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Separation of Valuable Fine Phosphate Particles from Their Slimes by Column Flotation

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ABSTRACT

Large quantities of fines are generated as slimes during beneficiation of phosphate ores. These slimes are discarded due to the lack of a suitable method for their treatment. This decreases the total recovery of phosphate. Moreover, the presence of these slimes causes an environmental problem in some localities. This paper studies the possibility of recovering the valuable fine phosphate particles ($<$45 μm) from their slimes through the application of the column flotation technique. Tests were performed using oleic acid as a collector for the phosphate minerals, and sodium silicate as a depressant for their associated gangues. The main operating parameters affecting the performance of column flotation were investigated. The results indicate that the best operating conditions for column flotation of phosphate slimes are: superficial gas velocity 0.84 cm/s, frother concentration 0.1 kg/ton, column height 230.5 cm, and superficial water velocity 2.2 cm/s. At these conditions a product assaying 25.3% P_2O_5 (~55.24% BPL, bone phosphate of lime) and 14.64% I.R. (insoluble residue) with a P_2O_5 recovery of about 51.52% was obtained from a feed containing 18.26% P_2O_5 (~39.87% BPL) and 24.03% I.R. Such a grade and recovery were not obtained by applying the conventional froth flotation technique, even after cleaning the rougher concentrate. Recovery of more than 50% of phosphate from disposed slimes will improve the economic viability of the beneficiation process for phosphate ores. It will also help in solving the environmental problems associated with the disposal of these slimes.

Key Words. Column flotation; Phosphate ore; Slimes; Fine particles; Frother; Washwater; Bubble size; Column height

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INTRODUCTION

Because of the extremely complicated physicochemicomechanical conditions existing in the flotation process, the problems associated with the presence of fine particles are most pronounced in flotation. There is general agreement that the flotation rate decreases with a decrease in particle size in the fine particle range. Two characteristics begin to dominate as the particle size is reduced: the specific surface becomes large and the mass of the particle becomes very small (1).

Also, because of the small mass and momentum of fine particles, they may be carried into the froth after either getting entrained in the liquid or being mechanically entrapped with the particles being floated. Some authors (2, 3) have shown that fine particles are carried into the froth as a mechanical carryover in layers of water attached to air bubbles. When such particles are of gangue minerals, the effect is a reduction in the grade of concentrate. The large specific surface of fine particles increases the adsorption capacity of reagents when considered on a mass basis. Thus, a significant proportion of reagent is consumed by small particles. When present in a large quantity, sufficient reagent may not be available for the flotation of larger particles, with a resultant decrease in recovery (1).

On the other hand, in the United States about one-third of Florida phosphate is lost as slimes due to the lack of a suitable method for their beneficiation (4). In Egypt, these phosphate slimes represent a larger ratio (~30–50 wt%) of Red Sea phosphates (5). Discarding a large percentage of fine phosphate particles as slimes represents a severe environmental problem besides making the beneficiation process uneconomical. A large portion of fines are rejected during beneficiation of Sebaiya West phosphates in Egypt. The quantity of these slimes is higher than the capacity of the main thickner of the beneficiation plant. This prevents the flotation plant from reaching its scheduled capacity. Moreover, the total recovery of the froth flotation process is usually low due to the losses of phosphates in the desliming and cleaning stages (6).

This paper studies the possibility of recovering valuable fine phosphate particles from their slimes by column flotation. The main operating parameters affecting a flotation column for these slimes were studied.

EXPERIMENTAL

Materials

A sample of Sebaiya West (Egypt) phosphate slimes (−0.045 mm) was used in this study. An x-ray diffractogram of the sample showed that the phosphate mineral is present as carbonate fluoro-apatite. The main gangue minerals present, in decreasing order, were quartz, calcite, and montmorillonite. The sample contained only 18.26% P_2O_5 (~39.87% BPL, bone phosphate of lime) due to the presence of large amounts of associated silica (24.24% SiO_2) and calcite as the XRD indicated. The phosphate sample also contained 2.56% Fe_2O_3 and 1.89% Al_2O_3 while it had a minor amount of MgO (0.19%).

