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BUBBLE GENERATION, DESIGN, MODELING AND OPTIMIZATION OF NOVEL FLOTATION COLUMNS FOR PHOSPHATE BENEFICIATION

Volume I

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September 2001

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BUBBLE GENERATION, DESIGN, MODELING AND OPTIMIZATION OF NOVEL FLOTATION COLUMNS FOR PHOSPHATE BENEFICIATION

FINAL REPORT: VOLUME I

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PERSPECTIVE

Column flotation was one of the research priorities identified in FIPR's *Strategic Initiatives and Applied Research Priorities*, prior to the 1998-2003 research plan. Currently, columns are mainly used for the coarse fraction (typically 16 by 35 mesh) of the flotation feed in some of the phosphate beneficiation plants in Florida. The recovery of phosphate from the coarse flotation feed used to be a major efficiency problem. Separate flotation of this feed using mechanical cells could recover only about 60% of the phosphate. The use of column flotation has brought the recovery up to 90%. However, maintenance costs and the problems associated with the air generation systems have prevented the industry from taking full advantage of the column flotation technology.

Columns are also believed to be more suitable than mechanical cells for floating finer (minus 35 mesh) feeds. This has been demonstrated in coal and metallic minerals processing. There are two potential applications for even finer (minus 100 mesh) flotation in processing phosphate minerals: recovery of phosphate from the phosphatic clays and separation of dolomite from the future dolomitic pebbles. Both applications are of significant importance in terms of industry efficiency and resource conservation.

Although a column is mechanically simpler, operation and maintenance costs for columns tend to be higher than mechanical cells due to the air sparger system. This is especially true in floating coarse feed. The most frequently encountered problem with an air generation system used in Florida phosphate plants is the dependence of airflow rate on waterflow, resulting in dilution of flotation pulp and loss of phosphate by washing. The Florida phosphate industry has been struggling with this problem since it first adopted column flotation for the coarse feed. Sparger plugging was also a problem.

In response to a growing interest in applying the column flotation technology to processing phosphate minerals, FIPR funded this major investigation on column flotation of phosphate. The project was composed of four major tasks: screening of frothers, evaluating air spargers, developing a column flotation model, and conducting pilot-scale column flotation tests.

Among the 28 commercially available frothers screened, CP-100 and F507 were found to be most suitable for column flotation of Florida phosphate. However, CP-100 proved to be more sensitive to water chemistry, leaving F507 to be the optimal choice.

Nine spargers were evaluated. Air holdup measurements were conducted for these spargers in a two-phase, air-water system for various frothers, and the bubble size was estimated for various operation conditions using the drift flux model. A comparison was made amongst spargers in terms of air holdup, estimated bubble size, and operational characteristics. The purpose of these analyses is to screen the effectiveness of eleven spargers and twenty-eight industrial frothers for phosphate column flotation.

Patrick Zhang Research Director - Beneficiation & Mining

ABSTRACT

To select frothers for sparger performance and phosphate flotation, 28 commercially available frothers were investigated by measuring air holdup under various operational conditions in an air/water system. The detailed description of operation characteristics for different commercially available spargers is given in this report. Generally speaking, an eductor sparger and two-phase ejector have strong air dispersion ability, simple operation, less clogging potential, and less energy consumption, compared with other external spargers. However, the addition of much more water to the eductor is required to aspirate atmospheric air into the sparger and to disperse into fine bubbles. For phosphate rougher flotation, the water added by the eductor meets the requirement for dilution of the dewatered reagentized feed. In applications where the feed is not dewatered, the eductor water may cause excess water addition to the flotation system. This problem can be overcome by properly selecting the eductor size to minimize the addition water amount. An economic performance measure was developed, which includes recovery, grade, and the reagent prices. A parametric study was conducted on both unsized and sized feeds to optimize column flotation. Another useful product of this project is a novel "intelligent" model for phosphate column flotation which combines a first-principles model with artificial neural networks. The model learns from column operational data: the more data presented to the model, the more accurate it becomes.

ACKNOWLEDGEMENTS

The Column Flotation Study was funded by the Florida Institute of Phosphate Research, managed by the University of Florida, and guided by a steering committee. Jacobs appreciated the good project management provided by Dr. Hassan El-Shall from the University of Florida's Department of Material Science and Engineering, and also the dedicated leadership of Dr. Patrick Zhang, Beneficiation Research Director for the Florida Institute of Phosphate Research. The diligent and dedicated efforts of each member of the steering committee are gratefully acknowledged. The committee's questions and suggestions contributed to the success of the project.

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- Cargill Fertilizer, Inc.
 - Fort Meade Plant
 - Hookers Prairie Plant
 - South Fort Meade Plant
- IMC-Agrico
 - Hopewell Plant

The following companies provided equipment and/or reagents to Jacobs free of charge for use during the project:

<u>Spargers:</u> The Deister Concentrator Company, Inc. EIMCO MULTOTEC

<u>Reagents:</u> Oreprep Chemicals O'Brien Industries Westvaco Chemical Division

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EXECUTIVE SUMMARY

The University of Florida in cooperation with Jacobs Engineering Group and BCD Technologies/ University of Alabama was granted funds to investigate the possibility of using flotation column to float phosphates of different feed sizes.

The proposed deliverables were:

- 1. A list of frothers that can be used for cost effective phosphate recovery.
- 2. A comparative analysis of the performance of sparger/frother systems with phosphate ore.
- 3. An economic model for evaluating rougher phosphate flotation using columns.
- 4. A user-friendly computer program that incorporates a column model and a suitable optimization algorithm for investigating "what if" scenarios and for determining starting values for operating variables for new columns or new column conditions.

To achieve these objectives, the work plan consisted of seven main tasks including:

- Task 1: Sample collection and dispatch.
- Task 2: Economic performance measure of operating variables.
- Task 3: Evaluation of aerators and frothers.
- Task 4: Parametric study of different feed sizes.
- Task 5: Comparative pilot testing of open columns.
- Task 6: Model development and program for optimizing the operation of open columns.
- Task 7: Final report.

This Volume I of the final report contains the data generated for tasks conducted by University of Florida and BCD Technologies including tasks # 3, 4, and 6. Data obtained for the tasks conducted by Jacobs Engineering involving tasks # 1, 2, the pilot plant parts in task 3, and 5, are presented in Volume II.

TASK 1: SAMPLE COLLECTION AND DISPATCH

Samples were collected by Jacobs Engineering and dispatched to University of Florida as described in Volume II.

TASK 2: ECONOMIC PERFORMANCE MEASUREMENT OF OPERATING VARIABLES

The economic performance measure is developed by Jacobs Engineering team (see Volume II). However, it is utilized by University of Florida's modeling team as follows:

Selectivity and separation efficiency do not include any economic input such as cost of the reagents. Therefore, an alternate performance measure was developed which includes recovery, grade, and the reagent prices. A scheme for penalizing lower grade rock has been developed. This scheme deducts differential costs, relative to 66% BPL, for transportation and acidulation. The acidulation scheme assumes soluble P_2O_5 losses increase in direct proportion to the amount of phosphogypsum. Thus, the procedure requires an estimate of the quantity of phosphogypsum that is produced.

The following assumptions are evoked:

- 1. The price of rock of 66% BPL = \$22.00
- 2. Zero insol % BPL = 73.33
- 3. Transportation cost = \$2.50 per ton.
- 4. Soluble P_2O_5 losses = 1.00%
- 5. Insoluble P_2O_5 losses = 6.00%
- 6. Increase in soluble P_2O_5 losses is proportional to the amount of phosphogypsum produced.

The transportation penalty is calculated as follows:

Base case: 66% BPL rock (dry basis) Freight cost per BPL ton = \$2.5/0.66 = \$3.79 Penalty: $\left(\frac{2.50}{B_L/100}\right) - 3.79$ per BPL ton Transportation penalty = $\left(\frac{2.50}{B_L/100} - 3.79\right) \frac{B_L}{100}$ per ton

where, $B_L = \% BPL$ when grade < 66%

The acidulation penalty is calculated as follows:

Base case: 66% BPL rock (30.21% P₂O₅, CaO:P₂O₅ = 1.49) Acid insol = $100\left(1 - \frac{B_L}{73.33}\right)$

Phosphogypsum components:

Acid insol = 1 ton rock
$$\times \left(1 - \frac{B_L}{73.33}\right)$$

Unreacted = 1 ton rock ×
$$\left(\frac{B_L}{73.33}\right)$$
 × 0.06
Dihydrate = 1 ton rock × $\frac{(B_L/100)}{2.184}$ × 1.49 × (172/56) × (1 - 0.06)

Total amount of phosphogypsum = Acid insol + Unreacted + Dihydrate

Soluble P₂O₅ losses =
$$\$300.0 \times \frac{(\% \text{ so lub le P}_2O_5 \text{ losses})/100}{2.184}$$
 per ton
= \$1.37 per ton

Acidulation Penalty = $\$62.0 \times \frac{(\text{Total amount of phosphogypsum})}{B_L} - 1.37$

The phosphate sales value increases with increasing grade, and the following relationship is assumed:

Sales value = Price of 66 % BPL rock * $(B_L/66)^{1.5}$

The adjusted sales value is calculated from:

Adjusted sales value = Sales value - Transportation penalty - Acidulation penalty

Let

Feed solid flow rate = F, ton per year Product solid flow rate = P, ton per year Feed grade = G_f , % Concentrate grade = G, % Product recovery = R, % Adjusted sales value of feed = C_f , \$ per ton Adjusted sales value of product = C_p , \$ per ton Reagent-i price = C_{ri} , \$/lb Reagent-i usage = U_i lb/ton feed

The feed flow rate and the product flow rate can be related by:

$$P = F\left(\frac{G_{f}}{100}\right)\left(\frac{R}{100}\right)\left(\frac{100}{G}\right)$$

The financial performance measure, representing dollars earned per year, is:

Financial Performance Measure = $C_pP - C_fF - F\sum_i U_iC_{ri}$, \$/year

TASK 3: EVALUATION OF AERATORS AND FROTHERS

Evaluation and Selection of Industrial Frothers

To select frothers for sparger performance and phosphate flotation, 28 commercially available frothers were involved in this investigation by measuring air holdup under various operational conditions in an air/water system.

Using air hold up measurements, the frothers in the present study can be divided into three groups: frothers with high, medium and low air dispersion ability. Frothers that have good air dispersion and high air holdup are OB-535 and OB-503 (from O'Brian), F-507, X-268, X-269, F-559, F-549 (from Oreprep), Percol F-948 and Percol F-940 (from Allied Colloids Inc), and H-230 (from Mineral Reagent International). Frothers that have moderate air holdup are H-205 and H-225 (from Mineral Reagent International), MIBC, Pine oil, Aerofroth 65, Percol F-941, Oreprep M-606 and F-571, and Aromox DMC9 (from Akzo Nobel). At low frother concentration and low superficial air velocity, some frothers from these two groups have very similar air holdup data. Frothers with poor air dispersion ability are: CP-100 (from Westvaco), F-551, WMX 6978-63, WMX 27-AR, Aromox C/12, Aromox DMHT, and Aromox NM16 (from Akzo Nobel).

In addition, the air holdup of lab water and that of plant water are compared. It was found that without adding frother, the air holdup in plant water was higher at high air flow rate, as compared to university lab water. With the addition of Oreprep F-507 at 16 and 30 ppm, the air holdup data was found to be similar.

Based on the suggestions of steering committee of this project, the next stage was to select four frothers that represent the three categories of air holdup performance for sparger performance evaluation and flotation tests as conducted by Jacobs Engineering research team (see Volume II). University of Florida investigators have concentrated their efforts on evaluation of flotation performance in presence of two frothers (F-507 and CP-100).

Two and three phase experiments indicated that CP-100 was sensitive to the water chemistry including the residual of fatty acid/fuel oil content in the conditioning water and pH, while F-507 was not significantly affected by these factors. Most of the initial testing was conducted in presence of tap water. However, the data showing the deterioration of CP-100 performance due to fatty acid/fuel oil presence in conditioning water is presented in later sections of this report

Evaluation of Spargers for Phosphate Column Flotation

In this investigation, nine spargers were evaluated, in addition to two more spargers tested by Jacobs Engineering, Inc. (see Volume II). Air holdup measurements were conducted for these spargers in a two-phase, air-water system for various frothers, and the bubble size was estimated for various operation conditions using the drift flux model. A comparison was made amongst spargers in terms of air holdup, estimated bubble size, and operational characteristics. The purpose of these analyses is to screen the effectiveness of eleven spargers and twenty-eight industrial frothers for phosphate column flotation.

As the first step, the University of Florida and Jacobs Engineering Inc. collected technical information on various air sparging systems including those fully developed and widely applied in industrial installations as well as those still in the stage of development. Direct contacts were established with the manufactures and suppliers. Nine spargers were collected or fabricated by the University of Florida, the manufactures or supplies are listed as follows:

Three spargers from Cominco Engineering Inc.:

- Cominco two-phase external sparger
- Cominco one-phase internal sparjet sparger
- One-phase internal porous sparger.

Two-phase eductor

Two external spargers from Pyramid Inc. (USBM sparger)

One sparger from Mott Metallurgical Corp., i.e., two-phase external porous sparger.

Three spargers were homemade. They are:

- two-phase ejector (external sparger).
- two-phase static mixing sparger (external sparger).
- one-phase perforated tube (internal sparger).

Deister two-phase sparger and Eimco duck-bell sparger.

The effects of operational conditions (i.e., superficial liquid velocity, superficial air velocity, sparger pressure, sparger water requirement, and frother type and concentration) on air holdup and bubble size for these spargers was discussed in the previous reports.

To investigate the effect of frother type on sparger performance, air holdup was also measured for a frother-containing air/water system (frother MIBC, X-268, OB-535, and Aerofroth-65 in addition to Westvaco CP-100 and Oreprep F-507), and the bubble size was estimated using drift flux model. The results showed that frother type has a significant influence on sparger performance For example, frothers F-507, X-268, and OB-535 showed higher air dispersion ability than frother CP-100 and MIBC. The typical bubble size for CP-100 frother was found to be in a range of 0.7-1.2 mm when the two-phase ejector was used. For the rest frothers tested, the typical bubble size was 0.3 to 0.6mm diameter.

The test results were arranged to allow direct comparison of the sparging systems, based on their air dispersion abilities and the amenabilities to operations under similar operation conditions.

1. Air dispersion ability. For the air/water systems containing 15 mg/L of F-507 frother, it is clearly illustrated that there are significant differences between spargers in air dispersion ability, which implies that the selection of sparger directly influences the air dispersion in the column, and consequently the flotation performance.

Based on the air holdup measurement and bubble size estimation under similar operation conditions and pressure at 40-50 psig, the air dispersion ability of spargers investigated can be arranged as follows:

Two-phase ejector, and Eductor > One-phase porous sparger > Cominco two-phase sparger > USBM sparger > Two-phase static mixing sparger ~ Two-phase porous sparger > Cominco one-phase sparger > Perforated tube.

For the system with 30 mg/L of CP-100 frother, a similar order of air dispersion ability for the spargers can be obtained. However, much bigger bubble size will be generated when frother CP-100 is used instead of F-507.

2. Sparger water requirement. An important feature of the external spargers is the use of clean frother-containing water. The water consumption is proportional to the sparger air pressure and is inversely proportional to the airflow rate. For any given air pressure and flow rate, there is one corresponding value of water flow rate giving a characteristic ratio of air to water. Water requirement for the five external spargers decreases in the following order:

Eductor > Two-phase static mixing sparger > Two-phase ejector > Cominco two-phase sparger > USBM sparger.

3. Operation Characteristics. The detailed description of operation characteristics for different commercially available spargers is given in Volume II of this report. Generally speaking, eductor sparger and two-phase ejector have strong air dispersion ability, simple operation, less clogging potential, and less energy consumption, compared with other external spargers. However, the addition of much more water to the eductor is required to aspirate atmospheric air into the sparger and to disperse into fine bubbles. For phosphate rougher flotation, the water added by the eductor meets the requirement for dilution of the dewatered reagentized feed. In applications where the feed is not dewatered, the eductor water may cause excess water addition to the flotation system. This problem can be overcome by properly selecting the eductor size to minimize the addition water amount.

TASK 4: PARAMETRIC STUDY OF DIFFERENT FEED SIZES

The objectives of the present study are to evaluate and select sparger and frother for phosphate column flotation, to investigate the effect of operational conditions (air flow rate, air pressure, discharge rate, and frother concentration) on phosphate flotation performance, and to optimize operational parameters to achieve a better separation efficiency. For these purposes, five different phosphate samples with various particle size ranges were prepared as the feed of column flotation, and the tests were conducted at the University of Florida (UF) and Jacobs Engineering Inc. (see Volume II). The flotation results for unsized (14x150 mesh), coarse (20x35 mesh), and fine (35x150 mesh) phosphate feeds are summarized as follows.

Unsized Phosphate (14x150 mesh)

- 1. The results of size-by-size recovery show that different particle sizes have different flotation responses, and the flotation recovery decreases as particle size increases for all frothers tested.
- 2. Bubble size has a significant influence on the flotation performance of unsized phosphate, and the optimum bubble size is found to be in the range of 0.8-1.0 mm.
- 3. To achieve a better flotation performance, there is an optimum combination between sparger and frother type. Generally speaking, for the sparger with stronger air dispersion ability, the weaker frother should be used to generate desirable bubble size; On the other hand, for the sparger with poorer air dispersion ability, the stronger frother should be added.

Coarse Phosphate (20x35 mesh)

- 1. The results show that, by using column flotation and properly selecting the operational parameters, coarse phosphate recovery can be significantly improved, for example, 99% recovery with 69% BPL content in the concentrate is expected to be achieved.
- 2. A net upward (negative bias) water flow prevalent in the column is helpful to improve the coarse phosphate flotation.
- 3. Bubble size has a significant influence on the coarse phosphate flotation, and there is an optimum bubble size range to obtain lower BPL content in the tailings and higher BPL recovery in the concentrates. From the flotation results obtained, it was found out the optimum bubble diameter for coarse phosphate flotation to be in the range of 0.8-1.0 mm.
- 4. In tap water, using CP-100 as frother yields better separation performance than F-507 when eductor is used as sparger. However, in presence of conditioning water (containing residual collector), F 507 shows superior performance over that of CP 100.
- 5. The order of significance for the effect of variables on BPL content in the concentrate is:

Slurry discharge rate > frother type > airflow rate > frother concentration.

The order of significance for the effect of variables on the recovery is: Frother type > frother concentration > slurry discharge rate > airflow rate

Fine Phosphate (35x150 mesh)

- 1. The results showed that frother type and operational conditions strongly affect the grade of flotation concentrate. Under practical conditions, (plant water containing residual collector) frother F-507 performs better than other frothers. However, using tap water, CP100 gives better grades in the phosphate concentrates.
- 2. It is interesting to note that, under the lab testing conditions, it is found that the frother type has no significant influence on phosphate recovery.
- 3. The order of significance for the effect of variables on BPL and A.I. contents in the concentrate can be determined as follows:

Frother type > airflow rate > frother concentration.

TASK 5: COMPARATIVE PILOT TESTING OF OPEN COLUMNS

The results of this task are given in Volume II of this report

TASK 6: MODEL DEVELOPMENT AND PROGRAM FOR OPTIMIZING THE OPERATION OF OPEN COLUMNS

The major accomplishments are:

- Development of a novel "intelligent" model for phosphate column flotation that combines a first-principles model with artificial neural networks that relate important model parameters to the column operating conditions. The model learns from column operational data: the more data presented to the model, the more accurate the model becomes. This is the first model to incorporate the effect of reagents (frother, collector, extender, and pH adjuster) on the performance of a flotation column. Furthermore, all previous models are for the conventional positive bias flotation columns instead of the negative bias columns used to float the larger phosphate particles. (In positive bias flotation wash water is added from the top, while in negative bias flotation water is added from the bottom).
- Development of several criteria for assessing the performance of the column. Among them is an economic performance criterion developed primarily by Jacobs Engineering that weighs recovery, grade, as well as the cost of reagents and utilities.
- A user-friendly windows program for the model that allows easy entry of operational data and automates model learning. The program outputs the predicted grade and recovery, as well as the economic performance measure developed by Jacobs Engineering.

INTRODUCTION

Traditionally, the Florida phosphate industry has effectively responded to the competitive pressures of the market place by implementing technological improvements to mining and beneficiation methods.

With further expansion of fertilizer production capacity worldwide, and depletion of Florida's high-grade deposits, the competitive pressures are expected to continue. If Florida is to remain the world leader, it is important to continue seeking out environmentally sound technological improvements in all areas of phosphatic fertilizer production.

In the beneficiation of Florida phosphate rock, flotation plays a predominant role because it is the most economical way to separate the phosphate values from the sand and other impurities contained in the matrix. Typically, the matrix is washed and deslimed at 150 mesh. The finer than 150 mesh is pumped to clay settling ponds. The rock coarser than 150 mesh is screened to separate pebbles (-3/4 + 14 mesh), which are of high phosphate content. Washed rock (-14 + 150 mesh) is, in many cases, sized into a fine (usually 35 x 150 mesh) and a coarse flotation feeds (usually 14 x 35 mesh) which are treated in separate circuits.

Flotation of phosphates from the fine feed (35×150 mesh) presents very few difficulties and recoveries in excess of 90% are achieved using conventional flotation cells. On the other hand, recovery of phosphate values from the coarse feed is much more difficult and flotation by itself usually yields recoveries of 60% or less.

The density of the solid, turbulence, stability and height of the froth layer, tenacity of the particle - bubble attachment, depth of the water column, viscosity of the froth layer are known to effect the flotation process in general. However, the exact reasons for low recovery of coarse particles in conventional flotation is not very well understood. There are several hypotheses about the flotation behavior of coarse particles. For instance, the floatability of large particles could be due to the additional weight that has to be lifted to the surface under the heavy turbulence conditions, and the difficulty to transfer and maintain these particles in the froth layer.

As mentioned above, flotation recovery of coarse fraction in mechanical flotation cells is much lower than the fine fraction. In this regard, the industry has developed innovative techniques to circumvent the problem of low floatability of coarse particles. For instance, such approaches are exemplified by the use of gravitational devices such as spirals, tables, launders, sluices and belts modified to perform "skin Flotation" of the reagentized pulp. Although a variable degree of success is obtained with these methods, they have to be supplemented by scavenger flotation. In addition, some of them require excessive maintenance, have low capacity or have high operating costs. Thus, a more efficient and cost effective technology is sought. In this regard, FIPR funded two projects through University of Florida (FIPR # 87-02-067) and Laval University (FIPR#

98-02-070). In the work done by University of Florida's research team, an increase in the recovery of coarse particles was obtained by adding frothers to the mechanical cells. In a different route, Laval University investigators designed a column cell and tested its performance both in the laboratory and the plant. In this case also, a frother was used. Results were extremely encouraging. Up to 99% BPL recovery was obtained with good concentrate grade (8+% insol). Currently, several companies such as Cargill, Agrifos, IMC-Agrico, etc are using columns to float coarse phosphates.

High recovery and grade and low operating cost depend largely on the optimal selection of operating variables such as the air flow rate, the feed flow rate, the frother concentration, and the wash water rate (if used). The search of the optimal conditions can considerably benefit by the availability of a flotation column model and a performance measure. The model can not only provide better understanding of the effect of the operating variables on column performance, but can also provide good starting points for the search of operating conditions that optimize the performance measure. Thus it can lessen the amount of experimentation required to determine optimal settings for important operating variables.

Considerable work has been done on modeling conventional flotation column designs. The collection zone has been frequently modeled by a one-dimensional dispersion model with a first order rate for flotation. The Peclet number (Pe) governs the degree of mixing. Froth zone recovery has been modeled as simply plug flow. Important model parameters (e.g., Pe or the flotation rate constant) depend on the operating variables, but also on particle characteristics (such as particle size), the chemical additives used, and the geometry of the column. For this reason a model that is successful with one column may be very poor in predicting the behavior of an apparently similar column.

