Publication No. 02-147-199

AMINE FLOTATION OF ROUGHER CONCENTRATE AND INTERMEDIATE PEBBLE IN A COLUMN CELL

Prepared by
The Pennsylvania State University

under a grant sponsored by

FIPR
Florida Institute of Phosphate Research

April 2003

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AMINE FLOTATION OF ROUGHER CONCENTRATE AND INTERMEDIATE PEBBLE IN A COLUMN CELL

FINAL REPORT

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FIPR Project Number: 00-02-147

April 2003

PERSPECTIVE

Patrick Zhang, Research Director - Beneficiation & Mining

In response to a growing interest in utilizing flotation columns for upgrading phosphate, FIPR has identified column flotation as one of its research priorities in the updated "Strategic Initiatives and Applied Research Priorities."

Flotation columns offer lower capital cost, better flotation recovery and lower energy consumption than mechanical flotation cells widely used in Florida.

Column cells have been used primarily on coarse particles in Florida's phosphate beneficiation plants. The recovery of phosphate from the coarse flotation feed (typically 16 by 35 mesh) used to be a major efficiency problem. Separate flotation of this feed using mechanical cells could recover only about 60% of the phosphate; column flotation has brought that recovery up to 90%.

Although columns are being adopted gradually for phosphate processing in Florida, current applications are limited to the rougher (fatty acid) flotation step, due to the lack of understanding and testing data. This is the first FIPR project to address amine flotation with a column. Pilot tests were carried out at five operating phosphate mines in Florida, and showed that the column performance met or exceeded the plant performance when the pilot unit was operated at capacities of 2-4 tons per hour. In many instances, the comparable performance in the column cell was achieved at much lower amine dosages (30-50% of those used in the plant).

Encouraged by the amine column flotation results, a supplementary funding was approved to conduct in-plant amine flotation testing in a column on the intermediate pebble (IP). In a typical phosphate beneficiation plant in Florida, phosphate matrix is first washed and deslimed at 150 mesh, generating three products: the waste clay (-150 mesh), a pebble product (16 by 35 mesh), and the flotation feed (16 by 150 mesh). The pebble product is usually of sufficient purity as a final product, and the flotation feed is subject to the standard Crago process to achieve low-insol product. However, in some Florida operations, the 16 by 150 mesh fraction is sized at 35 (or 28) mesh, with the undersize fraction floated and the oversize fraction (the IP) blended with the pebble. Blending the IP product with the pebble lowers the grade of the final product significantly, because IP contains higher insol. A typical IP analyzes well over 20% insol, versus 5-8% insol in various flotation concentrates. Depending on size distribution of the phosphate matrix, IP could account for as much as 1/3 of the total rock product.

ABSTRACT

Pilot plant studies were carried out in a 2 TPH Dual Extraction Column (DEC) at five operating mines to determine the efficacy of column cells in amine flotation rougher phosphate concentrate and intermediate pebble (IP) product. The DEC unit employed in the study had a diameter of 0.6 m with an overall height of 0.9 m. The study showed that the column was effective in processing fine and coarse rougher concentrates. The column capacity was determined to be approximately 3 TPH and 4 TPH for fine and coarse rougher concentrates, respectively. The optimum amine dosages for better or comparable separation results were determined to be approximately 40-60% of those currently employed in plant operations. The study showed that the use of columns will result in considerable savings in reagent costs and overall plant operating and maintenance costs. The column was successful in processing rougher concentrate feed having insol of 40% or higher and the use of process water had minimal effect on the performance of the column cell.

For IP upgrading, the use of amine provided by Arr-Maz was effective in producing a concentrate of sufficient purity that can be blended with the pebble. The study showed that with amine dosages of 0.5-1 lb/T, the IP can be upgraded from 40-45 BPL and insol of 38-45% to give a concentrate containing 58-62 BPL and insol of 16-18% with the product grade being limited by liberation issues. Owing to the inefficiency of the IP screens, they may be replaced by column cells.