Laboratory-grade oleic acid and technical-grade sodium silicate, supplied by El-Nasr for Medical Chemical Co., Egypt, were used in flotation column tests as a collector and a depressant, respectively. Analytical-grade sulfuric acid and sodium carbonate (B.D.H. Chemicals, UK) were used for pH regulation. Oleic acid was added as a mixture with kerosene at a ratio of 1:3. Pine oil (B.D.H. Chemicals, UK) was used as a frother to control bubbles size in column flotation.

Laboratory Flotation Column Equipment

A laboratory flotation column of 5.04 cm diameter by 361 cm total height was used in these tests. The column was made by using a 34-cm Prespex segment which allowed for visual inspection of sparger action at the bottom and the interface near the top. Unless otherwise mentioned, the active column height was 332.5 cm and the feed samples were introduced in the second section at about 60 cm from the top. Washwater was introduced through a perforated 2.8 cm copper tubing ring placed about 5 cm below the overflow lip. The washwater flow rate was controlled by a calibrated water flowmeter (Cole Parmer Co., USA). Air was purged through a porous stainless steel sparger of 25.12 cm^2 surface area at the bottom of the column which also allowed for tailings to exit. Air at a pressure of 2 kg/cm^2 was used through the sparger. The air flow rate was controlled by an air flowmeter (Cole Parmer Co., USA).

The feed sample was conditioned in a 50-L tank equipped with an impeller of fixed speed. The tank's bottom was designed to eliminate dead volumes which could lead to particle settling. The outlet of the feeding tank was connected to the feed point of the column through a variable speed peristaltic pump (Cole Parmer Co., USA). Another variable speed peristaltic pump (Cole Parmer Co., USA) was used to remove the nonfloated fraction (tail) at the bottom of the column.

Flotation Column Tests

In each experiment the flotation column was first filled with water and both the air and washwater flow rates were adjusted, unless otherwise mentioned, at superficial gas and washwater velocities of 0.84 and 0.32 cm/s, respectively. The frother concentration was fixed at a dosage of 67 g/ton. A sample of phosphate slimes was conditioned in the feed tank with the predetermined optimum amounts of reagents (i.e., 0.75 kg/ton of sodium silicate and 2 kg/ton of oleic acid) at pH 9.5. The froth (concentrate) was continuously removed from the column's top while the nonfloated product (tail) was discharged from the bottom. The flow rates of feed, air, and washwater were varied during the test program. The froth and tail samples were collected for chemical analysis and recovery calculations 10 minutes after reaching equilibrium (steady state). The retention time of the particles in the column was about 2 minutes.

RESULTS AND DISCUSSION

Effect of Superficial Air Velocity

Figure 1 shows the effect of air flow rate on grade and recovery of phosphate fines. The results indicate that as the superficial air velocity increases from 0.84 to 3.34 cm/s, the recovery of P_2O_5 increases from about 16.34 to 80.0%. Meanwhile, the P_2O_5 content decreases from about 22.80% at a superficial gas velocity of 0.84 cm/s to about 20.4% at 1.67 cm/s. The acid insoluble increases from 20.7% (at 0.84 cm/s) to 22.50% at 1.67 cm/s. The grade of concentrates did not change at the higher superficial gas velocity ($>$1.76 cm/s).

It is known that the air holdup in column flotation increases with rising superficial gas velocity. This may increase the probability of collision between particles and air bubbles and in turn will increase the number of particle-bubble aggregates which will be reported to the concentrate. However, the bubble size is linearly related to the applied superficial gas velocity (7–11). The higher air velocity produces bubbles of larger diameter which, in turn, provides a lower total surface area. Goodall and O'Connor (12) found that in-

