

## BENEFICIATION OF SAUDI PHOSPHATE ORES PART II: THE APPLICATION OF COLUMN FLOTATION

T.F. Al-Fariss\*, F.A. Abd El-Aleem, and M.S. Al-Qahtani  
King Saud University, College of Engineering,  
Chemical Engineering Department,  
P.O. Box 800, Riyadh 11421, Saudi Arabia.  
\* E-mail: [fariss@ksu.edu.sa](mailto:fariss@ksu.edu.sa)

### ABSTRACT

Phosphoric acid industry is expected to develop in Saudi Arabia in the near future. This is ascribed to the discovery of phosphate ores in the northern part of the kingdom, the availability of sulphur, "required for this industry", as a by-product from petrochemical industries in the kingdom and the construction of phosphate fertilizers required by the growing agricultural sector in Saudi Arabia.

The discovered Saudi phosphate ores are of sedimentary origin with carbonaceous impurities as calcite and dolomite. The beneficiation of this type of ores is a key factor for successful production of phosphoric acid by the wet process.

Froth flotation is one of the promising techniques acquiring large potential in the separation of calcite from such phosphate ores. In our previous work "Part I" of this project, an extensive experimental study using a conventional flotation cell for phosphate beneficiation was carried out and the optimum flotation parameters were achieved. During the next phase of our project "Part II", column flotation has been recognized in literature as one of the most significant advances in froth flotation technology in the last decade. These units, in a single separation step, can achieve a product recovery and purity equivalent to or even better than that obtained by multistage flotation in conventional flotation cells.

Hence, in the present work, a flotation column of 1.5 m height and 6.5 cm diameter has been designed and applied in the beneficiation of Al-Jalamid Saudi Phosphate ores of the calcareous type. The significant parameters like air flow rate, slurry feed rate, particle size of processed ore, wash water consumption and collector doses of flotation process are investigated in the present work to achieve the best recovery and quality of the beneficiated ores. Moreover, the optimum flotation parameters previously obtained in the single flotation cell were also applied in the present column flotation runs.

The results of this study revealed that column flotation technology is a promising tool for beneficiation of calcite phosphate ores by which a high purity ore of 35%  $P_2O_5$  can be

easily achieved at a high recovery value of 95% or even more than that obtained in the classical flotation cells.

*Keywords:* Column flotation, Beneficiation, Phosphate ores, Saudi Phosphate ores, Ore processing, Separation of Calcite from Apatite.

### INTRODUCTION

The development of column flotation is the most significant achievement in the area of mineral processing in the last decades.

In fact, these column flotation units have a wide range of uses due to many factors like high selectivity of separation, simple processing control, low energy consumption, absence of moving parts, low floor space requirements and simple design [1]. Moreover, the high efficiency of column flotation allows a reduction in the process time, a decrease in the number of stages and in the volume of the circulating load in the flotation circuit and as a result of this, an increase in the consistence and reliability of operations are expected.

Due to all these advantages, column flotation is applied in the present work for the beneficiation of Saudi phosphate ores. This ore was discovered in Saudi Arabia in the last decades at the Northern part of the Kingdom. The main problem of this ore is its high content of calcite mineral. The separation of this calcite from apatite "phosphate mineral" is the main target of this beneficiation process. Hence, this column flotation technology is investigated in the present work to account for its feasibility in this field.

For this purpose, a column flotation unit was designed and the main significant factors affecting its performance were thoroughly investigated.

### EXPERIMENTAL

The experimental set-up used in the present work is shown schematically in Fig. (1). It consists mainly of the designed flotation column which is made of Perspex with 1.5 m height

and 6.5 cm diameter. The column has a collection zone of 1.0 m and a cleaning zone of 30 cm. The column is also fitted with a mixing tank for slurry feed preparation and conditioning.