ACKNOWLEDGMENTS

Financial support for this work was provided by the Florida Institute of Phosphate Research (contract numbers 00-02-147R and 00-02-147S). The PI acknowledges with gratitude the support and helpful suggestions provided by Cargill Fertilizer, CF Industries, IMC, PCS and the FIPR Technical Advisory Committee. The in-plant studies would not be possible without the provision of the 2 TPH DEC pilot plant unit by Beneficiation Technologies and the operational assistance of Messrs. George Brooks and Mike Yencso. The support and provision of frother F-579 and a special amine formulation for coarse silica flotation by Arr-Maz were vital to the success of this study.

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EXECUTIVE SUMMARY

In the Florida phosphate industry, a two-stage flotation process is employed for the recovery of the phosphate mineral value. In both stages, conventional flotation cells are commonly used. In the present study, we have studied the effectiveness of column flotation in amine flotation of rougher phosphate concentrate and intermediate pebble (IP) product. Pilot plant studies carried out in a 0.61 m x 0.9 m dual extraction flotation column (DEC). The amine flotation of rougher phosphate concentrates was carried out at five operational mines in Florida while the IP studies were carried out at three operational mines in Central Florida. While the rougher concentrate studies employed plant amine, the IP studies used a special formulation from Arr-Maz.

The results show that column flotation technology can be successfully applied in cleaner duties and in the upgrading of IP product. For fine and coarse rougher concentrates flotation, the optimum amine dosages were determined to be lower than those employed in plant operations. In many instances, the column dosage rates for comparable performance were 40-60% of plant levels. The optimum amine dosage depends on the insol content of the feed. In general, 0.27 lb./T-0.6 lb./T and 0.7 lb/T-1 lb./T of amine are required for feed insol of 15-20% and greater than 30%, respectively. Our results show that recycled process water can be successfully used in amine flotation in the DEC unit with minimal loss in separation efficiency. At an air superficial velocity of 1.05 cm/s (13 CFM), our results show that the DEC unit can process 3 TPH and 4 TPH of fine and coarse rougher concentrates respectively.

Preliminary results show that direct flotation of the feed to the IP screens is feasible, thus eliminating the screens and fatty acid flotation steps. This change is expected to simplify the flow sheet. Our results show that with a feed containing 30-34 BPL and 50-56% insol, one stage of column flotation can produce a concentrate with 55-57 BPL and 18-20% insol at 75-80% BPL recovery. The tailings from this separation may either be scavenged and/or blended with the rougher flotation concentrate. However, more detailed studies are needed to optimize the flotation of the feed to the IP screen.

The DEC unit was effective in upgrading the IP product. Depending on the quality of the IP, amine dosage rates of 0.5-1 lb./T may be employed. With a feed containing 40-45 BPL and 38-45% insol, our results suggest that a concentrate with 58-62 BPL and 16-18% insol is possible at 85-90% BPL recovery. Our results suggest that the pilot DEC unit has a capacity of at least 5.3 TPH which was the maximum value tested in this study. The use of column flotation to upgrade the IP will simplify the process flow sheet. In addition, it should reduce transportation to, and grinding costs at the chemical plants as well as the build-up of the gypsum stacks.

PART I: ROUGHER CONCENTRATE FLOTATION

INTRODUCTION

In the beneficiation of Florida phosphate deposits, the clay minerals are separated by classification at 150 mesh. A coarse phosphate fraction (commonly referred to as pebble) is recovered by screening at 16 Tyler mesh and is usually of sufficient purity to be sold as product. Flotation technology is extensively used to recover the phosphate value from the remaining feed (16x150 Tyler mesh). However, in some Florida phosphate operations, the remaining feed is subjected to further classification to generate an intermediate pebble (IP) product (16x35 Tyler mesh) or coarse and fine flotation feed. For the operations where an IP product is generated, it is often blended with the pebble and shipped as final product.

The flotation feed contains a mixture of sand and phosphate mineral value. In a typical operation, the double-float process developed by Arthur Crago is employed (Oswald 1993). In the first stage, the phosphate mineral value is floated using a fatty acid collector. This process is not very selective and the rougher concentrate still contains high levels of silica. The rougher concentrate is then treated with sulfuric acid to remove the fatty acid coating and reconditioned with a cationic (amine) collector to selectively float the silica.