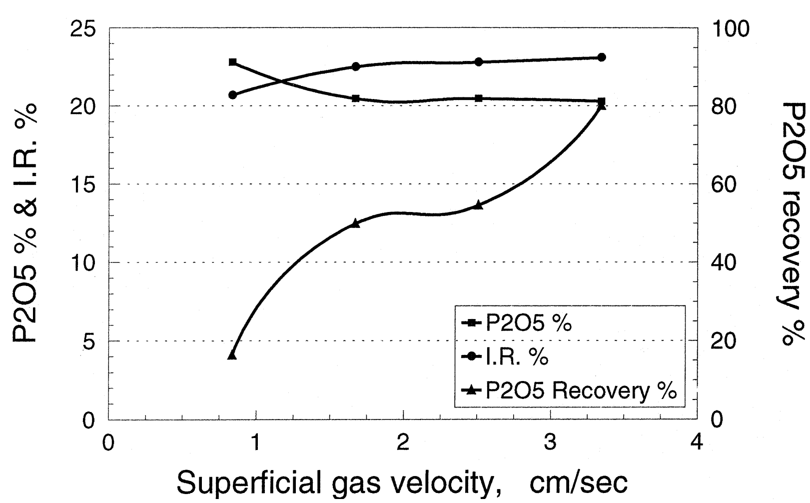

FIG. 1 Effect of superficial air velocity on the flotation of phosphate slimes.

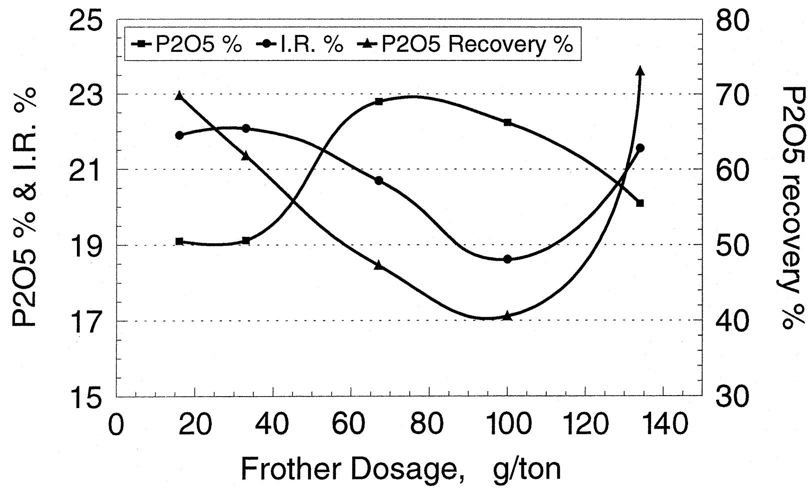

FIG. 2 Effect of frother dosage on the flotation of phosphate slimes.

creasing the superficial air velocity from 0.5 to 1.0 cm/s increased the bubble diameter from about 2 to 3 mm and, on the basis of a unit cross-sectional area, the total surface area of bubbles increased by a factor of 1.33 (12). These larger bubbles will entrain more liquid throughout the cleaning zone of column. Consequently, the presence of particles of lower grade in the froth product will increase. To avoid such behavior, the lower superficial gas velocity (0.84 cm/s) was used in the rest of the tests.

Effect of Frother Concentration

The results in Fig. 2 indicate that both grade and recovery of concentrates were affected by the frother dosage. Increasing the frother dosage from 0.014 to 0.1 kg/t increases the P_2O_5 content from 19.1 to 22.24% while the acid insolubles decreases from 21.9 to 18.61%. This, of course, is achieved at the expense of P_2O_5 recovery which sharply decreases from about 69.8 to 40.6%. At a higher frother dosage (0.134 kg/t), a sharp rise in recovery (~73.1%) can be seen at the expense of grade (~20.1% P_2O_5 and 21.6% I.R., insoluble residue).

It is expected that bubbles size will decrease with increasing frother dosage. The rising velocity of smaller bubbles is slower than that of larger ones. This significantly improves the air holdup, and in turn the air dispersion, in the column (9). This has been proved in some recent studies using different types of frothers (10, 11). Such an improvement in air dispersion will decrease the water recovered with concentrates and consequently will decrease the hydrophilic (silica) particles that can be collected with such water. This will improve the grade of the concentrate, as shown in Fig. 2. At the same time, the rate of collision between particles and small air bubbles will increase at the higher frother dosage. This may increase the collection rate (in terms of P_2O_5 recovery) at the expense of grade. For this reason, a dosage of 0.1 kg/t of frother was selected for all the experiments.