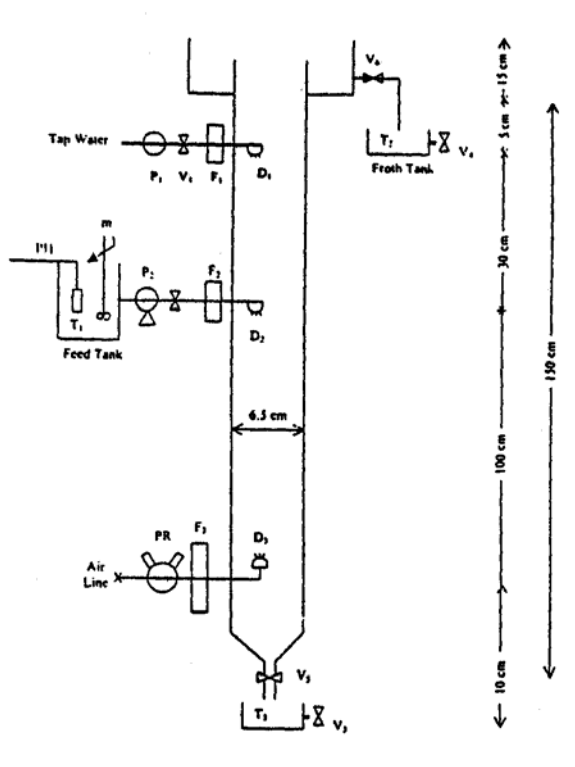


Fig. (1): The designed column flotation unit "The Experimental Set-up".

T<sub>1</sub> Mixing tank

T<sub>2</sub> Froth tank

T<sub>3</sub> Tailing tank

F<sub>1</sub> Water flow meter

F<sub>2</sub> Slurry flow meter

F<sub>3</sub> Air flow meter

D<sub>1</sub> Distributor for liquid

D<sub>2</sub> Distributor for liquid with wide holes

D<sub>3</sub> Air distributor

V<sub>1</sub> Valve

V<sub>2</sub> Valve

V<sub>3</sub> Valve

V<sub>4</sub> Valve

V<sub>5</sub> Valve

V<sub>6</sub> Valve

PR Pressure regulator

m Mixer

P<sub>1</sub> Wash water pump

P<sub>2</sub> Feed pump

pH pH meter

Air enters the column at the bottom through its sparger which is a porous stainless steel mesh with 50 μm pores. The air line is fitted also with a pressure regulator and a flow meter. The slurry feed is introduced to the upper part of the column via its feed pump and below the wash water inlet which is located at 5 cm from the top. The foam phase is collected at the top of the column while the tailings are collected at the bottom.

It is worth mentioning that the slurry feed distributor and wash water distributor are made from Plexiglass plates with 1.0 mm hole openings.

The slurry feed used in each experimental run was prepared from the crushed phosphate rock "of known particle size and P<sub>2</sub>O<sub>5</sub> content" and tap water according to the solids content required. Then, the flotation reagents were added "oleic acid in kerosene as collector and frother, Na<sub>2</sub>SO<sub>4</sub> as depressor" to the slurry mixture and its pH was adjusted as required by the flotation process [6].

After adjusting the feed mixture according to the required conditions, the experimental run was started by operating the feed pump, the wash water pump and the air valve at the required flow rates. Then allow the column to operate for a certain period until the flow pattern became stable and the column reaches its steady state operation. After that, the actual flow rates were measured and the discharged froth phase and tailings were sampled for P<sub>2</sub>O<sub>5</sub> analysis. These analyses were carried out spectrophotometry using unicam spectrophotometer "Model SP8-400" at a wavelength of 420 nm.

It is worth mentioning that the parameters investigated in the present work are:

- (i) Air flow rate.
- (ii) Wash water rate.
- (iii) Slurry feed rate.
- (iv) Particle size of phosphate ore.
- (v) The collector dose.

In order to account for the feasibility of this flotation technology for the beneficiation of the Saudi ores, both P<sub>2</sub>O<sub>5</sub> recovery and P<sub>2</sub>O<sub>5</sub> content of the product "or product purity" were obtained after each flotation run.

