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Separation and Concentration of Stibiconite

William Howard Painter

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SEPARATION AND CONCENTRATION OF STIBICONITE

by

A Thesis

Submitted to the Faculty of the Department of Mineral Dressing
in Partial Fulfillment of the Requirements for the Degree of Bachelor of Science in Metallurgical Engineering Mineral Dressing Option

> Montana School of Mines Butte, Montana
May 28, 1959

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TABLE OF CONTENTS

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INDEX TO FIGURES

THE AUTHOR

William Howard Painter was born in Livingston, Montana, October 16, 1937. He attended elementary schools in Livingston and was graduated from Park County High School in 1955. In 1959, he received a degree in Metallurgical Engineering (Mineral Dressing Option) from Montana School of Mines.

While attending Montana School of Mines, he was an active member of the student chapter of the AIME and a member of Theta Tau Fraternity. In the latter, he held the office of corresponding secretary.

Mr. Painter was employed by the Anaconda Company on a part time basis during the school year of 1955-56. During the summers, he was employed by the Montana Power Company as a laborer, draftsman, and during the summer of 1958 as a junior engineer. During his last three years at Montana School of Mines, he was employed in the engineering department of the Montana Power Company at Butte, Montana.

ACKNOWLEDGMENTS

The writer wishes to express his thanks to Professor Donald McGlashan, Research Professor, and head of the Department of Mineral Dressing; and Mr. George Gale, assistant head of the Metallurgical Research Department of the Anaconda Company, for their help and guidance in the development of this project. Special thanks is expressed to Equipment Engineers Incorporated for the Krebbs D4B Cyclone, and also to the Texas Smelting and Refining Company for the ore sample.

ANTIMONY IN INDUSTRY

The largest users of antimony in the United States are the producers of alloys used for bearing metals, printing type and battery construction. Antimony metal is alloyed with tin, lead and sometimes copper if a harder alloy is needed than the antimony alone can furnish. Antimony is also used in pewter and britannia metal but its content must be kept within the range of 6-10 percent, as antimony causes the alloy to be brittle and is for that reason used in the form of casting. The paint industry uses large quantities of the oxide as a pigment, its hite color being used as a base.⁸

Antimony occurs in a considerable number of minerals especially those belonging to a group known as the sulfo-salts: which are largely combinations of copper, lead, or silver, with antimony and sulfur. Deposits containing these minerals are mined, however, for the other metals that they contain and any antimony produced from them is in the nature of a by-product. Stibnite is practically the only mineral that is mined for its antimony and it has been found in the United States in comparatively few deposits. It has been mined on a ll scale in California, Nevada, and Idaho; but the chief source domestic antimony produced in the United States is from drosses in lead refining plants. The largest producer of antimony, as of 1948, s Bolivia; followed by Mexico, the United States, and China. 8

 1 ,

PURPOSE OF THE INVESTIGATION

Primarily, the purpose of this study is to separate stibiconite from associated gangue minerals. The ore containing antimony, as an oxide stibiconite, occurs in Mexico and its benefication would be an advantage to the mineral resources of that country. While the deposit from which this ore is derived is American owned, the resource is presently non-economic. A concentrate must be derived of suitable grade for its use in a smelting operation and from which metallic antimony would be produced.

Two approaches are considered in this report for the separation and concentration of the stibiconite, namely, flotation and cyclone separation. For both of these methods the stibiconite must be freed from the associated gangue minerals by crushing, grinding, and the auxiliary methods associated with these operations. On the one hand, flotation is an application of surface chemistry through which the desired mineral is made air avid and is separated from the gangue minerals as a froth. The problem in application to the stibiconitebearing ore is one of reagent selection and soluble ion control. On the other hand, cyclone cone separation is an application of centrifugal force to supplement that of gravity. The stibiconite, having a specific gravity greater than that of the associated minerals, is effected by this force situation to an extent that separation might be effected to a greater extent. In the body of this report, and at he two processes of benefication are described, they will be described in more detail.