Considering the above facts, The University of Florida in cooperation with Jacobs Engineering Group and BCD Technologies/ University of Alabama was granted funds to investigate the possibility of using flotation column to float Florida phosphates of different feed sizes and to deliver the following:

- 1. A list of frothers that can be used for cost effective phosphate recovery.
- 2. A comparative analysis of the performance of sparger/frother systems with phosphate ore.
- 3. An economic model for evaluating rougher phosphate flotation using columns.
- 4. A user-friendly computer program that incorporates a column model and a suitable optimization algorithm for investigating "what if" scenarios and for determining starting values for operating variables for new columns or new column conditions.

METHODOLOGY

FLOTATION COLUMN SETUP

The experimental set-up is shown in Figures 1 (A and B) and 2. A 5.75-inch inside diameter "Plexiglas" flotation column was constructed at the University of Florida. The height of the column can be varied from 4 ft to 6, 8, and 10 ft depending on the requirement of the process. In the present investigation, the height of the column was fixed at 6 ft. The bottom section can accommodate the installation of different spargers either from the side or at the base direction. The sampling ports are provided along the length of the column at the distance of every foot.

The sparger testing circuit consisted of the following elements: column, circulating tank, feed pump, discharge pump, air and/or sparger water gauges, air and/or sparger water flow meters, pressure regulators, and air valve, as shown in Fig. 1A.

TEST PROCEDURES FOR EVALUATION OF DIFFERENT FROTHERS AND SPARGERS IN TWO-PHASE EXPERIMENTS

Sparging systems were installed one at a time at the bottom section of the column and operated over a predetermined range of the operating variables. To provide test conditions similar to those present in an industrial column the circuit was built to vary downward liquid discharge flow rates by adjusting feed and discharge pumps speeds. Liquid discharged from the column through a 1/2 inch pipe, discharge pump, and flow meter, into a 230 L tank. A circulation (feed) pump delivers liquid back to the column at the location of 4.5 feet from the column bottom with the same rate as discharge. When an external type (two-phase sparger) was being tested, a portion of water from the tank was introduced into the sparger through a sparger pump and flow meter.

At the beginning of each series of test runs, the circulating tank was filled with fresh tap water to 100 liters, and the frother was added to meet to the prescribed concentration. The solution was mixed approximately 15 minutes to assure uniform distribution of frother in water. The column was then filled with frother-containing water, and the discharge pump was started at the same rate as feeding pump. Known volumetric flow rate of air was then introduced into the sparger to give the required superficial air velocity. For two-phase spargers, the air pressure was adjusted by altering the flow rate of sparger water. Sparger water with the same frother concentration as in the column was delivered into sparger by the pump and monitored by the flow meter. No solid was present in these experiments. The system was operated for couple of minutes to ensure equilibrium conditions before air hold measurement was taken. After a run was completed the variables were adjusted to new values and measurements were repeated.



Figure 1A. Schematic Diagram of Column Flotation Setup for 2-Phase Experiments.



Figure 1B. Schematic Diagram of Column Flotation Setup for 3-Phase Experiments.



Figure 2. Picture of UF Flotation Column.

EXPERIMENTAL SETUP FOR 3-PHASE EXPERIMENTS

The experimental setup, for 3-phase experiments, includes an agitated tank (conditioner) for reagentizing the feed, a screw feeder for controlling the rate of reagentized feed to the flotation column, and a flotation column, as shown in Figures 1B and 2.

Agitated tank:17.5 inches diameter x 29 inches highImpeller:two axial type impellers with 11 inches diameter.465 rpm rotation speed.1.5 inches clearance between impeller and tank bottom.

The feeder with one-inch diameter screw delivers the conditioned phosphate materials to the column; the feed rate was controlled by adjusting the screw rotation speed. For the present investigation, the screw rotation speed varies from 100 to 190 rpm, depending on the feed rate required.

Flotation tests were conducted using a 5.75-inch diameter by 6 feet high "Plexiglas" flotation column. The feeding point is located at 1 foot from the column top. A discharging valve underneath the column and an adjustable speed pump controlled the discharge flow rate. Three flow meters were used to monitor the flow rates for air, frother solution, and water, respectively.

EXPERIMENTAL PROCEDURE FOR 3-PHASE EXPERIMENTS

Two bags of feed material (22 kg each) were conditioned at 72% solid concentration by weight in an agitated tank for 15 second, 10% soda ash solution was added to the pulp to reach pulp pH (if otherwise not mentioned) of 9.4 and agitated for 45 second. Then, a mixture of fatty acid and fuel oil with a ratio of 1:1 by weight was added to the pulp at the predetermined dosages and mixed for another 2 minutes. The conditioned feed material was loaded in the feeder bin located at the top of the column.

Frother-containing water and air were first introduced into the column through the sparger at a fixed flow rate and frother concentration, and then the discharge valve and pump were adjusted to get the desired underflow and overflow rates. Air holdup was measured for the two-phase system using differential pressure gauge. After every parameter was set and two-phase system was in a steady state, the phosphate material with 66% solid concentration was fed to the column at a location of 1 ft from the column lip, and was further diluted in the column to a certain concentration. The feed rate can be adjusted by changing feeder's auger rotation speed. To insure that the samples were obtained from a steady-state column, the column was run for a period of three minutes with phosphate feed prior to sampling after a change in operating conditions. Timed samples of tailings and concentrates were taken. The collected samples were dried, weighed and analyzed for BPL and acid insoluble.

SURFACE TENSION MEASUREMENTS

A roller Smith precision balance (Figure 3) was used to measure the surface tension of different frothers at ten different concentrations: 2,4, 8, 12, 16, 20, 30, 40, 60, and 80 ppm. In each test, the force required to lift a platinum wire ring off the surface of the frother solution was compared to the force required for deionized water at 23° C.

MATERIALS

Phosphate Ore Samples

Five different phosphate samples, as shown in Table 1, were prepared by Jacobs Engineering, which were taken from Cargill's Fort Meade Mine, screened, and blended. The more detailed procedure for phosphate sample preparation is given in the report of Jacobs Engineering Inc.



Figure 3. Schematic Diagram of Surface Tension Measurement.

 Table 1. Phosphate Samples Used for Column Flotation Experiments.

Sample Name	Nominal size, mesh	% BPL	% A.I.
Unsized	14x150	18.3	74.2
Spiral feed	14x20	61.5	14.3
Belt feed	14x35	43.1	39.8
Coarse feed	20x35	39.4	45.4
Fine feed	35x150	12.8	82.3

Reagents Used in 3-Phase Experiments

Soda ash solution (10% by weight) was used to adjust pulp pH for phosphate conditioning. A mixture of fatty acid and fuel oil with a ratio (if otherwise not mentioned) of 1:1 by weight, supplied by Westvaco, was used as collector. The frothers selected for 3-phase experiments, based on the results of evaluation of different frothers in 2-phase tests, are listed below:

F-507	a mixed polyglycol by Oreprep.
CP-100	a sodium alkyl ether sulfate by Westvaco.
X-268*	a mixed polyglycol and diols by Oreprep.
OB-535	a glycol methyl ester by O'Brian

^{*} Recently, the producing company (Oreprep) changed its commercial name to F-579.

RESULTS AND DISCUSSIONS

EVALUATION AND SELECTION OF INDUSTRIAL FROTHERS FOR COLUMN FLOTATION OF PHOSPHATES

The addition of frothers during coarse phosphate flotation enhances phosphate recovery, but the mechanisms are not completely understood. The frother concentration, chemical composition, and sparging system all affect frother performance. The selection of a frother depends primarily on air dispersion, froth characteristics, physico-chemical interactions with other processing reagents, environmental impacts, and economic factors.

Air dispersion can be evaluated using the average bubble size and distribution, or the interfacial area between bubbles and liquid, or the air holdup in a flotation column. The measurement of air holdup is a reliable and most economic method for comparing the efficacy of air dispersion for many frothers. Air holdup is defined as the volume ratio of air to liquid phases, and this is expressed as a percentage. In addition, a frother's ability to increase air dispersion may be correlated to its ability to modify the interface properties.

A list of 28 commercially available frothers involved in the current investigation is presented in Table 2. The surface tension as a function of concentration in water for five of these frothers is studied. Using the air holdup data, ten frothers are selected that represent the different chemical families of industrial frothers. The performance of these ten frothers are examined at the University of Florida using different sparging systems in a lab scale, continuous feed flotation column. Five of these frother/sparger systems are submitted to Jacobs Engineering for examination on pilot plant scale. The Florida phosphate industry standard frother, Oreprep F-507, is used at all stages of the investigation for comparison purposes.

The effect of frother concentration on air holdup for seven different frothers is shown in Figure 4. As expected, air holdup increases as frother concentration increases. The frother reduces the surface tension of water so that air dispersion is increased, bubble coalescence is decreased, and the bubble rise velocity is decreased. Figure 4 also shows that there are differences between frothers in air holdup performance. Frothers that have high air holdup have good air dispersion characteristics, and may be good frothers for coarse phosphate flotation. However, the superficial air velocity also influences frother performance and air holdup; this is illustrated in Figures 5 and 6 for frother concentrations of 16 ppm and 30 ppm, respectively. These figures demonstrate that the air holdup increases as superficial air velocity increases.

Using air holdup measurements, the frothers in this study can be divided into three groups: frothers with high, medium and low air dispersion ability. Frothers that have good air dispersion and high air holdup are shown in Figures 7 and 8. These frothers are OB-535, F-507, X-268, X-269, F-559, F-549, OB-503 and H-230. Frothers

that have moderate air holdup, as shown in Figures 9-11, are H-205, C-99A, MIBC, H-225, Pine oil, F-515, Aerofroth 65, DMC, Allied Colloids Percol F-941; OrePrep M-606, F-571; Allied Colloids Percol F-948 & F-940. At low frother concentration and low superficial air velocity, some frothers from these two groups have very similar air holdup data.

Manufacturer	Commercial Name	Chemical Family
Oreprep	F-507	mixed polyglycols
	F-515	Alcohols, heavy aldehydes, esters and glycol
	F-551	
	F-549	Polyglycol ethers
	F-559	
	F-571	N/A
	M-606	Alkylated hydroxy polyethers
	X-268	N/A
	X-269	N/A
Mineral Reagent International (MRI)	Н-320	Alcohol
	H-225	alcohols, aldehyde, esters
	H-205	N/A
Akzo Nobel	Aromax C/12	amine oxide/alcohol
	Aromax DM16	amine oxide/alcohol
Manufacturer	Commercial Name	Chemical Family
	Aromax DMC	amine oxide/alcohol
	Aromax DMHT	amine oxide/alcohol
Cytec	Aerofroth 65	Polypropylene glycol
Shell	MIBC	Alcohol
Arizona Chemicals	Pine oil	
Allied colloids	Procol F-948	N/A
	F-940 B	N/A
	F-941 B	N/A
O'Brian	OB-535	N/A
	OB-503	N/A

Table 2. List of Frothers Used for	Column Flotation Experiments.
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Manufacturer	Commercial Name	Chemical Family
Westvaco	CP-99A	aliphatic glycols/esters
	CP-100	Sodium alkyl ether sulfate
	Custofloat 27-AR	Fatty acids soap and sulfates
	WMX 6978-63	Sodium sulfonates

 Table 2. List of Frothers Used for Column Flotation Experiments (Cont.).


Figure 4. Effect of Frother Concentration on Air Holdup.



Figure 5. Effect of Superficial Air Velocity on Air Holdup.



Figure 6. Effect of Superficial Air Velocity on Air Holdup.



Figure 7. Effect of Superficial Air Velocity on Air Holdup: Frothers Generating Higher Air Holdup at 16 mg/L Concentration.



Figure 8. Effect of Superficial Air Velocity on Air Holdup: Frothers Generating Higher Air Holdup at 30 mg/L Concentration.



Figure 9. Effect of Superficial Air Velocity on Air Holdup: Frothers Generating Moderate Air Holdup at 16 mg/L Concentration.



Figure 10. Effect of Superficial Air Velocity on Air Holdup: Frothers Generating Moderate Air Holdup at 30 mg/L Concentration.



Figure 11. Effect of Superficial Air Velocity on Air Holdup.

Figures 12-14 show the air holdup for the frothers with poor air dispersion ability: CP-100, F-551, WMX 6978-63, WMX 27-AR, Akzo Nobel Aromox DMHT, C-12 and DM-16.

In addition, the air holdup of lab water and that of IMC plant water are compared in Figure 15. Without adding frother, the air holdup in plant water is higher at high airflow rate, compared to university lab water. With the addition of Oreprep F 507 at 16 and 30 mg/L, the air holdup data is similar.

The results of the surface tension measurements for five frothers are presented in Figure 16. These data demonstrate that surface tension decreases as frother concentration increases, and some frothers, specifically Oreprep F-507, decrease surface tension more than others. However, frothers that are not water-soluble could not be compared using this technique because water insoluble components collect at the solution surface and interfere with surface tension measurement. Since many commercially available frothers contain water insoluble components, air holdup data provides a better indication of frother performance.



Figure 12. Effect of Superficial Air Velocity on Air Holdup: Frothers Generating Low Air Holdup at 16 mg/L Concentration.



Figure 13. Effect of Superficial Air Velocity on Air Holdup: Frothers Generating Low Air Holdup at 30 mg/L Concentration.



Figure 14. Effect of Superficial Air Velocity on Air Holdup.



Figure 15. Effect of Water Resources (University Water at Gainesville and IMC Plant Water) on Air Holdup.



Figure 16. Relationship Between Surface Tension and Frother Concentration.

EVALUATION OF SPARGERS FOR COLUMN FLOTATION OF PHOSPHATES

Background

Flotation columns are widely accepted in the mineral industry in recent year. The main advantages of flotation column over mechanically agitated conventional cells arise as a consequence of their ability to generate well-controlled hydrodynamic regimes and concentration gradients. These, in turn, aid in the collection of desired particles and the rejection of gangue. Further advantages arise from the relative ease by which flotation columns can be controlled, from savings in the energy and maintenance, as well as by the possibility of optimizing bubble size to the application at hand. The overall performance of flotation column is strongly affected by the type of sparger used, which has a decisive influence on the size distribution of the bubbles formed and, consequently, on air holdup, air/liquid interfacial area, and flotation performance. Therefore, selection and design of the sparger is a particularly important aspect of column flotation.

The spargers for column flotation can be broadly divided into two types: internal and external spargers. Internal spargers deliver air into the column through a material installed within the bottom of the column, such materials as sintered glass, porous rubber or filter cloth, or a single or-multi-nozzle sparger. With external spargers, air and liquid (either surfactant-containing water or slurry) are brought into contact outside the column before the mixture is introduced into the column. Generally, the spargers used for column flotation should be able to provide bubbles of the desired size and be capable of varying bubble size in order to meet processing requirements. The spargers should be robust, energy efficient, easily maintained and/or replaced, friendly operated, and exhibit little tendency to become plugged and worn out. The sparger tip velocity should be low in order to minimize back mixing and operational instabilities. However, the influence of sparger design and operational conditions on phosphate column flotation is not fully In this investigation, various spargers for column flotation were understood vet. evaluated and compared in order to select proper spargers meeting the above mentioned desirable features beneficiation.

Objectives

The objectives of this investigation are:

- Evaluate the critical operating variables, air dispersion abilities, and operating features of different spargers using an air/water system.
- Investigate the interaction between sparger and frother.
- Compare the sparger performance for phosphate beneficiation on the basis of the recovery-grade curve and utilize selectivity coefficient.
- Select spargers for phosphate column flotation.

Spargers Evaluated in Air/Water System

As the first step, University of Florida and Jacobs Engineering Inc. collected technical information on various air sparging systems including those fully developed and widely applied in industrial installations as well as those still in the stage of development. Direct contacts were established with the manufactures and suppliers. Nine spargers were collected or fabricated; the manufactures or supplies are listed as follows:

Three spargers from Cominco Engineering Inc.:

- Cominco two-phase external sparger
- Cominco one-phase internal sparjet sparger
- one-phase internal porous sparger

Two external spargers from Pyramid Inc. (USBM sparger) One sparger from Mott Metallurgical Corp., two-phase external porous sparger.

Three spargers were homemade. They are:

- two-phase ejector (external sparger)
- two-phase static mixing sparger (external sparger)
- one-phase perforated tube (internal sparger)

Spargers Description

Two-phase ejector: Two-phase ejector, as shown in Figure 17, consists of a liquid jet nozzle, an aspiration compartment, and a parallel throat 1.2 mm in diameter, and a divergent diffuser. Liquid phase is pumped to the ejector nozzle and the high-speed liquid jet formed by the nozzle enters the suction chamber into which compressed air is simultaneously fed due to the decrease in pressure at the nozzle outlet. The high-speed liquid jet, having a pulsating character, decomposes in the parallel throat (momentum exchange tube) and in the diffuser into fine droplets, which hit the air/liquid interface (bubble surface) causing its breakup. The small bubbles thus formed are then further disintegrated into even finer bubbles due to highly turbulent dynamic pressure in the parallel throat and in the diffuser.



Figure 17. Two-Phase Ejector.

USBM Two-phase spargers: Two USBM spargers utilize a air/water mixing chamber that disperses pressurized air in a packed bed of plastic beads prior to passing the air/water mixture through fine orifices or slotted rubble. Pressurized air and water containing frother enter this chamber filled with small (approximately 0.2 cm in diameter) plastic pellets. The purpose of this chamber, as stated by the supplier, is to assure intimate air/water contact and to provide easier control of the system (Figure 18). The air/water mixture enter the column through stainless steel tube perforated with 2 holes of 0.10 cm in diameter, or through stainless steel tube with a slotted rubber head. In this investigation, the former distributor was used. The USBM spargers are presently being marketed by Control International, and Pyramid Resources, Inc.



Figure 18. USBM Sparging Systems.

- Cominco Two-phase sparger: The Cominco two-phase sparger is similar to the USBM design except that the pressurized air and water are mixed at stainless steel tube rather than in the packed bed of plastic beads. The air/water mixture is injected into the column through two 0.10 cm diameter holes perforated in the tube.
- Two-phase static mixing sparger: Two-phase static mixing sparger is made of Konics static mixer with 7 1/2 inch long and 1/4 inch inside diameter (Figure 19). Air and water are introduced into the sparger at the end of static mixer through a T-joint, and mixed in the mixer. The air/water mixture exits the sparger from the other end of sparger, and is distributed in the column through a distributing ring located within the column.



Figure 19. Two-Phase Static Mixing Sparger.

Two-phase porous sparger: Two-phase porous sparger was supplied by Mott Metallurgical Corp. The sparger is 10 inch long and 1/4 inch inside diameter. Air and water under pressure enter the sparger where bubbles are generated by blowing air through micro porous media into sparger under very high shear forces provided by fast moving sparger water (Figure 20). The bubble water mixture is then distributed throughout the column by a perforated ring located within the column.



Figure 20. Two-Phase Porous Sparger.

Cominco One-phase sparjet sparger: The sparjet is a single-orifice, air-only sparger with a wear-protected nozzle and an adjustable needle valve with a T handle at the other end of the sparger, as shown in Figure 21. The needle valve allows for fine-tuning of the nozzle opening. The pressure of sparger depends on the airflow rate and the opening of the nozzle.



Figure 21. Cominco One-Phase Sparger.

Perforated tube: A perforated tube was made of a 1/2-inch diameter copper tube. 25 holes with 4 mm in diameter were perforated along the 3 3/4-inch diameter ring, as shown in Figure 22. This internal sparger directly distributes air across the column through these orifices.



Figure 22. Perforated Tube Sparger.

One-phase porous metal sparger: The one-phase porous metal sparger (Figure 23) was supplied by Cominco Engineering Services Ltd. (CESL). The sparger has 7/8 inch diameter and 10.5 inches length for porous section. The porous material was made of 316 stainless steel with porous holes.



Figure 23. Schematic Diagram of One-Phase Porous Metal Sparger.

Eductor sparger: The eductor sparger, also known as a jet pump, has a similar structure as two-phase ejector. The eductor used in the present investigation has a 3/8" in diameter for aspiration and discharge ends (3/8" Penberthy ELL jet pump). It consists of a liquid jet nozzle, an aspiration compartment, a parallel throat, and a divergent diffuser. Liquid phase containing frother is pumped to the eductor through the jet nozzle, air is sucked into the aspiration compartment, and then air is dispersed into fine bubbles in the parallel throat. Eductors are usually operated at 30 to 50 psig, depending on their sizes, and the required flow rates of air and water. For the eductor used for the present investigation, the relationship between water pressure, eductor water flow rate, and air aspiration rate, is shown in Table 3.

 Table 3. Operational Characteristics of the 3/8" Eductor.

Pressure Psig	Water flow rate L/min	Max. aspirated air flow rate L/min
20	8	3.5
30	12	8.7
40	14	12.1

Performance Evaluation

Performance of a sparger is assessed by its ability to disperse a required volume of air represented by the air holdup and by the size of the bubbles produced. These responses thus provided the basis for the evaluation study. In the present investigation, air holdup for various spargers under a wide operational conditions was measured using manometers, and is calculated by,

$$\varepsilon_{\rm g} = \Delta H/L \ge 100\% \tag{2.1}$$

where, $\boldsymbol{\varepsilon}_{g}$, air holdup, %;

 Δ H, difference in water level in manometers, cm; L, distance between manometers on the column, cm.

Experimental Conditions

The range of experimental conditions investigated was selected with a view of the industrial application of the column and operational characteristics of each individual sparger. Generally, four operating variables affect sparger performance, i.e., airflow rate, liquid discharge rate from the column, air pressure, and frother concentration. In industrial operation these variables are normally closely controlled to an established optimum, however they were tested over a wider range in this study.

In column flotation, air flow rate and liquid discharge rate from the column are usually presented as superficial air velocity and superficial liquid velocity respectively, and they can be expressed as the ratio of air flow rate or liquid flow rate over the cross section area of the column, i.e.,

 $V_{g} = Q_{a}/A \tag{2.2}$

 $V_{\rm L} = Q_{\rm L}/A \tag{2.3}$

where, V_g, superficial air velocity, cm/s

V_L, superficial liquid velocity, cm/s Qa, airflow rate, Q_L, liquid discharge flow rate, A, column across section area.

Bubble size was estimated using drift flux model. The concept of drift flux analysis was introduced by Wallis (1969) to relate phase flow rates, holdup and physical properties. From the drift flux analysis an estimate of terminal bubble rise velocity, U_T , is obtained which in turn can be used to calculate bubble diameter. This method was used to estimate bubble size in flotation column by Dobby and others (1988). The calculation of bubble diameter involves an iterative routine. The calculation steps are:

1. calculate terminal rising velocity of bubble

$$U_{\rm T} = \frac{V_{\rm g}}{\varepsilon_{\rm g} (1 - \varepsilon_{\rm g})^{\rm m}} - \frac{V_{\rm l}}{\left(1 - \varepsilon_{\rm g}\right)^{\rm m}}$$
(2.4)

where, V_g and V_L are superficial air velocity and superficial discharge velocity, respectively, cm/s.

 ε_g is air holdup, %

m is a function of Reynolds number. Here, m is fixed at 2.0, following the suggestion of Wallis (1969) for fine bubbles (d_b <2.0 mm).

2. calculate d_b iteratively

$$U_{T} = \frac{g d_{b}^{2} (\rho_{1} - \rho_{g})}{18 \mu_{1} (1 + 0.15 \text{ R e}_{b}^{0.687})}$$
(2.5)

$$\operatorname{Re}_{b} = \frac{d_{b}U_{T}\rho_{l}}{\mu_{l}}$$
(2.6)

where, g is acceleration due to gravity, cm/s² ρ_l and ρ_g are liquid and air density, g/cm³ μ_l is liquid viscosity, g/cm.s

Two frothers were used to evaluate the sparger performance and the interaction between spargers and frothers, i.e., OrePrep F-507 and Westvaco CP-100. The former has higher air dispersion ability, while the latter shows lower air dispersion ability as mentioned before. The frother concentrations were fixed at 15 ppm and 30 ppm, which are the typical frother concentrations in industrial flotation processes. The superficial liquid velocity varied from zero to 0.6, and 1.0 cm/s. The liquid retention time in the column was 5 and 3 minutes for superficial liquid velocity at 0.6 and 1.0 cm/s, respectively. The superficial air velocity and pressure for each sparger strongly depend on the design of the sparger, and the operation conditions are listed as shown in Table 4.

