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# Differential Flotation of Nigeria's Galena-Sphalerite Ore Using Xanthate Collector from Locally Sourced Caustic Potash

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**Abstract:** The Ishiagu galena (PbS)-sphalerite (ZnS) ore deposit was found to contain PbS and ZnS in commercial quantities. The differential flotation of the lead-zinc complex ore to separate the mineral components using potassium Ethylxanthate (KEX) prepared from locally available caustic potash was studied. Combinations of $C_3$ and $C_5$ alcohols as frother was used. The effect of $CuSO_4.5H_2O$ as an activator of sphalerite was studied at different activation times and at different pH values. Sphalerite was satisfactorily recovered using the activator. Maximum recovery of sphalerite was achieved, thereby establishing the zero-point-of-charge of sphalerite at this hydrogen ion concentration. Results obtained showed that high recovery of PbS (about 87%) was achieved at activation time of 20 min. Over 70% increase in the recovery of ZnS with copper sulphate pentahydrate as activator and prepared potassium ethylxanthate as collector was achieved. These results proves high collector ability of the local potassium ethylxanthate.

**Key words:** Galena, sphalerite, adsorption, activation, flotation recovery, Potassium ethyl xanthate, zero-point-of-charge (zpc), caustic potash

## INTRODUCTION

The minerals, galena (PbS) and sphalerite (ZnS) are the major sources of lead and zinc metals. The serve as raw materials for local smelting industries and great potentials for foreign exchange if processed (Abubakar, 2004). The method of recovery is by froth flotation. Froth flotation is an important and versatile mineral processing technique which utilizes the difference in the physical and chemical surface properties of minerals (Onyemaobi, 1990).

Many studies have shown that galena sphalerite (PbS-ZnS) Ore can be floated using xanthate ions and different collectors (Onyemaobi, 1990; Onyemaobi and Anyokwo, 1996; Ajayi, 2005). Different recovery using different raw material has been achieved (Sui *et al.*, 1999; Ofor, 1997). Sodium and potassium ethylxanthates has proved to be the most effective collector ion for the flotation of surphide minerals (Wills, 1980). The cost of sodium or potassium xanthate is high and are imported as fine chemicals. The present study aims at preparation of the potassium Ethylxanthate (KEX) using caustic potash from dry palm fruit bunch of Elaeis guineensis for the differential flotation of the ore at varying activation times and pH values.

## MATERIALS AND METHODS

**KEX preparation:** Sun dried chaff of palm fruit bunch was burnt to ash. Five hundred g of the ash leached with water. The filtrate evaporated to dryness and crystals obtained was assayed for potassium. The crystal was added to ethanol bought from local distillers (at source). Stirred to dissolve. Petroleum ether was added to form slurry. Carbon disulphide was added and stirred for five min and maintained at 40°C. After twelve hours the product was separated by filtration and dried in a dessicator.

**Ore preparation:** The galena-sphalerite ore was obtained from the Ishiagu mines in Ebonyi State of Nigeria. Representative sample of the ore as received were reduced to 100% minus 8 mesh size by consecutive crushing and grinding using the Blarke Jaw crusher and laboratory rodmill. The ground ore was passed through an 8 mesh screen between successive crushing and grinding steps.

**Chemical analysis:** Chemical analysis on the ore sample was carried out at National Metallurgical Development Center Analytical Laboratory, Jos, Nigeria using Atomic

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Absorption Spectrophotometer bulk 205 model; the representative sample was pulverized using a ball mill grinder and digested using hydrofluoric acid, perchloric acid.

**Sieve analysis:** The sieve analysis was performed on a 500 g representative sample of the reduced ore using a nest of sieves and a mechanical screen vibrator. The weight of ore retained on each screen was determined by weighing.

**Microscopic counting:** Portions of each screen portions were placed under a petrological microscope. The total mineral particles as well as the separate galena and sphalerite particles that were liberated up to 50% were counted. The degree of liberation of galena and sphalerite in each screen portion was then computed. From these values the Mesh-of-Grind (MOG) of the ore was determined by comparing the results of chemical analysis and sieve analysis.

