, spherical particles with a particular density and diameter are released into a viscous fluid to ascertain the velocity and time of settling. The present work is to carry out a sedimentation analysis of some sulphide ore concentrates and comparative studies on their leach-ability in butanoic acid solution.

2. Materials and Method

2.1 Beneficiation and Sedimentation Experiment

Solid minerals composed of Galena (PbS) and sphalerite (ZnS) were collected in lumps from deposits in South-eastern Nigeria. The crude Pb-Zn sulphide ore was pulverized and beneficiated (at National Metallurgical Centre, Jos) using sodium silicate, sodium xanthate and methl isobutyl ketone (MIBK) as depressant, collector and frother respectively. The pH of the liquid ore was regulated within 8-13. After the beneficiation process, each sulphide concentrate was homogenized prior to chemical analysis.

Following beneficiation of the ore, the concentrates were pretreated using a dispersing agent (sodium hexametaphosphate) to remove any gangue materials left.

Sedimentation analysis was carried out (at a test temperature of 30^0C) using the conventional procedure (BS1377:1975, Test 6(B)). Hydrometer type used was casagranda 20^0C. During the experiment, general corrections were done to facilitate accuracy of results. These corrections are meniscus correction cm, temperature correction Mt and dispersing agent correction X, and water density correction cw. The difference between the two scales readings multiplied by 1000 gives the meniscus correction;

$$Cm = (B-A) \times 1000 \qquad (1)$$

Where
Cm = Meniscus correction
B = Scale reading at the upper rim of the Meniscus
A = Reading at the surface of the liquid

True hydrometer reading Rh is calculated by

$$Rh = Rh^1 + Cm \qquad (2)$$

Where
Rh^1 = the first hydrometer reading

The correction X applied to Rh is given by

$$X = 2m_d \qquad (3)$$

Where
X = Dispersant correction
m_d = Mass of dispersing agent remaining after evaporation.

The X correction is subtracted from Rh value.

Water density correction used :1.8 was added to the true density Rh. The fully corrected hydrometer reading R is given by;

$$R = Rh^1 + Cm + Mt - X + 1.8 \qquad (4)$$

The equivalent particle diameter at a known and after a certain time interval from the start of sediment.The equivalent particle diameter, at a known and after a certain time interval from the start of the sedimentation process can be calculated from the equation:

$$D = 0.005531\sqrt{(2H/(G_0 - 1)t)} \qquad (5)$$

Where
D = Equivalent particle diameter (mm)
η=0.8909Viscosity of water at test temperature (Mpas)
H = Effective depth (mm)
G_o = Specific gravity of particles (determined using conventional procedure (BS1377:1975, Test 6(B))
t = Elapsed time (mins.)

The percentage by mass of particles smaller than the equivalent diameter D is denoted by K. This percentage is equivalent to the 'percentage passing' in sieve analysis. It is given by the equation;

$$K = \frac{G_o \times R \times 100\%}{m(G_o - 1)} \qquad (6)$$

Where
m = mass of dry particles after pretreatment
R = Fully corrected hydrometer reading

Also the effective depth H_R was calculated using the equation;

$$H_R = 219 - 4.1Rh \qquad (7)$$

The average grain size of the clay samples analyzed was calculated using the equation;

$$Dm = \frac{\sum[\Delta K \times D]}{\sum[\Delta K]} \qquad (8)$$

Where
Dm = Average grain size (μm)
ΔK = Actual percentage finer D

Sieve analysis of the residue from sedimentation analysis was carried out to verify the value of the average grain size obtain through sedimentation analysis.

2.2 Leaching Experiment

Weighed quantities of fresh samples of each of these sulphide ore concentrates were placed in cylindrical flask containing butanoic acid and distilled water (of ratio 100:2) and allowed for some time. Sodium hydroxide was used to vary the initial pH of the leaching solution. The leaching solution was tapped off after 2hrs and the filtrate analyzed to determine the concentration of lead and zinc dissolved. The leaching process was also carried using varied grain sizes of the ore concentrate. Furthermore, the final pH of the leaching solution measured in each case.