The flotation steps are almost exclusively carried out in conventional flotation cells, even though some plants (e.g., South Fort Meade and Hookers Prairie Mines) have employed flotation columns in the rougher flotation circuit. However, the amine flotation step is almost exclusively carried out in conventional cells. It is evident that mechanical cells are the mainstay of the Florida phosphate industry. However, mechanical cells have high operating and maintenance costs. By contrast, flotation columns do not have moving parts and therefore have lower operating and maintenance costs. Since columns also require less floor space, they are ideal candidates for retrofitting duties (Clingan and McGregor 1987). Thus, the objective of the present study is to determine the efficacy of column flotation for cleaner duty in the Florida phosphate industry.

EXPERIMENTAL METHODS

EQUIPMENT

The in-plant studies were carried out in a pilot plant Dual Extraction Column (DEC) unit owned by Beneficiation Technologies, LLC. The pilot plant unit was designed as a mobile, self-contained unit, suitable for both in-plant and remote site test work. The unit has a nominal capacity of 1-2 TPH depending on the density of the feed material. The pilot plant unit consists of an 0.51 m^3 stainless steel feed sump connected to a 2x1.5 inch pump, which has flexibility to deliver slurry to (i) a 0.15 m cyclone feeding a 0.4 m^3 stainless steel conditioner, equipped with a cruciform impeller driven by a 2 HP variable speed motor or (ii) directly to the DEC unit by-passing both cyclone and conditioner. The whole assembly is mounted on a trailer pad (see Figure 1).

The DEC consists of two identical tanks with overflow launder with one tank mounted directly above the other. Each tank has a diameter of 0.61 m and a height of 0.45 m. The height to diameter ratio employed in the pilot plant unit is slightly smaller than the 1.25 typically employed in the full size DEC unit. The level of fluid in the upper section of the unit was regulated by adjusting the opening of the drain pipe connecting the two sections. This was done with the aid of a conically shaped valve which could be manually raised or lowered as needed. With this arrangement, the underflow from the top section became the feed to the bottom section where additional aeration was introduced to recover more of the silica. The underflow discharge from the lower unit represents the final concentrate in the case of amine flotation. In conventional flotation, the top and lower cells operate as rougher and scavenger units, respectively. With this novel column design, two stages of flotation are combined in a single flotation column unit. While the froth from both sections may be combined or sampled separately, we adopted the former procedure in the present study.

Each section is equipped with one infuser block fed with 9.08 m^3/h (or 40 GPM) of water at a minimum recommended pressure of 207 kPa (30 psi) and up to 27.2 m^3/h (16 CFM) of air at 34 kPa (5 psi). The calibration curve of the blower is given in Appendix A. To ensure smooth operations, a 5 HP water pump was installed on the trailer pad to generate the necessary water pressure for the infusers. This modification was necessary when it became apparent that it might be difficult to locate a water source with the needed pressure at all locations. Air flow to the two infuser blocks is provided by a single positive rotary low pressure blower powered by a 1 HP variable speed motor. The combination of the air and water in the infuser block results in high energy dissipation rates thereby producing finely divided bubbles. Power supply to the trailer unit is standard 3 phase, 220/440 volts. Testing in remote areas utilizes the self-contained generator as power source.

EXPERIMENTAL PROCEDURE

During in-plant testing, the DEC unit received either conditioned or unconditioned feed from the plant circuit. In the case of the latter, the reagent dosage was regulated as required by the DEC and was the preferred mode for the current testwork. The use of unconditioned feed was preferred because the optimum amine dosages needed in the DEC unit were not known as this is not standard industry practice. Pre-conditioned feed was only available at one location.

Figure 1. Pilot Plant DEC Set-Up Employed in Rougher Concentrate and IP Flotation Studies.

The tests were carried out at five different operating mines owned by IMC (Four Corners and Fort Green), Cargill (South Fort Meade) , PCS and CF Industries. At each site, plant personnel would hook up the water supply, electrical circuit to the control panel mounted on the trailer pad, and run the hose from the washer box to the unit located on the ground floor. Except where indicated the feed supplied to the pilot plant unit was not reagentized, and this allowed us to vary the collector (amine) dosage. In these tests, the feed hose terminated inside a 4" PVC pipe which drained into the top section of the DEC unit. The required amine dosage was metered with the aid of a peristaltic pump (Masterflex Digital Console Drive Model 7523-20) and introduced inside the PVC pipe. The diluted amine solution used was taken from the plant feed line and varied in strength from 5 to 10% (v/v). With this arrangement, the amine conditioning time was limited to the time it took for the feed to travel the length of the pipe (typically less than three seconds). This arrangement was selected to mirror the plant practice where the amine is added either directly to the first cell or in the feed box. While depressants are sometimes employed in plant practice to generate high BPL concentrates, they were not evaluated in the present study.