**Flotation tests:** Two hundred grams of the pulverized ore sample (-300 µm or-52# BSS) was put in a 2-liter Denver Laboratory Flotation cell. Fifteen hundred cm$^3$ of tap water was run into the cell to obtain the desired dilution. The pulp was conditioned for 10 min and the ore floated using 3 g of the prepared potassium ethyl xanthate of Elaeis guineensis as collector and 3-drops of (combination of $C_3$-$C_5$ alcohols) frother were added to the pulp, agitated for 10 min and floated for 3 min by opening the air jet. The ore concentrate was collected, dried in an oven at 110°C and weighed. A representative sample of 10 g was used for the chemical analysis.

The flotation test was repeated on 200 g samples of the pulverized ore at different agitation times at 10 min interval up to 60 min. These tests were carried at the natural pulp pH of 4.6 and at ambient temperature of 30°C.

Two hundred g of the pulverized ore was floated as in (1) above, $CuSO_4.5H_2O$ solution ($2.5\times10^{-3}$ mole/ton ore) (Sui *et al*., 1999) was added as activator to the residual ore pulp (tailing) (Sui *et al*., 1999) pulp was agitated for 10 min and the ore then floated using the prepared potassium ethyl xanthate of Elaeis guineensis as collector and the frother (combination of $C_3$-$C_5$ alcohols).

This concentrate was dried, weighed and a representative sample of it was submitted for chemical analysis. Flotation tests were repeated at different activation times at 10 min intervals up to 60 min.

Flotation on 200 g samples were carried out at agitation time of 10 min at different pH-values. $CuSO_4.5H_2O$ activator was added to the residual ore pulp and floated at the optimum activation time of 10 min, at different pH-values of 4, 6, 8, 10 and 12. The concentrates were used for chemical analysis.

Table 1: Chemical analysis of pbs-zns ore

| Element | PbS | ZnS | $CaCO_3$ | $SiO_2$ | FeS |
|---|---|---|---|---|---|
| %Composition | 21.05 | 66.00 | 3.80 | 3.05 | 1.01 |

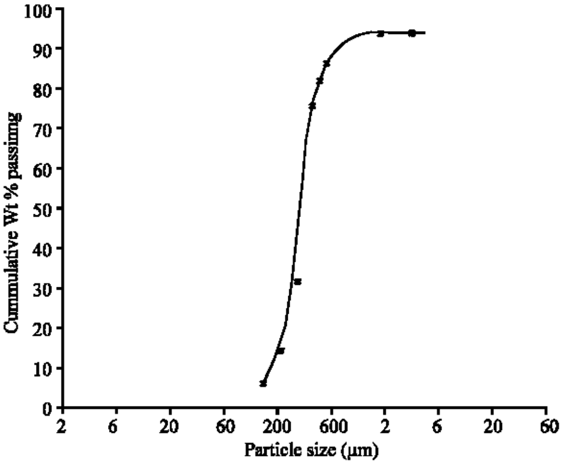

Fig. 1: The plot of sieve of PbS-ZnS Ore

## RESULTS

Table 1 represents result of the Chemical analysis of galena-sphalerite ore received from Ishiagu, Nigeria. Figure 1 is the graphical presentation of the sieve analysis of the minus 8 mesh reduced ore also shown in Table 2. The result of microscopic counting is shown in Table 3.

Figure 2 is the graphical presentation of the result of the flotation of the PbS-ZnS ore at varying agitation times using KEX prepared from caustic potash of elaeis guineensis in the absence of copper activation. The result of the flotation of residual PbS-ZnS ore at varying activation times using hydrated copper sulphate as activator is graphically presented in Fig. 3 and 4 is the graphical presentation of the results of the flotation of the residual PbS-ZnS ore at varying pH values using copper activation.

## DISCUSSION

The production of potassium Ethylxanthate (KEX) using palm fruit ash as source of alkali with the characteristic odour yielded good result.