Effect of Column Height

Figure 3 depicts the effect of changing column height on grade and recovery of phosphate fines. It is clear that column height plays a major role in determining grade and recovery of such fine phosphate particles. Increasing the column height from about 128.5 to 230.5 cm was accomplished by a successive improvement in the grade of concentrate where the P_2O_5 content reached its highest value (~23.91%) while the acid insoluble content was reduced to its minimum value (~16.86%). It should be mentioned that the flotation feed contains about 18.26% P_2O_5 and 24.03% I.R. The grade of concentrate deteriorated when the column height was higher than 230.5 cm. At the same time, a continuous increase in recovery from about 26.25 to 90.30% was noticed with increasing column height from 128.5 to 332.5 cm. A similar behavior had been noticed in column flotation of a sulfur ore (13).

There is a good relation between column height and residence time of particles (14). It is expected that the residence time will increase with increasing column height. Under these conditions, there will be a better chance for particle–bubble collision and subsequently forming more particle–bubble aggregates. This may lead to such a successive increase in recovery which, of course, will be at the expense of grade of concentrate, especially at the highest height of the column (i.e., 332.5 cm). However, at a suitable column height (e.g., 230.5 cm), hydrophobic particles will have relatively more time, in com-

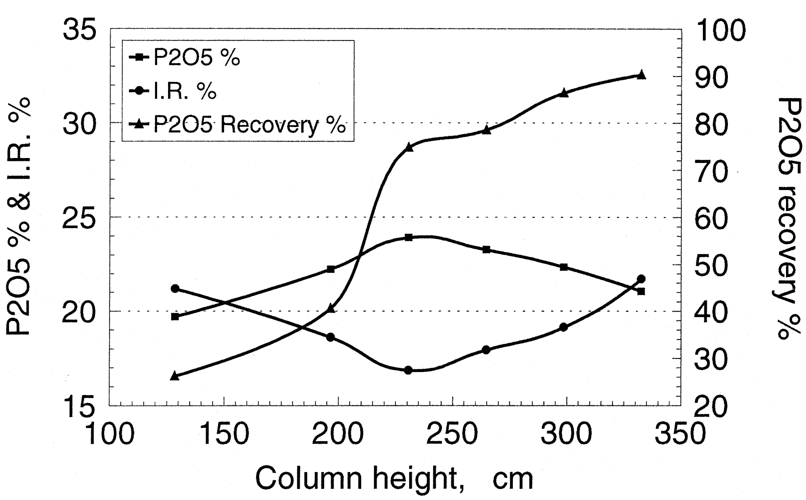

FIG. 3 Effect of column height on the flotation of phosphate slimes.

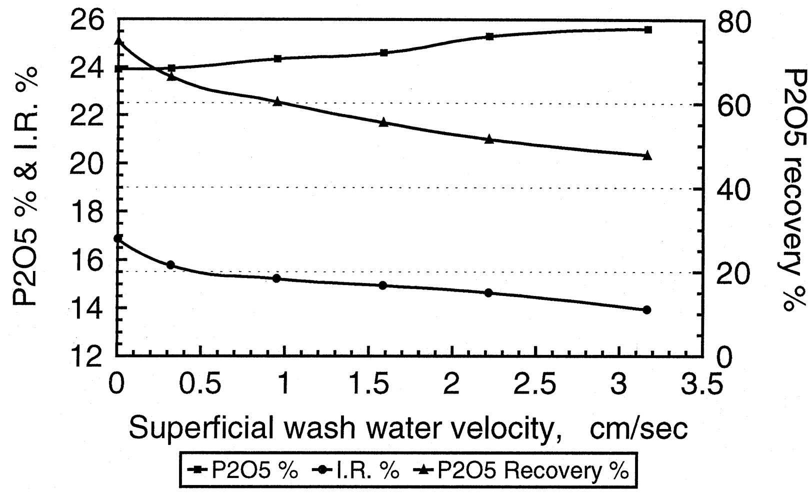

FIG. 4 Effect of superficial washwater velocity on the flotation of phosphate slimes.

parison with hydrophilic ones, to reach the top of the column. This produces a concentrate of better grade as shown in Fig. 3. Ityokumbul mentioned that increasing the recovery zone height does not necessarily result in increased recoveries (15). This would imply that the particle-collection process actually occurs in a relatively constant height that is independent of the actual recovery-zone height. For this reason, the height of the column was fixed at 230.5 cm (at which the best grade was obtained) in the next tests.