## RESULTS AND DISCUSSIONS

### Effect of Air Flow Rate

It is known that; aeration determines the separation performance to a great extent in the flotation columns. It also affects the gas hold-up, the bubble size and the flow pattern inside the column. Hence, this parameter was the first investigated one in the present experimental program. For this purpose, 4 air flow rate levels are tested in our work (i.e., 0.47, 0.56, 0.65, 0.75 L/Sec.). Other parameters are kept constant at their desired levels.

The results obtained are given in Table (1) and plotted in Fig. (2).

As shown in this figure, increasing the air flow rate (at first from 0.47 to 0.65 L/sec.) increases the  $P_2O_5$  recovery as expected due to the increase in the gas hold-up and also due to the increase in bubbles attached with the solid particles as well as the increase in the bubble-particle collision. This improvement in recovery continues until a value 0.65 L/sec. for air rate, beyond which the recovery decreases due "to the coalescence of gas bubbles formed leading to the formation of large bubbles which have low efficiency of flotation. Moreover, the capture efficiency between bubbles and solid particles is reduced at high flow rates resulting in lower  $P_2O_5$  recovery.

Table 1: Results of the air flow rate runs.

	Air Flow Rate (L/ sec. )	$P_2O_5$ %	Recovery %
Run # 1	0.47	24.01	79.07
Run # 2	0.56	26.12	54.79
Run # 3	0.65	29.01	91.09
Run # 4	0.75	20.71	78.57

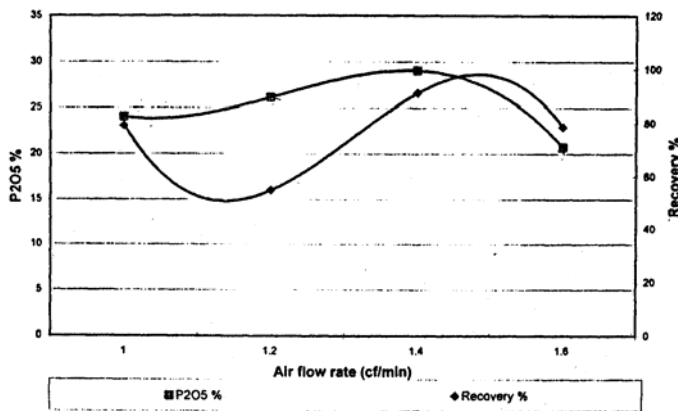


Figure 2: Effect of air flow rate.

**Constant Variables:**

- pH = 6.5
- Particle size = 180  $\mu$ m
- Pulp density = 4%
- Flotation time = 5 minute
- Collector quantity = 1.82 kg/ton
- Depressor quantity = 40 kg/ton
- Washing water flow rate = 200 ml/min
- Slurry flow rate = 956 ml/min

Figure (2) also indicates an optimum value for recovery (91.09%) at air flow rate 0.65 L/sec. This optimum value for air flow rate in the flotation column was also observed by other

workers in literature [1]. For example, in copper ore processing an optimum value for air flow rate was observed at a superficial air velocity of 2  $\rightarrow$  2.5 cm/sec while in coal processing a value of 1.8 cm/sec for optimum air velocity was reported. Concerning the  $P_2O_5$  content, as shown in Fig. (2); it also has an optimum value of (29.01%) at the same optimum air rate (0.65 L/sec.) which is equivalent to a superficial air velocity of 1.9 cm/sec. This optimum value (or 0.65 L/sec.) will be used in the next flotation runs.

**Effect of Washing Water Rates**

Wash water is usually introduced at the top of the flotation column below the froth phase overflow. It is used for washing out the entrained fine particles in the froth phase. Hence, it can help in producing cleaner products and can also affect the column performance and stability.

In the present work, wash water was introduced at 5 cm below the froth exit and its flow rate was changed from 150 mL/min. to 400 mL/min. at four levels as shown in the given Table (2). Four experimental runs were carried out with this parameter while the other parameters were kept constant at the desired levels as shown in Table (2).

