Physicochemical Problems of Mineral Processing, 34 (2000), 35–45
Fizykochemiczne Problemy Mineralurgii, 34 (2000), 35–45

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FACTORIAL DESIGN FOR COLUMN FLOTATION OF PHOSPHATE WASTES

Received March 15, 2000; reviewed and accepted May 15, 2000

A factorial design was used to study effects, and their interactions, of the main parameters affecting column efficiency to recover phosphates from their wastes. Three-phase experiments were performed using a mixture of fatty acid and fuel oil as a collector for phosphate while pine oil was used as a frother. Meanwhile, 2-phase experiments were conducted to correlate the results of 3-phase experiments with that of bubble diameter and air holdup.

The results show that application of statistical design reveals very interesting information about the interaction between the studied parameters. For example, it is shown that the interaction (X_1X_2) between superficial gas velocity (X_1) and frother concentration (X_2) has a beneficial effect on grade but aversely affect recovery.

Key words: column flotation, phosphate, frother, collector

INTRODUCTION

Large quantities of fines are generated, as slimes, during beneficiation of phosphate ores. Such slimes are discarded due to the lack of a suitable method for their treatment and consequently the total recovery of phosphate is decreased (Abdel-Khalek et al. 2000). For example, about 1/3 of run-of-mine of Florida phosphate, below 150 mesh, is discarded as slimes. In addition, the presence of these slimes causes, in some areas, an environmental problem.

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Meanwhile, column flotation is becoming more popular as a flotation technique (Dobby and Finch 1991, Tuteja et al 1995, Abdel-Khalek and Stachurski 1993). The performance of column flotation depends on a number of operating parameters. To get the best metallurgical performance from a column, it is necessary to optimize column parameters. The conventional practice in mineral processing research is to perform tests as a function of one - variable - at - a - time. However, this can be deceptive, because this method does not provide information on the interaction of variables within the system (Griffith 1962). Studying interaction between variables is useful for a good understanding of the flotation column variables (El-Shall et al 1999, Patil et al. 1996).

Among the several approaches that can be used to optimize experiments, the statistical designed approach is found to be the most useful. A recent review of mathematical models in column flotation processes shows that, compared to kinetic models, not much attention has been given to statistical models (Tuteja et al. 1994).

This paper discusses the results of column flotation of phosphate wastes using a statistical design. The main parameters that affect phosphate flotation were investigated. Also, 2-phase experiments were conducted to correlate the results of 3-phase experiments with that of bubble diameter and air holdup.

MATERIALS AND METHODS

A sample of phosphate wastes (-0.106 mm) was used in this study. The sample contained about 7.8 % P_2O_5 and 78.5 % acid insoluble. A mixture of fatty acids and fuel oil with a ratio of 1:1 by weight was used as a collector. Pine oil was used as a frother.

Flotation tests were conducted using a 14.6cm diameter by 1.8m high Plexiglas flotation column. The feeding point was located at 30 cm from the column top. In each 3-phase experiment, a sample was conditioned with the pre-determined dosage of collector at pH 9.5. Frother-containing water and air were first introduced into the column through the sparger (Eductor type) at a fixed flow-rate. After every parameter was set and two-phase system was in a steady state, the phosphate material was fed, at a fixed flow-rate, to the column. Timed samples of tailings and concentrates were taken after reaching steady state conditions. The collected samples were weighed and analyzed. In 2-phase experiments (air-water system), air holdup and bubble diameter were calculated. Air holdup was measured using manometers. The diameter of air bubbles was calculated using drift-flux method (Dobby et al. 1988).

RESULTS AND DISCUSSION

2^3 FACTORIAL DESIGN

In order to determine the effects and interactions between different parameters, a series of experiments using 2^3 factorial design has been performed. The variables are coded between "-" and "+", where "-" represents the low level and "+" represents the high level of the factor. The levels of the coding are indicated in Table 1. So, there are eight (2^3) possible combinations, each of which was replicated twice, as given in Table 2. Calculation of the effects and their interactions as well as analysis of variance (ANOVA) have been carried out using Yates' method (Myers and Montgomery 1995; Garcia-Diaz and Phillips 1995). This ANOVA technique can be used to determine whether several means are significantly different from one another when compared to the experimental error. The F-test is used to compare two variances, i.e. compare the precision of the two sets of data. $F=S_1^2/S_2^2$ where S_1^2 is always the larger variance. These F values are compared to standard tables of the F distribution at the 95% level. The null hypothesis is that there is no significant difference between variances with the alternate hypothesis that S_1^2 is greater than S_2^2. The main effect (E_i) of factor X_i is estimated as the difference between the two averages:

$$Y_i^+ = T_i^+/r2^{n-1} \text{ and } Y_i^- = T_i^-/r2^{n-1} \tag{1a}$$

that is:

$$E_i = 1/r2^{n-1}(T_i^+ - T_i^-) \tag{1b}$$

Where T_i is the sum of the $r2^{n-1}$ observations corresponding to the 2^{n-1} experimental conditions having X_I = +1 or -1 and r is the number of replications. The results of these analyses are shown in Table 3. The results in Table 2 show that it is possible to obtain concentrates with grade (23.4-29.0 % P_2O_5) and recovery (54.9-80.2%) depending on the applied operating conditions.

Table 1. Levels of variables for 3-phase experiments

Variable	Code	Level	
		(-)	(+)
Superficial Air Velocity, cm/s	A	0.46	0.94
Frother Concentration, ppm	B	5	25
Superficial Wash Water Velocity, cm/s	C	0.4	1.2

Table 2. Results of treatments combinations for 2^3 design for 3-phase experiments

#	Factors			% P_2O_5			% Recovery		
	A	B	C	Replications		Total	Replications		Total
	X_1	X_2	X_3	1	2		1	2	
1	-	-	-	25.8	25.4	51.2	56.3	54.85	111.15
2	+	-	-	23.4	23.8	47.2	70.1	72.7	142.8
3	-	+	-	25.24	26.0	51.24	65.6	63.6	129.2
4	+	+	-	26.91	26.6	53.51	80.2	78.8	159.0
5	-	-	+	27.83	27.97	55.8	50.3	48.95	99.25
6	+	-	+	25.75	26.1	51.85	65.3	66.0	131.3
7	-	+	+	28.62	29.0	57.62	60.53	59.5	120.03
8	+	+	+	26.8	26.13	52.93	70.63	69.86	140.49

Table 3. Analysis of variance for effects and interactions between parameters

Source of Variance	Grade		Recovery	
	Net Effect	Computed F	Net Effect	Computed F
Main Effect:				
X_1	-1.29±0.38	62.22	14.25±1.25	690.91
X_2	1.15±0.38	49.50	8.03±1.25	219.41
X_3	1.88±0.38	131.05	-6.39±1.25	138.81
2-Factor Interaction:				
X_{12}	0.69±0.38	17.71	-1.68±1.25	9.61
X_{13}	-0.86±0.38	27.62	-1.12±1.25	4.24
X_{23}	-0.43±0.38	6.89	-0.54±1.25	0.97
3-Factor Interaction:				
X_{123}	-0.88±0.38	28.41	-1.22±1.25	5.05
$F_{(0.05,1,8)} = 5.32$				

EFFECT OF SUPERFICIAL AIR VELOCITY

Table 3 shows that the effect of (X_1) superficial air velocity (J_g) is statistically significant with respect to grade and recovery. Changing this parameter from its lower level (0.46 cm/s) to higher level (0.94 cm/s) causes a remarkable improvement in recovery of P_2O_5 by 14.2 %. This is at the expense of reducing the grade by about 1.3 %. The effects of changing superficial air velocity on air holdup and bubble diameter are shown in Figures 1-2 respectively. Air holdup, at different levels of frother concentration, is expected to increase with rising superficial gas velocity (Figure 1). 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 results in Figure 2 reveal that bubble size is linearly related to the superficial gas velocity, at different levels of frother concentration. Similar results have been mentioned by other authors (El-Shall et al. 1999; El-Shall et al. 2000; Dobby and Finch 1986; Yianatos et al. 1986). Thus, the higher air rate produces larger bubbles, which implies a lower total surface area. For example, increasing J_g from 0.24 to 0.94 cm/s, at a frother concentration of 5 ppm, increased the bubble diameter from about 0.44 to 1.01 mm. Such larger bubbles will entrain more liquid throughout the cleaning zone of column. Consequently, the occurrence of particles of lower grade in the froth product will increase.