3. Results and Discussion

3.1 Sedimentation analysis

The chemical composition of the homogenized sulphide ore concentrates used is given in Table 3.

Experimental corrections were made to ensure precision in the results obtained. These corrections were evaluated from the equation already stated. Corrections done includes the meniscus correction Cm = +0.5, temperature correction Mt = +2.3, dispersant correction X = 3.5 and water correction Cw = 1.8 for both galena and sphalerite.

The percentage pretreatment loss was calculated for both sulphide ore concentrates using the equation ;

$$PTL = \frac{IDM - DMP}{IDM} \times 100 \qquad (9)$$

Where
PTL = Pretreatment loss (%)
IDM = Initial dry mass (g)
DMP= Dry mass after pretreatment (g)
LM = Loss of mass due to pretreatment (g)
SS = Sieve size (mm)
MR = Mass retained (g)

Table 1 indicates that the pretreatment loss percent evaluated for galena and sphalerite concentrates is the same and amounts to 23%. The pretreatment loss is suspected to have resulted from further removal of left-over gangue materials from the ore concentrates.

Table 1: Result of Pretreament process carried out

Sulphide ore	IDM	DMP	LM	PTL
Galena	50	38.5	11.5	23
Sphalerite	50	38.5	11.5	23

The specific gravities of Ishiagu galena and sphalerite were determined as 7.50 and 4.10 respectively.

The results of the sedimentation analysis (Tables 4 and 5) indicate that the average grain sizes of Ishiagu galena and sphalerite are 102.72 and 148.8 µm respectively. This implies that galena is finer than sphalerite. This is strongly believed to be so because galena is softer than sphalerite due presence of lead and this characteristic will likely enhance easier loosening of the contact between the gangue materials during size reduction of galena lumps and grains.

Table 2: Size analysis of residue from sedimentation analysis

Galena		Sphalerite	
SS (mm)	MR (g)	SS (mm)	MR (g)
2.00	0.00	2.00	0.00
1.18	0.00	1.18	0.00
0.600	0.80	0.600	0.50
0.425	0.50	0.425	0.30
0.300	1.50	0.300	1.10
0.150	8.20	0.150	23.60
0.100	24.20	0.100	9.60
0.075	3.60	0.075	3.10
Pan	0.25	Pan	0.30

Table 2 shows that out of 38.5g, (being separate masses of residue from the sedimentation analysis of both galena and sphalerite) 24.2 and 23.6g were retained in sieve sizes 0.1 and 0.15mm respectively.

This is a clear confirmation of the results of the sedimentation analysis.

3.2 Metal dissolution per unit mass of ore concentrates

The metal dissolution per unit mass of ore concentrates resulting from leaching galena and sphalerite at a mass range: 2-20g and initial pH 3.2 were determined. Metal dissolution C_m per unit mass of ore concentrates (mg/Kg/g) is calculated from the equation;

$$C_m = C/M \quad (10)$$

Therefore, a plot of concentration of metal dissolved C (from galena and sphalerite) against mass of ore concentrates, m at the leaching temperature; 25^0C (as in Figures 1 and 2) gives a slope, S at points (4, 165.03) & (16, 38.13) and (4,79.58) & (16, 18.66) for galena and sphalerite respectively, following their substitution into the mathematical expression;

$$S = \Delta C/\Delta M \quad (11)$$

Eqn. (11) is detailed as

$$S = C_2 - C_1/ M_2 - M_1 \quad (12)$$

Where

ΔC = Change in the concentration of metal dissolved C_2, C_1 at two mass values M_2, M_1.

