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Gunnard Edmund Johnson

Department: Materials Science and Engineering

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LEACHING EXPERIMENTS ON AN ARIZONA COPPER ORE.

BY

GUNNARD EDMUND JOHNSON

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A

THESIS

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Degree Of

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Approved by Horace T. Mann

Professor of Metallurgy.

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INTRODUCTION.

The leaching of low grade copper ores is a problem that has always been attractive in theory, and has been the object of much experimental work. In recent years large scale experiments on oxidized cappings and mill tailings have added considerable knowlege to the subject and lead to a further study of the possibilities. Laist's experiments on the leaching of copper tailings, and the very complete experiments of Croasdale on the Ajo ores have shown that under favorable conditions leaching is preferable to other methods of recovery.

Scope of Thesis.

The object of this thesis was to obtain a general knowledge of the problems to be solved in leaching a typical carbonate copper ore or oxidized capping of the New Cornelia Copper Co's ore. The experimental work previously performed far exceeds any that might be attempted in the short time allotted to this work, and the few results obtained indicate that the ore is particularly favorable for leaching.

DESCRIPTION OF ORE.

The following description of the ore is given by L. D. Ricketts[1] The Ajo ore has a matrix of granite with a large percentage of secondary quartz replacing feldspar. There is little calcium present in soluble form, and the oxidized ore seems to be best adapted to a leaching process. Preliminary laboratory tests showed that the ore crushed with the production of remarkably little slime and that the copper will dissolve in dilute sulphuric acid quite freely when the crushed ore contains fragments no larger than 6 mm cubes. Tests also showed that a little cuprite is present, which is only partly soluble in sulphuric acid. There are also other soluble salts, notably salts of iron and aluminium.

Following is a typical analysis of the ore as given by Croasdale[2]-

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1. Some Problems in Copper Leaching by L. D. Ricketts. Trans. American. Electrochemical Society, Vol.XXVII, pp. 35-74, 1915.
2. Leaching experiments on the Ajo Ores by Stuart Croasdale, Bull. A. I. M. E., Aug. 1914, p. 1911.

SiO_2	63.29%
Fe	4.20
Al_2O_3	14.30
CaO sol in acid	0.90
MgO	0.80
MnO	0.14
S total	0.27
S as sulphate	0.10
Cu total	1.45
Cu sol in 10% H_2SO_4	1.32
CO_2	1.26
Au aunces	0.01
Ag "	0.18

SAMPLING AND ANALYSIS OF ORE.

Approximately 200 lbs of ore was received for the experiments. It had been crushed to pass 1/4" screen and contained all the fines produced by crushing. No screen analysis was made, as it was desired to treat the entire bulk of the ore as received from the mine. The fines did not seriously interfere with the percolation of the lixiviant, though perhaps a better extraction could have been made on the sized product.

The entire lot was thoroughly mixed and split into 20 portions of about 10 lbs each. One of the 20 portions was further cut down to a 1 lb sample which was crushed through 100 mesh and used as a representative sample for analysis.

The representative sample was then run for total and soluble copper. The total copper obtained by the usual solution in HNO_3 and HCl and titration with a standard thiosulphate solution of the iodide method. The soluble copper was obtained by treating the pulp one hour in a 10% H_2SO_4 solution. The results obtained were as follows:

Analysis of Ajo Ore for Total Copper.

	% Cu	
#1	1.499	Average = 1.496 % Cu.
#2	1.494	

Analysis for Soluble Copper.

	%Cu	
#1	1.490	Average = 1.490 % Cu.
#2	1.490	

$$\% \text{ Sol. Cu} = \frac{1.49}{1.496} \times 100 = 99.6\%$$

Owing to the limited time available no determinations other than for copper were made. The unusually high soluble copper content indicates that the lot of ore was thoroughly oxidized and that practically no sulphides or cuprite was present. The analyses were made on 5 gm. portions and the results agreed very closely; hence the addition of the third figure in the decimal place, in order to get the percentage of soluble copper. From this it can be seen that a slight error in analysis will materially change the percentages concerned; a fact which must be kept in mind when dealing with laboratory experiments.

LEACHING METHODS AND EXPERIMENTS.

Sulphuric acid was used as the solvent. Though other solvents could be used, they do not offer the combined advantages of cheapness and possible production from sulphide ores, usually available at lower levels of ore deposits.

For the leaching experiments, 5 lb. lots of the ore were used. For the leaching vats, the bottom was removed from several bottles (see sketch p. 16) and a rubber stopper and glass tube with a rubber tube and clamp, for drainage placed in the neck of the bottle. The bottle was then inverted and placed in a support as shown in the sketch. This arrangement permitted easy removal of the solutions and also a rapid removal of the residue after washing. By means of the clamp any desired rate of percolation could be obtained.

The ore was placed in the vat dry, no particular pains taken to distribute the fines uniformly, the object being to approximate actually working conditions. The lixiviant was then poured on top of the charge and allowed to percolate through the ore. The solution rapidly reached the bottom, the fines apparently not being detrimental. The ore proved to be well adapted for this method; the solution of the carbonates produced CO_2 which circulated the solution and permitted rapid diffusion. The evolution of CO_2 continued from 5 to 7 hours after charging. No attempt was made to introduce air into the vat, though it was arranged for a compressed air connection.

Approximately 600 c.c. of the lixiviant were required for the complete immersion of the ore.

Theoretically only 290 c.c. of the 10% H_2SO_4 solution would be required to dissolve all the copper present, though the other soluble constituents would probably require at least another 100 c.c. Thus a certain amount of free acid would be present in each solution taken from the ore. Following is the data obtained from the leaching experiments.-

Data from Leaching Experiments.