 $-2 -$

MINERALOGY

The mineral, stibiconite, is sometimes found in connection with stibnite. In the antimony sulfide deposits, it can be found in the upper layer which has been exposed to weathering. The composition and the crystal system of stibiconite are not definitely known, but ccording to Dana, its chemical composition is thought to be Sb306)(0H) and its crystal system is thought to be Isometric; Hexoctahedral.

The mineral has a hardness within the range of $4.0-5.5$ and the specific gravity of the pure mineral is about 5.58. The mineral has a color ranging from pale yellow to yellow white, red white. The mineral usually contains calcium oxide (CaO) but the role of the calcium is uncertain.²

CONCENTRATION BY FLOTATION

An extensive research program has been carried out at the Montana School of Mines during the past few years and it would appear that flotation could be used as a means of concentration of this ore, if the proper reagent could be found to control the soluble calcium ions, which tend to interfere with the collector.

Undesirable Characteristics

The collectors most generally used for the flotation of oxides, such as stibiconite, are the carboxylates, or fatty acids, but presence of the calcium ion tends to precipitate this type of collector as an insoluble soap as exemplified below.

$$
Ca2 \times 2 CH3(CH2)10CO2H = \frac{(CH3(CH2)10CO2)2Ca}{Calcium Laurente (Insol.)}
$$

"The insoluble soap formed has the tendency to smear all of the particles with the gummy soap and carry them into the froth." To have a successful flotation process, the collector must be kept unaffected by the calcium ion by using a modifying reagent which could tie the calcium ion up as will be discussed below.

Chelation

Chelation might be considered to be a reaction between a metal and an electron donor, which has two or more groups, and which will form a complex with two or more ring structures. It could be contrasted to a simple complex in which a metal reacts with a simple electron donor.⁸

 $-4 -$

The chelating agent most frequently used is ethylenediaminetetraacetic acid (EDTA) or one of the salts of this acid. The Dow Chemical Company produces a whole series of these chelating agents under the trade name of "versene". When the name versene is used without any other designation, it is assumed to be the tetra-sodium salt of ethylenediaminetetraacetic acid. Chelating agents have been in use for some time in industry where the presence of certain metals, as free ions, would be quite harmful. Among these are the rubber producers, who require the removal and control of copper and manganese ions; the soap manufacturers, who require the removal of calcium ions; and the manufacturers of iron products who cannot allow any surface oxidation of their iron products.¹

An example of ion control has been demonstrated in a series of experiments performed on living animals in an attempt to control the calcium level in blood serum.

Protection by Ca and Mg against a lethal dose of EDTA to rabbits. The solid line represents intravenous lethal dose; the dashed line represents lethal dose \neq 1 equivalent of Ca I.V. (additional lethal dose at 6 minutes).

 $-6-$

The results of the experiments by Popvivi, Geshickter, Reinovsky, and Rubin⁹ indicate that with proper administration of the chelating agent, the effect of free calcium ions can be made to approach zero within the time range of 2.5-2.7 minutes. To insure enough EDTA in solution, it is necessary to know the rate of dissolution of the calcium. If this is impossible, it becomes necessary to "swamp" the pulp with EDTA. A method for the quantitative analysis of calcium ion dissolution is outlined further along in this report.

At a high pH, the effect of the calcium ion become negligible but the cost involved in maintaining this pH and the cost of maintaining equipment at this pH is quite costly. To summarize, the chelating agent produces the same effect as a high pH, but does so at a much lower cost.

The reaction between the calcium ion and the versene could be shown thus,

 $-7-$

The stability constants of chelating agents with different metals may be calculated through thermodynamic data but the results are largely imperical and can at best, give an indication of the stability constants. the following is a discussion of stability constants and their thermodynamic derivations.⁹

 $aA + bB + \cdots = cC + dD + \cdots$

If the activities of A and B are a_A and a_B at the end of the reaction and,

If the activities of C and D are a_C and a_D at the end of the reaction then,

The free energies/mole at any temperature T are given by

 $F_A = F_A^O + RT \ln a_A$ $F_B = F_B^0 \div RT \ln a_B$
 $F_C = F_C^0 \div RT \ln a_C$
 $F_D = F_D^0 \div RT \ln a_D$

Therefore, the free energy change of the reaction is

 $\Delta F = (cF_C + dF_D) - (aF_A + bF_B)$ and $\Delta F = \Delta F^{\circ} + RT \ln \frac{a_C^{\circ} a_D^{\circ}}{a_A^{\circ} a_B^{\circ}}$

This is known as the reaction isotherm because, for any reaction F^o is constant at any given temperature and is completely independent of pressure.