At each site, preliminary experiments were carried out to determine the optimum collector dosage. These experiments were used to establish approximate amine dosage levels for the steady state tests. For the steady state tests, the operation of the DEC was set (air flow rate and amine dosage) while froth and tailing samples were collected over an extended period (at least one hour) to ascertain the attainment of steady state results. During the steady state test, plant feed, concentrate and tailing samples were also taken, where possible, for direct comparison with the DEC performance. At the end of the test period the samples were handed over to the respective plant laboratories for drying and chemical analysis. Since the turnaround time for the analyses at three of the sites was rather long, some samples were sent to an external laboratory to provide preliminary results on the effectiveness of the conditions employed during the test-work. This allowed us to change the operating parameters so as to cover a broader spectrum of test conditions. From the composition of the feed, tailings and froth samples, the recovery was computed. In plants where fine and coarse amine feeds are produced, every effort was made to test both feed streams.

RESULTS AND DISCUSSION

PLANT A

Preliminary Evaluation of Column Flotation

Preliminary experiments were carried out at Plant A to determine the effects of air flow rate and amine dosage on the flotation response. The tests were carried out on both fine and coarse amine feed. For the fine feed, the variation of product grade with air flow rate and amine dosage is shown in Figure 2. The feed rate for these tests was approximately 2.5 short tons per hour (TPH). Our results show that the product grade is dependent on both the air flow rate and amine dosage. In general, the effect of amine dosage was more pronounced at the lowest air flow rate. At low amine dosage, the product grade increases with collector dosage, however, the increase was more pronounced at the higher air flow rates. When the optimum amine dosage was exceeded, the product grade decreased with further increases in collector dosage. By contrast, this effect appeared to be more pronounced at the lower air flow rate. For all the air flow rates tested, there appears to be an optimum amine dosage in the range of 0.37-0.5 lb/T of amine feed, which tends to increase with airflow rate. The results from the preliminary tests show that the maximum product grade appeared to increase with air flow rate. For comparison, the plant amine dosage during the test period was 0.56 lb/T and the average concentrate grade was 68.3 BPL, which is lower than the maximum values observed at the three air flow rates employed in the DEC unit.

Figure 2 also shows the variation of tailings BPL with amine dosage and air flow rate. In general, in the tailings grade decreased with increasing amine dosage and air flow rate, thus confirming that that more of the silica was being floated. This observation was expected since flotation is a surface phenomenon and the available bubble surface area for silica flotation increases with airflow rate. However, when the optimum amine dosage was exceeded, the selectivity of the separation decreases, resulting in a higher BPL in the tailings stream. For comparison, the plant amine tailings averaged 13.8 BPL, which was considerably higher than the worst results from the DEC unit. A comparison of the DEC and average plant performance is shown in Table 1. The results show that at the optimum amine dosage, the BPL recovery in the column cell was about 1.3-2.8% higher that that in the plant. The preliminary results for the fine amine feed suggest that column flotation may be a viable alternative to the conventional cells in use the phosphate industry.