Sparger	Superficial air Velocity, cm/s	Pressure, psi
two-phase ejector	0.24, 0.46, 0.70, 0.94	20, 30, 40, 50, 60
Cominco two-phase sparger	0.24, 0.46, 0.70, 0.94	20, 30, 40, 50, 60
USBM sparger	0.24, 0.46, 0.70	20, 30, 40, 50, 60
two-phase static mixing sparger	0.24, 0.46, 0.70, 0.94, 1.18, 1.41	20, 30, 40, 50
Two phase porous sparger	0.24, 0.46, 0.70, 0.94, 1.18, 1.41, 1.66	5 to 8
Cominco one-phase (sparjet) sparger	0.24, 0.46, 0.70, 0.94, 1.18, 1.41, 1.66	2 to 52
perforated tube	0.24, 0.46, 0.70, 0.94, 1.18, 1.41	2

Table 4. Operation Conditions for Various Spargers.

Effect of Superficial Liquid Velocity

The effect of liquid downward discharge rate from the column on air holdup is shown in Figures 24 and 25 for Cominco two-phase sparger and two-phase ejector, respectively. In general, for all spargers investigated, it was found that air holdup increases slightly with superficial liquid velocity. This may be attributed to the fact that higher downward liquid flow rate reduces the bubbles rising velocity in the column and causes a slight increase in the air holdup.



Figure 24. Effect of Superficial Liquid Velocity on Air Holdup for Cominco Two-Phase Sparger.



Figure 25. Effect of Superficial Liquid Velocity on Air Holdup for Two-Phase Ejector (30mg/L, F-507).

Effect of Superficial Air Velocity

Figures 26 through 32 show the effect of superficial air velocity on air holdup for seven spargers at 30 ppm of F-507. It can be seen that air holdup strongly depends on superficial air velocity for all spargers except two-phase ejector. Amongst five external spargers, two-phase ejector and Cominco two-phase sparger are less responsive to the airflow rate. Although the two internal spargers (Cominco sparjet and one-phase perforated tube) are more responsive to the airflow rate increase, the resulting holdups are lower than those obtained from the external systems for a given superficial air velocity. For sparjet air-only sparger, higher air holdup can be expected only at high superficial air velocity and at high pressure, however, these conditions may be associated with strong back mixing and bigger bubble size in the column.



Figure 26. Effect of Superficial Air Velocity on Air Holdup for Two-Phase Ejector (30 mg/L F-507).



Figure 27. Effect of Superficial Air Velocity on Air Holdup for Cominco Two-Phase Sparger (30 mg/L).



Figure 28. Effect of Superficial Air Velocity on Air Holdup for USBM Two-Phase Sparger (30 Mg/L, F-507).



Figure 29. Effect of Superficial Air Velocity on Air Holdup for Two-Phase Static Mixing Sparger (30 mg/L, F-507).



Figure 30. Effect of Superficial Air Velocity on Air Holdup for Two-Phase Porous Sparger (30 mg/L F-507).



Figure 31. Effect of Superficial Air Velocity and Sparger Pressure on Air Holdup for Cominco One-Phase Sparger (30 mg/L, F-507).



Figure 32. Effect of Superficial Air Velocity on Air Holdup for Perforated Tube Sparger (30 mg/L, F-507).

Effect of Operating Pressure

The operating pressure for two-phase sparger depends on air and sparger water flow rates. Operating pressure directly influences the energy dissipation rate within the sparger, which in turn determines air dispersion characteristics. Figures 33 to 36 show the effect of pressure on air holdup for four external spargers. It was found that the air holdups almost linearly increase with the increase in pressure.



Figure 33. Effect of Pressure on Air Holdup for Two-Phase Ejector (30 mg/L, F-507).


Figure 34. Effect of Pressure on Air Holdup for Cominco Two-Phase Sparger (30 mg/L, F-507).



Figure 35. Effect of Air Pressure on Air Holdup for USBM Sparger (30 mg/L, F-507).



Figure 36. Effect of Air Holdup for Two-Phase Static Mixing Sparger (30 mg/L, F-507).

Effect of Frother Concentration and Frother Type

The effect of frother concentration and frother concentration on air holdup is shown in Figures 37 to 43. It is generally accepted that air holdup is strongly influenced by the air/water interfacial characteristics (frother type and concentration) due to its ability to promote air dispersion, hinder bubble coalescence, and decrease bubble rise velocity. As shown in Figures 37 and 38, air holdup decreases as frother concentration decreases from 30 to 15 ppm. This can be attributed to the fact that the presence of less frother causes an increase in bubble size in the column consequently decreasing air holdup. In Figure 41, air holdup rapidly increases as frother concentration increases from 5 to 10 ppm, particularly at higher airflow rate, and then this tendency becomes less pronounced. Figure 42 shows the bubble size at various airflow rates and frother concentration from 5 to 10 ppm. Beyond 10 ppm of F-507 the bubble size becomes slightly smaller, even increasing frother concentration at lower airflow rate.

To investigate the effect of frother type on the sparger performance, air holdup was also measured for the air/water systems containing MIBC, X-268, OB-535, and Aerofroth-65 in addition to Westvaco CP-100 and OrePrep F-507, and the bubble size was estimated using drift flux model. Figures 39, 40 and 43 reconfirm the results mentioned before, that frother F-507, X-268, and OB-535 has higher air dispersion ability than frother CP-100 and MIBC. The typical bubble size for CP-100 frother is found to be in a range of 0.7-1.2 mm when the two-phase ejector is used. For the rest frothers tested, the typical bubble size is 0.3 to 0.6 mm diameter.

It is clear from these results that frother type have more significant influences on two-phase spargers than one-phase internal spargers. The internal spargers (Cominco one-phase sparger and perforated tube) show a limited influence of the frother used on air holdup, whereas the external spargers revealed a stronger relationship. It is concluded that, in general, when the external spargers are operated at a suitable pressure, the air holdup can be controlled by the frother type and concentration over a wide range.



Figure 37. Effect of Frother Concentration on Air Holdup.



Figure 38. Effect of Frother Concentration on Air Holdup.



Figure 39. Effect of Frother Type on Air Holdup for Various Spargers.



Figure 40. Effect of Frother Type on Air Holdup for Various Spargers.



Figure 41. Effect of Frother Concentration on Air Holdup.



Figure 42. Effect of Frother Concentration on Bubble Size.



Figure 43. Effect of Frother Type and Superficial Air Velocity on Bubble Size with Two-Phase Ejector Sparger.

Water Requirements for Two-Phase Spargers

An important feature of the external spargers is the use of clean frother-containing water. The water consumption is proportional to the sparger air pressure and is inversely proportional to the airflow. For any given air pressure and flow rate, there is one corresponding value of water flow giving a characteristic ratio of air to water. It can be seen from Figs. 44 to 47 that for all spargers, sparger water requirements depend on the airflow rate and the pressure settings, and water flow rate decreases with an increase in airflow rate at a given pressure.



Figure 44. Sparger Water Requirements at Different Air Flow Rates Under Various Operating Pressures (30 mg/L, F-507).



Figure 45. Sparger Water Requirements at Different Air Flow Rates Under Various Operating Pressures (30 mg/L, F-507).



Figure 46. Sparger Water Requirements at Different Air Flow Rates Under Various Operating Pressures (30 mg/L, F-507).



Figure 47. Sparger Water Requirements at Different Air Flow Rates Under Various Operating Pressures (30 mg/L F-507).

Sparger Comparison

The test results were arranged to allow direct comparison of the sparging systems, their air dispersion ability and the amenability to operation under similar operation conditions.

Air Holdup

Figures 48 to 50 compare the air holdup and bubble size generated by various spargers for the air/water systems containing 15 ppm of F-507 frother. It is clearly illustrated that there are significant differences between spargers in air dispersion ability, which implies that the selection of sparger directly influences the air dispersion in the column, and consequently the flotation performance.

Based on the air holdup measurement and bubble size estimation under similar operational conditions and pressure at 40~50 psig, the air dispersion ability of spargers investigated can be arranged as follows:

Two-phase ejector, and Eductor > One-phase porous sparger > Cominco twophase sparger > USBM sparger > Two-phase static mixing sparger ~ Two-phase porous sparger > Cominco one-phase sparger > Perforated tube.

Use of two-phase ejector and eductor in which air is exposed to very high local energy dissipation rates in the throat and diffuser, were found to be particularly beneficial to air dispersion. Cominco two-phase sparger has a superior air dispersion ability as compared to USBM sparger at the operating pressure ≤ 60 psig. On the other hand, Cominco two-phase sparger has a simple design compared with USBM sparger. Two-phase static mixing sparger and two-phase porous sparger have poorer air dispersion ability than two-phase ejector, Cominco two-phase sparger, and USBM sparger, due to less local energy dissipation rate presented in these types of spargers; The air dispersion ability for Cominco one-phase sparger is more responsive to the superficial air velocity and the corresponding pressure. Perforated tube yields lowest air holdup and coarse bubbles due to low local energy dissipation rate in the sparger.



Figure 48. Comparison of Air Holdup for Various Spargers.



Figure 49. Comparison of Bubble Size for Various Spargers.



Figure 50. Comparison of Air Holdup for Various Spargers.

Sparger Water Requirements

An important feature of the external spargers is the use of clean frother-containing water. The water consumption is proportional to the sparger air pressure and is inversely proportional to the airflow. For any given air pressure and flow rate, there is one corresponding value of water flow giving a characteristic ratio of air to water. The sparger water flow rate to achieve the required operational pressure for four external spargers are given in Figure 51. The sparger water requirement decreases as the following order:

Eductor > Two-phase static mixing sparger > Two-phase ejector > Cominco two-phase sparger > USBM sparger.

This can be explained in terms of the sparger orifice size through which air/water mixture is delivered into the column from the sparger and the packing extent within the sparger in which air and water are mixed. USBM sparger needs less amount of sparger water than Cominco two-phase sparger, although they have identical orifice diameter (1.0 mm) for the distribution of air/water mixture into the column. This is because USBM sparger uses a plastic pellets packed generator (mixing chamber) which provides a high resistance to air and water flow and has a limited air and water throughput capacity. Since two-phase ejector has bigger orifice holes (1.2 mm) than Cominco two-phase sparger and USBM sparger (1.0 mm in diameter), it requires more water than Cominco two-phase sparger and USBM sparger. Two-phase static mixing sparger has the biggest inside diameter (1/4" inside diameter) as compared with the orifice size of other spargers, which demands larger amount of water to build up the required pressure within the sparger.



Figure 51. Comparison of Sparger Water Requirements at Various Air Flow Rates and Operating Pressures for Different Two-Phase Spargers (30 mg/L F-507).

Operation Characteristics

The detailed description of operation characteristics for different commercially available spargers was given by the report prepared by Jacobs Engineering, Inc. Generally speaking, eductor sparger and two-phase ejector have strong air dispersion ability, simple operation, less clogging potential, and less energy consumption, compared with other external spargers. However, the addition of much more water to the eductor is required to aspirate atmospheric air into the sparger and to disperse into fine bubbles. For phosphate rougher flotation, the water added by the eductor meets the requirement for dilution of the dewatered reagentized feed. In applications where the feed is not dewatered, the eductor water may cause excess water addition to the flotation system. This problem can be overcome by properly selecting the eductor size to minimize the addition water amount.

A PARAMETRIC STUDY OF COLUMN FLOTATION FOR PHOSPHATE BENEFICIATION

Unsized Phosphate Column Flotation

Experimental Conditions

Table 5 shows the experimental conditions for column flotation of unsized phosphate feed. The experimental conditions for frother concentration, superficial air velocity, and sparger pressure were selected with a view of the industrial application of the column and operational characteristics of each individual parameter.

The superficial discharge velocity for the flotation of this feed was adjusted to ensure non-formation of a froth layer at the top section of the column, unlike conventional columns featured by forming froth bed and adding washing water. However, the discharge rate for unsized feed flotation was kept at 0.96 cm/s to maintain less overflow in order to minimize the entrainment of fine gangue particles into the concentrate product. With this discharge rate, up to 70% of water introduced into the column was reported to the tailings stream. No conditioning water was used in these experiments. The chemical dosage for conditioning was selected after consulting with IMC, Cargill, and Jacobs Engineering Inc.

For the unsized phosphate flotation, the effect of collector dosage, frother type, frother concentration, and interaction between sparger and frother on the flotation performance was investigated. The samples collected were subsequently screened to determine the size-by-size BPL recovery under different operation conditions. An attempt was also made to correlate the bubble size with flotation performance.

Parameter	Conditions
Frother concentration, mg/L	3, 5, 10 15, 25
Sparger pressure, psig	40
Superficial air velocity, cm/s	0.46
Superficial discharge rate, cm/s	0.96
Fatty acid, Kg/t	0.32, 0.66
Fuel oil, Kg/t	0.32, 0.66
Soda ash, Kg/t	0.21
Pulp pH	9.5
Feed rate, Kg/min	3.5
Overall solid concentration, wt% in the column	22

 Table 5. Experimental Conditions for Column Flotation of Unsized Feed Sample.

Effect of Collector Dosage

The effect of collector dosage on column flotation was investigated under two reagent dosage levels using frother F-507 and CP-100, i.e., fatty acid at 0.38 Kg/t and 0.66 Kg/t, as shown in Table 6. It was found that for both collector dosage levels there was no significant difference in BPL recovery in presence of 15 ppm of F-507. However, a slightly higher BPL content in the concentrate can be obtained when higher collector dosage is applied. When frother CP-100 was used, higher BPL recovery (>96%) was achieved at higher collector dosage, compared with 92% recovery obtained at lower collector dosage, but an increase of collector amount resulted in a slight reduction in the BPL content of concentrate products. Based on the results obtained, fatty acid and fuel oil at a dosage of 0.66 Kg/ton for each were selected for the subsequent tests for unsized phosphate feed.

Table 6.	Effect of	Collector	Dosage on	Unsized	Phosphate	Column Flotation.
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Sparger: Eductor, 40 psig

Frother Type	Frother Conc.		<u>Concentrate</u>			
	mg/L	wt.%	BPL%	Recov.%	BPL%	
Fatty acid: 0.38	Kg/t; Fuel Oil: 0.	38 Kg/t				
F-507	15	46.1	31.2	92.0	2.3	
CP-100	15	27.6	58.3	92.0	2.1	
CP-100	5	31.3	60.0	92.7	2.2	
Fatty acid: 0.66	Kg/t; Fuel Oil: 0.	66 Kg/t				
F-507	15	47.2	33.4	92.9	2.3	
CP-100	15	31.5	56.0	96.3	1.0	
CP-100	5	32.3	56.8	96.8	0.9	

Effect of Frother Type

The effect of frother type on unsized phosphate column flotation is summarized in Table 7. It can be seen that frother has a significant influence on the flotation separation. Under the present experimental conditions, amongst the four frothers tested, it was found that CP-100 showed superior flotation performance to the others, for example, 96% recovery with 56% BPL content in the concentrate can be achieved. The performance of X-268 and F-507 was very similar in terms of recovery and concentrate quality, and frother OB-535 yielded lowest BPL recovery.

Table 7. Effect of Frother Type on Unsized Phosphate Column Flotation.

Frother Type	Frother Conc. mg/L	wt.%	Concentrate BPL%	Recov.%	Tailings BPL%
OB-535	15	39.3	41.7	86.3	4.3
X-268	15	52.2	33.2	91.8	3.2
F-507	15	47.2	33.4	92.9	2.3
CP-100	15	31.5	56.0	96.3	1.0

Fatty acid: 0.66 Kg/t; Fuel oil: 0.66 Kg/t.

To find out the effect of frother on the recovery of different particle size, the timed tailings and concentrate samples collected for those four frothers were subsequently screened to determine the size-by-size BPL recovery. The results are shown in Tables 8 to 10 and in Figure 52. The data in Tables 8 to 10 show a good agreement between the composite results calculated from size fractions and the results calculated directly from concentrate and tailings head samples, which indicates that sampling, sample preparation, and chemical analyses had low experimental error. Tables 8 to 10 indicate that high recovery can be obtained for fine particle sizes for all those frothers, however, as particle size increases, the recovery decreases accordingly. This tendency becomes more pronounced for frothers OB-535 and X-268, and the recovery drops off quickly for coarser particle sizes. For frother CP-100, BPL recovery is more evenly distributed across the range of particle sizes, as shown in Figure 52. Based on the results obtained, the order of phosphate particle recovery for the four frothers can be arranged as follows: CP-100 > F-507 > X-268 > OB-535.



Figure 52. Size-By-Size BPL Recovery for Unsized Phosphate Column Flotation.

Table 8. Size-By-Size BPL Recovery for Unsized Phosphate Column Flotation.

Frother: OB-535, 15 ppm

Tyler mesh	Conce wt.%	entrate BPL%	<u>Tai</u> wt.%	lings BPL%	Recovery to C weight	Concentrate (%) BPL
+20	2.9	69.0	2.5	49.1	1.1	50.1
20/28	10.4	68.5	7.3	20.8	4.0	74.6
28/35	8.3	66.4	7.5	7.9	3.2	85.3
35/48	5.6	65.5	13.2	2.8	2.2	86.1
48/65	24.4	49.8	47.4	0.9	9.4	94.8
65/100	45.1	25.4	20.7	1.2	17.4	96.7
100/150	3.3	25.8	1.4	2.5	1.3	93.9
Composite	100	42.8	100	4.4	38.5	85.8
Analyzed		41.7		4.3	39.3	86.3

Table 9. Size-By-Size BPL Recovery for Unsized Phosphate Column Flotation.

Frother: X-268, 15 ppm

Tyler mesh	Conce wt.%	entrate BPL%	<u>Tai</u> wt.%	lings BPL%	Recovery to C weight	Concentrate (%) BPL
+20	2.7	69.5	1.9	45.4	1.4	70.5
20/28	7.9	70.4	7.5	12.5	4.1	86.8
28/35	6.4	66.2	9.7	3.9	3.3	92.4
35/48	4.3	43.7	9.3	2.0	2.2	91.6
48/65	32.8	28.2	46.9	0.8	17.2	96.3
65/100	40.4	15.3	22.0	1.0	21.3	96.9
100/150	5.5	22.2	2.6	2.2	2.9	95.8
Composite	100	30.2	100	3.1	52.5	91.6
Analyzed		33.2		3.2	52.2	91.9

Tyler Mesh	Conce wt.%	entrate BPL%	<u>Tai</u> wt.%	lings BPL%	Recovery to C weight	Concentrate (%) BPL
+20	5.2	68.3	0.5	16.4	1.9	95.9
20/28	13.5	68.8	3.6	3.8	4.8	97.4
28/35	10.6	68.3	5.2	1.2	3.8	98.5
35/48	6.0	64.8	9.7	0.32	2.1	98.5
48/65	22.7	45.2	59.7	0.2	8.1	98.2
65/100	36.0	40.3	19.6	0.1	12.8	99.7
100/150	6.0	34.0	1.7	0.2	2.1	99.6
Composite	100	50.8	100	0.4	35.6	98.4
Analyzed		50.4		0.5	35.7	98.1

Table 10. Size-By-Size BPL Recovery for Unsized Phosphate Column Flotation.

Frother: CP-100, 15 ppm

Effect of Frother Concentration

The effect of CP-100 concentration on unsized phosphate flotation is given in Table 11. It can be seen that when CP-100 concentration varies in a range of 5 to 25 ppm, there is no significant influence on the flotation performance. On the contrast, when F-507 concentration decreases from 15 to 3 ppm, the BPL recovery can be increased from 93% to 97% (Table 12).

Table 11. Effect of Frother Concentration on Unsized Phosphate Flotation.

Frother Type	Frother Conc.		Concentrate		
	ppm	Wt%	BPL%	Recov.%	BPL%
CP-100	5	32.3	56.8	96.8	0.9
CP-100	15	31.5	56.0	96.3	1.0
CP-100	25	34.4	54.9	96.8	0.9

Fatty acid: 0.66 Kg/t; Fuel oil: 0.66 Kg/t.

Tyler mesh	Conce wt.%	entrate BPL%	Tai wt.%	lings BPL%	Recovery to C weight	Concentrate (%) BPL
+20	3.3	70.7	1.0	19.2	1.9	94.3
20/28	8.4	70.7	7.0	1.3	4.8	98.9
28/35	6.4	68.5	8.6	5.9	3.7	92.0
35/48	3.8	60.3	9.6	2.8	2.2	92.2
48/65	35.8	23.2	45.7	0.5	20.6	97.8
65/100	38.3	17.3	25.6	0.6	22.0	98.5
100/150	4.0	25.3	2.4	0.8	2.3	98.5
Composite	100	30.9	100	1.5	57.5	96.6
Analyzed		31.9		1.3	57.4	97.0

Table 12. Size-By-Size BPL Recovery for Unsized Phosphate Column Flotation.

Frother: F-507, 3 ppm

Effect of Bubble Size

An attempt was made to correlate the bubble size with flotation performance under various operational conditions. The bubble size was estimated using drift flux model as described before. Table 13 shows that both recovery and BPL content in the concentrate strongly depend on the bubble size generated in flotation column. Frothers OB-535, X-268, and F-507, which have strong air dispersion ability, give poor flotation separation compared with CP-100 when eductor is used as sparger. This may be attributed to the fact that the former three frothers generate much fine bubbles (about 0.5 mm) than CP-100. Although it is not clear yet why finer bubbles deteriorate the flotation separation, it was observed that fine bubbles cause the formation of bigger aggregates of particles/bubbles due to hydrophobic hetero-coagulation, which drastically reduces the rising velocity of particle/bubble aggregate, and resulting in an decrease of flotation rate. Figure 53 indicates that optimization of bubble size plays an important role to achieve a better metallurgical performance.

Table 13. Effect of Bubble Size on Unsized Phosphate Column Flotation.

Frother Type	Frother Conc.	Concentrate		Estimated
	ppm	BPL%	Recov.%	Bubble size,
				mm
OB-535	15	41.7	86.3	0.51
X-268	15	33.2	91.8	0.50
F-507	15	33.4	92.9	0.52
F-507	3	31.9	97.0	0.81
CP-100	25	54.9	96.8	0.83
CP-100	15	56.0	96.3	0.90
CP-100	5	56.8	96.8	1.02

Fatty Acid: 0.66 Kg/t; Fuel oil; 0.66 Kg/t.



Figure 53. Effect of Bubble Size on BPL Recovery and BPL Content in Tailings for Coarse Phosphate Flotation.

Interaction Between Frother and Sparger Type

To investigate the interaction between frother and sparger type, the flotation tests were conducted using two different spargers and frothers. The two spargers used are Cominco two-phase sparger and eductor, the later has a higher air dispersion ability; the two frothers tested are F-507 and CP-100, and the later produces a relative coarser bubbles, as shown before. The result is summarized in Table 14. It can be seen that there is an interaction between frother and sparger used, resulting in affecting the flotation performance. This is because frother directly determines the interfacial properties of air and liquid, and sparger determines the local energy dissipation rate for air dispersion, the combination of those two factors decides the bubble size generated. When 15 ppm of frother F-507 is used, Cominco sparger gives better concentrate quality and slightly higher recovery than eductor sparger. When CP-100 is used as frother, eductor sparger yields higher recovery than Cominco sparger, but with comparable concentrate quality. These results imply that to achieve a better flotation performance, there is an optimum combination between sparger and frother, which is believed to affect the bubble size. For the sparger with stronger air dispersion ability, the weaker frother should be used to generate desirable bubble size. On the other hand, for the sparger with poorer air dispersion ability, the stronger frother should be added.