> at equilibrium, $\Delta F = 0$ and $\triangle F^0$ = -RT ln $\frac{a_C^c}{a_A^a} \frac{a_D^0}{a_B^0}$

Since the activities are now at equilibrium, it may be said that,

$$
a_C^c a_D^d = K_a
$$

\n
$$
a_A^a a_B^b
$$

\nor,
$$
\Delta F^o = -RT \ln K_a
$$

Ka is the equilibrium constant expressed in activities and is called the thermodynamic equilibrium constant. This may also be called the stability constant for the chelating agents.

Concentration or partial pressures may be substituted for activities in the equation but the results can, at best, approach the true equilibrium constant.

For a typical chelation reaction,

 $M + xKe = MKe_x$

where $M = a$ metal and,

 $Ke = a$ chelating agent

Thus,

$$
K_{a} = \frac{a(MKe_x)}{a(M)a(Ke)^{X}}
$$

The "apparent" equilibrium constant may be calculated by substituting the molar concentrations for the activities but this calculated value of the stability constant will, at best, yield a close approximation of the true stability constant. Concentrations are substituted for activities because they are much easier to handle and determine at equilibrium. 10 Thus,

$$
K_{c} = \frac{mMKe_{X}}{mM \ mKe^{X}}
$$

and,

 $K = K_c$ am $K e_X$
am akex

where,

 K_c : the "apparent" equilibrium constant K = the thermodynamic formation constant m = molar concentration

a = activity coefficient

For successful chelation, the stability constant must be large; that is, the concentration of the metal ions in equilibrium with the chelating agent must be very small.⁹

 $-10 -$

Quantitative Determination of Calcium ions

If a versene is used to remove the calcium ion from the pulp, it is necessary to determine, quantitatively, the number of calcium ions present in the pulp. If this is not possible, the pulp must be "swamped" with versene to insure calcium removal.

A method for determining water hardness by titrating with a chelate has been suggested by Biedermann and Schwarzenbach.⁹ Since the calcium ion is the principle offender in hard water, this method may be used for calcium ion determination in an ore pulp such as stibiconite.

In a solution where both calcium ions and magnesium ions are present, the chelating agent will preferentially remove the calcium ions first and then the magnesium ions. To take advantage of this phenomenon, a small number of magnesium ions are added to a solution of Eriochromeschwarz-T and the pulp titrated. After the free magnesium ions are removed, the EDTA will remove magnesium from the dye and a color change will occur. "Since the wine-red chelate of the indicator dye is less stable than the blue chelate of the EDTA it can be used as a quantitative indicator." The following equation will illustrate the chemical reaction

$$
MgF^{-2} + H = HF^{-2} + MgEDTA^{-2}
$$

Red Blue

where,

EDTA represents the chelating agent and, HF-2 represents an ionized form of Eriochromeschwarz-T

 $-11-$

Metal Chelate of Erichromeschwarz-T (Red)

The reagents used and the method used to prepare them is listed

below.

- 1. Buffer Solution 1 part of 1.0M (Molar) NH₄Cl was mixed with 5 parts of 1.0M NH₄OH and a solution with a pH of just over 10 resulted.
- Standard Calcium Chloride Solution 1.000 g. of pure $2.$ CaCO₃ was dissolved in dilute HCl, neutralized with NH_LOH until slightly alkaline and then diluted to 1000 cc.
- Indicator 2.00 g. of Eriochromeschwarz-T is dissolved $3.$ in 500 cc. of alcohol.
- Standard Versene Solution 3.00 g. of purified versene 4. was dissolved in water containing 1.5 g. of N_aOH. 0.100 g. of MgCl₂.6H₂O was dissolved in water, added to the versene solution and the mixture diluted to 1000 cc. After primary standardization, enough water is added to make 1 cc. of versene equivalent to 1.00 mg. of calcium carbonate.