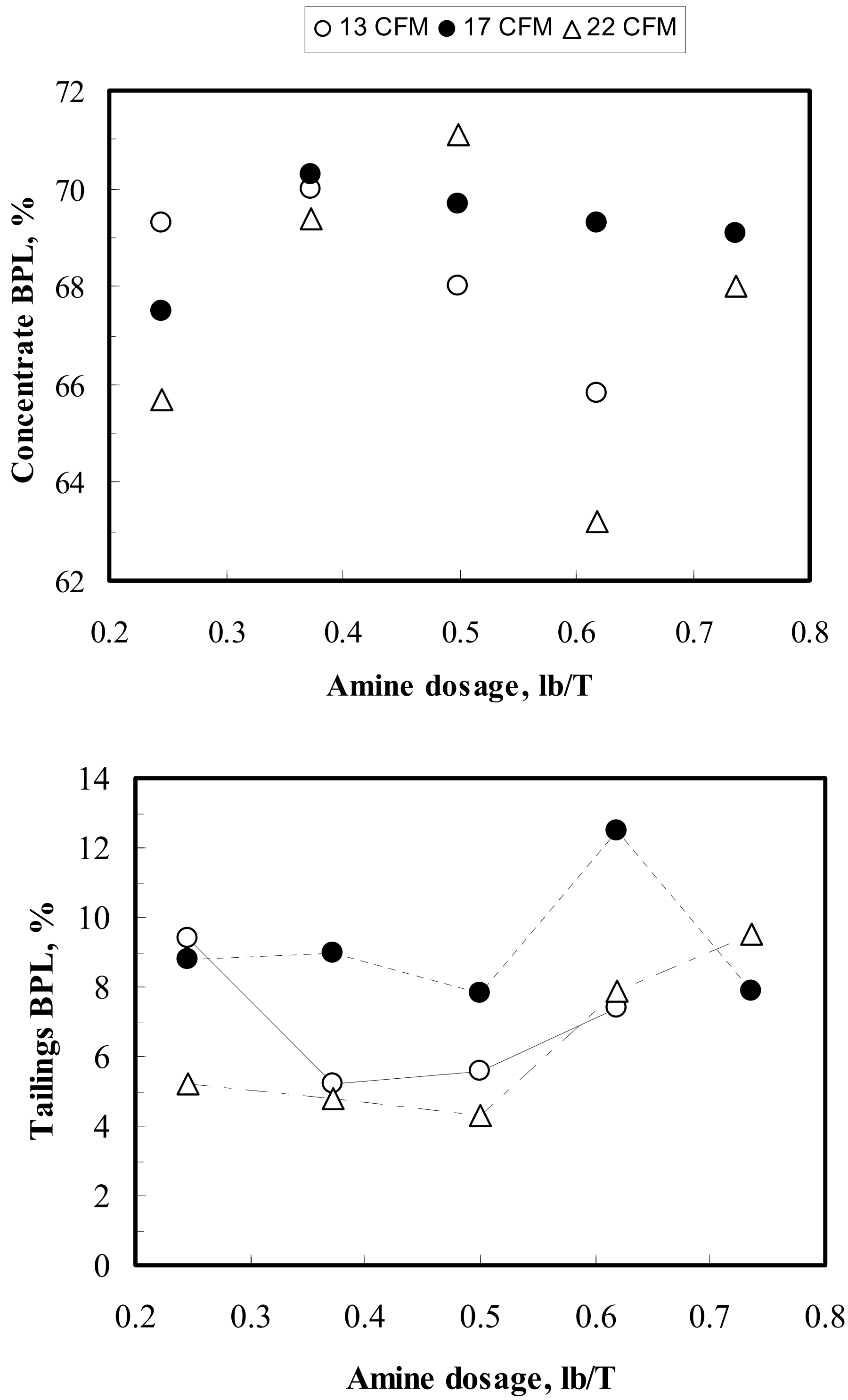


Figure 2. Effect of Amine Dosage and Air Flow Rate on Fine Rougher Concentrate Flotation.

Table 1. Dependence of Optimum Amine Dosage on Air Flow Rate.

DEC Air Flow Rate (CFM)	Amine Dosage, (lb./T)	Concentrate BPL	Tailings BPL	BPL Recovery
9	0.37	70.0	5.2	98.4%
11	0.37	70.3	9.0	97.1%
13	0.50	71.1	4.3	98.6%
Plant*	0.56	68.3	13.8	95.8%

*Plant performance data was provided by plant management.

One of the problems normally encountered with in-plant test work is the variability in the feed characteristics. In order to normalize the data, we have plotted the yield index as a function of insol rejection and the results are shown in Figure 3. By definition, the yield index is the product of the BPL recovery and the insol rejection. The objective of most separation processes is to maximize the yield index, high recovery with little contamination of the product. Ityokumbul (1994) has shown that a maxima exists when the separation is characterized using such an index. It is interesting to note that the separation results for the three airflow rates and different amine dosages lie on the same line. As expected the highest values of the yield index were obtained at the highest airflow rate. Since the DEC unit was relatively shallow further increases in the airflow rate were not evaluated due to increased turbulence in the cell, resulting in high BPL losses. Thus, the maximum airflow rate for these tests was fixed at 13 CFM.