Table 14. Effect of Sparger and Frother Type on Unsized Phosphate Column Flotation.

	Sparger Type		Concentrate	Tail	ings
-		wt.%	BPL%	Recov.%	BPL,%
F-507, 15 mg/L	Cominco two-phase sparger	37.1	42.5	93.5	1.7
	Eductor	47.2	33.4	92.9	2.3
F-507, 3 mg/L	Cominco two-phase sparger	33.8	39.3	74.6	6.8
	Eductor	57.4	31.9	87.3	1.3
CP-100, 15 mg	/L Cominco two-phase sparger	23.7	58.4	91.0	1.8
	Eductor	31.5	56.0	96.3	1.0

Fatty acid: 0.66 Kg/t; Fuel oil: 0.66 Kg/t; Sparger pressure: 40 psig;

Coarse Feed Phosphate Column Flotation

The conditions of column flotation of coarse feed are listed in Table 15. The experimental conditions for frother concentration, superficial air velocity, and sparger pressure were selected with a view of the industrial application of the column and operational characteristics of each individual parameter.

The discharge rate for coarse feed flotation was fixed at 0.3 and 0.5 cm/s, which is much lower than that for unsized feed, in order to maintain a net upward water flow (negative bias) in the column to help floating coarse phosphate particles. At the superficial discharge velocity of 0.3 to 0.5 cm/s, it is estimated that 70% and 50% of water was reported to the concentrate product, respectively. The chemical dosage for conditioning was selected after consulting with IMC, Cargill, and Jacobs Engineering Inc. No conditioning water was used in these experiments.

Parameter	Conditions
Frother concentration, mg/L	5, 25
Sparger pressure, psig	40
Superficial air velocity, cm/s	0.46, 0.70
Superficial discharge rate, cm/s	0.30, 0.50
Fatty acid, Kg/t	1.70
Fuel oil, Kg/t	1.70
Soda ash, Kg/t	0.21
Pulp pH	9.5
Feed rate, Kg/min	1.8
Overall solid concentration, wt% in the column	15

 Table 15. Experimental Conditions for Coarse Feed.

Factorial Design for Some Parameters Affecting Column Flotation of Coarse Feed

For the coarse size fraction (20x35 mesh), experiments were conducted using 2^{4-1} fractional factorial design. Here, four factors (frother type, air flow rate, discharge/upward flow rate, frother concentration) at two levels each were used for the factorial design. The variables were coded between "-" and "+", where "-" represents the low level and "+" represents the high level of the factor, however, for the factor of frother type, "-" stands for the frother F-507 and "+" for the frother CP-100. The levels of the coding are indicated in Table 16.

Table 16. Levels of Variables.

Variable	Code	Level		
		(-)	(+)	
Frother type	А	F-507	CP-100	
Superficial air velocity, cm/s	В	0.46	0.70	
Superficial discharge velocity, cm/s	С	0.3	0.5	
Frother Concentration, mg/L	D	5	25	

There are eight possible combinations, as given in Table 17. The calculation of the effects and analysis of variance were carried out according to Yates' method. The results of the eight experiments corresponding to the factorial design are also shown in Table 17. Yates' analysis for the grade of phosphate concentrate and BPL recovery are given in Table 18.

				Concentrate					Tailings	
				BPL%	A.I %	Mass Wt.%	BPL Rec. %	A.I. Rec. %	BPL %	A.I. %
1	-	-	-	35.2	50.8	30.2	95.6	18.2	0.7	98.4
2	+	-	-	54.8	22.6	20.5	95.4	5.6	0.7	98.4
3	-	+	-	36.6	49.5	33.8	96.6	20.4	0.7	98.5
4	+	+	-	46.2	34.8	22.8	95.7	9.4	0.6	99.0
5	-	-	+	33.5	53.8	34.7	96.8	22.4	0.6	99.1
6	+	-	+	51.5	27.8	23.2	96.0	7.8	0.6	98.7
7	-	+	+	36.1	49.2	32.4	93.9	19.2	1.1	98.7
8	+	+	+	48.4	33.7	24.4	94.7	9.8	0.9	98.9

 Table 17. Treatment Combination and the Experimental Results of the 2⁴⁻¹ Design for the Coarse Phosphate Flotation.

A: Frother Type

B: Superficial air velocity

C: Frother concentration

The results shown in Table 17 indicate that frother type and operational conditions affect coarse phosphate flotation. For example, the BPL content in the tailing products varies in a wide range from 0.6 to 7.9%, and the corresponding BPL recovery in the concentrate changes from 99.5 down to 87.2%, depending on the experimental conditions applied. The tests show that it is possible to achieve a 98-99% recovery with reasonable good concentrate quality up to 68-69% BPL content. The effect of each individual variable on the flotation performance is discussed as follows.

The average changes in the concentrate grade and recovery for a change of frother type from F-507 to CP-100 are 1.8 and 5.2, respectively, i.e., using CP-100 frother instead of F-507 results in increasing phosphate recovery and improving concentrate quality under the present experimental conditions, Table 18. This may be contributed to the fact that F-507 frother has stronger air dispersion ability and produces finer bubbles than CP-100 does. An attempt was made to correlate the bubble size estimated from drift flux model with flotation performance, and the results will be discussed later on.

The effect of superficial air velocity on the recovery is negligible, as indicated by the average change of 0.4 % in recovery. However, the average change in BPL content in the concentrate for a change in superficial air velocity from 0.7 to 0.5 cm/s is -1.2, which shows that higher airflow rate slightly reduces the BPL content in the concentrate.

Source of variation	Concentrate		
parameter	BPL, %	Recovery, %	
Frother type, A	1.8	5.2	
Superficial air velocity, B	-1.2	0.4	
Superficial discharge velocity, C	-2.0	-1.8	
Frother concentration, D	-0.6	-2.9	

 Table 18. Effect of Different Parameters on Coarse Phosphate Flotation.

The average change of BPL content and recovery for an increase of superficial discharge velocity from 0.3 to 0.5 cm/s, which reduces a net upwards flow of water (negative bias) in the column from 7.6 to 5.6 L/min, is -2.0 and -1.8. This shows the tendency that a reduction of the net upward water flow within the column will decrease the concentrate's BPL content and the recovery. It means that using a net upwards water flow countercurrent to the downward particles flow will be a beneficial for coarse phosphate recovery, instead of the co-current flow featured by conventional columns, as confirmed by Laval University in 1992. The introduction of an upward water flow (negative bias) is essential to elevate the phosphate particles and bubbles aggregates, to eliminate the froth layer at the top of the column, and to facilitate the removal or transportation of phosphate particle from the column tip to the froth launder. This upward
water flow may also dampen the turbulence and back mixing within the column, which can deteriorate the coarse particles flotation.

It is believed that the frother concentration strongly influences air dispersion characteristics (i.e., bubble size, air holdup, and air/liquid interfacial area) in the column, consequently flotation performance. The effect of frother concentration in a range of 25 to 5 ppm on the BPL content in the concentrate may be negligible since the average change is -0.6, however, when the frother concentration is reduced from 25 to 5 ppm, the recovery can be significantly improved. This is particularly true when F-507 frother is used, as shown in Table 17. The recovery can be reduced from 97.1 to 92.1%, at the discharge velocity of 0.3 cm/s, when frother concentration is increased from 5 to 25 ppm, and the corresponding bubble diameter is changed from 0.7 down to 0.5 mm. Similarly, the recovery reduces from 94.9 to 87.2%, at a discharge velocity of 0.5 cm/s, while F-507 concentration is increased from 5 to 25 ppm.

Figure 53 shows the effect of bubble size on the BPL content in the tailings and the recovery in the concentrate. It clearly demonstrates that the bubble size has a significant influence on the coarse phosphate flotation, and there is an optimum bubble size range to obtain lower BPL content in the tailings and higher BPL recovery in the concentrates. It can be seen that fine bubble size yields higher BPL content in the tailings and lower recovery. The mechanism of this phenomenon is not clear. However, it was observed that fine bubbles cause the formation of bigger aggregates of phosphate particles and bubbles due to hydrophobic forces. These flocs have lower rising velocity within the column and lower flotation rate. On the other hand, too big bubbles also reduce the flotation efficiency. This may be attributed to the low attachment efficiency between particles and bubbles. From the flotation results obtained, it can be found out the optimum bubble diameter for coarse phosphate flotation to be in the range of 0.8-1.0 mm.

From the absolute values of the average change for different variables listed in Table 18, the order of significance for the effect of variables on BPL content in the concentrate can be determined as follows:

Slurry discharge rate > frother type > airflow rate > frother concentration.

The order of significance for the effect variables on the recovery is:

Frother type > frother concentration > slurry discharge rate > airflow rate

Parameters Affecting Flotation Chemistry of Coarse Feed

Based on the above discussion, it can be seen that parameters such as frother type and its concentration play a very important role in determining the efficiency of column flotation of coarse feed. For this reason, it has been decided to study further these parameters in addition to collector dosage and pH, i.e., parameters responsible for the chemistry of flotation. The approach used is a statistical design for the parameters: collector dosage, frother concentration and pH using each frother separately. No conditioning water was used in these experiments. The aim of that is to determine the region of optimum conditions for flotation of coarse feed with different frothers. Three frothers are investigated: F-507, F-579 and CP-100.

Experiments were conducted using 2^3 fractional factorial design. Here, three factors (collector dosage, frother concentration and pH), at two levels each, were used for the factorial design. The variables were coded between "-" and "+", where "-" represents the low level and "+" represents the high level of the factor, Table 19. Such fractional factorial design was repeated with the following frothers: F-507, F-579 and CP-100. Three more experiments were performed at the mid-point (coded as 0) to calculate the experimental error.

Parameters	Code		Levels	
		(-)	(+)	(0)
Collector Dosage, lb/t	А	1.5	3.5	2.5
Frother Concentration, ppm	В	10	30	20
рН	С	8.5	9.5	9.0

Table 19. Levels of the Studied Parameters.

The results of these experiments corresponding to the factorial design are also shown in Tables 20 and 22. Yates' analysis was used to analyze the data.

#	A	В	С	% BPL	% A.I.	Recovery BPL	Distribution A.I.
1	-	-	-	52.73	22.81	52.16	4.67
2	+	-	-	57.67	15.80	73.95	4.43
3	-	+	-	51.57	22.96	57.16	5.56
4	+	+	-	48.67	26.24	94.89	10.28
5	-	-	+	57.38	14.80	68.57	4.32
6	+	-	+	43.44	31.58	98.51	15.17
7	-	+	+	56.21	17.12	84.82	6.52
8	+	+	+	43.81	22.52	84.44	12.12
9	0	0	0	55.67	19.59	92.08	7.77
10	0	0	0	54.18	19.10	92.57	7.22
11	0	0	0	55.05	18.98	91.74	7.85

Table 20. Results of Concentrates of Factorial Design for Flotation of Coarse Feedwith F-507.

Table 21. Results of Concentrates of Factorial Design for Flotation of Coarse Feedwith F-579.

#	Α	В	С	%	%	Recovery	Distribution
				BPL	A.I.	BPL	A.I.
1	-	-	-	57.38	16.04 35.43		2.22
2	+	-	-	54.76	22.24	86.89	7.53
3	-	+	-	55.92	20.85	50.88	4.00
4	+	+	-	55.92	18.25	89.96	7.35
5	-	-	+	55.93	16.65	78.66	5.08
6	+	-	+	45.04	31.61	98.33	13.15
7	-	+	+	61.15	18.49	51.04	3.54
8	+	+	+	45.63	19.72	92.59	12.08
9	0	0	0	54.47	18.98	87.41	6.66
10	0	0	0	55.05	19.18	88.57	6.86
11	0	0	0	56.38	19.85	87.20	7.04

#	Α	В	С	%	%	Recovery	Distribution
				BPL	A.I.	BPL	A.I.
1	-	-	-	64.92	14.20	44.69	2.09
2	+	-	-	57.38	16.41	96.06	5.53
3	-	+	-	61.15	14.47	42.22	2.17
4	+	+	-	62.02	14.95	85.77	4.68
5	-	-	+	58.83	15.94	36.76	1.93
6	+	-	+	68.19	19.99	90.42	6.45
7	-	+	+	57.08	19.38	56.05	4.51
8	+	+	+	68.40	16.71	78.51	4.57
9	0	0	0	57.72	19.95	83.48	5.57
10	0	0	0	57.67	20.22	82.95	5.69
11	0	0	0	56.79	20.42	82.88	6.02

 Table 22. Results of Concentrates of Factorial Design for Flotation of Coarse Feed with CP-100.

The results shown in Tables 20-22 indicate that frother type and operational conditions strongly affect the grade and recovery of flotation concentrate. When frother F-507 is used, phosphate concentrates contain about 54.2-55.7 % BPL and 19.1% acid insoluble with recovery of 91.7-92.6 % are obtained at the mid-point. Under the same operating conditions, concentrates of similar grade (55-57.7% BPL) but of lower recovery (83-88.6 %) are obtained with F579 or CP-100. The recovery can reach to 98.5 % at the expense of grade (43.4 % BPL). Similar grade (45.0% BPL) and recovery (98.33 %) are obtained with F579 as a frother.

The results in Figures 54-56 show the net effect of changing collector dosage from lower to higher levels on grade and recovery of concentrates. The results show that, more or less, a similar trend where the recovery increases with changing collector dosage from lower to higher one. This is, of course, at the expense of grade where % BPL is slightly decreased.



Figure 54. Effect of Collector on Coarse Feed Using CP-100.



Figure 55. Effect of Collector on Coarse Feed Using F-507.

99



Figure 56. Effect of Collector on Coarse Feed Using F-579.

100

Meanwhile, the results of changing pH (Figures 57-59) reveal a very interesting behavior where a higher recovery is obtained at the higher level of pH 9.5 while using either F-507 or F-579 as a frother. However, with CP-100, the reverse trend is true where the higher recovery is obtained at the lower level of pH 8.5.

The interaction between each pair of the studied parameters are studied, i.e., interaction between collector and frother, interaction between collector and pH, and interaction between frother and pH. However, it is found that the most important interaction is that between frother and pH (Figures 60-62). For both frothers F-507 and F-579, the results indicate that the higher pH (9.5), the better the recovery. Meanwhile, the lower concentration (10 ppm) of each of these two frothers at high level of pH is enough to maximize the recovery. However, with CP-100 as a frother, a reverse trend is obtained where the highest recovery is obtained at the lower level of each of pH and frother concentration. This shows the strong interaction between this type of frother (CP-100) and pH where it works better at lower pH. This may be correlated with its nature as an anionic (alkyl ether sulfate) frother. At such lower pH, the ionization of this frother will be less and in turn its efficiency would be better. This has been confirmed with fine feed, in three phase experiments, as well as in two-phase experiments. These results will be shown next.

On the other hand, the contours of recovery between frother concentration and pH are shown in Figures 63-65. Again, these contours show that lower level (10 ppm) of frother is enough to maximize the recovery. Higher pH (9.5) is better for both F-507 and F-579 as frothers while CP-100 works better at lower pH (8.5). These findings are, also, confirmed with flotation of the fine feed samples as will be shown below.



Figure 57. Effect of pH on Coarse Feed Using F-507.



Figure 58. Effect of pH on Coarse Feed Using F-579.



Figure 59. Effect of pH on Coarse Feed Using CP-100.



Figure 60. Effect of Interaction on Coarse Feed Using F-507.



Figure 61. Effect of Interaction on Coarse Feed Using F-579.



Figure 62. Effect of Interaction on Coarse Feed Using CP-100.



Figure 63. Effect of Collector Dosage on Grade and Recovery of Fine Feed with F-507.



Figure 64. Effect of Collector Dosage on Grade and Recovery of Fine Feed with CP-100.



Figure 65. Effect of Frother Concentration (F-507) on Grade and Recovery of Fine Feed.

Fine Feed Phosphate Column Flotation

For the fine size fraction (35 x 150 mesh), experiments were conducted using 2^3 fractional factorial design. Here, three factors (frother type, air flow rate, and frother concentration) at two levels each were used for the factorial design. The variables were coded between "-" and "+", where "-" represents the low level and "+" represents the high level of the factor, however, for the factor of frother type, "-" stands for the frother F-507 and "+" for the frother CP-100. No conditioning water was used in these experiments.

The results of the eight experiments corresponding to the factorial design are also shown in Table 23. Yates' analysis for the BPL and acid insoluble of phosphate concentrate, and BPL recovery are given in Table 24.

Test	А	В	C	D		Concentrate					
					BPL%	Mass Wt. %	Recovery,%	BPL %			
	-	-	-	+	68.4	59.1	97.1	3.0			
2	+	-	-	+	69.8	60.0	99.5	0.6			
3	-	+	-	+	63.0	54.4	92.1	6.5			
4	+	+	-	-	68.8	61.5	98.1	2.1			
5	-	-	+	+	65.7	45.1	87.2	7.9			
6	+	-	+	-	64.6	58.3	96.9	2.9			
7	-	+	+	-	65.4	59.1	94.9	5.1			
8	+	+	+	+	66.4	56.5	97.1	2.6			

 Table 23. Treatment Combination and the Experimental Results of the 2⁴⁻¹ Design for the Fine Phosphate Flotation.

A: Frother type

B: Superficial air velocity

C: Superficial discharge velocity

D: Frother concentration

The results shown in Table 23 indicate that frother type and operational conditions strongly affect the grade of flotation concentrate. When frother F-507 is used, phosphate concentrates contain about 50% acid insoluble and only 34-37% BPL, compared with 22-34% acid insoluble and 46-55% BPL in concentrates for frother CP-100. However, it was found that the frother type has no significant influence on phosphate recovery. The effect of each individual variable on the flotation performance is discussed as follows.

The average changes in BPL and acid insoluble content in the concentrate and recovery for a change of frother type from F-507 to CP-100 are 14.9, -21.1 and -0.3, respectively, as shown in Table 24. It is clear that using CP-100 frother instead of F-507 results in significantly improving concentrate quality, i.e., the BPL content will increase by 14.9% and acid insoluble will reduce by 21.1%. It is also found that frother type has no significant influence in fine phosphate recovery, since the average change of the recovery is negligible (-0.3%).

The effect of superficial air velocity on the recovery is negligible, as indicated by the average change of 0.4% in recovery. However, the average change in BPL and A.I. contents in the concentrate for a change in superficial air velocity from 0.7 to 0.5 cm/s are -1.9 and 3.1, respectively, which shows that higher air flow rate slightly reduces BPL content and increase amount of A.I. in concentrates.

The effect of frother concentration in a range of 25 to 5 ppm on BPL content in the concentrate and recovery may be negligible since the average change are -0.8 and - 0.5. However, an increase of frother concentration from 5 to 25 ppm results in a slightly increase of A.I. content in the concentrate.

Source of variation		Concentrate	2	
parameter	BPL%	A.I.%	BPL Reco. %	A.I. %
Main effects:				
Frother type, A	14.9	-21.1	-0.3	-11.9
Superficial air velocity, B	-1.9	3.1	-0.7	1.2
Frother concentration, C	-0.8	1.7	-0.5	1.4
Two factor interaction:				
AxB	-3.9	6.0	0.2	1.7
AxC	0.3	0.4	0.3	-0.1
BxC	1.7	-2.4	-1.4	-1.8

Table 24. Effect of Different Parameters on Average Change of Fine Phosphate Flotation.

The average values for a change in frother type from CP-100 to F-507 at a superficial air velocity 0.46 to 0.7 cm/s shows that the interaction between frother type and air flow rate is significant with respect to BPL and A.I. contents in the concentrate. The interaction between airflow rate and frother concentration has also significant influence on BPL and A.I. contents in the concentrate, as well as on BPL recovery. It was also found that there is no significant interaction between frother type and frother concentration, which indicates that the interaction between the levels of these two variables do not affect the process.

The order of significance for the effect of variables on BPL and A.I. contents in the concentrate can be determined as follows:

Frother type > airflow rate > frother concentration.

The order of significance for the effect of variables on A.I. recovery in the concentrate is:

Frother type > frother concentration > airflow rate.

The order of significance of interactional effects of the variables on BPL and A.I. contents in the concentrate is:

Frother type and airflow rate (AxB) > airflow rate and frother concentration (BxC) > frother type and frother concentration (AxC).

The order of significance of interactional effects of the variables on BPL and A.I. recovery in the concentrate is:

Airflow rate and frother concentration (BxC) > Frother type and airflow rate (AxB) > frother type and frother concentration (AxC).

Central Composite Design for Parameters Affecting Flotation Chemistry of Fine Feed

Based on the above discussion, it can be seen that parameters such as frother type and its concentration play a very important role in determining the efficiency of column flotation of coarse feed. For this reason, and as we did before with the coarse feed, it has been decided to study further these parameters in addition to collector dosage and pH, i.e. parameters responsible for the chemistry of flotation. The approach used, this time, is a central composite design for the parameters: collector dosage, frother concentration and pH using each frother separately. Each of these parameters is studied at 5 different levels. No conditioning water was used in these experiments. The aim of that is to determine the region of optimum conditions for flotation of fine feed with each frother (F-507 and CP-100).

The central composite design consists of:

- A complete 2³ factorial design, where the levels are coded as -1 and +1 values. This is called the factorial portions of the design.
- n_0 center points where $(n_0 \ge 1)$. In this study, $n_0 = 3$.
- Two axial points on the axis of each design variable at a distance of
 (∝) from the design center. This portion is called the axial portion of
 the design.

Thus, the total number of design experiments is $N=2^k + 2k + n_o = 2^3 + 2(3) + 3 =$ 17. This central composite design (17 experiments) is conducted with each frother type (F-507 and CP-100). A special software called DOE-PC IV (Quality America Inc., USA) is used for analysis of data from these statistically designed experiments. Using this software the effect of the main parameters and their interactions as well as the surface response contours can be determined.

Table 25 shows levels of variables of the experimental central composite design for the three studied parameters: collector dosage (A), frother concentration (B) and pH (C) used with each type of frother. The variables are coded "+1.68", "+1", "0", "-1", and "-1.68". The codes "-1" and "+1" represent the low and high levels respectively. The code "0" represents the mid-point while the codes "-1.68" and "+1.68" are the axial low and high points respectively.

Table 25. Levels of Variables for Central Composite Design of Fine Feed PhosphateSample with Each Frother (F-507 and CP-100).

Variable	Code	Level						
		-1.68	-1	0	+1	+1.68		
Collector Dosage, lb/t	Α	0.396	0.6	0.9	1.2	1.404		
Frother Concentration, ppm	В	6.6	10	15	20	23.4		
pH	C	8.16	8.5	9.0	9.5	9.84		

There are 17 possible combinations according to this statistical central composite design. The results of these experiments corresponding to each type of frother are shown in Tables 26 and 27 for F-507 and CP-100 respectively.

Effect of the Studied Parameters on Column Flotation of Fine Feed

According to the central composite design, five different levels (or points) are calculated for each of the studied variables. The minimum and maximum points of these levels are chosen to cover the range of variation of these parameters that might occur during the flotation of this fine feed (35×150 mesh) in the beneficiation phosphate plants. No conditioning water was used in these experiments. The effects of these variables on grade and BPL recovery are shown below.