The dissociation of KEX in shown is this order

$$C_2H_5OCS_2K \longrightarrow C_2H_5OCS_2^- + K^+ \qquad (1)$$

Table 2: Sieve analysis of the #8 reduced ore

| Mesh number | Weight of ore retained | Weight % retained | Nominal aperture (µm)"x" | Cumulative weight retained (% oversize) | Cumulative weight passing (% under size) "y" |
|---|---|---|---|---|---|
| +8 | 6.20 | 1.24 | 2000 | 1.24 | 98.76 |
| -8+25 | 30.00 | 6.00 | 600 | 7.24 | 92.76 |
| -25+30 | 19.95 | 3.99 | 500 | 9.99 | 90.01 |
| -30+36 | 12.55 | 2.51 | 425 | 12.50 | 87.50 |
| -36+52 | 49.95 | 9.90 | 300 | 22.40 | 77.60 |
| -52+150 | 237.95 | 47.59 | 106 | 69.99 | 30.01 |
| -150+200 | 81.05 | 16.21 | 75 | 86.20 | 13.80 |
| -200+240 | 30.00 | 6.00 | 63 | 92.20 | 7.80 |
| -240 | 31.00 | 6.20 | | | |

Table 3: Microscopic counting

| Particlesize mesh no. | Nominal aperture size µm | | Total No. of mineral particle | No. of galena | % galena particle | No of sphalerite | % sphalerite | No. of silica particles | No. of locked particles |
|---|---|---|---|---|---|---|---|---|---|
| 25 | 600 | | 10 | 2 | | 8 | | | |
| | | | 14 | 2 | | 10 | | | 2 |
| | | | 13 | 3 | | 9 | | | 1 |
| | | Total | 37 | 7 | 8.91 | 30.27 | 72.97 | | 3 |
| 30 | 500 | | 35 | 5 | | 25 | | 5 | |
| | | | 26 | 5 | | 20 | | 1 | |
| | | | 37 | 3 | | 24 | | 10 | |
| | | Total | 98 | 13 | 13.30 | 69 | 70.40 | 16 | |
| 36 | 425 | | 25 | 2 | | 20 | | 3 | |
| | | | 22 | 4 | | 15 | | 3 | |
| | | | 32 | 5 | | 25 | | 2 | |
| | | Total | 79 | 11 | 13.90 | 60 | 75.94 | 8 | |
| 52 | 300 | | 9 | 5 | | 4 | | | |
| | | | 15 | 1 | | 14 | | | |
| | | | 34 | 11 | | 21 | | 2 | |
| | | Total | 58 | 17 | 29.30 | 39 | 67.20 | 2 | |
| 150 | 106 | | 36 | 9 | | 26 | | 1 | |
| | | | 29 | 8 | | 19 | | 2 | |
| | | | 39 | 8 | | 23 | | 8 | |
| | | Total | 104 | 25 | 24.00 | 68 | 65.40 | 11 | |
| 200 | 75 | | 160 | 20 | | 130 | | 10 | |
| | | | 181 | 34 | | 115 | | 22 | |
| | | | 126 | 37 | | 72 | | 17 | |
| | | Total | 467 | 91 | 19.49 | 317 | 67.88 | 49 | |
| 240 | 63 | | 63 | 12 | | 29 | | 22 | |
| | | | 100 | 20 | | 42 | | 38 | |
| | | | 110 | 14 | | 46 | | 50 | |
| | | Total | 273 | 40 | 16.80 | 117 | 42.80 | 110 | |

Also in the aqueous medium, the following reactions occur

$$PbS + 2C_2H_5OCS^-_2 + H_2O \longrightarrow Pb(C_2H_5OCS_2)_2 + HS \quad (2)$$

Insoluble lead ethyl xanthate and

$$Zns + 2C_2H_5OCS^-_2 + H_2O\ Zn \longrightarrow (C_2H_5OCS_2)_2 + HS \quad (3)$$

Insoluble Zinc ethyl xanthate

Table 1 indicate that the Ishiagu ore is predominantly sphalerite (66%), with a substantial amount of galena (21.03%) and suppressed amounts of calcite (3.80%), silica (3.05%) and feldspar (1.01%).