Effect of Superficial Washwater Velocity

The main objective of applying a downward flow of washwater in column flotation is to minimize the entrained and entrapped nonfloatable particles from the floatable bubble–particle aggregates (16, 17). The use of washwater is a unique feature of column flotation used to overcome the entrainment or/and entrapment that occurs in the conventional flotation cell, where the frothing zone is very short. Figure 4 shows the effect of countercurrent washwater addition on grade and recovery of P_2O_5. The concentrate grade increases significantly, at the expense of recovery, by increasing the superficial washwater velocity from 0 to 3.16 cm/s. The P_2O_5 increases in concentrates from about 23.91% in a nonwashed sample to 25.62%, while the acid insolubles decrease from 16.86 to 13.94% by merely increasing the superficial washwater velocity to 3.16 cm/s. It appears that the grade of concentrate can be controlled by altering the amount of washwater added. It should be noticed that the grade of washed concentrates (23.95–25.62% P_2O_5 and 15.77–13.94% I.R.) exceeds those obtained from column flotation without using washwater (23.91% P_2O_5 and 16.86% I.R) or even from a conventional flotation cell, 21.7% P_2O_5 and 20.06% I.R. (18). The recovery of washed concentrates varied between 47.81 and 66.30%. Similar tests with a conventional flotation cell produced a rougher concentrate of 21.70% P_2O_5 with a recovery of 67.51% which, after its cleaning at lower impeller's speed, gave a final concentrate of 24.00% P_2O_5 and 16.4% I.R. with a recovery of only 38.15%. It is clear that the flotation column gave better results in terms of grade and recovery than those obtained with a conventional flotation cell even after cleaning stages (18).

The results shown in Fig. 4 also indicate that P_2O_5 recovery is significantly decreased by increasing the washwater rate. For example, the recovery decreases to about 47.81%, at the highest superficial washwater velocity (3.16 cm/s) compared to 74.8% in the absence of washwater.

It seems that at low superficial washwater velocity, hydrophilic particles of gangue minerals are washed in the froth zone and then returned to the recovery zone. The resulting product is of good grade with considerable recovery. However, a higher washwater flow rate may affect both the hydrophilic particles and the less hydrophobic particles. This may produce a concentrate of high grade but of lower recovery, as shown in Fig. 4.

It is clear from this study that the best operating conditions for column flotation of phosphate slimes of grain size 100% below 45 μm are: superficial gas velocity 0.84 cm/s, frother concentration 0.1 kg/ton, column height 230.5 cm, and superficial washwater velocity 2.2 cm/s. At such conditions a concentrate assaying about 25.3% P_2O_5 (~55.24% BPL) and 14.64% I.R., with a P_2O_5 recovery of about 51.52%, would be obtained from a feed containing 18.26% P_2O_5 (~39.87% BPL) and 24.03% I.R. This concentrate may represent a value added to the beneficiation of phosphate ores. At the same time, it may help to decrease the environmental problems associated with dumping larger amounts of slimes as final tailings since at least 50% of the phosphate minerals lost in these slimes could be recovered by using the column flotation technique.

CONCLUSIONS

Column flotation studies of the phosphate slimes of Sebaiya West, Egypt, indicated that the best operating conditions of the column were: superficial gas velocity 0.84 cm/s, frother concentration 0.1 kg/ton, column height 230.5 cm, and superficial washwater velocity 2.2 cm/s. At these conditions a product assaying about 25.3% P_2O_5 (~55.24% BPL) and 14.64% I.R. with a P_2O_5 recovery of 51.52% was obtained from a feed containing 18.26% P_2O_5 (~39.87% BPL) and 24.03% I.R. Such a grade and recovery are not obtained by applying the technique of froth flotation, even after cleaning the rougher concentrate. Meanwhile, the grade of column concentrate can be improved by altering the amount of washwater added, which helps to minimize the entrained, nonfloatable particles from the floatable bubble–particle aggregates.