#	Α	В	С		Co	oncentra	ite			Tail			
	Collector	Frother	pН	Wt	BPL	A.I.	Dist	%	Wt	BPL	A.I.	D	ist. %
	Dosage	Dosage		%	%	%	BPL	A.I.	%	%	%	BPL	A.I.
1	-1	-1	-1	1.82	43.56	39.63	4.80	0.93	98.18	16.01	78.18	95.20	99.07
2	-1	-1	+1	14.09	62.80	12.44	44.92	2.40	85.91	12.63	82.87	55.08	97.60
3	-1	+1	-1	6.70	53.43	26.56	20.11	2.37	93.30	15.24	78.44	79.89	97.63
4	-1	+1	+1	12.98	61.23	13.45	48.22	2.37	87.02	9.89	82.78	51.78	97.63
5	+1	-1	-1	23.45	55.78	24.63	73.41	7.78	76.55	5.19	89.40	26.59	92.22
6	+1	-1	+1	26.65	52.89	21.76	79.91	7.97	73.35	4.84	91.34	20.09	92.03
7	+1	+1	-1	26.37	52.80	14.91	77.79	5.62	73.63	5.40	89.67	22.21	94.38
8	+1	+1	+1	29.70	50.94	19.69	81.67	8.11	70.30	4.83	94.22	18.33	91.89
9	-1.68	0	0	3.07	45.77	45.01	7.95	1.73	96.73	16.79	80.83	92.05	98.27
10	+1.68	0	0	27.12	58.12	20.92	84.13	7.95	72.88	4.08	90.09	15.87	92.05
11	0	-1.68	0	17.79	58.12	20.78	52.61	4.80	82.21	11.33	89.19	47.39	47.39
12	0	+1.68	0	15.72	61.23	17.01	58.18	3.40	84.28	9.66	90.06	41.82	96.60
13	0	0	-1.68	16.81	59.94	20.33	58.11	4.42	83.19	8.73	88.77	41.89	95.58
14	0	0	+1.68	16.49	56.03	23.78	53.08	5.11	83.51	9.78	87.21	46.92	94.89
15	0	0	0	18.34	58.32	23.19	59.92	5.35	81.66	8.76	92.15	40.08	94.65
16	0	0	0	17.56	58.69	24.23	60.80	5.34	82.44	8.08	91.44	39.20	94.66
17	0	0	0	17.62	58.57	21.20	60.47	4.82	82.38	8.19	89.62	39.53	95.18

 Table 26. Results of Central Composite Design for Column Flotation of Fine Feed Phosphate with F-507.

#	Α	В	С		(Concenti	rate				Tail		
	Collector	Frother	pН	Wt	BPL	A.I.	Recover	ry %	Wt	BPL	A.I.	Dis	t. %
	Dosage	Dosage		%	%	%	BPL	A.I.	%	%	%	BPL	A.I.
1	-1	-1	-1	1.22	43.26	49.36	2.90	0.78	98.78	17.89	77.12	97.10	99.22
2	-1	-1	+1	4.51	62.90	23.97	21.84	1.28	95.49	10.63	87.15	78.16	98.72
3	-1	+1	-1	0.44	19.72	28.68	0.49	0.16	99.56	17.58	78.90	99.51	99.84
4	-1	+1	+1	8.20	70.46	11.37	29.75	1.22	91.80	14.86	82.50	70.25	98.78
5	+1	-1	-1	11.16	64.11	19.74	38.71	2.84	88.84	12.75	84.91	61.29	97.16
6	+1	-1	+1	9.82	66.68	10.38	55.55	1.27	90.18	5.81	87.64	44.45	98.73
7	+1	+1	-1	15.89	66.83	15.70	67.42	3.24	84.11	6.10	88.49	32.58	96.76
8	+1	+1	+1	12.69	65.27	15.62	48.63	2.57	87.31	10.02	86.03	51.37	97.43
9	-1.68	0	0	1.30	48.70	55.47	3.41	0.96	98.70	18.19	75.67	96.59	99.04
10	+1.68	0	0	23.86	62.00	26.36	93.86	8.20	76.14	1.27	92.44	6.14	91.80
11	0	-1.68	0	13.80	60.97	9.27	54.28	1.68	86.20	8.22	86.61	45.72	98.32
12	0	+1.68	0	11.94	68.95	14.52	55.13	2.23	88.06	7.61	86.36	44.87	97.77
13	0	0	-1.68	10.33	67.44	18.36	41.57	2.47	89.67	10.92	83.48	58.43	97.53
14	0	0	+1.68	9.26	66.83	14.75	35.95	1.76	90.74	12.15	84.20	64.05	98.24
15	0	0	0	16.37	68.64	12.60	61.19	2.73	83.63	8.52	87.80	38.81	97.27
16	0	0	0	19.32	67.74	13.87	61.09	3.69	80.68	10.33	86.58	38.91	96.31
17	0	0	0	18.66	65.61	13.88	59.30	3.59	81.34	10.33	85.76	40.70	96.41

 Table 27. Results of Central Composite Design for Column Flotation of Fine Feed Phosphate with CP-100.

Effect of Collector Dosage on Column Flotation of Fine Feed

The dosage of collector mixture (1:1 fatty acids to fuel oil) is varied between ~ 0.4 to 1.4 lb/t (i.e. 0.18 to 0.637 kg/t). Frother concentration and pH are at their midpoints, i.e. 15 ppm of each frother and pH 9.0. Figures 63 and 64 depict the effect of collector dosage on grade and BPL recovery using F-507 and CP-100 respectively. The results show, relatively, a similar behavior with both frothers. The recovery is progressively improved with increasing the collector dosage from ~ 0.4 to 1.4 lb/t. It is expected that the hydrophobicity of phosphate particles will be improved with adding more collector and, in turn, the recovery will increase. Meanwhile, the grade, in the region between 0.4 - 0.9 lb/t collector, is successively improved as indicated by the gradual increase in the % BPL and a continuous reduction in the % A.I. Such grade is decreased after adding more collector specially with using CP-100 as a frother, Fig. 64.

Effect of Frother Concentration on Column Flotation of Fine Feed

The concentration of frothers is varied between 6.6 to 23.4 ppm while collector dosage and pH are kept at their midpoints, i.e. 0.9 lb/t collector and pH 9.0. Figures 65 and 66 show the change in grade and BPL recovery of fine feed phosphate sample with changing concentration of F-507 and CP-100 respectively. The results indicate that the BPL recovery is gradually improved with increasing the concentration of frother from 5 ppm up to 15 ppm at which the highest recovery is obtained. The results also show that the % BPL recovery is almost the same at higher concentration (15-23.4 ppm) of F-507 while a remarkable reduction is noticed with CP-100 at the same range of concentration. On the other hand, Figures 65 and 66 reveal that the grade of concentrates is slightly affected at higher frother concentration. These results show that a concentration of 15 ppm of each frother is enough for flotation of the fine feed phosphate sample. This behavior might explain the results of changing frother concentration shown above in Table 24 for fine feed, where the net effect of increasing concentration of frother from 5 to 25 ppm caused a slight decrease in both grade (%BPL) by -0.8 % and recovery (-0.5 %). This is because the region of optimum condition lies in the middle (at 15 ppm) between the lower and higher limits of that statistical design (5-25 ppm).

Meanwhile, the estimated bubble diameter, using Drift Flux method, indicates that the diameter of bubbles while using F-507 is significantly smaller that with CP-100, Table 28. With F-507, the bubbles diameter decreases from 0.624 mm (at 5 ppm) to 0.585 (at 15 ppm) and it is nearly constant (0.585 - 0.567 mm) at higher concentration. With CP-100, larger bubble diameters (~ 1.032 mm) are generated at lower concentration (6.6 ppm), which slightly decreases to about 0.888 mm at 15 ppm.

Then, the bubble diameter is slightly decreased to 0.754 mm at the highest concentration (23.4 ppm). It is clear that F-507 helps in producing smaller bubbles in comparison to CP-100. This is confirmed in 2-phase experiments shown before.



Figure 66. Effect of pH on Grade and Recovery of Fine Feed with F-507.

Frother concentration	Estimated bubble diameter, mm					
ppm	F-507	CP-100				
6.6	0.624	1.032				
10	0.614	0.916				
15	0.585	0.888				
20	0.579	0.871				
23.4	0.567	0.754				

 Table 28. Average Values for the Estimated Bubble Diameter at Different Frother Concentrations.

Effect of pH on Column Flotation of Fine Feed

The pH is varied in a range between 8.2 to 9.8 while dosage of collector mixture (0.9 lb/t) and frother concentration (15 ppm) is kept at their midlevels. The results are shown in Figures 67 and 68 for F-507 and CP-100 respectively. The results depict that the grade, in general, can be improved at higher pH with both types of frothers. Again, CP-100 produces concentrates of higher grade in comparison with F-507. For example, at pH 9.5 the % BPL is about 61.86 and 55. 14 % for CP-100 and F-507 respectively. At higher pH of 9.8 a better grade is obtained with both frothers. However, the curves of % BPL recovery show different trends with changing pH for both frothers, Figures 67 and 68. With F-507 as a frother, the BPL recovery is successively improved from about 39.21 % to 56.11 % merely by raising pH from 8.2 to 9.8. However, with CP-100 the BPL recovery is significantly increased from 8.1 to 41.95 % at the beginning with increasing pH from 8.2 to 9.0. Above pH 9.0 a dramatic reduction in BPL recovery is noticed with CP-100. It should be noticed that the BPL recovery while using F-507 is always higher at any pH in comparison with CP-100. These results indicate that F-507, as a frother, gives better results at alkaline pH (9.5 to 9.8) while CP-100 is effective at pH 9.0. This is in agreement with the previous results of column flotation of coarse feed.



Figure 67. Effect of pH on Grade and Recovery of Fine Feed with F-507.



Figure 68. Effect of pH on Grade and Recovery of Fine Feed with CP-100.

Interactions Between the Studied Parameters

Figures 69 and 70 depict the effect of interaction between frother concentration and pH on the fitted % BPL recovery for F-507 and CP-100 respectively. The dosage of collector is 0.9 lb/t. These results show a different behavior for each frother. For F-507, there is a successive improvement in BPL recovery with increasing its concentration especially at lower pH values (8.2 -9.0). At higher pH (9.5-9.8) the best recovery is noticed at the mid-point concentration of 15 ppm. These results indicate that the best combination of frother concentration and pH is to add 15 ppm of F-507 at higher pH (~ 9.5 - 9.8). This, again, is in agreement with the previous results. Meanwhile, the best combination of frother concentration of CP-100 and pH is at 15 ppm and pH 9.0 where the highest BPL recovery is obtained. This is, also, in agreement with the previous results.

The interaction between collector dosage and frother concentration is also studied at the mid-point of pH (pH 9.0). The results are shown in Figures 71 and 72 for F-507 and CP-100 respectively. These results depict that, at any concentration level of both frothers, the % BPL recovery increases progressively with increasing collector dosage from ~ 0.4 - 1.40 lb/t (i.e. ~ 0.18 - 0.64 kg/t). However, the recovery obtained at any level of collector and frother with F-507 is always higher than its respective with CP-100. This is in agreement with the results shown before in Figures 63 and 64.

The results in Figures 71 and 72 also reveal that, at a given collector dosage, the value of % BPL recovery depends on the concentration of frother used. With F-507, the change in recovery is small when higher dosage (1.2-1.4 lb/t) of collector is used. The reverse trend is correct with CP-100 as a frother. Moreover, the BPL recovery is found to increase with raising the frother concentration from about 6.6 to 15 ppm where the highest recovery is obtained. These results show that a concentration of 15 ppm of each frother is enough to get the best recovery at any level of collector. It is clear that changing the dosage of F-507 has a little effect on BPL recovery while with CP-100; the BPL recovery becomes a function of its concentration and the quantity of collector added. Higher dosage of collector together with higher concentration of CP-100 has a negative effect of the BPL recovery. This might give an indication for the interaction between the collector and CP-100, which affect adversely the BPL recovery. Similar behavior had been noticed with column flotation of the belt feed phosphate sample as well as with the statistical design for two-phase experiments, the results of which are shown next.



Figure 69. Effect of Interaction Between F-507 and pH on BPL Recovery at Mid-Point of Collector Dosage (0.9 Kg/T).



Figure 70. Effect of Interaction Between CP-100 and pH on BPL Recovery at Mid-Point of Collector Dosage (0.9 Kg/T).



Figure 71. Effect of Interaction Between Collector Dosage and Concentration of F-507 on BPL Recovery at Mid-Point of pH (9.0).



Figure 72. Effect of Interaction Between Collector Dosage and Concentration of CP-100 on BPL Recovery at Mid-Point of pH (9.0).

Surface Response Contours for Fine Feed

Response surface contours are generated for % BPL grade and recovery as a function of the studied variables (i.e. collector dosage, frother concentration and pH) for each of F-507 and CP-100. Figures 73 and 74 depict the contours of BPL recovery between pH and concentration of both frothers. All contours are drawn at the mid-point of collector dosage (0.9 lb/t). These contours indicate that the area of highest recovery is attained within the present levels of pH and frother concentration. With F-507, this region is expanded along the whole range of concentrations (i.e., 6.6-23.4 ppm) and the higher pH, the better recovery. With CP-100, the area of highest recovery is between ~ 13-18 ppm of this frother and at pH around 9.0. This is in agreement with the previous results.

The contours of BPL recovery between collector dosage and concentration of both frothers are, also, analyzed. The results are shown in Figures 75 and 76 for F-507 and CP-100 respectively. All contours are drawn at the mid-point of pH (i.e., at pH 9.0). These contours suggest that the area of the highest recovery is not attained within the applied levels (~0.4-1.4 lb/t) of collector. This means that more collector is needed to maximize the recovery.

Meanwhile, the contours of grade (% BPL) for the collector dosage with pH for both F-507 and CP-100 show a similar trend where two areas for maximum grade are appeared at the same pH and dosage of collector, Figures 77 and 78 respectively. The first area of highest grade is at higher pH (~9.8) with low collector dosage while the second at higher collector dosage (above 1.1 lb/t) but at lower pH. This similarity in the general trend of these contours with both frothers might indicate that the area of highest grade is not related to the frother type rather than the chemistry of fatty acids, as collector, with changing pH. It is expected that at lower collector dosage, the phosphate particles of high hydrophobicity (i.e. of high grade) will be floated. Meanwhile, raising pH above ~ 8.6 is accompanied with changing the fatty acid from the form (R-COO⁻) to (R-COO)₂²⁻. But at lower pH, fatty acid will be changed from NaH (RCOO)₂ to R-COOH ^{and} in turn the recovery will be low. This produces concentrates of high grade at the expense of recovery.



Figure 73. Contour of BPL Recovery for Concentration of F-507 and pH at Mid-Point of Collector Dosage (0.9 Lb/T).



Figure 74. Contour of BPL Recovery for Concentration of CP-100 and pH at Mid-Point of Collector Dosage (0.9 Lb/T).


Figure 75. Contour of RPI Recovery for Collector Dosage and Concentration of F-507 at Mid-Point of pH (9.0).



Figure 76. Contour of BPL Recovery for Collector Dosage and Concentration of CP-100 at Mid-Point of pH (9.0).



Figure 77. Contour of % BPL for Collector Dosage and pH Using F-507 (15 ppm).



Figure 78. Contour of % BPL for Collector Dosage and pH Using CP-100 (15 ppm).

Column Flotation of Belt Feed Phosphate

For the belt feed phosphate flotation, parameters such as frother type, frother dosage, collector dosage, the ratio of oleic acid to fuel oil, and conditioning water were investigated. The conditioning water is the decanted water from the feed after conditioning with reagents. In some tests it is added at a specified rate with the feed. In other tests, it is substituted with tap water (i.e. no conditioning water is added). A set of experiments were also conducted using a fractional factorial design to study the main effects and interactions between frother type, frother concentration, collector concentration and conditioning water

Effect of Frother Type

The effect of frother type (F-507 vs. CP-100) experiments were performed at a superficial air velocity of 0.7 cm/s and using about 4.41 lb/ton of 1:1 mixture of fatty acid and fuel oil, Table 29. The collector dosage was chosen based on discussions with personnel from the phosphate industry. No conditioning water was used in these experiments. Instead tap water was added to the feed. Under these experimental conditions, it was found that the frother CP-100 showed superior results, in terms of grade and recovery, in comparison with that of F-507. For example, at a frother dosage of 15 ppm, a concentrate of 67.5 % BPL and 7.8 % A.I. with a BPL recovery of 99.5 % can be obtained with the CP-100 in comparison with that obtained using F-507 (63.6 % BPL, 13.00 % A.I. and BPL recovery of 99.4 %). Similar results were obtained at different frother concentrations.

It seems that, regardless of the concentration of the frother, the concentrates obtained with CP-100 have, in general, higher grade and BPL recovery in comparison with F-507. These results are, also, similar to that obtained in the column flotation of the unsized, coarse and fine phosphate samples. This could be attributed to the fact that F-507 has strong air dispersion ability, and in turn generates smaller bubbles in comparison with CP-100. It has been observed, by naked eye, that such fine bubbles form aggregates with solid particles including silica. Such air / solid aggregates become buoyant and float with phosphate particles leading to lower grade concentrates. It should be noted that the recovery does not get affected much, which may confirm the aggregation / entrapment observation discussed above.

Froth	er		Conce	entrate		Tail			
Туре	ppm	BPL%	A.I.%	Rec.% BPL	Dist% A.I.	BPL%	A.I.%	Rec.% BPL	Dist.% A.I.
CP-100	15	67.54	7.79	99.46	8.97	0.46	98.86	0.54	91.03
F-507	15	63.58	13.00	99.43	10.7	0.33	99.08	0.57	89.30
CP-100	25	66.42	8.53	99.71	8.25	0.20	99.29	0.30	91.75
F-507	25	64.58	12.00	99.34	9.12	0.39	95.96	0.66	90.88
CP-100	50	67.60	6.90	99.24	6.97	0.55	98.63	0.76	93.03
F-507	50	64.57	11.55	99.03	9.22	0.55	98.67	0.97	90.78

 Table 29. Effect of Frother Type on Belt Feed Phosphate Column Flotation.

Effect of Frother Dosage

The effect of using different concentrations of CP-100 or F-507 on belt feed phosphate flotation is given in Table 30. The experiments were performed at a superficial air velocity of 0.7 cm/s and using about 4.41 lb/ton of 1:1 mixture of fatty acid and fuel oil. No conditioning water was used in these experiments. It can be seen that when concentration of frothers varies in a range of 15 to 50 ppm, there was no significant influence on the flotation performance where both the BPL% and its recovery are almost constant. Again, these results are similar to that of the unsized phosphate flotation mentioned earlier. The absence of any effect of frother concentration may be attributed to a masking effect produced by the high dosage of collector, which has frothing characteristics. This also has been confirmed by conducting further testing using even higher dosage of collector as discussed below.

Frother			Conc	entrate		Tail			
Туре	ppm	BPL%	A.I.%	Rec.% BPL	Dist% A.I	BPL%	A.I. %	Dist.% A.I.	Dist% BPL
CP-100	15	67.54	7.79	99.46	8.97	0.46	98.86	91.03	0.54
	25	66.42	8.53	99.71	8.25	0.20	99.29	91.75	0.29
	30	67.12	7.45	99.65	12.79	0.46	98.86	87.21	0.35
	50	67.60	6.90	99.24	6.97	0.55	98.63	93.03	0.76
F-507	15	63.58	13.00	99.46	10.70	0.33	99.08	89.30	0.54
	25	64.58	12.00	99.34	9.12	0.39	95.96	90.88	0.66
	50	64.57	11.55	99.03	9.22	0.55	98.67	90.78	0.97

 Table 30. Effect of Frother Concentration on Belt Feed Phosphate Column Flotation.

Effect of Collector Dosage and Its Fatty-Acid-to-Fuel-Oil Ratio

The effect of collector dosage, and its fatty acid:fuel oil ratio, on belt feed phosphate column flotation was investigated over a relatively wide range (total dosage 4.4 - 8.8 lb/ton). Such extremely high dosage of collector, in comparison with that actually used in industry, was performed to study the influence of collector on the performance of the flotation column and its possible interaction with the type of frother applied. This is because it is well known that fatty acids, as collectors, have self-frothing power in addition to their collecting action. So, their possible influence or interaction with the type of frother applied, at such extremely high dosage, might be determined. These experiments were performed at a superficial air velocity of 0.7 cm/s and using different concentration of frothers (15 - 25 ppm of CP-100 or F-507), the results of which are shown in Table 31. It should also be emphasized here that no conditioning water was used in these tests.

Collector, lb/t		Frother (ppm)	Concentrate				Tail				
Fatty acid	Fuel oil	Total		BPL%	A.I .%	Rec. BPL%	Dist % A.I.	BPL %	A.I .%	Dist. % BPL	Dist.% A.I.
2.2	2.2	4.4	CP-100 15 ppm	67.54	7.79	99.46	8.97	0.46	98.86	0.54	91.03
4.4	4.4	8.8		66.75	7.97	99.66	10.98	0.35	99.04	0.34	89.02
2.2	2.2	4.4	CP-100 25 ppm	66.42	8.53	99.71	8.25	0.20	99.29	0.29	91.75
4.4	4.4	8.8		67.95	6.47	99.35	10.46	0.79	98.30	0.65	89.54
2.2	2.2	4.4	F-507 15 ppm	63.58	13.00	99.43	10.70	0.33	99.08	0.57	89.30
4.4	4.4	8.8		65.22	10.33	99.00	16.11	1.20	97.72	1.00	83.89
2.2	2.2	4.4	F-507 25 pm	64.58	12.00	99.34	9.12	0.39	95.96	0.66	90.88
4.4	4.4	8.8		66.88	7.73	98.91	11.04	1.14	96.10	1.09	88.96

 Table 31. Effect of Collector Dosage and Its Fatty-Acid-to-Fuel-Oil Ratio on Belt Feed Phosphate Column Flotation.

The results in Table 31 indicate that increasing the collector (mixture of 1:1 fatty acid and fuel oil) dosage from 4.4 to 8.8 lb/t had nearly no significant influence on the BPL recovery which was reached to about 99.7 % regardless the type and concentration of frother applied. However, a better grade, in terms of BPL and A.I. contents, could be obtained when higher dosage is applied, especially with F-507. Similar data were obtained with the column flotation of the unsized phosphate. Also, these results indicate that a dosage of 4.4 lb/t of 1:1 fatty acid: fuel oil as a collector is enough to recover over 99% of BPL.

Effect of frother concentration at lower collector dosages is an important point, which has been raised by the steering committee. Therefore, testing these frothers at different conditions including collector dosage is discussed below.

Another important point was raised by members of the steering committee is the difference in the performance of CP-100, and F 507 frothers as obtained in this study in comparison to the results obtained by industry. Further examination of the experimental procedure indicated that the use of the screw feeder necessitated decanting some of the conditioning water. Such water was substituted by tap water in the previous tests. It should be noted that flotation feed to industrial columns includes the conditioning water. Such water may contain residual collector, which may influence frother performance. Therefore, it was decided to investigate the role of the conditioning water.

Effect of Conditioning Water

In order to study the influence of conditioning water on the performance of flotation column, a series of experiments was performed using conditioning water. The results of flotation tests of belt feed in presence and absence of conditioning water are given in Tables 32 and 33. The flow rate of both types of water was fixed at 1.0 L/minute. The experiments were conducted using different dosages of collector (1:1 fatty acid: fuel oil) and frothers (CP-100 vs. F-507). It should be noted that the results in this case represent data before the steady state has been attained after conditioning water addition. Later in this report, it will be shown that the steady state has never been reached in case of CP-100/ conditioning water supply. As a matter of fact, recovery keeps declining as a function of time. At longer times, flotation is nil. Thus, the data presented in this section should be taken to indicate the trend of conditioning water effect.

The results in Table 32 show that the presence of such conditioning water while using CP-100 as a frother had a negative influence on the efficiency of the flotation column. It dramatically reduced the % BPL recovery irrespective of the dosage of each of collector and frother used although a little improvement in the grade was noticed. For example, at a dosage of collector of 4.4 lb/t and in presence of 15 mg/L of CP-100, the BPL recovery was decreased from 99.5% (in absence of conditioning water) to 87.5% although the BPL (~ 67.5%) was almost the same. This reduction in BPL recovery was obtained as a result of the significant decrease in the weight % floated (~ 38.2%) in comparison with that in the absence of conditioning water (55.6%). Increasing the

dosage of collector to 8.8 lb/t and that of CP-100 to 25 ppm gave similar trend although the difference in BPL recovery was slightly improved to become about 92.7 % as compared to 99.4 % in absence of conditioning water. Further addition of CP-100 to 75 ppm, in an attempt to compensate the negative effect of this conditioning water, did not improve the BPL recovery.