The plot of cumulative percent passing ("y") versus the particle size ("x") was carried out. The graph of cumulative weight percent passing ("y") versus particle size (microns) ("x") represented in Fig. 2 shows the 80% passing size or the mesh-of-grind (MOG) is at 300 µm or 52 mesh (BSS 410). This is the ore particle size as established by sieve analysis at which the mineral components of the ore are optimally liberated.

Microscopic counting (Table 3) gave MOG of 52 mesh for galena and 36 mesh for sphalerite. For flotation tests therefore the 8 mesh ore was pulverized to 100% minus 52 mesh.

Figure 2, shows the optimum impeller agitation time for maximum recovery of both PbS-ZnS is 10 min. That is, in the absence of copper activation, the prepared and

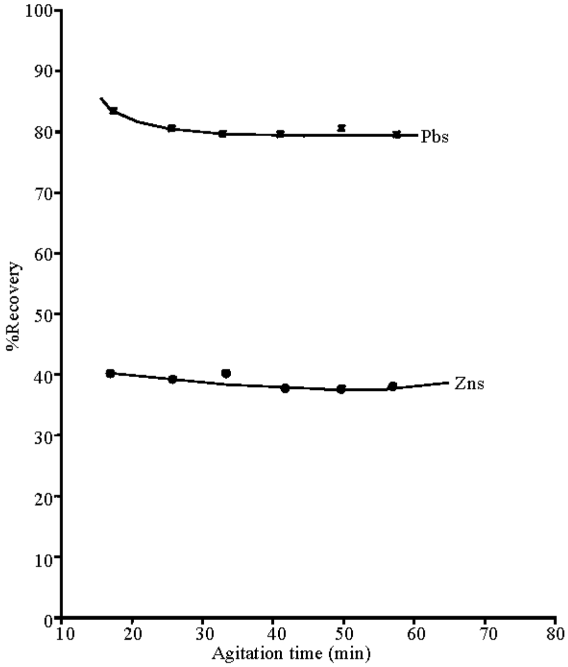

Fig. 2: Flotation of PbS-ZnS Ore at Varying agitation time without copper

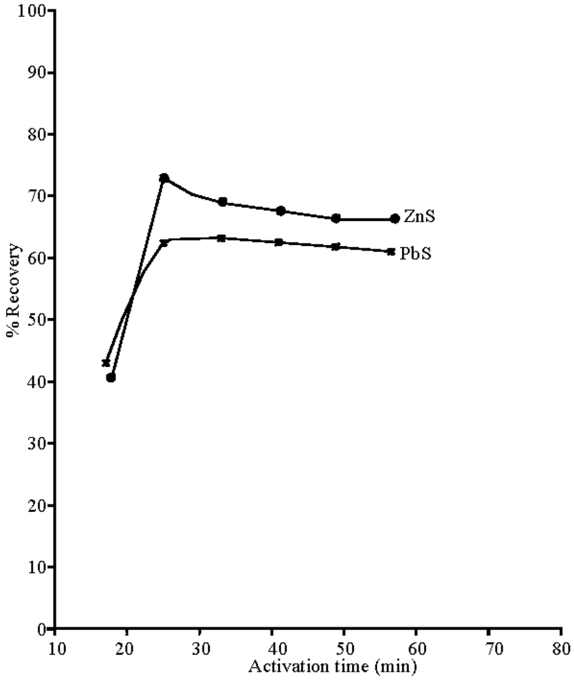

Fig. 3: Flotation of residual PbS-ZnS Ore at Varying activation time using copper activation