Table 2: Results of wash water runs.

	Wash Water Rate ml/min	$P_2O_5$ %	Recovery %
Run # 1	150	35.2	94.73
Run # 2	200	29.01	91.09
Run # 3	300	31.44	89.79
Run # 4	400	33.21	73.53

The collected samples from these runs were dried, weighed and analyzed for  $P_2O_5$  content and recovery calculations as usual. Data obtained from these runs for  $P_2O_5$  recovery and  $P_2O_5$  content are given in table (2) and plotted in Figure (3).

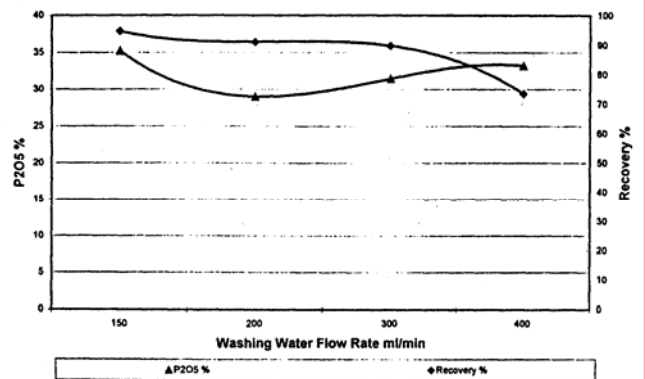


Figure 3: Effect of washing water flow rate.

**Constant Variables:**

- pH = 6.5

Particle size = 180  $\mu\text{m}$   
Pulp density = 4%  
Flotation time = 5 minute  
Collector quantity = 1.82 kg/ton  
Depressor quantity = 40 kg/ton  
Air flow rate = 1.4 ft<sup>3</sup>/min. (0.65 L/sec.)  
Slurry flow rate = 956 ml/min

As shown from table (2) and Fig. (3), the smaller value of wash water (150 mL/min.) gives the best recovery (94.73%) and the best P<sub>2</sub>O<sub>5</sub> content (35.2%). Hence, the increase of wash water rate has negative effect on both recovery and purity which can be ascribed to the destruction of the froth layer formed inside the column. [1]. Moreover, the increase in wash water can also enhance the detachment of solid particles from the froth resulting in less product quality and less recovery. Hence, small wash water rates are preferable. Other workers in this field observed also similar trends for wash water during the application of column flotation for siliceous phosphate rocks [3,4]. They reported also that, wash water rates of about 13% of the slurry feed rate is preferred for phosphate flotation. Meanwhile, in the present work, our best results are achieved at 150 mL/min. which is about 15% of the used slurry feed rate (956 mL/min.). Hence, the present results are in good agreement with those workers on column flotation.

### Effect of Slurry Flow Rate

The feed throughput "or the slurry flow rate" is a major operating parameter for any column flotation equipment. In order to elucidate the effect of this parameter, its value was varied from 956 to 2630 mL/min, at four levels "as shown in table (3) while keeping the other parameters at the desired levels "at the same time, air flow rate and wash water rate were kept at their optimum values achieved in the previous runs". The results of these runs are given in Table (3) and plotted in Figure (4).

Table 3: Results of the slurry flow rate runs.

	Slurry Flow Rate ml/min	P <sub>2</sub> O <sub>5</sub> %	Recovery %
Run # 1	956	35.2	94.73
Run # 2	1348	32.31	68.5
Run # 3	2023	30.2	41.327
Run # 4	2630	32.97	62.54

As shown from this table and that figure, both P<sub>2</sub>O<sub>5</sub> recovery and P<sub>2</sub>O<sub>5</sub> content decrease by increasing the slurry flow rate from 956 to 2023 mL/min. This decrease in recovery and grade can be ascribed to the decrease in the retention time of slurry inside the column. This reduction in the solids retention time means less flotation time inside the column and hence less recovery and less grade as expected. But, after slurry

rate of 2023 mL/min., more increase in feed rate starts to improve recovery and grade as shown in Fig. (4). This improvement can be ascribed to the effect of slurry rate on the rise velocity of bubbles as recommended by other workers [1]

