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INVESTIGATION OF HALLIMOND-TUBE FLOTATION OF LOW GRADE PHOSPHATE MATERIAL

December 1966 Progress Report
Minerals Research Laboratory
Lab. No. 1869 - Book 212
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Introduction

Large reserves of low grade phosphate ore exist in North Carolina that are not being utilized. The gangue materials are quartz, clays, and soft limestone, chiefly calcite. Known methods are available for removal of quartz and clays. As yet, no satisfactory economical method exists for a calcite-calcium phosphate separation. Methods such as screening and differential grinding will remove only part of the calcite.

This is a study of flotation separation employing a small experimental flotation cell, known as a Hallimond tube. A few reagent combinations suggested in patents and published articles were tried with limited success.

The Cell

The flotation cell is shown in Figure 1. This unit has several advantages, among which are: a small amount of sample is used (2-3 grams), a large number of conditions and reagent schedules may be employed in a short time, and neither the reagent concentration nor the amount of liquid varies during the test.

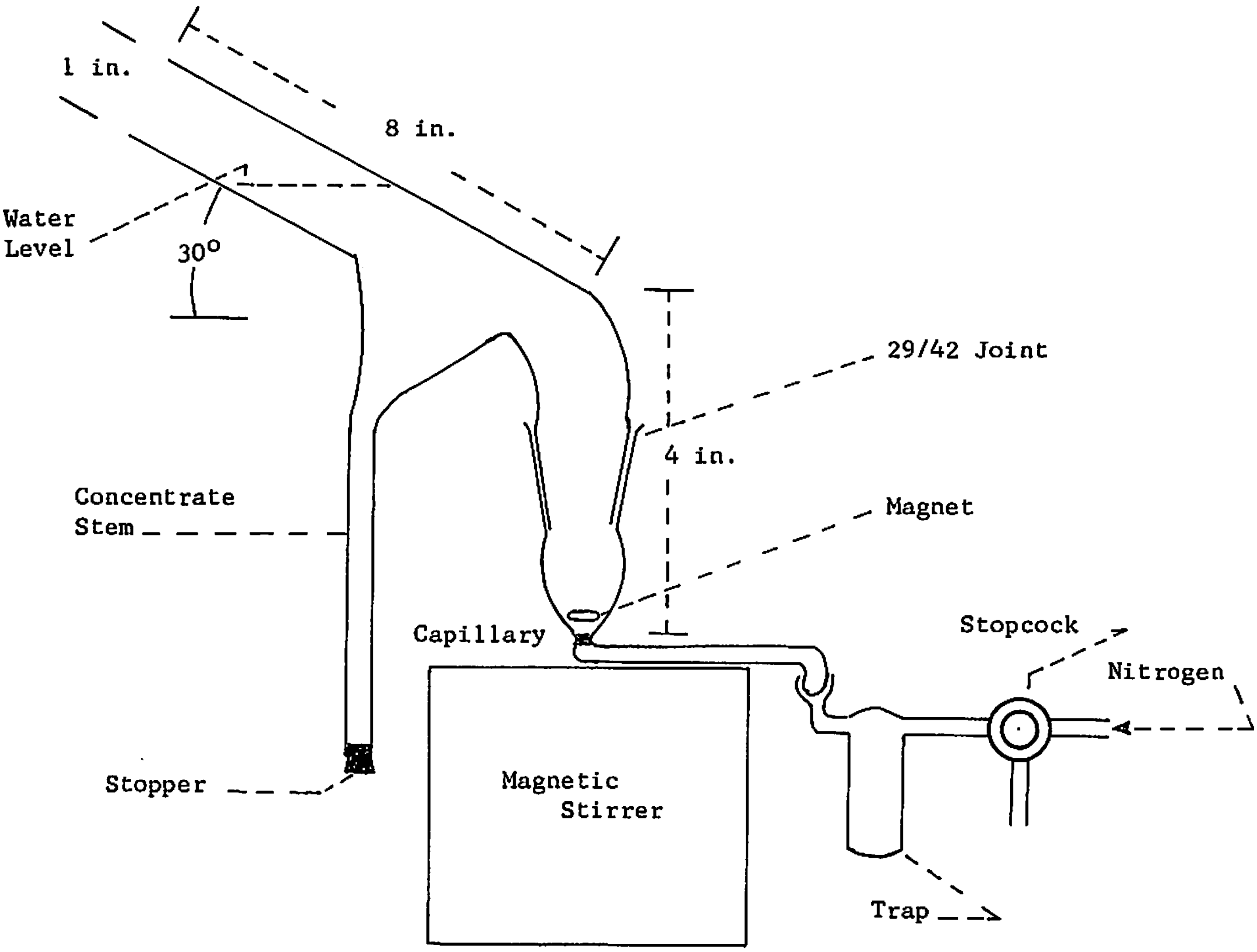
The flotation volume of the cell is about 100 ml and purified nitrogen gas is used for bubble formation. The cell has a magnetic stirrer, a fritted glass bottom for admitting gas, and a ground glass joint for ease of handling. The well of the cell, made from a 29/42 Pyrex joint is constructed so that unfloated particles always lie over the fritted glass bottom. The cell can be clamped over a magnetic stirrer and a polyethylene-coated magnet is used to stir the particles.

The flotation variables include reagent concentration, conditioning time, flotation time, degree of aeration, and degree of agitation within the cell.