Considering the points (4, 165.03) & (16, 38.13) and (4, 79.58) & (16, 18.66) for galena and sphalerite respectively for (C_1, M_1) and (C_2, M_2) respectively, and substituting them into eqn. (12), gives the slope as -10.575 and -5.077 mg/Kg/g respectively. These are the Pb and Zn dissolutions per unit mass of their ore concentrates respectively. A comparison of these two metal dissolutions per unit shows that the leach-ability of PbS in butanoic acid solution to produce Pb is twice that of ZnS to produce Zn. This implies that the reaction between PbS and butanoic acid is twice that between ZnS and same acid. This is confirmed by the fact that the concentration of Pb dissolved in the acid is twice the concentration of Zn dissolved in the same acid.

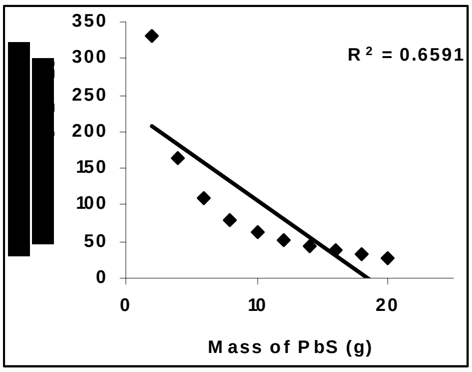

Figure 1: Variation of mass of PbS with the concentration of dissolved lead

The negative values of C_m is an indication of a negative slope which implies that the concentration of metal dissolved decreases with increase in the mass-input of ore concentrates. This is because the H^+ (from the acid) present for the leaching reaction is fixed but the other reacting specie (ore concentrates) increases. As a result the limited H^+ cannot guarantee maximum interaction with the ore, leading to counter productivity.

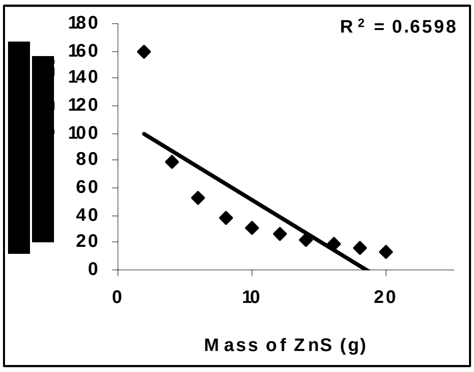

Figure 2: Variation of mass of ZnS with the concentration of dissolved zinc

Point to comparison of the leach-ability of PbS and ZnS shows that PbS has greater tendency to leach-out Pb in butanoic acid solution than ZnS would leach-out Zn.

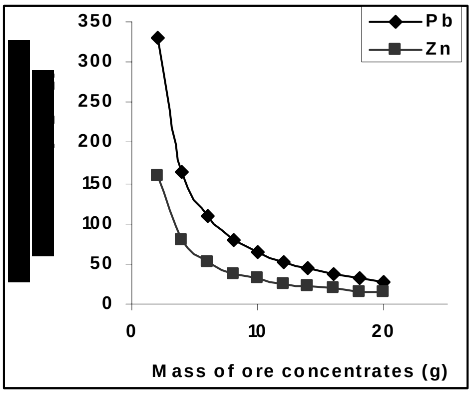

Figure 3 Point to point comparison of the leach-ability of PbS and ZnS to extract Pb and Zn respectively

3.3 Effect of initial pH of leaching solution on the concentration of metal dissolved

Increase in the initial pH of the leaching solution results to decrease in the concentration of metal dissolved. (Figure 4). Increase in the pH of the leaching solution using sodium hydroxide, implies increasing the concentration of OH^- in the leaching solution. Therefore to maintain the ionic equilibrium of water, H^+ in the leaching solution decreases and so the reduction of PbS and ZnS to Pb and Zn respectively by H^+ decreases. This invariably translates to decrease in the concentrations of Pb and Zn dissolved.