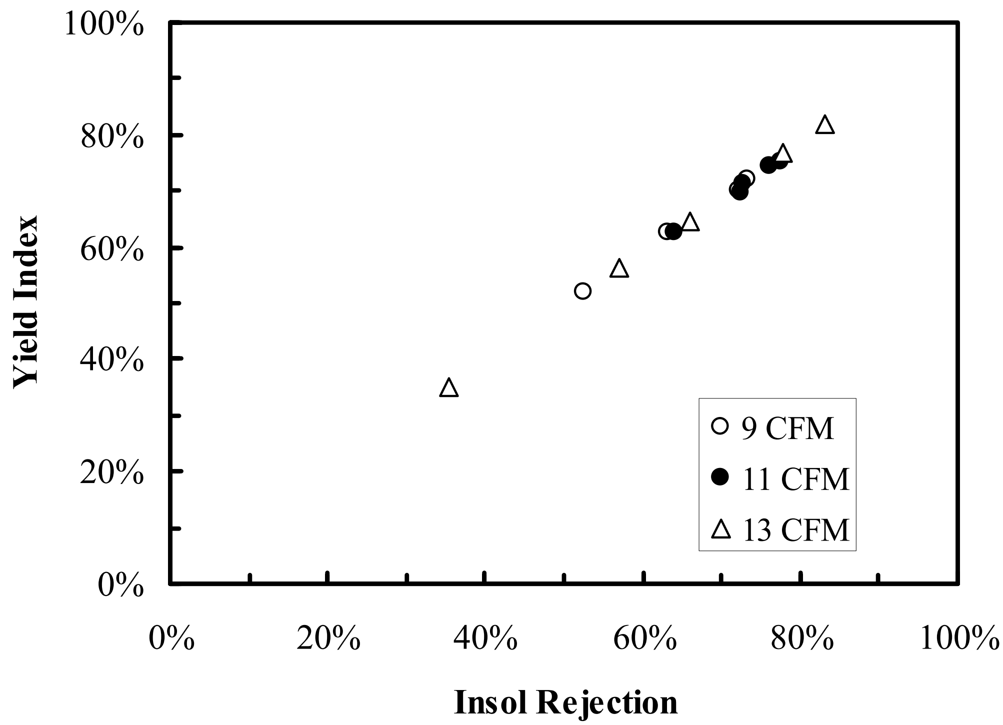


Figure 3. Loci of Separation at Different Air Flow Rates.

Plant A produces both fine and coarse amine feed so additional preliminary tests were carried out on the coarse feed. However, the production of coarse feed on the test date was low so the feed rate was limited to 0.87 TPH. Figure 4 shows the variation of product grade with air flow rate and amine dosage. It is noted that the test conditions were not optimum as the feed was very dilute, but since the plant was also processing the same feed, a comparison of results may be used to provide an indication of the efficacy of columns in coarse amine flotation. For the coarse feed, the optimum amine dosage appears to be in the range of 1.46-1.83 lb/T. The amine dosage is considerably higher than that determined for the fine feed. By comparison, the plant dosage was 1.26 lb/T. For the column and plant operations, the maximum concentrate grade (66.2-66.5) and BPL recoveries (99.5-99.8%) were approximately the same. As indicated earlier, the high amine dosages determined for the coarse feed is attributed to the dilute nature of the feed. However, our results confirm that column flotation is a viable technique for amine flotation of rougher phosphate concentrate.

As indicated above, the maximum airflow rate employed in these tests was 13 CFM. If we assume that the air was equally split between the two cells, the air superficial velocity in each section was approximately 1.05 cm/s. This limitation is only applicable in the pilot unit employed in this study as the full-scale unit will be much deeper the effect of turbulence will be minimized. In scale-up operations, the correlation provided by Ityokumbul (1993) may be used to establish the upper limit of air velocities to be employed in the amine flotation.