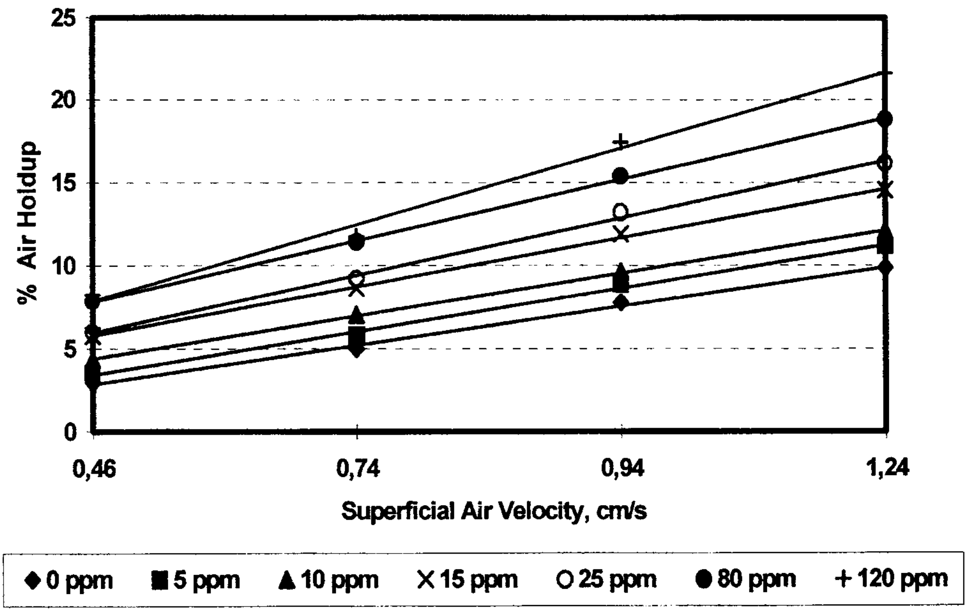

Fig. 1. Effect of superficial air velocity on air holdup at different frother dosages

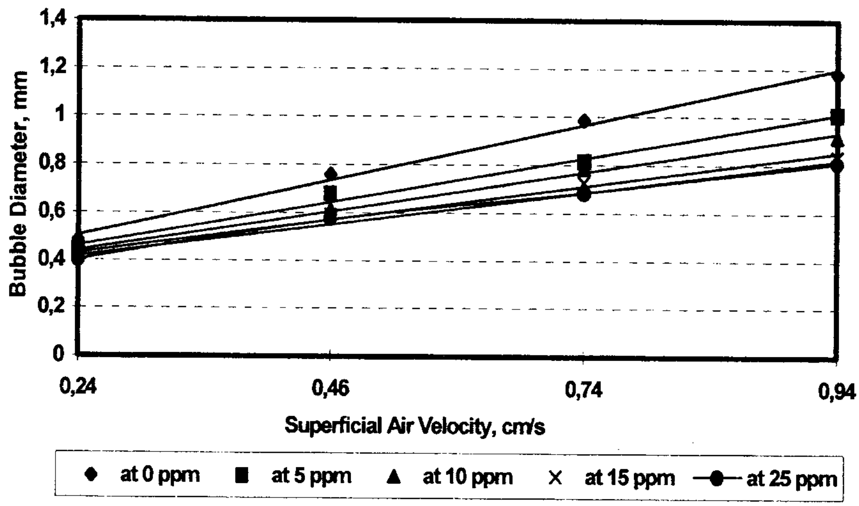

Fig. 2. Effect of superficial air velocity on air bubble diameter

EFFECT OF FROTH CONCENTRATION

The results in Table 3 depicts that increasing frother dosage (X_2) from 5.0 to 25.0 ppm, is accompanied by an improvement in grade of concentrate where the net increase in P_2O_5 content is +1.2. Also, it is interesting to note that the P_2O_5 recovery is also increased by about 8.0 %. It seems from these results that increasing concentration of frother in flotation of such fine phosphate particles is beneficial. On the contrary, it has been shown that in flotation of coarse (belt feed –1.18+0.425, coarse feed -0.85+0.425, and unsized feed –1.18+0.106 mm) particles of Florida phosphate, excessive addition of frother may affect adversely the recovery of concentrates (El-Shall et al 1999, El-Shall et al 1998). To investigate such behavior, the effect of frother concentration on air holdup and bubble diameter was studied in 2-phase experiments, the results of which are shown in Figures 3 and 4.