Meanwhile, the results in Table 32 indicate that such decrease in % BPL recovery in presence of conditioning water was, also, accompanied with a significant reduction in holdup %, of the 3-phase, especially at higher dosages of collector (8.8 lb/t) and frother (50 - 75 ppm). The % holdup was reduced under these experimental conditions by about 3.0 - 3.3%.

DOS	DOSAGE		Cond. Water		Concentrate				Tail			
Collector lb/t	Frother ppm	_ phase	Yes/ No	BPL %	A.I. %	Rec. BPL%	Dist % A.I.	BPL%	A.I. %	Dist.% A.I.	Dist.% BPL	
4.4	15	9.58	No	67.54	7.79	99.46	8.97	0.46	98.86	91.03	0.54	
		7.25	Yes	67.49	6.73	87.52	5.04	5.94	78.30	94.96	12.48	
8.8	25	11.82	No	67.95	6.47	99.38	10.46	0.79	98.30	89.54	0.62	
		10.68	Yes	68.06	5.71	92.71	5.56	4.92	89.19	94.44	7.29	
8.8	50	14.18	No	67.87	6.35	99.60	9.90	0.47	98.79	90.10	0.40	
		11.20	Yes	68.44	5.73	93.57	7.07	5.69	91.11	92.93	6.43	
8.8	75	14.79	No	67.83	6.30	99.62	9.36	0.42	98.63	90.64	0.38	
		11.51	Yes	68.50	5.70	92.68	6.42	5.92	90.90	93.58	7.32	

 Table 32. Effect of Conditioning Water on the Belt Feed Phosphate Using CP-100.

On the other hand, the results of conducting the same experiments while using F-507 as a frother, Table 33 depicted that application of conditioning water had a little effect on the performance of the column flotation in comparison with CP-100. The BPL recovery decreased slightly (2.2 - 2.8 %) irrespective of collector dosage (4.4 - 8.8 lb/t) or the frother concentration (15 - 25 ppm). Also, a minor reduction in the % air holdup, for the 3-phase, (0.13 - 0.8 %) was noticed while applying conditioning water instead of tap water. It should be noted that the values of % holdup, obtained while using F-507, are significantly higher than that recorded in presence of CP-100 (Tables 32 and 33).

The above-mentioned results indicate clearly that the presence of conditioning water deteriorates the performance of phosphate column flotation in presence of CP-100 as a frother while it has a minor effect while using F-507. This could be attributed to an interaction between the CP-100 as a frother and the residual concentration of collector (fatty acid and fuel oil) present in such conditioning water. It is well known that fatty acids have frothing power in addition to their collecting action. Some authors have mentioned that slimes associated with water adversely affect the performance of carbonate - silicate column flotation using commercial fatty acid by enhancing coalescence and froth collapse (Gomez and others 1988). However, this could be responsible partially for this phenomena because if this is the case in the present study, the deterioration in the phosphate column flotation should occur, to the same degree, while using any fatty acid - frother system and not only with application of CP-100.

Meanwhile, the % solids of generated slimes in conditioning water is very small (~ 0.4 - 0.45 wt % solids). Also, the size analysis of such slimes of conditioning water, using Coulter laser LS particle size analyzer, indicates that they have about 50 wt % below 1.5-1.7 um in size with a mean of 2.3 - 3.8 um, as seen in Figure 79. It seems that the effect of such very small amount of slimes (0.4 - 0.45 wt %) in conditioning water on the deterioration of flotation performance may not be significant. This may suggest that the interaction of the residual concentration of fatty acid with CP-100 could be related to the nature of chemical structure of the latter as an ionic compound (sodium alkyl ether sulfate) in comparison with the non ionic nature of F-507 (mixed polyglycol). It is expected that the residual concentration of fatty acid (or its mixture with fuel oil) in conditioning water would be significant at higher dosage of collector and consequently a severe deterioration in the phosphate column flotation would be noticed as shown in the above results (Table 32).

In order to understand the mechanisms responsible for the effect of collector on frother performance, a further study involving 2-phase and 3-phase systems containing the collector mixture with CP-100 and F-507 has been conducted as discussed below.

DOSAGE		Hold up % 3-phase	Cond. Water		Conc	entrate			Tail			
Collector lb/t	Frother ppm		Yes/ No	BPL %	A.I. %	Rec. BPL%	Dist % A.I.	BPL %	A.I. %	Dist.% BPL	Dist.% A.I	
4.4	15	17.97	No	63.58	13.00	99.43	10.70	0.33	99.30	0.57	89.30	
		17.84	Yes	65.37	9.97	96.69	4.92	1.14	97.95	3.31	95.08	
8.8	15	16.25	No	65.22	10.33	99.00	16.11	1.20	97.72	1.00	83.89	
		15.73	Yes	67.75	7.53	96.75	10.72	2.69	95.29	3.25	89.28	
8.8	25	17.36	No	66.88	7.73	99.04	12.36	1.14	96.10	0.96	87.64	
		16.56	Yes	66.72	7.54	96.89	10.76	3.25	94.80	3.11	89.24	

Table 33. Effect of Conditioning Water on the Belt Feed Phosphate Using F-507.

COULTER	L	S Particle Size Analyzer	
FILE NAME:	FLOTATION	GROUP ID:	
Sample ID:	110		
Operator:	Rajesh	Run number:	1
Comments:	Conditioning Water		
Optical model	: Fraunhofer PIDS included		
LS 230	Small Volume Module		
Start time:	16.22 5 Jan 1998	Run length:	90 Seconds
Obscuration:	4%		
PIDS Obscure	: 45%		



Calculations f	rom 0.04	40 μm to 2000 μm			
Volume		100.0 %			
Mean		3.787 μm	S.D.	5.15 μm	
Median		1.652 μm			
Mode		0.999 µm			
% <	10	25	50	75	90
Size µm	0.537	0.845	1.652	4.499	9.765

Figure 79. Size Analysis of Slimes Present in Conditioning Water.

Flotation Kinetics of Belt Feed

Several experiments were performed to study the kinetics of column flotation process for the belt feed phosphate sample in presence of conditioning water while using each of CP-100 and F-507. The experiments were conducted using a fixed dosage of collector mixture (1:1 fatty acid: fuel oil) of about 8.8 lb/t and in presence of different concentrations of frother. The results are shown in Figures 80- 81.

These results indicate that the column flotation process is nearly stable (in terms of grade and recovery) while using tap water, and with any frother, irrespective the sampling time of the froth (concentrates) and tails. However, once conditioning water is applied to the column, instead of tap water, and in presence of CP-100, the efficiency (in terms of weight % floated) deteriorates depending on the concentration of frother applied. For example, at lower concentration of CP-100 (10 - 25 ppm) flotation continues for 8.0 minutes, after which a complete deterioration occurs and all the feed sample reports to the tailings. This is also accompanied with a gradual decrease in the % holdup of the 3-phase. Increasing the concentration of CP-100 to 50 ppm increases the flotation time to about 10 minutes. Addition of more CP-100 to about 75 ppm increases further the total flotation time to about 19 minutes (Figure 80).

However, this phenomenon is not noticed while using F-507 as a frother (Figure 81) although the concentration of the latter was significantly lower (7.5 - 25 ppm) than that of CP-100. The stability of the column flotation is, nearly, constant and the total time of the experiment varies between 17.5 - 19 minutes depending on the weight of the flotation feed sample (Figure 81).

These results, again, demonstrate that there is some kind of interaction between CP-100 and the residual concentration of fatty acid (or its mixture with fuel oil) present in conditioning water. Such interaction prevents the role of CP-100, as a frother, from controlling the bubble diameter as indicated by the gradual decrease in the % holdup and consequently deteriorates the efficiency of the column flotation process.



Figure 80. Performance of Column Flotation as a Function of Sampling Time in Presence of Conditioning Water and Using CP-100.



Time of sampling after initial stability of column, min

Figure 81. Performance of Column Flotation as a Function of Sampling Time in Presence of Conditioning Water and F-507.

Factorial Design for Studying the Role of Conditioning Water in 3-Phase Experiments

In order to determine the effects and interactions between frother concentration, dosage of collector mixture and the conditioning water in a 3-phase flotation system for the belt feed phosphate using each type of frother (CP-100 and F-507), a series of experiments using 2^3 factorial design has been performed. For each frother type, three factors (frother concentration, collector dosage and conditioning water), at two levels each, are used for the factorial design. 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 34.

Variable	Code	L	evel
		(-)	(+)
Frother Concentration, ppm	А	15	25
Collector Dosage, lb/ton	В	4.4	8.8
Conditioning Water, (No/Yes)	С	No	Yes

Table 34. Levels of Variables for Belt Feed Phosphate Column Flotation with EachType of Frother (CP-100 & F-507).

There are eight possible combinations. The results of these experiments corresponding to the factorial design using each type of frother are shown in Tables 35 and 36 for CP-100 and F-507 respectively. The calculation of the effects and their interactions as well as analysis of variance has been carried out according to Yates' algorithm. The calculated effects are depicted in Tables 37 and 38 for CP-100 and F-507 respectively. It should be noted that the results of CP-100 represent data before the steady state has been attained after conditioning water addition. As shown before, the steady state has never been reached in case of CP-100/ conditioning water supply. As a matter of fact, recovery keeps declining as a function of time. At longer times, flotation is nil. Thus, the data presented in this section with CP-100 should be taken to indicate the trend of conditioning water effect.

The results shown in Table 35 indicate that conditioning water, frother concentration as well as the dosage of collector, affect belt phosphate column flotation. For example, it is possible to achieve a 99 % recovery with a reasonable good concentrate quality of BPL% up to 68 % and of lower A.I. (5.7 - 5.9 %) content depending on the experimental conditions and type of frother applied. The effect of each individual variable, as well as interactions between them, on the column flotation performance for each frother type are given in Tables 37 and 38.

#	А	В	С	BPL %	A.I. %	Rec. % BPL	Dist. % A.I.
1	-	-	-	67.54	7.79	99.46	8.97
2	+	-	-	66.42	8.53	99.71	8.25
3	-	+	-	66.75	7.94	99.66	10.98
4	+	+	-	67.95	6.47	99.35	10.46
5	-	-	+	67.49	6.73	89.67	6.16
6	+	-	+	67.72	6.35	90.32	4.80
7	-	+	+	68.44	5.75	93.57	7.07
8	+	+	+	68.06	5.71	84.67	5.56

Table 35. Treatment Combination and the Results of the Factorial Design Using
CP-100 Frother.

Table 36. Treatment Combination and the Results of the Factorial Design UsingF-507 Frother.

#	А	В	С	BPL %	A.I. %	Rec. % BPL	Dist. % A.I.
1	-	-	-	63.58	13.00	99.43	10.70
2	+	I	-	64.58	12.00	99.34	9.12
3	-	+	-	65.22	10.33	98.48	11.19
4	+	+	-	66.88	7.73	98.91	11.04
5	-	I	+	65.37	9.97	96.69	16.67
6	+	-	+	66.10	8.53	95.70	12.64
7	-	+	+	67.75	7.53	95.18	9.16
8	+	+	+	66.72	7.54	96.89	10.76

Considering the three-factor interaction effect is a manifestation of the experimental error, the results in Tables 37 and 38 indicate that the net effect of each of the studied parameters varies with the type of frother applied. For example, increasing the frother concentration of CP-100 from 15 to 25 ppm has a negligible effect on both recovery and concentrate grade (Table 37). Considering the experimental error, the same argument can be said on the effect of F-507 concentration (Table 38).

Effect & Interactio	n	BPL %	A.I. %	Recovery BPL %
Frother Concentration (A)		-0.018	-0.288	-2.078
Collector Dosage	(B)	+0.508	-1.883	+0.478
Conditioning Water	(C)	+0.763	-1.548	-9.988
(A) x (B)		+0.428	-0.468	-2.528
(A) x (C)		-0.058	+0.078	-2.048
(B) x (C)		+0.138	+0.073	-0.398
(A) x (B) x (C)		-0.733	+0.638	-2.248

 Table 37. Effects and Interactions of Different Parameters on Belt Feed Phosphate

 Flotation Using CP-100.

Table 38.	Effects and Interactions of Different Parameters on Belt Feed Phosphate
	Flotation Using F-507.

Effect & Interacti	on	BPL %	A.I. %	Recovery BPL %
Frother Concentration (A)		+0.590	-1.258	+0.265
Collector Dosage	(B)	+1.735	-2.593	-0.425
Conditioning Water	(C)	+1.420	-2.373	-2.925
(A) x (B)		-0.275	-0.038	+0.805
(A) x (C)		-0.740	+0.543	+0.095
(B) x (C)		-0.235	+0.878	+0.265
(A) x (B) x (C)		-0.605	+0.763	+0.545

Also, the results in Table 37 indicate that increasing collector dosage from 4.4 to 8.8 lb/t does not affect flotation performance to a significant degree in the presence of CP-100 as frother. With F-507, however, an improvement in the grade (+1.74 in BPL and -2.59 in A.I.) is noticed as shown in Table 38. Once more, it should be emphasized that high dosage of collector is only used to determine the role of collector and its presence in conditioning water on the performance of the column. Lower dosage of collector, comparable with that used in industry, will be used, next, with coarse and fine feed phosphates.

Most interesting is the effect of conditioning water as obtained in this series of tests, which shows the same trends as obtained in the previous tests as discussed earlier in this report. For instance, using CP-100 as a frother, (Table 37), under the present experimental conditions, the application of conditioning water has the most negative influence on the performance of the column. The average reduction in BPL recovery was about -9.99, although a minor improvement in the grade was obtained. However, in presence of F-507 as a frother, a small reduction (-2.93) in BPL recovery was noticed with an improvement in the grade (Table 38).

This clearly indicates that, the effect of such conditioning water, while using F-507, is not significant. However, in presence of CP-100 as a frother, the application of conditioning water, which contains a residual concentration of collector (fatty acid and fuel oil), causes deterioration for the column efficiency, and not its selectivity, probably by interfering with the role of frother. At such conditions, it is expected that only particles with sufficient hydrophobicity will float as concentrates. This may explain the slight improvement in the grade of the obtained concentrates while using such conditioning water.

Generally, from the absolute values of the average change for different variables listed in Tables 37 and 38, the order of significance for the effect of variables on BPL recovery and content in the concentrate while using CP-100 may be stated as follows:

Conditioning water > collector dosage > frother concentration

For F-507 such effects on BPL recovery and concentrate content can be arranged as follows:

Conditioning water = Collector dosage > frother concentration

This shows that BPL recovery is mainly affected by conditioning water and frother type. Application of conditioning water and replacing F-507 by CP-100 as a frother have the most negative influence on reducing the BPL recovery and deterioration of the efficiency of the column flotation.

These results clearly show that for column flotation of each specific mineral commodity, the applied collector - frother system should be studied carefully to prevent any possibility of their interactions. This phenomenon has been studied further in 2-phase experiments as shown below.

Factorial Design for Studying the Role of Conditioning Water in 2-Phase Experiments

In order to determine effects and interactions between frother type and its concentration, pH of the medium and the residual concentration of the collector mixture present in the conditioning water, a series of experiments using 2^3 factorial design is performed. Three factors (frother concentration, pH, and concentration of the collector mixture present in water) for each frother type (CP-100 vs. F-507) at two levels each are used for the factorial design. 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 39.

Variable	Code	Level		
		(-)	(+)	
pH of the medium	А	8.0	9.5	
Collector Dosage, mg/L	В	4.0	10.0	
Frother Concentration, mg/L	С	5.0	20.0	

Table 39. Levels of Variables for Two-Phase Design for Each Frother Type.

There are eight possible combinations, as given in Tables 40-41. Each of these eight experiments is conducted at different superficial air velocity (0.24 to 0.94 cm/s). The results of these experiments corresponding to the factorial design are shown in Tables 40 and 41 for CP-100 and F-507 respectively.

#	A	В	С	%	% Holdup at Different Vg				ble Diam Differ	eter in m ent Vg	m, at
				0.24	0.46	0.70	0.94	0.24	0.46	0.70	0.94
1	-	-	-	1.974	2.456	3.347	4.033	1.136	1.839	2.108	2.408
2	+	-	-	1.841	2.406	3.260	3.954	1.223	1.883	2.172	2.463
3	-	+	-	2.177	2.823	3.618	4.209	1.028	1.579	1.939	2.296
4	+	+	-	1.936	2.512	3.388	4.051	1.159	1.794	2.080	2.396
5	-	-	+	2.259	2.973	4.030	5.382	0.991	1.493	1.719	1.757
6	+	-	+	2.316	2.826	4.078	5.184	0.967	1.577	1.697	1.828
7	-	+	+	2.279	2.818	3.936	4.786	0.982	1.581	1.763	1.992
8	+	+	+	2.205	2.716	3.762	4.585	1.016	1.647	1.853	2.089

Table 40. Results of the Experimental Design of the Two-Phase Using CP-100.

Table 41. Results of the Experimental Design of the Two-Phase Using F-507.

#	A	В	C	%	% Holdup at Different Vg				ble Diam Differ	neter in mr rent Vg	n, at
				0.24	0/46	0.70	0.94	0.24	0.46	0.70	0.94
1	-	-	-	4.849	7.183	9.477	13.078	0.507	0.652	0.766	0.788
2	+	-	-	5.203	6.696	10.85	13.041	0.480	0.699	0.692	0.789
3	-	+	-	4.851	7.123	10.717	13.533	0.505	0.656	0.698	0.769
4	+	+	-	4.911	7.004	10.001	13.670	0.501	0.665	0.735	0.763
5	-	-	+	7.779	9.332	13.812	18.760	0.370	0.539	0.0589	0.632
6	+	-	+	7.801	12.574	17.676	21.926	0.369	0.450	0.518	0.591
7	-	+	+	7.618	10.297	14.212	17.309	0.374	0.506	0.580	0.659
8	+	+	+	7.754	11.825	18.308	23.699	0.370	0.465	0.511	0.576

Comparison of the data in Tables 40 and 41 clearly shows that the values of the % holdup obtained with F-507 are significantly higher than that obtained using CP-100 under the same experimental conditions. Also, such % holdup is found to increase with raising the superficial air velocity (Vg) from 0.24 to 0.94 cm/s. These results are reflected on the values of the estimated bubble size, which is significantly (2 - 3 times) smaller in presence of F-507 than that in presence of CP-100. Bubble diameters obtained with F-507 vary between 0.37 to 0.789 mm in comparison to 0.967 to 2.463 mm diameter bubbles obtained with CP- 100.

The calculation of the effects of different parameters (pH, concentration of collector mixture in water and frother type) and their interactions as well as analysis of variance are carried out according to Yates' method, the results of which are depicted in Tables 41 and 42 for CP-100 and F-507 respectively.

It is interesting to note the results in Tables 42 and 43 show a completely different behavior for the main effects, of the studied parameters, and their interactions depending on the type of used frother. The data can be explained as follows (considering the three-factor interaction $X_1X_2X_3$ is a manifestation of the experimental error).

- 1. Changing the pH of the medium from natural (~ 8) to that used in phosphate column flotation (~9.5) has a negative effect on the % holdup while using CP-100 as a frother. This reduction in % holdup is small (- 0.098) at low superficial air velocity (0.24 cm/s) and then it becomes more significant (-0.16) with raising the latter to 0.94 cm/s. However, with the frother F-507 the change in pH increases the % holdup, which is found, as expected, to be a function of the applied superficial air velocity. In other words, CP-100 works better at lower pH. The reverse trend is noticed with F-507.
- 2. The effect of presence of minor concentration of collector (1:1 fatty acid and fuel oil) in the water of the column on % holdup is not significant at lower rate of superficial air velocity (0.24 to 0.46 cm/s).
- 3. The effect of increasing frother concentration indicates that both CP-100 and F-507 have positive effect on the values of % holdup. However, the effect of F-507 on % holdup (+2.79 to + 7.1) is significantly higher in comparison with CP-100 (+0.28 to +0.92). The higher values of % holdup, the lower the bubble diameter produced as shown before in Tables 40 and 41.

On the other hand, studying the interaction between the studied parameters suggests the following:

4. There is an interaction between CP-100 and the collector (X_2X_3) in Table 42). This interaction had a negative effect on reducing the % holdup, and consequently, increasing the bubble diameter. Such interaction is, also, found to be more significant at higher superficial air velocity. On the other hand, such interaction between frother and collector is not significant with F-507 (X_1X_3) in Table 43). This clearly indicates that there is a mutual interaction between CP-100 and the residual concentration of collector mixture in the water of the column. Such interaction prevents the role of frother (CP-100) from controlling the bubble diameter and consequently it deteriorates the superior results obtained with CP-100 in absence of conditioning water.

Table 42.Main and Interaction Effects of Different Parameters on % Holdup
at Various Vg Values in Presence of CP-100.

Vg cm/s	X ₁ (A) pH	X ₂ (B) collector	X ₃ (C) CP-100	X ₁ X ₂	X ₁ X ₃	X ₂ X ₃	X ₁ X ₂ X ₃
0.24	-0.098	+0.052	+0.283	-0.060	+0.089	-0.097	-0.006
0.46	-0.153	+0.052	+0.284	-0.054	+0.028	-0.185	+0.077
0.70	-0.111	-0.028	+0.548	-0.091	+0.048	-0.202	-0.020
0.94	-0.159	+0.231	+0.923	-0.021	-0.041	-0.367	+0.019

Table 43. Main and Interaction Effects of Different Parameters on % Holdupat Various Vg Values in Presence of F-507.

Vg cm/s	X ₁ (A) pH	X ₂ (B) collector	X ₃ (C) F-507	X_1X_2	X ₁ X ₃	X ₂ X ₃	$X_1 X_2 X_3$
0.24	+0.143	-0.125	+2.785	-0.045	-0.064	+0.021	+0.102
0.46	+1.109	+0.048	+3.937	-0.405	+1.276	+0.061	-0.452
0.70	+2.154	+0.356	+5.741	-0.464	+1.826	+0.160	+0.582
0.94	+2.414	+0.352	+7.093	+0.850	+2.364	-0.191	+0.763

MODELING

BACKGROUND

Models can be divided into white-box, black-box, and gray-box (hybrid) depending on the amount of prior knowledge used for developing the model. White-box or first-principles modeling strategies are mainly knowledge driven. Black-box modeling strategies are mainly data driven and the resulting models often do not have reliable extrapolation properties. Black-box strategies have been applied to many chemical processes; especially since convenient black-box modeling tools like artificial neural networks have become available (Hornik and others 1989; Bhat and McAvoy 1990; Psichogios and Ungar 1992a). Gray-box or hybrid modeling strategies are potentially very efficient if the black-box and white-box components are combined in such a way that the resulting models have good interpolation and extrapolation properties.

There are two types of gray-box modeling approaches in which a neural network is combined with a black-box model: the parallel and the serial approach. In the parallel approach, the neural network is placed parallel with a white-box model. In this case, the neural network is trained on the error between the output of the white-box model and the actual output. Su and others (1992) demonstrated that the parallel approach resulted in better interpolation properties than pure black-box models. Johansen and Foss (1992) also used a parallel structure where the output of the hybrid model was a weighted sum of a first-principles and a neural network model. In the serial hybrid modeling strategy, the neural network is placed in series with the first-principles model. Various researchers (Psichogios and Ungar 1992a; Thompson and Kramer 1994) have shown the potential extrapolation properties of serial hybrid models. Psichogios and Ungar (1992b) used this approach for parameters that are functions of the state variables and manipulated inputs. Liu and others (1995) developed a serial hybrid model for a periodic wastewater treatment process by using ANNs for the bio-kinetic rates of a first-principles model. Cubillo and Lima (1997) also used this approach to develop a hybrid model for a rougher flotation circuit.