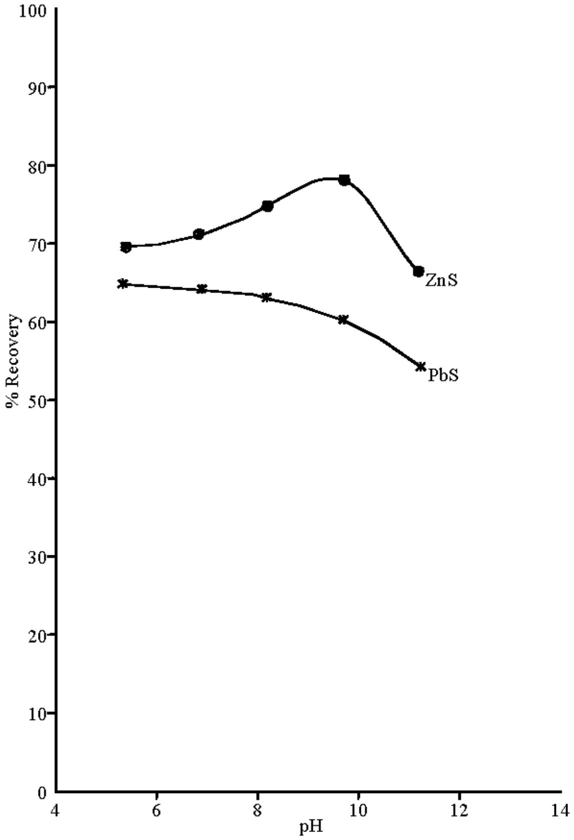

Fig. 4: Flotation of residual PbS-ZnS Ore at Varying activation time using copper activation

unpurified potassium ethylxanthate collector is optimally adsorbed onto the mineral surface within 10 min of agitating the ore pulp. Figure 3, gave the optimum activation time for the maximum percent recovery of both galena and sphalerite at 20 min. That is the collector, potassium ethylxanthate is optimally adsorbed onto both mineral surfaces within 20 min of activation. This activation time of 20 min was kept constant through out the flotation tests.

Figure 3 shows a substantially higher recovery of sphalerite (about 73% average max) than Fig. 2 (42% maximum). These figures represents over 70% increase in recovery and further show that $CuSO_4 . 5H_2O$ is effectively activating the sphalerite to enhance its flotation. Figure 3 further shows that at any given activation time, more sphalerite is recovered than galena showing further that sphalerite is being activated by copper well and above galena.

Figure 2 and 3 show higher recoveries of galena without copper activation (about 87% maximum) than with copper activation (about 65% maximum, Fig. 3). This indicates that the bulk of galena floats directly with xanthate collector and does not require copper activation.

Figure 4 shows that recovery of sphalerite increases gradually with increasing pH to a maximum recovery of

83% at pH 10; at higher pH values recovery falls. The opposite trend is observed with galena where recovery is highest at low pH values and decreases gradually towards higher pH values with a rapid drop beyond pH 10.

The recovery of activated sphalerite rises to a maximum at pH 10 beyond which pH it falls, indicates that the Zero-Point-of-Charge (ZPC) of sphalerite is in the region of pH 10. A further reason for the fall in recovery beyond pH 10 would be that the preponderance of hydroxyl ions above pH 10 would lead to OH-ions competing with xanthate collector ions for adsorption at the mineral-water interface.

## CONCLUSION

A Nigerian galena-sphalerite complex ore in which the minerals galena (PbS) and sphalerite (ZnS) are present satisfactorily separated into different concentrates of galena and sphalerite by differential flotation using potassium ethylxanthate prepared using caustic potash from dry palm fruit bunch of *Elaeis guineensis* with a combination of $C_3$ and $C_5$ alcohols as frother. Sphalerite was readily floated by the prepared potassium ethyl xanthate with activation by copper: over 70% increase in the percent recovery of sphalerite was obtained with copper activation.

The recovery of activated sphalerite rises to a maximum at pH 10, beyond which pH recovery falls. This indicates zpc of 10 for sphalerite. It also suggests at pH 10 and above there is a preponderance of hydroxyl ions competing with xanthate ions for adsorption onto the mineral-water interface.

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