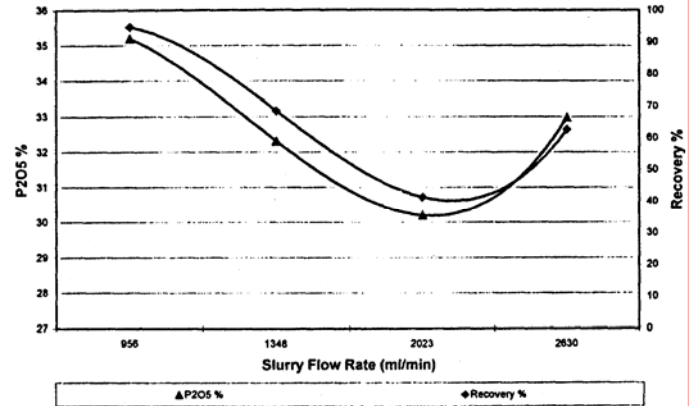


Figure 4: Effect of slurry flow rate.

### Constant Variables:

pH = 6.5  
Particle size = 180  $\mu\text{m}$   
Pulp density = 4%  
Flotation time = 5 minute  
Collector quantity = 1.82 kg/ton  
Depressor quantity = 40 kg/ton  
Washing water rate = 150 ml/min  
Air flow rate = 1.4 cf/min (0.65 L/sec.)

in this field. They reported that, increase of slurry rate after some extent can reduce the bubble rise velocity inside the column and hence can increase the bubble retention times or increase the bubble loading (or increase the gas hold-up inside the column) resulting in improving both recovery and grade as shown by fig. (4). But still, best results are achieved at the smaller slurry rate, i.e. the highest recovery (94.73%) and the best grade (35.2% P<sub>2</sub>O<sub>5</sub>) are at the smallest slurry rate (956 mL/min.).

In brief, the recommended value for the slurry flow rate in the present work is 956 mL/min.

### Effect of Collector Quantity

The collector dosage used in the present work is oleic acid dissolved in kerosene as it revealed promising results with the conventional flotation cell for phosphate beneficiation in the previous work carried out by Abd EI-Razik et al. [6].

For better understanding of the collector effect on the performance of the present designed column flotation, various collector dosages are investigated in these runs. Four levels of collector dosages were used "1.82, 2, 3, 4 kg/tons" together with the suitable amounts of kerosene (which equals to 2.5

times the collector used as reported in previous work of Abd EI-Razik [6]. The obtained results from these runs for  $P_2O_5$  content and recovery are given in Table (4) and plotted in Figure (5).

Table 4: Results of the collector quantity runs.

	Collector Quantity (kg/ton)	$P_2O_5$ %	Recovery %
Run # 1	1.82	35.2	94.73
Run # 2	2	31.13	94.6
Run # 3	3	32.51	94.77
Run # 4	4	34.62	95.91

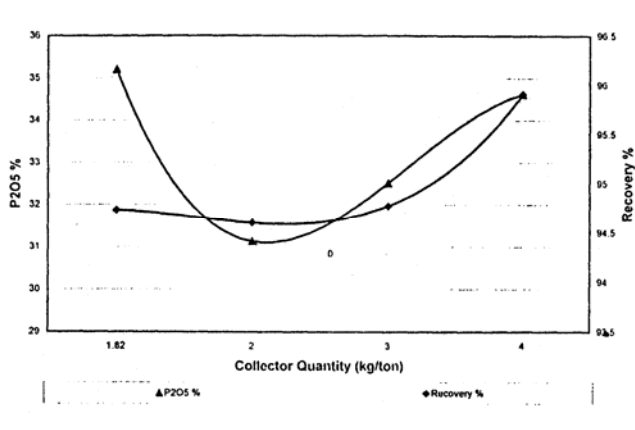


Figure 5: Effect of collector quantity.