Leach No.	Amt. of Ore taken	Length of Leach	No. of Washings.	Final Volume	Total gms. Cu in Ore	gm. Cu extracted.	gms. Cu per liter in final solution	% Extraction.
1	5 lb.	22 hr.	4	2100 cc.	34.02	25.52	12.1	75.0
2	"	20 "	1	650 cc.	34.02	20.19	31.0	62.0
3	"	120 "	1	660 cc.	Used as lixiviant for No. 4.			
4	"	24 hr.	0	340 cc.	68.04	16.65	49.0	——

Method of Determining Extraction.

The extraction obtained was determined by calculating the total copper present in the ore used for each charge and comparing this with the grams of copper found in the solution obtained from leaching. The copper content of the solution was determined by analyzing a small portion (10 or 20 c.c.) and calculating the quantity in the volume obtained.

The method eliminated considerable work in sampling and crushing the residue, and for small scale experiments appears to be the most convenient, though perhaps not quite as accurate. A small error in analysis of the solution would be multiplied by 50 or 60, which would affect the percent extraction obtained materially.

Discussion of Results.

The first two experiments were made to get an idea of the extraction possible with a fair degree of washing. With four washings a 75% extraction was obtained, while with only one wash the extraction was reduced to 62%. The concentration of the final solution is reduced considerably by increased washing, and in view of the fact that the solutions obtained were to be used for electrolytic deposition, the concentration (12 gms. per liter) becomes too low for the electrolyte desired.

To find the maximum concentration possible, the solution obtained from leach No. 3 was used as the lixiviant for No. 4. This gave a concentration of 49 gm. Cu per liter without any dilution by washing.

From the results obtained it was planned to use the spent electrolyte from the electrolytic cells as the lixiviant for a new charge, adding to it whatever fresh acid or other constituents that might be required. This arrangement would also permit the use of the ferric sulphate, produced by electrolysis, as a solvent for any sulphides of copper present in the ore; and also make a nearly perfect cycle of operations.

RECOVERY OF COPPER FROM SOLUTION.

Two methods for recovering the copper from solution were considered; (1) deposition upon iron, and (2) electrolytic deposition. In many respects the deposition on iron appears the better of the two, were it not for the fact that a final product is not obtained, as the cement copper must be further refined before it is marketable. For this reason it was decided to deposit the copper by electrolysis direct from the solution obtained from the leaching vats.

Electro-deposition of Copper.

The electrolysis of copper sulphates liquors using insoluble anodes has been the subject of a very complete investigation by Lawrence Addicks.[1] The experiments were conducted in conjunction with the leaching of Ajo ores, hence the research work was available for reference in the experiments conducted. The conditions outlined by Mr. Addicks were adhered to as nearly as possible, and the few results obtained coincided completely with those he obtained.

Before electrolyzing the solutions obtained from leaching a few preliminary experiments were

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1. The Electrolysis of Copper Sulphate Liquors using Carbon Anodes. Met. and Chem. Eng., Vol. XIII pp. 748 etc., 1915.

conducted upon a pure copper sulphate solution (40 gms. to a liter) and gradually increasing the amount of ferric sulphate. No record was kept of the increase in voltage, though with an increase of ferric sulphate the voltage drop also increased. Addition of aluminium sulphate was also tried, but no data was obtained on the corrosive effect upon the copper cathode.

Apparatus for Electro-deposition

In the preliminary experiments a small cell was connected with an Edison storage battery, delivering a maximum of two amperes at a pressure of six volts. The cathode was made (4" X 2") of sheet copper, the anode of graphite 1/4" thick and of the same size as the cathode. Compressed air was introduced from the bottom of the cell against the anode by means of a perforated glass tube. This method proved very satisfactory for reducing the polarization.

For the electrolysis of the leaching solutions three cells of the same type and size as used in the preliminary experiments were placed in multiple across an ordinary lighting circuit. To obtain the different current dinsities desired, a bank of incandescent lamps was placed in the circuit as shown in the sketch. An ammeter registered the current delivered. and a voltmeter connected to the anode and cathode gave the voltage drop through the electrolyte.

Results.

The difficulties encountered in arranging the apparatus for the deposition, prevented further work on the leaching. The apparatus was used several times on the solutions obtained from leaching, but no data taken as the usual "teething" difficulties made any data taken erratic. However, if further work is desired, with a few trial runs some very interesting data should be secured.

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Sketch of

Leaching Apparatus

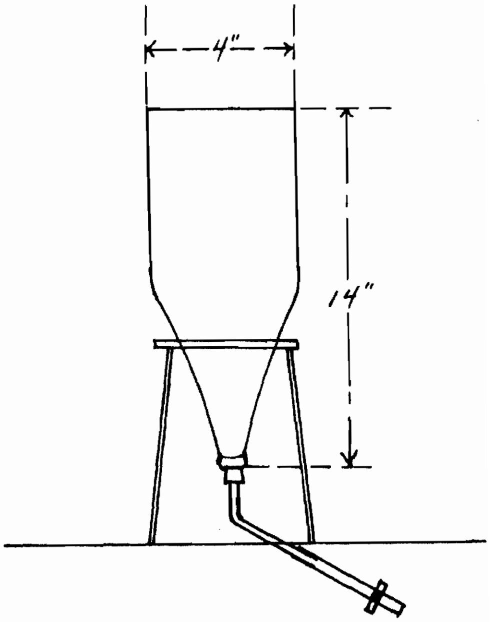

Diagram of Connections for Deposition of Copper.