The concentrate obtained can either be used for direct fertilizer application or partially acidulated. Also, it can be blended with the Sebaiya West phospho-concentrate to increase the recovery of the beneficiation process. This may represent a value added to the beneficiation of phosphate ores. At the same time, it will help to decrease the environmental problems associated with dumping large amounts of slimes as final tailings since at least 50% of the lost phosphate minerals could be recovered from these slimes.

REFERENCES

1. D. W. Fuerstenau, "Fine Particle Flotation," in P. Somasundaran (Ed.), *Fine Particles Processing*, *Proceedings of the International Symposium on Fine Particles Processing*, *Las Vegas*, *Nevada*, *Vol*. *2* (Chap. 35, pp. 669–705), SME-AIME, New York, NY, 1980.
2. V. I. Klassen and S. A. Tikhonov, "On the Mechanical Separation of Slime Particles during Flotation," *Tsvetn*. *Met*., *5*(9), 4 (1964).
3. A. Waksmundzki, J. Neczaj-Bruzewica, and M. Planik, "Mechanism of Carryover of Gangue Slimes during Flotation of Sulfur Ore," *Trans*. *Inst*. *Min*. *Metall*., *80*, C249 (1971).
4. T. V. Subrahmanyam and K. S. E. Foressberg, "Fine Particles Processing: Shear-Flocculation and Carrier Flotation—A Review," *Int*. *J*. *Miner*. *Process*., *30*, 265–286 (1990).
5. A. A. Yousef (Principal Investigator), *The Hamrawein Phosphate Project*, Final report, Presented to Misr Phosphate Co. from Central Metallurgical R & D Institute (CMRDI), Egypt, November 1981.
6. T. R. Boulos, S. N. Boulis, and N. A. Abdel-Khalek, *Investigating the Performance of the Phosphate Beneficiation Plant at Sebaiya West*, Submitted from Central Metallurgical R & D Institute (CMRDI) to Abu Zaabal Fertilizers and Chemicals Co., Internal Report, October 1990.
7. G. S. Dobby and J. A. Finch, "Flotation Column Scale-up and Modeling," *CIM Bull*., *79* (889), 89–96 (1986).
8. J. B. Yianatos, J. A. Finch, and A. R. Laplante, "Holdup Profile and Bubble Size Distribution of Flotation Column Froths," *Can*. *Metall*. *Q*., *25*(1), 23–29 (1986).
9. J. A. Finch and G. S. Dobby, *Column Flotation*, Pergamon Press, New York, NY, 1990.
10. H. El-Shall, Y. H. Cheng, S. Gupta, S. Svornos, N. A. Abdel-Khalek, and R. Sharma, *Sparger Evaluation Study for Phosphate Flotation*, Engineering Foundation Conference, Cairo, November 16–21, 1997.
11. H. El-Shall (Principal Investigator), *Bubble Generation*, *Design*, *Modeling and Optimization of Novel Flotation Columns for Phosphate Beneficiation*, Report to Florida Institute of Phosphate Research, USA, 1998.
12. C. M. Goodall and C. T. O'Connor, "Pulp–Froth Interactions in a Laboratory Column Flotation Cell," *Miner*. *Eng*., *4*(7–11), 951–958 (1991).
13. N. A. Abdel-Khalek and J. Stachurski, "Beneficiation of Sulfur Ore by Conventional and Column Flotation," *Miner*. *Metall*. *Process*., *10*(3), 135–138 (August 1993).
14. J. B. Yianatos, R. Espinosa-Gomez, J. A. Finch, and A. R. Laplante, "Effect of Column Height on Flotation Column Performance," *Ibid*., pp. 11–14 (February 1998).
15. M. T. Ityokumbul, "What Really Determines the Height in Column Flotation," *Ibid*., pp. 36–40 (February 1996).

16. J. W. Choung, G. H. Luttrell, and R. H. Yoon, "Characterization of Operating Parameters in the Cleaning Zone of Microbubble Column Flotation," *Int. J. Miner. Process.*, *39*, 31–40 (1993).
17. G. S. Dobby and J. A. Finch, in *17th Canadian Mineral Processors Operators Conference*, January 1985, pp. 22–24.
18. N. A. Abdel-Khalek, F. Hassan, and M. A. Arafa, "Recovery of Valuable Phosphates from their Slimes by Froth Flotation," *Afinidad*, To Be Published.

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