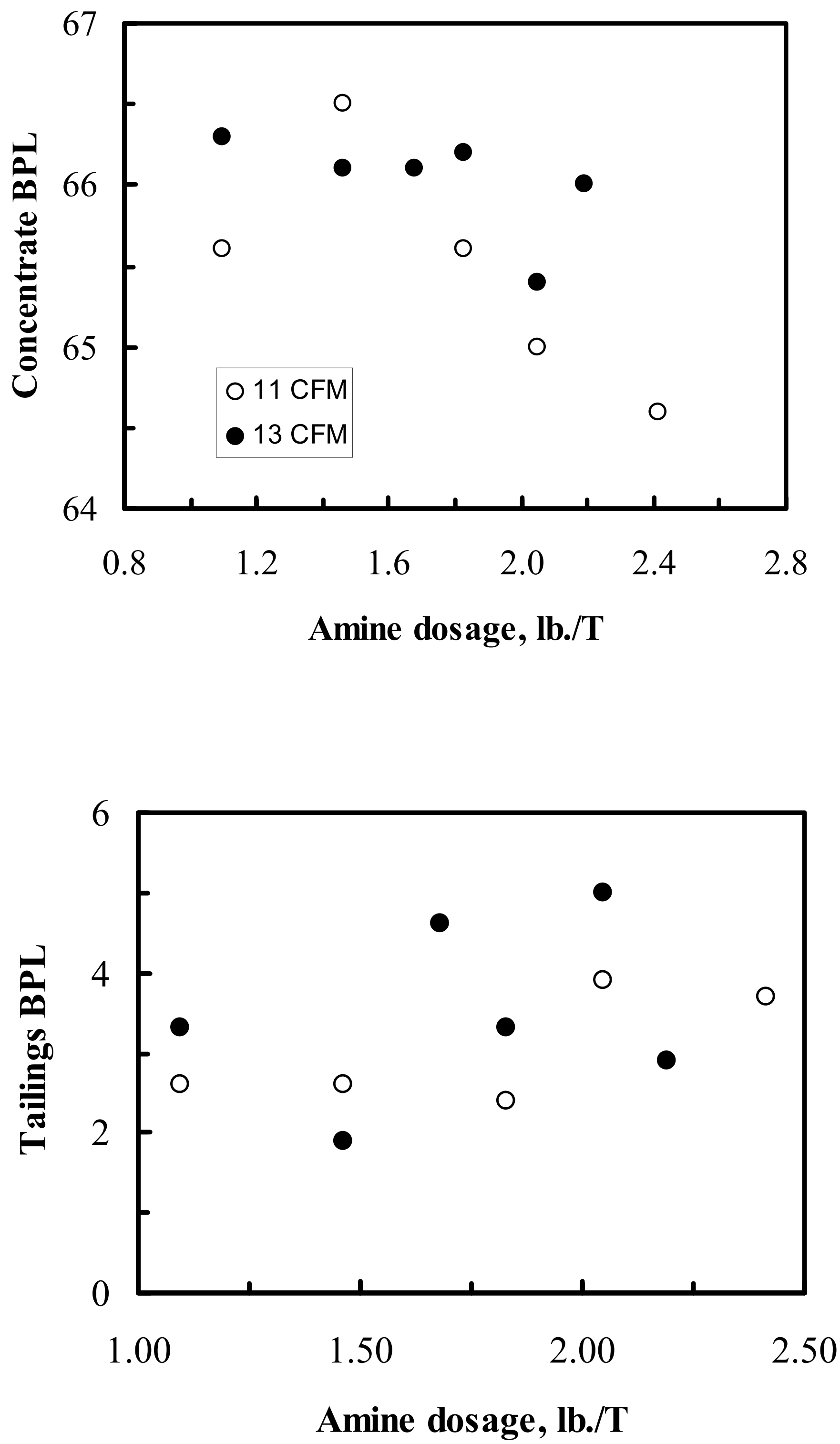


Figure 4. Effect of Amine Dosage and Air Flow Rate on Coarse Rougher Concentrate Flotation.