A sample of the ore with a pulp density of about 50 per cent seems to be most satisfactory for analysis. Addition of the chelating agent can be done either quickly or slowly if the reaction is allowed to go to equilibrium.

CONCENTRATION BY CENTRIFUGAL METHODS

The first cyclones were used for dust collection, but in more recent times cyclones have been used for desliming, for classification, and most recently, for concentration. The first cyclones used were quite similar, in construction, to those in use today; that is, a vortex finder and an adjustable appex orifice were incorporated in the construction. In about 1936, M. G. Driessen⁴, of the Dutch State Mines, developed a cyclone which used no vortex, but instead a knife edge blade which surrounded the overflow. Later cyclones went back to the vortex finder until now it is standard with cyclone construction.

Cyclones, as classifiers, are used as closed circuit classifiers in place of conventional methods, or in conjunction with these methods, by the producers of copper, lead, zinc, uranium, dement, chemicals, and many others. When used in conjunction with the spiral or the rake classifier, the cyclone serves to remove extreme fines which result in a cleaner and denser overflow from the classifier to flotation or whatever the next concentration process might be.

Cyclones are used to desand oil well mud, desilt water supply systems, dewater tailings, or to deslime such materials as uraniumbearing sands or mine fill. One specific example of successful concentration or upgrading with a cyclone is at the Acoje Mining Company where a tailing, weighing about 65 tons per day and assaying about 20% Cr_2O_3 , is pumped through a bank of three cyclones. The concentrated product from the cyclone yields about 21 tons per day of concentrate which assays 50% Cr₂0₃ for a recovery of approximately 81%.

 $-14 -$

The taconite deposits of northern Minnesota and Michigan present a problem because to be competitive with the rich iron ores, the taconite must be concentrated from its present 18-25% iron to over 64% iron. Magnetic separators are used to concentrate the ore. A two step concentration process is used; that is, the ore is fed to a rougher magnetic separator and from there, the concentrate is fed to ball mills. The discharge from the ball mill is then sent to the cleaner magnetic separators and from there the concentrate is demagnatized and sent to cyclones. The overflow from the cyclone is sent to further processing while the underflow is recirculated to the ball mill for further liberation.⁷

Separation in a cyclone is affected by, all other things being equal, particle size and pecific gravity. If the particle size were controlled, it would be possible to expect that concentration using the differences in specific gravity could be affected. Figures 1 and 2 illustrate the forces acting upon a particle and the working principle of the cyclone.

 $-15 -$

Fig. 2-Forces working on suspension particles in cyclone Full lines; path of liquid; dotted lines; path of suspension particles

Figure 3-Illustration showing the working principle of the cyclone.

Enlarged Section A-A

Laboratory Cyclone

The laboratory cyclone is a four inch, 42° model with an overall length of 10 inches. The inlet is $3/8$ inches in diameter while the overflow is $1/2$ inch in diameter. The underflow is $3/8$ inches in diameter, but, it has an adjustable orifice which will allow a range of sizes from full open to closed. The adjustable orifice is of the valve type instead of the conventional rubber lined, uniform diameter type control; for this reason, the orifice has a tendency to clog. The vortex is adjustable in length only and not in diameter, as on the Krebbs model cyclone.

The cyclone is fed with a Fairbanks-Morse centrifugal pump which can deliver about 20-25 gallons per minute at 20 pounds per square inch. The laboratory cyclone is illustrated in figure 4 , and the laboratory cyclone circuit is illustrated in figure 5.4

Krebbs Cyclone

The Krebbs cyclone operates on the same principle as the laboratory model cyclone but with this model, the apex orifice is controlled with a lever operated device instead of the valve type control which is used on the laboratory cyclone, and the vortex on the Krebbs cyclone may be replaced with vortexes of different diameters; this is to be contrasted with the vortex of the laboratory cyclone which is adjustable in length only.