Feed Material

With the exception of a few runs with high grade phosphate (Lab. No. 3038) to standardize operating procedure, the samples were obtained from low grade ore (Lab. No. 1869). The ore was first passed through a free-fall electrostatic separator to remove quartz, and then was screened to obtain a minus 48 plus 100 mesh fraction. Sample 143-B-3, which was used for most of the work, contained approximately 57 percent collophane, 40 percent calcite and about three percent quartz. The

Figure 1



HALLIMOND TUBE

material had been scrubbed and deslimed before electrostatic treatment to remove slimes from the surfaces of the particles.

Procedure

A dry three-gram sample was conditioned in the bottom of the cell, or in a 30 ml beaker at 60 percent solids with the appropriate reagents for two minutes. Water was added to increase the volume of slurry in the cell to 100 ml and the slurry was stirred for 20 seconds with the magnet to ensure uniformity of reagent concentration. The nitrogen gas was turned on and allowed to flow at 3/4 cc/sec, usually for two minutes of float time. The floats fell down into the concentrate stem and were drained off. The pH of this solution was recorded. The concentrates and tails were filtered, dried, weighed, and the mineral content of each was estimated by microscope. Table 1 shows the reproducible results of fatty acid flotation of high grade phosphate (Lab. No. 3038). Three grams of material were conditioned with one ml of 0.5 percent solution of saponified M-28 fatty acid, and one ml of water in the cell for two minutes and then floated as described above.

Table 1

Hallimond Tube Phosphate Flotation

Run No.	Float Time	Float pH	% Float	% Quartz, Floats*
36	2 min	7.2	61.4	5
37	2 min	7.2	62.8	5
38	2 min	7.3	58.8	5
39	2 min	7.3	63.0	5
40	2 min	7.3	66.3	5

* Estimated by microscope.

The investigation on calcite-phosphate separation was in three main areas: first, a high pH amine float, with various calcite depressants, second, oleic acid float with phosphate ion as a phosphate depressor and, thirdly, flotation of calcite with short-chain saturated acids, principally capric acid as suggested by Hoffmann and Mariacher of Israel (Mining Engineering, May 1961).

Results

No absolute, sharp separation was achieved, although some trends are evident. Table 2 gives the results of several tests applying Armac-T amine with various calcite depressants and pH regulators. The feed material was approximately 57 percent phosphate pebble, 40 percent calcite and three percent quartz.

Table 2

Basic Amine Floats

Test No.	Pre-Float Conditions	Reagent Conc. (Grams/liter)			pH	% Wt. Floats	%Phos. Floats	% Recov.**
		Armac-T	Ca(OH)$_2$	Dextrine				
65	Condition in beaker	0.040	0.040	0.090	9.6	78	73	87
67	Condition in beaker	0.040	0.020	0.090	9.1	93	60	99
68	Condition in beaker	0.025	0.020	0.060	9.2	95	63	99
128	Leach 5 ml 5 g/l H_2SO_4*	0.040	0.040	0.090	10.1	7.5	70	10
				Quebracho				
136	Condition in beaker	0.050	0.040	0.020	10.0	no float		
			NaOH					
126	Condition in beaker	0.015	0.035	-	9.7	63	70	82
130	Leach 5 ml 5 g/l H_2SO_4	0.015	0.035	-	10.0	no float		
				Na_2SiO_3				
131	Condition in beaker	0.015	0.035	0.025	10.0	68	65	90

* Sample leached with acid before conditioning

** Recovery = $R = \frac{100\,c(f-t)}{f(c-t)}$, estimated percentages

From Table 2 it can be seen that preconditioning with H_2SO_4 is detrimental. From the figures in this table and from other work $Ca(OH)_2$ is the best pH regulator and dextrine the best depressant.

Table 3 gives the results of the best tests with M-28 fatty acid and PO_4^{-3} ion. A non-float product containing 80 percent phosphate pebble was the best attained. Leaching the material briefly before fatty acid conditioning, appeared to improve selectivity of the collector toward calcite. Tests 111 & 114 gave a very clean calcite float product.

Table 3

Fatty Acid Floats

Test No.	Pre-Float Conditions	Reagent Conc. (Grams/liter)		pH	% Wt. Non-Floats	% Phos. Non-Floats	% Phos. Recov.
		Na_2HPO_4	M-28				
70	Condition in beaker	10.0	0.40	6.6	90	70	98
107	Condition in beaker	9.0	0.08	6.6	80	80	88
85	Condition in beaker	2.25	0.04	6.6	74	80	89
84-A	Condition in beaker	2.25	0.04	6.8	83	78	93
88	Condition in beaker	2.25	0.053	6.6	60	75	72
89	Condition in beaker	2.25	0.053	6.3	92	60	98
111	Leach 5 ml 10 g/l H_2SO_4	2.25	0.04	6.1	83	75	98
114	Leach 5 ml 10 g/l H_2SO_4	9.0	0.05	6.4	86	75	99
		$(NH_4)_2$ HPO_4					
74	Condition in beaker	10.0	0.40	6.5	88	75	97

Since the concentration of phosphate ion is high, such a process would be very costly if the flotation solution were discarded. With this in mind attempts were made to reuse the liquid and these were successful only if fine calcite particles were first removed by filtration.

Exhaustive attempts to float calcite from phosphate pebble with short chained saturated acids such as caprylic, capric and lauric acids proved almost entirely negative. Such reagents, although somewhat selective towards calcite, possess a low collecting power. Float products were almost entirely calcite but only about five percent by weight.

Summary

Hallimond tube tests indicate that each of the three reagent combinations attempted does not give satisfactory calcium carbonate-calcium phosphate separations. Such a separation by flotation is hampered by the softness of the calcite which slimes and coats the phosphate pebble. This property of the calcite suggests that further work should include conventional methods such as differential grinding and screening in combination with a strong cationic flotation system.