Flotation columns have been previously modeled using first-principles models. particle transport in the collection zone is usually modeled as axial convection coupled with axial dispersion. The peclet number (pe), or its inverse, the dispersion number, governs the degree of mixing. Most models only consider the slurry phase (Finch and Dobby 1990; Luttrell and Yoon 1993), in which case particle collection is viewed as a first order net attachment rate process. A model that considers both slurry and air phase was developed by Sastry and Loftus (1988). In this case particle attachment and detachment are modeled separately with first order rates. Luttrell and Yoon (1993) used a probabilistic approach to relate the particle net attachment rate constant to some operating variables (e.g., air flow rate). However, their approach involves empirical parameters and it cannot be used to predict the effect of certain operating variables such as the frother and collector concentrations.

In this work, we developed a serial hybrid model that predicts the effect of operating variables, including frother, collector, and extender concentration, on the performance of an anionic phosphate flotation column. We integrate a first-principles model for the slurry phase with neural networks that determine the dependence of the phosphate and gangue flotation rate constants on the operating variables. The model uses measurements of recovery and grade and inverts the first-principles model to calculate corresponding phosphate and gangue net flotation rate constants. The neural networks are then trained on the errors of calculated model parameters instead of the errors of the output of the first-principles model, as is the case with previous hybrid models.

The First-Principles Components

The basic equations representing the flotation of solid particles in a flotation column can be written by making a material balance for the solid particles in the slurry phase. This results in the following partial differential equations for the section above and below the feed point, respectively:

$$\frac{\partial C_{p_1}^{j}}{\partial t} = \left(\frac{U_p}{1 - \varepsilon_g} - U_{sl}^{j}\right) \frac{\partial C_{p_1}^{j}}{\partial z} + D \frac{\partial^2 C_{p_1}^{j}}{\partial z^2} - k_p (d_p^{j}) C_{p_1}^{j}$$
(1)

$$\frac{\partial C_{p_2}^{j}}{\partial t} = \left(\frac{U_t}{1 - \varepsilon_g} + U_{sl}^{j}\right) \frac{\partial C_{p_2}^{j}}{\partial z} + D \frac{\partial^2 C_{p_2}^{j}}{\partial z^2} - k_p (d_p^{j}) C_{p_2}^{j}$$
(2)

where,

- $C_{p_1}^j$ = Phosphate concentration of jth mesh size particles for the section above the feed point
- $C_{p_2}^j$ = Phosphate concentration of jth mesh size particles for the section below the feed point

 U_{p} = Superficial liquid velocity above the feed point

$$= Q_p / A_c$$

 U_t = Superficial liquid velocity below the feed point

$$= (Q_t - Q_e)/A_e$$

- D = Dispersion coefficient
- Q_p = Product volumetric flow rate
- Q_t = Tailings volumetric flow rate
- Q_e = Elutriation volumetric flow rate
- A_c = Cross-sectional area of the column
- U_{sl}^{j} = Slip velocity of jth mesh size particles

 ϵ_{g} = Air holdup

 $k_p(d_p^j) =$ Flotation rate constant for phosphate for j^{th} mesh size particles

The following assumptions are made in deriving the above equations:

- 1) The concentration of solid particles in the slurry phase is a function of height, z only, and variations of the concentration in radial and angular directions can be neglected.
- 2) The air holdup is constant throughout the column.
- 3) All the air bubbles in the system are of a single size.
- 4) Rate of detachment is either negligible or is a function of conditions in the slurry phase. This assumption allows us to treat the net attachment rate with just one flotation rate constant.

The slip velocity is calculated using the expression of Villeneuve and others (1996):

$$U_{sl}^{j} = \frac{g d_{p}^{j^{2}} (\rho_{s} - \rho_{1}) (1 - \phi_{s})^{2.7}}{18\mu_{1} (1 + 0.15R_{ep}^{j^{0.687}})}$$
(3)

where the particle Reynolds number is defined as

$$R_{ep}^{j} = \frac{d_{p}^{j} U_{sl}^{j} \rho_{s} (1 - \phi_{s})}{\mu_{1}}$$
(4)

where

	, ,	
g	=	Acceleration due to gravity (m/s^2)
μ_l	=	Water viscosity (kg/ms)
ρ_l	=	Water density (kg/m ³)
ρ_s	=	Solid density (kg/m ³)
φs	=	Volume fraction of solids in slurry
d_p^j	=	Particle diameter (m)
•		

Since R_{ep}^{j} is a function of U_{sl}^{j} , an iterative procedure is used to calculate the slip velocity. The procedure starts with an initial guess for U_{sl}^{j} and corresponding value of R_{ep}^{j} is plugged in Equation 3 and new value of U_{sl}^{j} is found. This new value is then used in Equation 2 and this procedure is continued till convergence is achieved. The axial dispersion coefficient is calculated by a modified expression of Finch and Dobby (1990):

$$D = 0.063 (1 - \varepsilon_g) d_c \left(\frac{J_g}{1.6}\right)^{0.3}$$
(5)

where

Equations 1 and 2 can be solved analytically for the concentration profile of the solid particles at steady state. The resulting analytical expressions for the concentration profile are

$$C_{p_{1}}^{j} = K_{1}^{j} \exp\{-\frac{1}{2}(a^{j} - \sqrt{a^{j^{2}} + 4b^{j}})z\} + K_{2}^{j} \exp\{-\frac{1}{2}(a^{j} + \sqrt{a^{j^{2}} + 4b^{j}})z\}$$
(6)

$$C_{p_{2}}^{j} = K_{3}^{j} \exp\{-\frac{1}{2}(d^{j} - \sqrt{d^{j^{2}} + 4b^{j}})z\} + K_{4}^{j} \exp\{-\frac{1}{2}(d^{j} + \sqrt{d^{j^{2}} + 4b^{j}})z\}$$
(7)

where

$$a^{j} = \frac{\left(\frac{U_{p}}{1-\varepsilon_{g}} - U_{sl}^{j}\right)}{D}; \quad d^{j} = \frac{\left(\frac{U_{t}}{1-\varepsilon_{g}} + U_{sl}^{j}\right)}{D}; \text{ and } b^{j} = \frac{k_{p}(d_{p}^{j})(1-\varepsilon_{g})}{D}$$

 K_1^j , K_2^j , K_3^j , and K_4^j are the constants of integration to be determined by using appropriate boundary conditions.

We determine boundary conditions from material balances at the top of the column, point of feed, and bottom of the column. A material balance at the top (z = L) results in the following equation:

$$A_{c}\Delta z \frac{dC_{p_{1}}^{j}}{dt} = A_{c} \left(\frac{U_{p}}{1 - \varepsilon_{g}} - U_{sl}^{j} \right) \left(C_{pl}^{j} \Big|_{L - \Delta z} - C_{pl}^{j} \Big|_{L} \right) + A_{c}D \frac{C_{pl}^{j} \Big|_{L - \Delta z} - C_{pl}^{j} \Big|_{L}}{\Delta z} - k_{p}(d_{p}^{j}) A_{c}\Delta z C_{p_{1}}^{j}$$

which in the limit as $\Delta z \rightarrow 0$ reduces to the boundary condition:

$$\frac{\mathrm{d}C_{p_{1}}^{j}}{\mathrm{d}z}\Big|_{z=L} = 0 \tag{9}$$

Continuity of the concentration profile at the feed location $(z = L_f)$ gives

$$C_{p_1}^{j}\Big|_{z=L_f} = C_{p_2}^{j}\Big|_{z=L_f}$$
 (10)

A similar material balance at the feed inlet gives for the solid particles in the slurry phase

$$Q_{f}C_{f}^{j} = A_{c}\left(\frac{U_{p}}{1 - \varepsilon_{g}} - U_{sl}^{j}\right)C_{p_{1}}^{j}\Big|_{z = L_{f}} - A_{c}D\frac{dC_{p_{1}}^{j}}{dz}\Big|_{z = L_{f}} + A_{c}\left(\frac{U_{t}}{1 - \varepsilon_{g}} + U_{sl}^{j}\right)C_{p_{2}}^{j}\Big|_{z = L_{f}} + A_{c}D\frac{dC_{p_{2}}^{j}}{dz}\Big|_{z = L_{f}}$$

where

At the bottom of the column (z = 0), due to the elutriation flow, the derivative of the concentration profile reduces to the following expression:

$$D\frac{dC_{p_{2}}^{j}}{dz}\Big|_{z=0} = -\frac{Q_{e}}{(1-\varepsilon_{g})A_{c}}C_{p_{2}}^{j}\Big|_{z=0}$$
(12)

The four boundary conditions can be solved in conjunction with Equations 6 and 7 for K_1^j , K_2^j , K_3^j , and K_4^j . The resulting expressions for the constants of integration are:

$$K_{4}^{j} = \frac{\left(Q_{f}C_{f}^{j}/A_{c}D\right)}{m^{j}(a^{j}-\alpha^{j})p^{j}\exp\{\alpha^{j}L_{f}\} + (d^{j}-\gamma^{j})q^{j}\exp\{\gamma^{j}L_{f}\} + m^{j}(a^{j}-\beta^{j})\exp\{\beta^{j}L_{f}\} + (d^{j}-\delta^{j})\exp\{\delta^{j}L_{f}\}}$$

(13)

$$\mathbf{K}_{3}^{j} = \mathbf{q}^{j} \mathbf{K}_{4}^{j} \tag{14}$$

$$\mathbf{K}_{2}^{j} = \mathbf{m}^{j} \mathbf{K}_{4}^{j} \tag{15}$$

$$\mathbf{K}_{1}^{j} = \mathbf{p}^{j} \mathbf{m}^{j} \mathbf{K}_{4}^{j} \tag{16}$$

where

$$\alpha^{j} = -\frac{a^{j}}{2} + \frac{1}{2}\sqrt{a^{j^{2}} + 4b^{j}}$$
(17)

$$\beta^{j} = -\frac{a^{j}}{2} - \frac{1}{2}\sqrt{a^{j^{2}} + 4b^{j}}$$
(18)

$$\gamma^{j} = -\frac{d^{j}}{2} + \frac{1}{2}\sqrt{d^{j^{2}} + 4b^{j}}$$
(19)

$$\delta^{j} = -\frac{d^{j}}{2} - \frac{1}{2}\sqrt{d^{j^{2}} + 4b^{j}}$$
(20)

$$p^{j} = \frac{\left\{-\frac{Q_{p}}{A_{c}D} + a^{j} - \beta^{j}\right\} exp(\beta^{j}L)}{\left\{\frac{Q_{p}}{A_{c}D} - a^{j} + \alpha^{j}\right\} exp(\alpha^{j}L)}$$
(21)

$$q^{j} = \frac{\left\{-\frac{Q_{t}}{A_{c}D} + d^{j} - \delta^{j}\right\}}{\left\{\frac{Q_{t}}{A_{c}D} - d^{j} + \gamma^{j}\right\}}$$
(22)

$$m^{j} = \frac{q^{j} \exp(\gamma^{j} L_{f}) + \exp(\delta^{j} L_{f})}{p^{j} \exp(\alpha^{j} L_{f}) + \exp(\beta^{j} L_{f})}$$
(23)

The recovery (%) of the phosphate particles of the j^{th} mesh size can be expressed in terms of the feed and tailings flow rates and concentration as

$$R_{p}^{j} = \left(\frac{Q_{f}C_{f}^{j} - \left[Q_{t} + A_{c}(1 - \varepsilon_{g})U_{sl}^{j}\right]C_{p_{2}}^{j}\Big|_{z=0}}{Q_{f}C_{f}^{j}}\right) * 100$$
(24)

Grade can be calculated as the ratio of the weight of phosphate to the sum of the weight of phosphate and gangue in the concentrate stream:

$$G^{j} = \left(\frac{Q_{f}C_{f}^{j} - \left[Q_{t} + A_{c}(1 - \varepsilon_{g})U_{sl}^{j}\right]C_{p_{2}}^{j}\Big|_{z=0}}{(Q_{f}C_{f}^{j} - \left[Q_{t} + A_{c}(1 - \varepsilon_{g})U_{sl}^{j}\right]C_{p_{2}}^{j}\Big|_{z=0}) + (Q_{f}C_{f_{g}}^{j} - \left[Q_{t} + A_{c}(1 - \varepsilon_{g})U_{sl}^{j}\right]C_{g_{2}}^{j}\Big|_{z=0})} \right) * 73.3$$
(25)

where $C_{g_2}^{j}$ is the gangue concentration of the jth particle size and $C_{f_g}^{j}$ is the gangue feed concentration of jth particle size. The multiplication factor is 73.3 because pure Florida phosphate rock measures at about 73.3 %BPL.

Calculation of Model Parameters

Since air-holdup ε_g is measured experimentally, the above first principles model has only two unmeasured model parameters for each particle size, namely, the flotation rate constants for phosphate (k_p) and for gangue (k_g). The experimental analysis usually available in industrial flotation columns is in terms of grade and recovery of phosphate. The recovery of gangue can then be readily calculated from measurements of grade and recovery of phosphate using the following relationship:

$$R_{g}^{j} = \frac{W_{g}^{j}}{W_{fg}^{j}} = \frac{R_{p}^{j}G_{f}^{j}(73.3 - G^{j})}{G^{j}(73.3 - G_{f}^{j})}$$
(26)

where G_{f}^{j} is the grade of the feed material.

The recovery of phosphate R_p^j is only a function of the flotation rate constant for phosphate, $k_{p},$ and air holdup, $\epsilon_{g}.$ Similarly, the recovery of gangue $R_{g}^{\,j}$ is only a function of flotation rate constant for gangue, $k_g,$ and air holdup, $\epsilon_g. \$ Since air holdup is measured, we can invert the model to determine the value of k_p that results in the measured recovery of phosphate R_p^j and the value of k_g that yields the measured recovery of gangue R^j_g. As shown in Figure 82, a one-dimensional search is performed to determine the values of flotation rate constants when supplied with the recovery of phosphate and gangue, respectively. The search for k_p is initialized with two values that yield errors in the corresponding recovery R_p^j of opposite sign. Since typically $0 \le k_p \le$ 10 min⁻¹ the values of 0 and 100 min⁻¹ are used. Then the method of false position (Chapra and Canale 1988) is used to iterate until the magnitude of the error in R_n^j drops to less than 10^{-3} . It is possible that the calculated recovery has a higher value than the experimental even for $k_p = 0$. In these cases k_p is set equal to zero. The above procedure is also used to determine k_g , except that the high initial value is set to 10 min⁻¹. The algorithm converges to unique solutions since recovery for both phosphate and gangue increases monotonically with the corresponding flotation rate constant.





The Hybrid Model

The hybrid model utilizes back propagation neural networks (Rumelhart and McClelland 1986) to predict the values of the flotation rate constants, k_p and k_g , and air holdup, ε_g . The overall structure of the hybrid model is shown in the Figure 83. Neural network NNI correlates the flotation rate constant for phosphate, k_p , with phosphate particle size, superficial air velocity, frother concentration, collector concentration, extender constant for gangue, k_g , with gangue particle size, superficial air velocity, frother concentration air velocity, frother concentration, performing the flotation rate constant for gangue, k_g , with gangue particle size, superficial air velocity, frother concentration, and pH.

Neural network NNIII correlates the air holdup, ε_g , with superficial air velocity and frother concentration. In this structure, all three neural networks are specific to the type of frother or sparger used.



Figure 83. Overall Structure of the Hybrid Model.

Neural Network Structure and Training

Single output feed forward back propagation neural networks are used with a single layer of hidden nodes. A unit bias is connected to both the hidden layer and the output layer. Both the hidden layer and the output layer used a logistic activation function (Hertz and others 1992) and the input and the output values were scaled from 0 to 1.

During the training mode, training examples are presented to the network. A training example consists of scaled input and output values. For NNI and NNII, the output values are the flotation rate constants calculated from one-dimensional searches for phosphate and gangue, respectively. For NNIII, the output value is the experimentally measured air holdup.

There are two approaches towards updating the weights (neural network parameters). In one approach, the input-output examples are presented one at a time and after each presentation the weights are updated using rules such as the delta rule (Rumelhart and McClelland 1986). This method is attractive for its simplicity but is restricted to rather primitive optimization algorithms. In contrast, the batch training approach employed here allows use of powerful methodology for nonlinear optimization. It processes each input-output example individually but updates the weights only after the whole set of input-output examples has been processed. In this case, the gradient is cumulated for all presentations, then the weights are updated, and finally the sum of the squared errors is calculated.

For the neural networks in our hybrid model the training process is started by initializing all weights randomly to small non-zero values. The random number is generated between -3.4 and +3.4 with standard deviation of 1.0 following the procedure recommended by Masters (1993). The optimal weights are determined using simulated annealing (Kirkpatrick and others 1983) and a conjugate gradient algorithm (Polak 1971). The simulated annealing algorithm is used for eluding local minimum. It perturbs the independent variables (the weights) while keeping track of the best (lowest error) function value for each randomized set of variables. This is repeated several times, each time decreasing the variance of the perturbations with the previous optimum as the mean. The conjugate gradient algorithm is then used to minimize the mean-squared output error. When the minimum is found, simulated annealing is used to attempt to break out of what may be a local minimum. This alteration is continued until networks can not find any lower point. We then hope that the local minimum is indeed the global minimum.

Model Validation

To validate the model, experiments were conducted in our laboratory column with CP-100 as the frother and feed obtained from Cargill. A total of 35 experiments were conducted with a fractional factorial design. Some of the experiments were conducted with coarse feed (14 x 35 Tyler mesh), some with fine feed (35 x 150 Tyler mesh), and some with unsized feed (14 x 150 Tyler mesh). Frother concentration ranged from 5 to
25 ppm, collector and extender concentrations from 0.27 to 1.7 kg/t, pH from 8.2 to 9.9, and superficial air velocity from 0.46 to 0.7 cm/s. The experimental protocol, conditions, and results were described earlier in the report.

The performance of the three artificial neural networks (ANNs) is shown in Figures 84-92. Figure 84 compares the flotation rate constants for phosphate (k_p) corresponding to the experimentally measured recoveries with those predicted by NNI. As shown in this figure, NNI captures the dependence of the flotation rate constant on particle size, superficial air velocity, frother concentration, collector and extender concentration, and pH. Similarly, Figure 85 compares flotation rate constant for gangue (kg) determined from experimental data with those predicted by NNII. As shown, NNII successfully predicts the flotation rate constant for gangue. Figure 86 presents the air holdup values (ε_{g}) predicted using NNIII against those measured experimentally. A satisfactory match is seen. The hybrid model integrates NNI, NNII, and NNIII as shown in Figure 83. Predictions of the hybrid model are shown in Figures 87-92. Figures 87 and 88 compare the experimental recovery (%) and grade (%BPL) with those predicted by the hybrid model, respectively, for the coarse feed size distribution (14X 35 Tyler mesh). As shown in these figures, the hybrid model successfully predicts both recovery and grade. Figures 89 and 90 compare the experimental recovery (%) and grade (% BPL) with those predicted by the hybrid model, respectively, for the fine feed size distribution. The hybrid model fails to successfully predict both recovery and grade. This is attributed to the fact that fine feed has a very wide size distribution (35X150 Tyler mesh size) and only the overall recovery and grade were measured experimentally. It is therefore desirable to utilize narrower ranges of feed size in the model and to analyze for recovery and grade according to each size range instead of just one recovery and grade for the entire particle size distribution. This was implemented for the unsized feed which has even a wider size distribution (14X150 Tyler mesh). Figures 91 and 92 compare the experimental recovery (%) and grade (%BPL) predicted by the hybrid model, respectively, for the unsized feed after it has been sized and grade and recovery were determined for each size. As can be seen from these figures, the hybrid model successfully predicts both recovery and grade.



Figure 84. Performance of NNI: Model Versus Experimental Flotation Rate Constant for Phosphate (kp).



Figure 85. Performance of NNII: Model Versus Experimental Flotation Rate Constant for Gangue (kg).



Figure 86. Performance of NNIII: Model Versus Experimental Air Holdup for Frother CP-100.



Figure 87. Performance of the Overall Hybrid Model: Predicted Versus Experimental Recovery (%) for Coarse Feed Size Distribution.



Figure 88. Performance of the Overall Hybrid Model: Predicted Versus Experimental Grade (%BPL) for Coarse Feed Size Distribution.



Figure 89. Performance of the Overall Hybrid Model: Predicted Versus Experimental Recovery (%) for Fine Feed Size Distribution.



Figure 90. Performance of the Overall Hybrid Model: Predicted Versus Experimental Grade (%BPL) for Fine Feed Size Distribution.



Figure 91. Performance of the Overall Hybrid Model: Predicted Versus Experimental Recovery (%) for the Unsized Feed After It Has Been Sized.



Figure 92. Performance of the Overall Hybrid Model: Predicted Versus Experimental Grade (%BPL) for the Unsized Feed After It Has Been Sized.

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Two-Level Hybrid Model

An alternative hybrid model architecture to that of Figure 83 is shown in Figure 93. The neural networks are structured in two levels. The first level consists of the ANNs for predicting k_p (NNI) and k_g (NNII) and receives as an input the inferred bubble size. This is the output of one of the ANNs of the second (top) level, NNIII. The second level also includes NNIV, which predicts air holdup. The advantage of this structure is that NNI and NNII are independent of the type of frother and sparger used, and therefore would not need retraining if these change.

As bubble size is not measured in industry, we infer it from the two-phase (air/water) air holdup, J_g , and U_t using the well-known Drift-flux analysis (Yianatos and others 1988). The output required to train NNIV is the (two-phase) air holdup. Air holdup is relatively easy to obtain, so after a change of frother or sparger the hybrid model of Figure 93 can become functional in a short interval of time.

The two-level hybrid model was validated with experimental from four frothers (F-507, CP-100, OB-535, and F-579) and 14 x 150 feed that was sized. Recovery and grade were measured for each size. Predictions of the hybrid model are shown in Figures 94 and 95. Figure 94 presents the predicted recovery (%) against the experimental recovery for frother CP-100 (square points), F-507 (circles), OB-535 (triangles), and F-579 (diamonds). Similarly, Figure 95 compares the predicted grade (%BPL) against the experimental grade for CP-100, F-507, OB-535, and F-579. It can be seen from these figures that predicted recovery and grade for OB-535.



Figure 93. Performance of the Two-Level Hybrid Model: Predicted Versus Experimental Grade (% BPL) for the Four Frothers.



Figure 94. Performance of the Two-Level Hybrid Model: Predicted Versus Experimental Recovery (%) for the Four Frothers.



Figure 95. Performance of the Two-Level Hybrid Model: Predicted Versus Experimental Grade (%BPL) for the Four Frothers.

Performance Measures

The performance of a flotation column is affected by both recovery (%) and grade (%BPL). To guide optimization it is necessary to combine the two outputs (grade and recovery) in a single performance measure. Several performance measures are possible, and some are presented below.

Selectivity

Selectivity is defined as

$$S = \left[\frac{R}{R_{b}} / \frac{R_{t}}{R_{tb}}\right]^{\frac{1}{2}}$$
(27)

where

R	=Recovery of phosphate in the product stream.
R _b	=Recovery of gangue in the product stream.
\mathbf{R}_{t}	=Recovery (or Reject ability) of phosphate in the tailings
R _{tb}	=Recovery (or Reject ability) of gangue in the tailings

stream.

We developed the following expression that relates selectivity to the recovery and the grade of the product stream

$$S = \left[\frac{\frac{G}{G_{f}} - G(1 - R) - R}{(1 - G)(1 - R)}\right]^{\frac{1}{2}}$$
(28)

where

G = Grade (%BPL) of phosphate in the product stream. $G_f = Grade$ (BPL) of phosphate in the feed.

Separation Efficiency

Separation efficiency is defined as follows:

$$\mathbf{E} = \mathbf{R} - \mathbf{R}_{\mathbf{b}} \tag{29}$$

In terms of the recovery and grade of the product stream

$$E = \frac{R(G - G_f)73.3}{G(73.3 - G_f)}$$
(30)

Economic Performance Measures

Selectivity and separation efficiency do not include any economic input such as cost of the reagents. Therefore, an alternate performance measure was developed which includes recovery, grade, and the reagent prices. A scheme for penalizing lower grade rock has been developed. This scheme deducts differential costs, relative to 66% BPL, for transportation and acidulation. The acidulation scheme assumes soluble P_2O_5 losses increase in direct proportion to the amount of phosphogypsum. Thus, the procedure requires an estimate of the quantity of phosphogypsum that is produced.