The installation of the Krebbs cyclone necessitated dismantlent of the laboratory model cyclone in order to salvage the

 $-18-$

centrifugal pump and motor. It was also necessary to construct a table upon which the cyclone and the pump-motor could be mounted. An agitator tank was used which was standing idle in the mineral dressing department's pilot mill, however, a new pulley system had to be built to convey power from the motor to the agitator drive unit. Figure 6 shows the laboratory installation of the Krebbs cyclone unit.

Operation of the Cyclone

The operation of the cyclone is, in both types, dependent largely upon the type of feeding to the cyclone; that is, the size feed, and less emphasis should be put on the adjustable parts of the cyclone.

The effect of a vortex finder adjustment is, generally, the larger the cross-sectional area, the coarser the cut and the smaller the cross-sectional area, the finer the cut. Out might be termed efficiency in making a clean cut at a certain specific gravity if the device is being used for concentration, or in making a clean cut at a certain particle size if the device is being used for sizing. It might be pointed out that too much emphasis on a fine cut might cut down the volume of discharge to such an extent that the cyclone operation might become non-economical. The apex orifice adjustment regulates the air column as well as the underflow discharge but the usual adjustment is to regulate the fineness of the spray.

 $-19-$

Figure 6--Krebbs Cyclone Circuit

Legend:

- Motor $1.$
- Centrifugal Pump $2.$
- Krebbs Cyclone $3.$
- Adjustable Apex Orifice Handle 4.
- 5. Replaceable Vortex Finders
- 6. Replaceable Apex Liners
- 7. Overflow
- 8. Inlet Pressure Gage
- 9. Agitator Tank

Calculation Formulae and Examples

The following formulae and examples are taken from the Krebbs Cyclone Bulletin #822, and they have been derived through actual industrial calculations:

Let $x =$ specific gravity (sp. gr.) of the pulp

 $y = per$ cent dry solids in the pulp

 $z =$ cubic feet (ft3) in one ton of dry solids for a given specific gravity.

Per cent solids in the pulp = $\frac{sp. gr. \cdot 100 (x-1)}{(sp. gr. -1) x}$ l.

Specific gravity of the pulp-
 $\frac{sp. gr. \cdot 100}{sp. gr. \cdot 100 - (sp. gr. -y)}$ $2.$

Cubic feet of pulp needed to make one dry ton of solids = $3.$ $\frac{2(sp. gr. \cdot 100) - (sp. gr. - y)}{y}$

Tonnage (gallons per minute (gpm) and per cent solids known) 4. $gpm \cdot g = ft^3$ of pulp/min.

Dry Tons (DT) = ft^3 pulp
 ft^3/ton

- Cubic Feet of pulp = $\frac{gpm \cdot 8}{tph}$ $5.$
- Gallons per minute = $\frac{\text{tph} \cdot \text{ft}^3 \text{ pulp}/\text{dry}}{8}$ $6.$

The following examples are used to illustrate the calculations involved in a typical cyclone operation and they are taken directly from the Krebbs Cyclone Bulletin #822. In the examples a specific gravity of 2.6 is assumed for the solids.

1. Overflow

cyclone measuring 200 gpm with a density of 10% solids It can be found that at 10% solids, it would take 300.31 ft³ to make one dry ton of solids.

> 200 gpm \cdot 8 = 1600 ft³ ¹⁶⁰⁰*=* 5 .33 dry ton/hour 300.31

2. Underflow

A cyclone measures 20 gpm with a density of 68% solids. It can be found that at 68% solids, it would take 27.37 ft³ to make one dry ton of solids.

```
20 gpm • 8 = 160 \text{ ft}^3160= 5.84 TPH 
27.37
```
3. Feed

To calculate the feed, the overflow and the underflow gallonages and tonnages are dded together to make 220 gpm and 11.17 TPH feed rate.

> $220 \cdot 8 = 1760 \text{ ft}^3$ ¹⁷⁶⁰*=* 157.56 ft3 of pulp/ton 0f solids 11.17

Through further calculations, it is possible to find that the pulp is made up of about 18% solids.