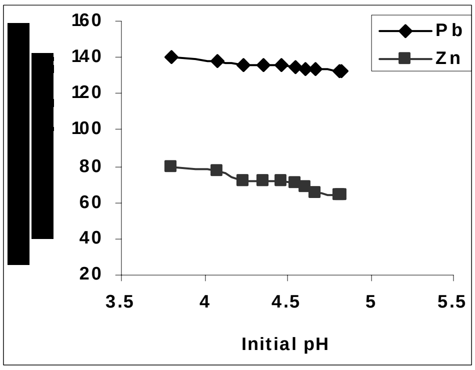

Figure 4: Variation of initial pH of leaching solution with the concentration of dissolved lead and zinc

3.4 Effect of final pH of leaching solution on the concentration of metal dissolved

Figure 5 shows that the concentrations of lead and zinc dissolved drop with increases in the final pH of the leaching solution. This is so because as H^+ from the butanoic acid is consumed during the leaching process, the pH increases. Therefore, steady decline in the concentration of the hydrogen ions, (implying steady increase in the final pH) decreases the reduction of PbS and ZnS to Pb and Zn respectively since H^+ is the reducing agent.

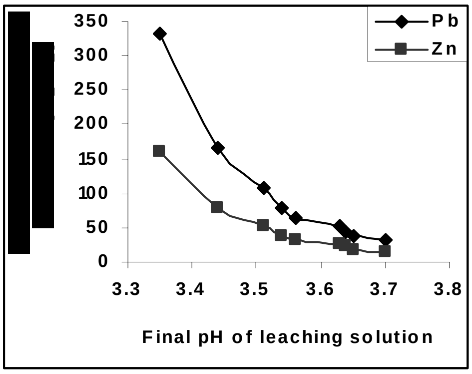

Figure 5: Variation of final pH of leaching solution with the concentration of dissolved lead and zinc

3.5 Effect of varied grain sizes of PbS and ZnS concentrate on the concentration of metal dissolved

Figure 6 shows that increase in the grain sizes of PbS and Zns concentrates decreases the concentrations of metal dissolved. This is because increase in the grain size decreases the surface area of the ore interacting with hydrogen ions. This invariably decreases the reduction of PbS and ZnS by hydrogen ions due to the minimized interaction between H^+ and the ore.

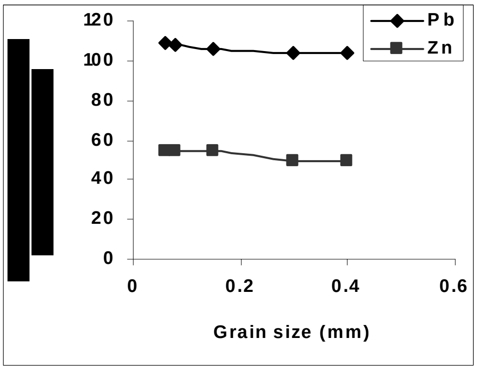

Figure 6 Variation of grain sizes of PbS and ZnS concentrate with the concentration of metal dissolved

It is also believed that higher concentration of Pb dissolved compared to the concentration of Zn due to the smaller grain size characterizing PbS.