The following assumptions are evoked:

- 1. The price of rock of 66% BPL = 22.00
- 2. Zero insol % BPL = 73.33
- 3. Transportation cost = \$2.50 per ton.
- 4. Soluble P_2O_5 losses = 1.00%
- 5. Insoluble P_2O_5 losses = 6.00%
- 6. Increase in soluble P_2O_5 losses is proportional to the amount of phosphogypsum produced.

The transportation penalty is calculated as follows:

Base case: 66% BPL rock (dry basis)
Freight cost per BPL ton =
$$$2.5/0.66 = $3.79$$

Penalty: $\left(\frac{2.50}{B_L/100}\right) - 3.79$ per BPL ton
Trensportation penalty = $\left(\frac{2.50}{B_L} - 2.70\right) B_L$ pen top

Transportation penalty = $\left(\frac{2.50}{B_L/100} - 3.79\right) \frac{B_L}{100}$ per ton (31) where $B_L = 0^{\circ} B_L$ when grade $< 66^{\circ}$

where, $B_L = \% BPL$ when grade < 66%

The acidulation penalty is calculated as follows:

Base case: 66% BPL rock (30.21% P₂O₅, CaO:P₂O₅ = 1.49)
Acid insol =100
$$\left(1-\frac{B_L}{73.33}\right)$$

Phosphogypsum components:

Acid insol = 1 ton rock ×
$$\left(1 - \frac{B_L}{73.33}\right)$$

Unreacted = 1 ton rock × $\left(\frac{B_L}{73.33}\right)$ × 0.06
Dihydrate = 1 ton rock × $\frac{(B_L/100)}{2.184}$ × 1.49 × (172/56) × (1 - 0.06)

Total amount of phosphogypsum = Acid insol + Unreacted + Dihydrate

Soluble P₂O₅ losses = $\$300.0 \times \frac{(\% \text{ so lub le P}_2O_5 \text{ losses})/100}{2.184}$ per ton = \$1.37 per ton

Acidulation Penalty =
$$\$62.0 \times \frac{(\text{Total amount of phosphogypsum})}{B_L} - 1.37$$
 (32)

The phosphate sales value increases with increasing grade, and the following relationship is assumed:

Sales value = Price of 66 % BPL rock *
$$(B_L/66)^{1.5}$$
 (33)

The adjusted sales value is calculated from:

Adjusted sales value = Sales value - Transportation penalty - Acidulation penalty (34)

The relationship between adjusted value and %BPL is shown in Figure 96.

Let

Feed solid flow rate = F, ton per year Product solid flow rate = P, ton per year Feed grade = G_f , % Concentrate grade = G, % Product recovery = R, % Adjusted sales value of feed = C_f , \$ per ton Adjusted sales value of product = C_p , \$ per ton Reagent-i price = C_{ri} , \$/lb Reagent-i usage = U_i lb/ton feed

The feed flow rate and the product flow rate can be related by:

$$P = F\left(\frac{G_{f}}{100}\right)\left(\frac{R}{100}\right)\left(\frac{100}{G}\right)$$
(35)

The financial performance measure, representing dollars earned per year, is:

Financial Performance Measure =
$$C_p P - C_f F - F \sum_i U_i C_{ri}$$
, \$/year (36)



Figure 96. Value of Phosphate Rock as a Function of % BPL.

The Model Program

A user-friendly Windows program was developed that incorporates the hybrid model and the financial performance measure. The program consists of more than 10,000 lines of original C/C++ code, and allows the user to enter data, to modify the data file, to update the neural network parameters (i.e., train the networks), and to change column geometry, operating conditions, and reagent prices. The program outputs predicted grade, recovery, and the annual profit or loss.

When the user runs the program, the main screen shown in Figure 97 loads up. The menu bar at the top offers the options of printing instructions, adding data to the file that is used for teaching the model, updating the model parameters, modifying the data file, specifying the inputs that affect column performance, running the model, and exiting the program.

Typical usage of the program involves the following steps:

- 1. From the inputs menu, specify the column geometry
- 2. Add data. At least 25 sets of data should be added and a minimum of 100 is recommended. Very important: Always set the geometry to that of the column used to collect data before adding or modifying data.
- 3. Update the model. In turn, update the phosphate flotation rate constant, the gangue flotation rate constant, and the air holdup.
- 4. From the inputs menu, specify the conditions for which you wish to predict column performance.
- 5. Run the model.



Phosphate Flotation Column Simulator Figure 97. The Program's Main Screen.

Selecting to add data (the geometry of the column must be set first) brings up the screen shown in Figure 98. The user should click on a mesh size for the feed, then click the mouse on the box for feed %BPL, enter a value, then click on the box for feed flow, enter a value, and so on until all the boxes have been filled. If the user clicks the "click when done" box without having given values for all inputs, then nothing is written to the data file, and the user receives a warning to that effect. This is done because after the data entry the program not only saves the entered data to the file, but also calculates the corresponding phosphate and gangue flotation rate constants and saves these on file as well. This calculation requires a complete data set. If the product has been sized and grade and recovery have been calculated for each size (something highly recommended), the user should treat each size as a separate run, i.e. enter all input conditions for each size.

If the user needs to modify some previously entered data (the geometry of the column must be set first), selecting from the main screen to "modify data" brings up the data file of the training set as shown in Figure 99. Highlighting a number and printing a new value changes it, and when clicking "click to exit" the changes are saved. Before saving the changes the program also recalculates the phosphate and gangue flotation rate constants (not shown to the user), and for this reason, if the data set is large, the user may have to wait a while for this process to be completed. One difference between the "add data" screen and the "modify data" screen is that in the former the particle size is entered as Tyler mesh, while in the latter as microns. This is the geometric mean of the maximum and minimum size of the mesh that the user had selected. The "modify data" screen could also be used to enter new data, but care should be taken that each column is filled and that nothing is printed to the right of the cells with text "NOPASSING."

TYLER MES	H WEIGHT %	RECOVER	IY.	GRADE
20/28	0.00	41.3%		66.3%
28/35	0.00	48.7%		69.9%
35/48	0.00	60.4%		55.3%
48/65	0.00	72.7%		61.8%
65/100	0.00	82.3%		43.9%
100/150	0.00	88.9%		26.9%
20/35	0.00	42.3%		68.7%
35/150	0.00	77.9%		60.1%
16/28	0.00	40.9%		62.1%
28/150	100	72.7%		52.1%
16/35	0.00	42.5%		60.6%
	OVERALL GRADE		52.1 %	
	OVERALL RECOVE	RY	72.7 %	
	ANNUAL PROFIT (million \$)	0.0162	

Figure 98. The Screen for Adding Data to the File that Contains the Training Set.

	basedata dat				
FEED %BPL	14.040	14.040	14.040	14.040	14.840
FEED FLOW (gpm)	0.198	0.418	0.126	0.284	0.376
TAUNGS FLOW (game	2.014	1.779	1.432	2.062	1.897
ELUTRIATION FLOW (gpm)	2.409	2.351	2.423	2.919	2.893
FEED SOLIDS (%)	59.370	35.110	48.530	35.030	49.750
AIR FLOW BATE (solv)	0.093	0.371	0.093	0.371	0.232
RECOVERY (%)	68.310	40.040	34.600	45.900	67.720
GRADE (SBPL)	55.950	55.360	40.070	61.860	51.040
SPECIFIC GRAMITY	2.600	2.600	2.600	2.600	2.600
FEED PARTICLE SIZE(microne)	321.040	321.040	321.040	321.040	321.040
AR HOLDUP (11)	29.100	21.450	17.610	23.130	31.610
FROTHER CONC. (ppm)	5.000	5.000	25.000	25.000	5.000
COLLECTOR CONC. (BH/N)	0.600	0.600	0.600	0.600	0.600
EXTENDER CONC. (bi/l)	0.600	0.600	0.600	0.600	0.600
pH	9.500	9.500	9.500	9.500	9.500
CUCK TO DOT	1		P. 101010-10	C at white	ch 2005th

Figure 99. The Screen that Displays the File that Contains Data for Training the Neural Networks.

Once sufficient data have been entered (at least 25 sets, but a minimum of 100 is recommended), the user should train the networks by selecting "update model." There are three neural networks, one for the phosphate flotation rate constant, one for the gangue floatation rate constant, and one for the air holdup. The user should select to train each of the three. As network training can take a considerable amount of time, this is a task that should be performed only periodically and only if a significant amount of data have been added since the last training.

Once the model has been updated, the user can run the model to obtain predictions. But first, the inputs that affect column performance must be specified. Selecting the "inputs" menu brings four submenus: "geometry," "feed characterization," "operating conditions," and "financial." The screen for specifying column geometry is shown in Figure 100. The user must enter the diameter, the height, and the location of the feed. Values can be entered either by typing in the boxes or by moving the scroll bars. The values cannot exceed the specified maxima or be below the specified minima. If the user wishes to expand them, he/she should contact Dr. Svoronos at the University of Florida (Tel: (352) 392-9101; E-mail: svoronos@ufl.edu). The screen for specifying the feed is shown in Figure 101. A variety of Tyler mesh sizes are available, and the user can select to specify that 100% of the feed is of one size (e.g., 100% is 28/150 as shown in the figure) or to distribute the feed among several sizes (e.g., 30% 20/35 and 70% 35/150). If for a particular mesh size the weight percent is greater than zero, the %BPL must be entered. For sizes with zero weight %, it doesn't matter what is entered in the %BPL column. The program immediately calculates the composite %BPL and displays it at the bottom of the second column. The screen for specifying operating conditions is shown in Figure 102. Values can be entered either by typing them in the boxes or by moving the scroll bars. They cannot be outside the specified range. The last input screen is needed for calculating the financial performance of the column and is shown in Figure 103. It allows the user to change the assumptions of the financial performance measure (e.g., soluble and insoluble losses), and to specify the price and consumption of reagents. The amount of collector, extender, and frother consumed cannot be entered since these values are calculated from dosage entries in the "operating conditions" screen.

Once all the inputs have been specified, the user can select the menu "Run Model" to obtain predictions of the column performance. The program output is shown in Figure 104. Grade and recovery are displayed for all mesh sizes, and the overall grade and recovery are displayed in the bottom. The program also uses the financial performance measure to output the estimated annual profit in millions of dollars.



Figure 100. The Screen for Specifying Column Geometry.

Tyler Mesh	96W1		%BPL	
16/20	0.00		36.10	
20/28	0.00		22.30	
28/35	0.00		40.00	
35/48	0.00		13.30	
48/65	0.00		24.90	
65/100	0.00	-	12.20	
100/150	0.00		12.00	
20/35	0.00	1	31.05	
35/150	0.00		25.00	
16/28	0.00		14.00	
28/150	100.00		14.04	
16/35	0.00	1	14.00	
Total	100.00	Composite	14.04	Click When Dor

Figure 101. The Screen for Specifying the Feed.

	Minimum		Maximum	
Feed Weight % Solids	0.00	1 <u> </u>	<u>></u>]00.00	52.34
Specific Gravity of Solids	¥.08	<u>ع</u>	3.00	2.60
Feed Flow Rate (gpm)	0.00	1	17.62905	0.229600
Tailings Flow Rate (gpm)	0.05	<u>.</u>	▶ 352.5810	1.646900
Elutriation Flow Flate (gpm)	0.00		352,5010	2.615600
Frother Conc. (ppm)	0.00		25.00	5.00
Air flow rate (scfm)	0.0758000	11	1 0.7730250	0,100000
Collector Conc. (Ibs/ton)	0.20	1	.00	0.60
Extender Conc. (Ibs/ton)	1.00		• [s.nu	0.60
pН	1 8.00	1 I	10.00	9.50
		1		

Figure 102. The Screen for Specifying Operating Conditions.

Market price of	of 66%BPL pho	sphate rocks	22.00		
Transportatio	n cost of phosp	hate rock per ton	3.50		
Zero insols %	BPL		73.34		
Soluble losse	18				
Insoluble los	965		6.00		
	Anionic Flota	tion Reagents		Cationic Flot	ation Reagents
	Cost \$/lb	Consumptions lbs/ton		Cost 9/16	Consumptions lbs/ton
Collector	0.12	notneedled		0.15	0.20
Extender	0.08	not-needed		0.08	0.10
pH Modifier	0.08	0.16		0.00	0.00
Frother	0.20	unt.needed		0.00	0.00
Sulturic acid	0.00	0.00		0.06	2.50
Other	0.00	0.00		0.00	0.00

Figure 103. The Screen for Calculating the Financial Performance of the Column.

EED SIZE (mesh	ENTER INPUTS FO	R TRAINING MODE
16/20 C		
20/20 🔿	FEED %BPL	RECOVERY PQ
28/35 🔿	FEED FLOW (gpm)	GRADE (NBPL)
35/48 🕥		
48/65 C	TAILINGS FLOW (gpm)	AIR HOLDUP PG
65/100 C	ELUTRIATION FLOW (gpm)	FROTHER CONC.[ppm]
100/150 🔿	FEED SOLIDS ING	COLLECTOR CONC RhsM
20/35 C		
35/150 C	AIR FLOW (scfm)	EXTENDER CONC.(Ibs/t)
16/28 🤇	pH	SPECIFIC GRAVITY
28/150 0		
16/35 C		

Figure 104. The Output of the Phosphate Flotation Column Simulator.

CONCLUSIONS

Study of Frothers and Spargers

- Selection of the proper frother and its combination with sparger type for a particular flotation feed are ones of the main criteria, which determine the efficiency of column flotation of Florida phosphate. Air holdup data for twenty-eight commercially available frothers, of different chemistry, was studied. The performance of these frothers can be divided into three distinct categories: high, medium and low air holdup. The air holdup data is not influenced significantly by the source of water used in the experiments.
- The results of the two-phase experiments showed that, with each type of frother, the % holdup increased gradually with raising the superficial air velocity from 0.24 to 0.94 cm/s. The increase in %holdup was more pronounced at higher superficial air velocity. Meanwhile, the estimated bubble diameter was greatly affected by the applied superficial air velocity. Increasing the superficial air velocity from 0.24 to 0.94 cm/s was accompanied by a gradual increase in bubble diameter regardless of the type of frother used. The generated air bubbles while using F-507 were smaller in diameter than those obtained with CP-100 under the same experimental conditions.
- It is generally accepted that air holdup is strongly influenced by the air/water interfacial characteristics (frother type and concentration) due to its ability to promote air dispersion, hinder bubble coalescence, and decrease bubble rise velocity. Air holdup rapidly increases as frother concentration increases from 5 to 20 ppm, particularly at higher airflow rate, and then this tendency becomes less pronounced. Meanwhile, bubble size rapidly decreases with increasing frother concentration.
- The external spargers produced higher air holdup than the internal spargers (perforated tube and Cominco one-phase sparger at lower superficial air velocity and low pressure). Based on the air holdup measurements under similar operational conditions for 9 spargers, the air dispersion ability of spargers investigated can be arranged as follows:

Two-phase ejector, and Eductor > One-phase porous sparger > Cominco two-phase sparger > USBM sparger > Two-phase static mixing sparger ~ Two-phase porous sparger > Cominco one-phase sparger > Perforated tube.

- The air dispersion ability for Cominco one-phase sparger is more responsive to the superficial air velocity and the corresponding pressure. The air holdup rapidly increases with an increase in the airflow rate. When the superficial air velocity is more than 1.0 cm/s and pressure is greater than 40 psi, Cominco one-phase sparger generated higher air holdup than two-phase static mixing sparger and two-phase porous sparger.
- A common feature of all spargers is that generation of higher air holdup or small bubbles relies on the shear rate and air/liquid interfacial properties. In general, air holdup can be increased or bubble size can be reduced through one of the following ways. 1- by increasing the shear rate, which can be accomplished by increasing the energy dissipation rate in the system. 2- by reducing the liquid surface tension, which can be accomplished by adding more frother. 3- by using more efficient frother. However, excessive use of frother can be costly and detrimental to flotation selectivity. Increasing air holdup or reducing bubble size by increasing the shear rate or using more efficient frother is a better choice.
- Air holdup increases with superficial air velocity for all spargers. However, the maximum airflow that can be injected into the column is limited by a transition from uniform bubbly flow to churn-turbulent flow.
- Frother type has a significant influence on sparger performance. The results of air holdup measurement for two different frothers (F-507 and CP-100) showed that F-507 has higher air dispersion ability than frother CP-100 for various spargers. This confirms that for all spargers used, the frothers can be divided into three distinct categories: high, medium and low air holdup. It was found that frother type have more significant influences on two-phase spargers than one-phase internal spargers. The internal spargers show a limited influence of the frother used on air holdup, whereas the external spargers revealed a stronger relationship. It is concluded that in general when the external spargers are operated at a suitable pressure, the air holdup can be controlled by the frother type and concentration over a wide range.
- The sparger water requirement for the five two-phase spargers decreases in the following order:

Eductor > Two-phase static mixing sparger > Two-phase ejector > Cominco two-phase sparger > USBM sparger.

• Based on the results obtained, the eductor sparger or two-phase ejector is recommended to be used for phosphate column flotation. This kind of sparger has several advantages over the others, such as, strong air dispersion ability, simple operation, less clogging potential, and less energy consumption, compared with other external spargers.

Column Flotation of Unsized Feed Phosphate (14x150 Mesh)

- Column flotation of unsized feed was studied while using four different types of frothers and in absence of conditioning water. Amongst the four frothers (CP-100, F-507, X-268 same as F-579, and OB535) tested, it was found that CP-100 shows superior flotation performance to the others, for example, 96% recovery with 56% BPL content in the concentrate can be achieved, when eductor is used as sparger.
- The results of size-by-size recovery show that different particle sizes have different flotation responses, and the flotation recovery decreases as particle size increases for all frothers tested. The results also show that better coarse phosphate recovery can be obtained using CP-100 as frother and eductor as sparger.
- It was found that bubble size has a significant influence on the flotation performance of unsized phosphate, and the optimum bubble size was found to be in the range of 0.8-1.0 mm.
- To achieve a better flotation performance, there is an optimum combination between sparger and frother type. Generally speaking, for the sparger with stronger air dispersion ability, the weaker frother should be used to generate desirable bubble size. On the other hand, for the sparger with poorer air dispersion ability, the stronger frother should be added.

Column Flotation of Coarse Feed Phosphate (20x35 Mesh)

- Results of column flotation of coarse feed show that by properly selecting the operational parameters, phosphate recovery can be significantly improved, for example, 99% recovery with 69% BPL content in the concentrate is expected to be achieved.
- A net upwards water flow prevalent in the column is helpful to improve the coarse phosphate flotation.
- Bubble size has a significant influence on the coarse phosphate flotation, and there is an optimum bubble size range to obtain lower BPL content in the tailings and higher BPL recovery in the concentrates. From the flotation results obtained, it was found out the optimum bubble diameter for coarse phosphate flotation to be in the range of 0.8-1.0 mm.
- Using CP-100 as frother, in absence of conditioning water, yields better separation performance than F-507 when eductor is used as sparger.
- The order of significance for the effect of variables on BPL content in the concentrate is:

slurry discharge rate > frother type > airflow rate > frother concentration.

Meanwhile, the order of significance for the effect of variables on the recovery is:

Frother type > frother concentration > slurry discharge rate > airflow rate.

Column Flotation of Fine Phosphate (35x150 Mesh)

- For column flotation of fine phosphate (35x150 mesh), the results showed that frother type and operational conditions strongly affect the grade of flotation concentrate. When frother F-507 is used, phosphate concentrates contain about 50% acid insoluble and only 34-37% BPL, compared with 22-34% acid insoluble and 46-55% BPL in concentrates for frother CP-100.
- For column flotation of fine phosphate, the order of significance for the effect of variables on BPL and A.I. contents in the concentrate can be determined as follows: Frother type > air flow rate > frother concentration. Meanwhile, the order of significance for the effect of variables on A.I. recovery in the concentrate is: Frother type > frother concentration > air flow rate. The order of significance of interactional effects of the variables on BPL and A.I. contents in the concentrate is: Frother type and air flow rate (AxB) > air flow rate and frother concentration (BxC) > frother type and frother concentration (AxC). The order of significance of interactional effects of the variables on BPL and A.I. recovery in the concentrate is: Air flow rate and frother concentration (BxC) > Frother type and air flow rate and frother concentration (AxC).
- The results of central composite design for column flotation of fine phosphate showed that a concentration of 15 ppm of frother (F-507 or CP-100) is enough to maximize the recovery. Meanwhile, a pH of 9.5 is close to optimum for F-507 in comparison to pH 9.0 when CP-100 is used. But slightly more collector (above 1.4 lb/t) is needed to get the maximum recovery (above 90 %).

Column Flotation of Belt Feed Phosphate

The results of the parametric study of the belt feed phosphate column flotation indicated the following:

• In absence of conditioning water and regardless of the concentration of the frother, the concentrates obtained with CP-100 have, in general, marginally higher BPL and BPL recovery in comparison with F-507. Also, increasing the collector dosage from 2.2 to 8.8 lb/t (i.e. 2 to 4 kg/t), and in absence of conditioning water, has nearly no significant influence on the BPL recovery or grade. For both frothers (F-507 and CP-100) recoveries were consistently above 99 % and the grade (BPL %) was above 66 % for CP-100 and above 63 % for F-507.

- On the contrary, the presence of conditioning water deteriorates the superior performance of phosphate column flotation in presence of CP-100 as a frother while it has a minor effect when using F-507. Also, the results of studying the kinetics of column flotation, in presence and absence of conditioning water, indicated that the flotation process is nearly stable (in terms of grade and recovery) while using tap water, and with any frother, irrespective of the time of sampling the froth (concentrates) and tails. However, once conditioning water was applied to the column in the presence of CP-100 the efficiency (in term of weight % floated) of the flotation process started to deteriorate depending on the concentration of frother applied.
- Application of factorial design for the belt feed phosphate column flotation gave an insight into the behavior of column parameters on metallurgical performance. For example, by properly selecting the operational parameters, it is possible to achieve a 99 % recovery with a reasonable good concentrate quality of BPL% up to 68 % and with a low A.I. (5.71 %) content. However, application of conditioning water and replacing F-507 by CP-100 as a frother have the most negative influence on reducing the BPL recovery and deterioration of the efficiency of the column flotation. On the other hand, the results indicated that the most significant binary interactions, which adversely affect on the recovery of the produced concentrates, are those between frother concentration of CP-100 and conditioning water. Such interaction does not exist in presence of F-507. These results clearly show that for column flotation of each specific mineral commodity, the applied collector - frother system should be studied carefully to prevent any possibility of their interactions.
- To confirm the above conclusion on the role of interaction between frother and collector at different pH, a statistical design for 2-phase system was performed. The results showed that changing the pH of the medium from 8 to that used in phosphate column flotation (~9.5) has a negative effect on the % holdup while using CP-100 as a frother. However, with the frother F-507 the change in pH increased the % holdup that was found, as expected, to be a function of the applied superficial air velocity. In other words, CP-100 works better at lower pH. The reverse trend is noticed with F-507. The effect of increasing frother concentration indicated that both CP-100 and F-507 have positive effect on the values of % holdup. However, the effect of F-507 on % holdup is significantly higher in comparison with CP-100. The higher is % holdup, the lower will bubble diameter be. On the other hand, studying the interaction between the studied parameters showed that there is an interaction between CP-100 and the collector. This interaction had a negative effect on reducing the % holdup, and consequently, increasing the bubble diameter. Such interaction is, also, found to be more significant at higher superficial air velocity. On the other hand, this interaction between frother and collector was not seen with F-507 at different superficial air velocities. This clearly indicates that there is a mutual interaction between CP-100 and the residual concentration of collector mixture in the water of the column which prevents frother CP-100 from controlling the bubbles diameter and consequently deteriorates the superior results obtained with CP-100 in the absence of conditioning water.

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