 $-24 -$

EXPERIMENTAL WORK

The experimental work on the separation and concentration of stibiconite was hampered by the fact that the chelation indicator did not arrive in time for analysis and the fact that the Krebbs cyclone came in time for partial installation only. After the arrival of the Krebbs cyclone, the laboratory model had to be dismantled to make use of its pump and motor.

Size Reduction

Preliminary investigation indicated that part of the ore had a tendency to powder while the rest of it was more resistant to crushing. To overcome this difficulty and keep the production of fines to a minimum, the ore was run through a vibrating screen before each successive crushing step. A rod mill was chosen for the final grinding step due to the rod mills' inherent characteristic of preferential grinding; that is, grinding the larger particles before grinding the fines.

Chelation

The chelating solutions were prepared and the apparatus for determining, uantitatively, the number of calcium ions present in a solution was set up but since some of the chemicals did not arrive, no experimental research was carried on.

 $-25 -$

Cyclone

An artificial ore was made up of galena and quartz in an attempt to become familiar with the operation of the cyclone, and, at the same time, conserve the stibiconite ore, which might have been worn out as a result of recirculation in the metal laboratory cyclone.

Five samples of the stibiconite ore were run through the laboratory cyclone, but only one was assayed because in the other tests, the cyclone clogged and the material passed through in spurts. The cause of this clogging was not definitely determined but it is felt that it is caused by pulp jamming around the pipe valve. This difficulty could be relieved by using a control valve, such as those recommended by the Equipment Engineers Incorporated.¹ This valve is similar to a pinchcock except that it pinches uniformly around the tube and leaves a smooth bore.

The Krebbs cyclone was set up but no stibiconite ore was run because time ran out before the large pilot rod mill could be set up to handle the large volume of ore required for operation of the four inch cyclone.

SUMMARY AND CONCLUSIONS

Although there is little experimental data available, literature surveys have indicated certain factors that should be considered when attempting to choose between flotation and centrifugal concentration. On the one hand is flotation, in which chelation is a consideration of prime importance. The cost factor involved in arresting the interfering calcium would appear to be quite large, unless a method **were** found to carefully control both the time of arrestment and the time required for the collector to react with the mineral surface. There is much need for experimental study along this line before any decision as to use of a chelating agent is made. On the other hand, centrifugal concentration has the advantage of having been, in some cases, found a useful tool for concentration. It is well to keep in mind the following favorable points when considering the cyclone for concentration: "lower capital investment, less overall space requirement, more flexibility of operation in the event of changes in ore characteristics and/or plant capacities", and, this large unfavorable point: an inherently high operating cost is involved in cyc lone use.⁷ The author recommends further experimental research on each of these types of concentration before a decision is made as to which is the most satisfactory for this type of ore.

 $-27 -$

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Appendix. I

Cyclone tests on ground artificial ore

CYCLONE TEST No. 100 L

ASSAY

% DISTRIBUTION

REMARKS: The test was not used due to the fact that the pulp had the tendency to clog and the eyclone ran in spurts.

CYCLONE TEST No. 101 L

DATE 11-March-1959

ASSAY

% DISTRIBUTION

REMARKS: This test was not assayed due to the fact that the pulp clogged and did not flow into the cyclone smoothly. This also accounts for the high input pressure.

 \mathbf{I}

% DISTRIBUTION ASSAY b HEAD ÷. **CONCENTRATE** TAILS

V

REMARKS:

The test indicated that a pulp density of about 25% with a lower imput pressure might result in a concentration, however, the cyclone still had the tendency to clog and a better method
of feeding will have to be investigated. Perhaps a greater head with no pump action would result in a more even flow.

ASSAY

.

% DISTRIBUTION

REMARKS: The cyclone was allowed to run with a natural head from the agitator but it could not supply the necessary input pressure. Appendix. II

Cyclone tests on ground stibiconite ore

CYCLONE TEST No. 104 L

DATE 17-April-1959

ASSAY

% DISTRIBUTION

REMARKS: The assay indicated that even tho small, a separation could be made.