**Constant Variables:**

- pH = 6.5
- Particle size = 180  $\mu$ m
- Pulp density = 4%
- Flotation time = 5 minute
- Air flow rate = 1.4 cf/min. (0.65 L/sec.)
- Depressor quantity = 40 kg/ton
- Wash water rate = 150 ml/min
- Slurry flow rate = 956 ml/min

As shown in Fig. (5) and Table (4), slight increase in the  $P_2O_5$  recovery (from 94.73 to 95.91) is observed by increasing the collector dosage which can be ascribed to the improvement in the hydrophobicity of the solid particles of the ore used. But at the same time the  $P_2O_5$  content of the product decreases from 35.2% to 31.13% at first, then starts to increase again until it reaches a value of 34.62% at a collector dosage of 4 kg/ton.

The initial decrease in  $P_2O_5$  content can be ascribed to the improved hydrophobicity of both calcite and apatite present in the phosphate rock enhancing the flotation of both minerals resulting in less  $P_2O_5$  content in the final product. But with

increasing the collector dosage (>2kg/ton) the selectivity for calcite separation increases giving more  $P_2O_5$  purity in the final product.

Concerning the collected results of table (4) and figure (5), still, the lower value of the collector dosage (1.82 kg/ton) is the best level at which both recovery and grade reach their best values (94.73% for recovery and 35.2%  $P_2O_5$  content). Hence the 1.82 kg/ton collector is selected to be the recommended value for this parameter.

**Effect of Particle Size of Phosphate Ores**

In the present work, this parameter is studied by investigating three levels of particle size (125  $\mu$ m, 180  $\mu$ m and 250  $\mu$ m) and conducting three column flotation runs keeping the other parameters constant at their desired levels (as shown in table (5)). The collected results from these runs are given in Table (5) and plotted in Fig. (6).

Table 5: Results of particle size runs.

	Particle Size $\mu$ m	$P_2O_5$ Before Flotation	$P_2O_5$ After Flotation	Recovery %
Run # 1	125	23.92	30.02	80.67
Run # 2	180	25.103	35.2	94.73
Run # 3	250	26.55	33.94	69.57

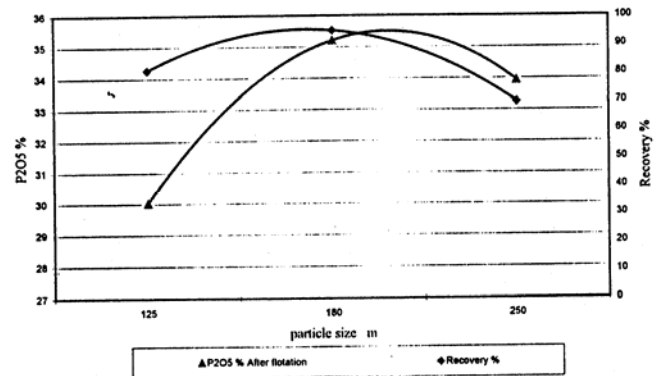


Figure 6: Effect of particle size.

**Constant Variables:**

- pH = 6.5
- Air flow rate = 1.4 cf/min. (0.65 L/sec.)
- Pulp density = 4%
- Flotation time = 5 minute
- Collector quantity = 1.82 kg/ton
- Depressor quantity = 40 kg/ton
- Wash water rate = 150 ml/min

Slurry flow rate = 956 ml/min

As shown from this table (5) and fig. (6), an optimum recovery of 94.73% was achieved at a particle size of 180  $\mu\text{m}$ . The  $\text{P}_2\text{O}_5$  content of this size has also an optimum  $\text{P}_2\text{O}_5$  content (35.2%). It can be stated that, larger particles than this optimum have lower recovery and grade due to their lower retention time inside the column. On the other hand ultra fine particles (smaller than this optimum size) have weak attachment to the surface of the bubble phase in the collection zone and may be carried out easily with the streams of wash water used. Special designs of the column flotation units are required to handle either ultra fine ores or; coarse ore particles as recently published in literature [5,7,8].