The results in Figure 3 show that there is a gradual increase in air holdup with increasing frother concentration in the range 5-120 ppm. The increase in air holdup is noticed at different J_g with increasing frother concentration. Such improvement in air holdup is accompanied by a significant reduction in bubbles diameter as shown in

Figure 4. Thus, bubble size will decrease with increasing frother dosage. The rising velocity of these smaller bubbles will be slower than that of larger ones. This will decrease the water recovered with concentrate and consequently will decrease the hydrophilic particles that can be reported with the concentrate. This might improve the grade (Table 3). At the same time, the rate of collision between particles and small air bubbles will increase at the higher frother dosage. This may lead to an increase in the collection rate (in terms of P_2O_5 recovery).

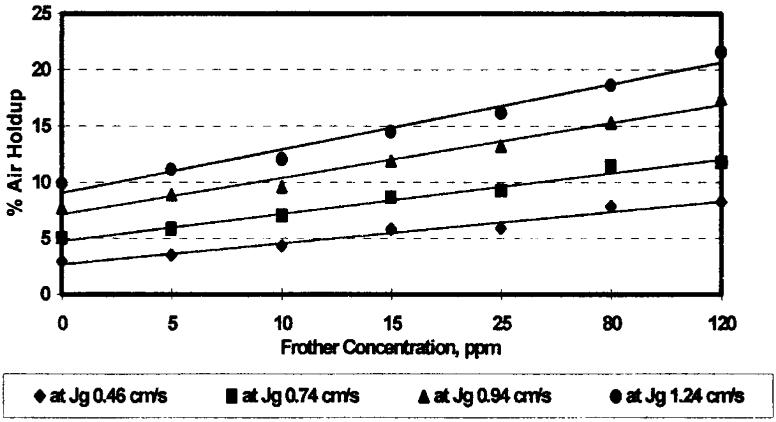

Fig. 3. Effect of frother concentration on air holdup at different superficial air velocity

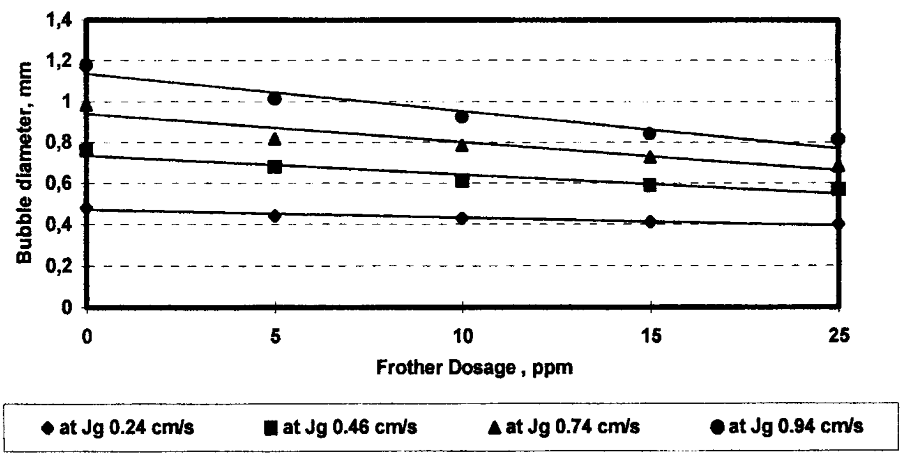

Fig. 4. Effect of frother.dosage on air bubble diameter

This is because the collection rate constant (k) depends on bubble diameter (d_b) and superficial air velocity (J_g) according to the following equation (Finch and Dobby 1990).

$$K=1.5\ J_g\ E_K/\ d_b \tag{2}$$

Where E_K is the collection efficiency.

EFFECT OF SUPERFICIAL WASH WATER VELOCITY

The main objective of applying the downward flow of wash water in the column is to minimize the entrained and entrapped non-floatable particles from the floatable bubble-particle aggregates (Choung et al 1993; Dobby and Finch 1985). This cleaning action is proved in Table 3. The net increase in P_2O_5 content is about +1.9 with increasing the superficial wash water velocity (X_3) from 0.4 to1.2 cm/s. This cleaning action has, also, been proved in column flotation of Egyptian phosphate ores (Abdel-Khalek et al. 2000).