Table 3: Result of hydrometer analysis of sphalerite

(1) Serial No	(2) t	(3) Rh^1	(4) Rh	(5) H_R	(6) R	(7) D	(8) K	(9) ΔK	(10) ΔK.D	Calculations
	16/8/2005									
1	1	1.0	1.5	207.85	0.3	42.75	1.03	1.72	73.53	
2	4	0.5	1.0	209.90	-0.2	21.48	-0.69	1.03	22.12	
3	8	0.2	0.7	211.13	-0.5	15.23	-1.72	0.68	10.36	
4	15	0.0	0.5	211.95	-0.7	11.15	-2.40	1.72	19.18	
5	30	-0.5	0	214.00	-1.2	7.92	-4.12	1.72	13.62	
6	60	-1.0	-0.5	216.05	-1.7	5.63	-5.84	3.44	19.37	Dm=∑ΔK.D
7	120	-2.0	-1.5	220.15	-2.7	4.02	-9.28	0	0	∑ΔK
8	180	-2.0	-1.5	220.15	-2.7	3.28	-9.28	1.71	5.61	
9	240	-2.5	-2.0	222.20	-3.2	2.85	-10.99	1.03	2.94	153.27
10	300	-2.8	-2.3	223.43	-3.5	2.56	-12.02	0	0	1.03
12	17/8/2005									= 148.8 µm
	1380	-2.8	-2.3	223.43	-3.5	1.19	-12.02	0	0	≈ 0.150 µm
	1440	-2.8	-2.3	223.43	-3.5	1.17	-12.02	0	0	
	1500	-2.8	-2.3	223.43	-3.5	1.14	-12.02	0	0	
	1560	-2.8	-2.3	223.43	-3.5	1.12	-12.02	-12.02	-13.46	
								1.03	153.27	

Table 4: Result of hydrometer analysis galena

(1) Serial No	(2) t	(3) Rh^1	(4) Rh	(5) H_R	(6) R	(7) D	(8) K	(9) ΔK	(10) ΔK.D	Calculations
	16/8/2005									
1	1	1.0	1.5	207.85	0.3	29.52	0.09	1.50	44.28	
2	4	0.5	1.0	209.90	-0.2	14.83	-0.60	0.90	13.35	
3	8	0.2	0.7	211.13	-0.5	10.52	-1.50	0.60	6.31	
4	15	0.0	0.5	211.95	-0.7	7.72	-2.10	1.50	11.55	
5	30	-0.5	0	214.00	-1.2	5.47	-3.60	1.50	8.21	
6	60	-1.0	-0.5	216.05	-1.7	3.89	-5.10	3.00	11.67	Dm=∑ΔK.D
7	120	-2.0	-1.5	220.15	-2.7	2.77	-8.10	0	0	∑ΔK
8	180	-2.0	-1.5	220.15	-2.7	2.26	-8.10	1.50	3.40	
9	240	-2.5	-2.0	222.20	-3.2	1.97	-9.60	0.90	1.77	92.45
10	300	-2.8	-2.3	223.43	-3.5	1.77	-10.50	0	0	0.9
12	17/8/2005									= 102.72 µm
	1380	-2.8	-2.3	223.43	-3.5	0.82	-10.50	0	0	≈ 0.100 µm
	1440	-2.8	-2.3	223.43	-3.5	0.81	-10.50	0	0	
	1500	-2.8	-2.3	223.43	-3.5	0.79	-10.50	0	0	
	1560	-2.8	-2.3	223.43	-3.5	0.77	-10.50	-10.50	-8.09	
								0.9	92.45	

Table 5: Chemical composition of sulphide ore concentrates used **(%)**

Sulphide ore	Zn	S	Fe	Pb	Cu	Ag	Au	CaO	SiO_2
Galena	1.97	22.20	14.52	51.53	2.18	1.26	0.18	0.38	5.12
Sphalerite	59.06	22.13	9.52	1.28	2.12	0.98	0.11	0.34	4.31

Conclusion

Results of the sedimentation analysis indicate that the average grain sizes of Ishiagu galena and sphalerite are 102.72 and 148.8 µm respectively. This implies that galena is finer than sphalerite. Increase in the mass-input of PbS and ZnS, initial and final pH of the leaching solution decreases the concentration of dissolved metal as a result of minimized reductive tendency of H^+ under this condition. The concentration of lead and zinc dissolved per unit mass of PbS and ZnS concentrates leached in butanoic acid solution was evaluated as 10.575 and 5.077 mg/Kg/g respectively. This implies that the concentration of dissolved lead in butanoic acid is approximately twice that of zinc. This also implies that the reaction between PbS and butanoic acid is twice that between ZnS and the same acid (experimental process and conditions being the same).

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