In brief, the optimum particle size for this work is 180  $\mu\text{m}$ .

Finally, all optimum values obtained in the present work were used in the final experimental run of this work and its results are given here below in Table (6) which gives the best achieved results in this work, i.e. a high recovery level of 97.5% and a high  $\text{P}_2\text{O}_5$  content of 36% with a lower CaO:  $\text{P}_2\text{O}_5$  ratio of 1.53, which is a suitable beneficiated Saudi phosphate ore for further processing towards phosphoric acid production by the wet process.

### Optimum Conditions

- (i) Air flow rate = 0.65 L/sec.
- (ii) Wash water rate = 150 mL/min.
- (iii) Slurry flow rate = 956 mL/min.
- (iv) Depressor quantity = 40 kg/ton
- (v) Collector quantity = 1.82 kg/ton
- (vi) Particle size = 180  $\mu\text{m}$

### Optimum Results

Table 6: Optimum Results.

$\text{P}_2\text{O}_5$ %	CaO%	Recovery %	Separation Efficiency (S.E) %
36	55.12	97.52	39

$\text{P}_2\text{O}_5$  % in rock before flotation = 25.103%.

CaO% in rock before flotation = 52.68%.

CaO:  $\text{P}_2\text{O}_5$  ratio before flotation = 2.1

CaO:  $\text{P}_2\text{O}_5$  ratio after flotation = 1.52

### CONCLUSIONS

The experimental results obtained in this study led to the following conclusions:

1. It is possible to beneficiate the AI-Jalamid phosphate rock by column flotation to obtain a product concentrated

enough and suitable to the wet process for phosphoric acid production.

2. This flotation technology gives good beneficiation results, i.e.  $\text{P}_2\text{O}_5$  concentration above 35% and recoveries above 95% using AI-Jalamid rock of about 25%  $\text{P}_2\text{O}_5$ .
3. Our result showed that the lowest wash water is recommended to give the suitable amount necessary to purify the froth, but high wash water rate is not recommended due to loss in  $\text{P}_2\text{O}_5$  recovery.
4. The slurry flow rate has to be kept at its lowest level to increase its retention time and improve recovery and purity of products.
5. The actual optimum conditions achieved in this work are:
  - (i) Air flow rate is 0.65 L/sec.
  - (ii) Washing water flow rate = 150 ml/min.
  - (iii) Slurry flow rate = 956 ml/min
  - (iv) Depressor quantity = 40 kg/ton
  - (v) Collector quantity = 1.82 kg/ton.
  - (vi) Particle size = 180  $\mu\text{m}$ .

### REFERENCES

1. Robinstein, J.B. *Column Flotation, Processes, Design and Practices*, Gordon and Beach Science Publishers, 1995.
2. Gerald H. Luttrell and Roe-Hoan Yoon, *Column Flotation – A Review*, Center for coal and Minerals Processing, Virginia Polytechnic Institute and State University, Pages 361-369.
3. Patil, D.P. et al. *Column Flotation of Siliceous Rock Phosphate*, Minerals and Metallurgical Processing V. 13, p. 147 – 150, 1996.
4. Polinsky, S. et al., *Column Flotation of Western Phosphate Ores*, Proceedings of Engineering Foundation by AIME, Littleto, Co. U.S.A., p. 154 – 163.
5. EI Shall, et al., recent work on column flotation, Florida, 1999. (Private Communication)
6. Abdulrazik M.H., *Beneficiation of Saudi Phosphate Rock by Flotation*, M.Sc. Thesis, 1990, King Saudi University.
7. Von Holt, S.T. and Franzidis, J.P., *Column Flotation of South Africa Cooking Coal*, Journal of Coal Preparation, V. 14, No. 3-4, p. 147 – 166, 1994.
8. Honaker, R.Q., et al. *Enhanced Column Flotation Performance for Fine Coal Cleaning Minerals Engineering*, V. 9, p. 931 – 945, 1996.