The results shown in Table 3 also show that the recovery is significantly decreased by – 6.4 units with increasing the rate of wash water addition. It seems that at a low superficial wash-water velocity, hydrophilic particles of gangue minerals are washed at the froth zone and then returned to the recovery zone. The resulting product has good grade and high recovery. However, a higher superficial wash-water velocity may affect both the hydrophilic particles and the less hydrophobic particles. This may lead to a high-grade concentrate with lower recovery.

INTERACTIONAL EFFECTS

The interaction (X_1X_2) between superficial air velocity (J_g) and frother concentration is shown to be statistically significant for both grade and recovery as indicated from their average change. The computed F value also suggests the same conclusion. It is expected that the grade of concentrate can be positively affected due to the reduction of bubble diameter as a result of increasing frother dosage (Fig. 4).

On the contrary, the interaction (X_1X_3) between superficial air velocity (J_g) and superficial wash water velocity (J_{ww}) is statistically significant for grade but not for recovery. The computed F value also suggests the same conclusion. It is expected that wash water and air rates have more effect on the bias rate, because variations in these parameters significantly affect the performance of column flotation, as indicated by the main effect. This may lead to such slight reduction in grade (Table 3).

The interaction (X_2X_3) between frother concentration and superficial wash water velocity (J_{ww}) may or may not be significant with respect to grade but it is not significant to recovery. The computed F value also shows the same trend. This is probably due to an increase of mixing in the cleaning zone as a result of increasing wash water flow rate. It may be concluded that there is no significant interaction between wash water flow-rate and frother concentration. In other words, whatever the level of frother concentration, when the wash water flow rate is increased from lower to higher level, the change in grade or recovery is not significant.

The interaction between the three parameters ($X_1X_2X_3$) is not significant to the recovery and may be significant to the grade as computed from F values. The change in grade is about –0.88 unit.

It is clear from the above discussion that application of such statistical design reveals very interesting information about the interaction between the studied parameters. It is shown that the interaction (X_1X_2) between superficial gas velocity and frother concentration has a beneficial effect on grade but aversely affect recovery. Meanwhile, interaction between superficial wash water and either frother concentration (X_2X_3) or superficial air velocity (X_1X_3) adversely affects the grade but it does not affect recovery.

Based on the above results the effects of the main parameters on the grade can be arranged in the following order: wash water > air flowrate > frother concentration. The order of significance of the main effects of variables for recovery is as follows: air flow rate > frother concentration > wash water.

CONCLUSIONS

Flotation column can be used to recover phosphate mineral from their wastes. It is possible to obtain concentrates with considerable grade and recovery depending on the applied operating conditions.

The effect of wash water or frother concentration is found to be statistically significant where each of them improves the grade of concentrate. On the contrary, effect of superficial air velocity decreases the grade. On the other hand, the effect of each of the studied parameters is found to be statistically significant with respect to the recovery. Both superficial air velocity and frother concentration increases the recovery. However, wash water decreases the recovery.

The interaction (X_1X_2) between superficial gas velocity and frother concentration has a beneficial effect on grade but aversely affect recovery. The interaction between superficial wash water and either frother concentration (X_2X_3) or superficial air velocity (X_1X_3) adversely affects the grade but it does not affect recovery.

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Metoda planowania czynnikowego została zastosowana do opisania wpływu i współzależności miedzy podstawowymi parametrami procesu flotacji kolumnowej zastosowanej do odzysku fosforytu z

odpadów fosforytowych. Doświadczenia flotacyjne zostały przeprowadzone z wykorzystaniem kwasu olejowego i oleju napędowego jako kolektorów oraz oleju sosnowego jako spieniacza. Planowanie typu 2^3 zostało przeprowadzone w celu znalezienia korelacji między wynikami eksperymentu a średnicą pęcherzyków powietrza i parametrami urządzenia, które jej wytwarza. Otrzymane wyniki wskazują, że zastosowana metoda planowania dostarcza interesujących informacji o wzajemnej korelacji miedzy analizowanymi parametrami procesu. Dla przykładu , zostało pokazane, że korelacja X_1X_2 czyli miedzy prędkością przepływu gazu (X_1) a stężeniem spieniacza (X_2) na korzystny wpływ na wychód a niekorzystny wpływ na uzysk procesu flotacji.