Steady State Studies

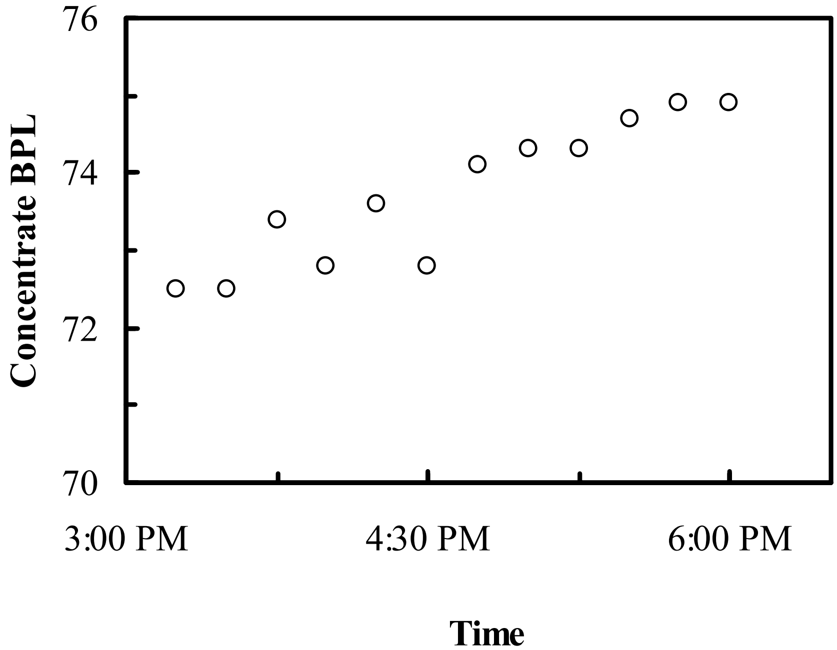
As indicated above, the performance of the DEC unit was comparable to that of the plant. On the basis of these promising results, FIPR Technical Advisory Committee (TAC) recommended that we proceed with the steady state studies. The steady state studies were initiated approximately one month after the conclusion of the preliminary studies. In these operations the amine dosage was fixed while several concentrate and tailings samples were taken over a longer period (at least one hour) to ensure that steady state conditions were attained. For these tests the airflow rate was fixed at 13 CFM. In order to avoid problems encountered during the preliminary tests, the plant management authorized the operators to make the necessary adjustments to avoid supplying a dilute feed to the DEC unit. With these adjustments, we received steady feed rates in the range of 1.8-4.0 TPH. In general, test conditions at this site were ideal and the results may be taken to be representative of amine flotation of rougher phosphate concentrate.

Fine Amine Feed

After the unit was set-up for the steady state tests at Plant A, the feed rate and amine dosage were fixed while the froth depth was varied. This test was carried out to establish the grade recovery profile for the DEC unit. Figure 5 shows the variation of concentrate and tailings grade with time. In general, the tailings grade increases with product grade. The results show that it is possible to produce a high-grade concentrate with an Insol of 2-3%. However, this is generally accomplished at the expense of high BPL in the tailings stream. It is noted that the BPL in the tailings stream was generally below 10% and was comparable to the reported amine plant tailings on the test date (6.8% BPL).

Figure 6 shows the grade-recovery curve for the flotation of fine amine feed with the DEC unit operating at approximately 2 TPH. Also shown in Figure 6 is the average plant performance. We note that the even though the amine dosages were comparable (0.617 lb./T in the DEC compared to 0.604 lb./T in the plant), the concentrate grades were higher in the DEC unit. Similarly, for the same BPL recovery, the concentrate grade in the DEC was approximately 1.5-3.5 BPL higher, thus showing that deployment of flotation columns in amine flotation of rougher phosphate concentrate will produce comparable (if not better) separation results.

For the test described above, the plant concentrate was not sampled. Thus, it was not possible to carry out a real-time comparison of the column and plant performances. Subsequently, we carried out additional tests in which the amine dosage was varied while taking concentrate and tailings samples from the plant and DEC unit. Figure 7 shows a real-time comparison of the product and tailings grade from the plant and DEC unit. The feed rate to the DEC unit was 1.95 TPH while the amine dosage ranged from 0.33-0.65 lb./T. By comparison, the average plant amine dosage in the plant was 0.59 lb./T. In general, the concentrate grade in the DEC unit mirrors that of the plant, the only difference being that the former was of a higher grade. Since the feed was not sampled, it is not possible to determine whether the variation in the product grade was due to changes in the feed. The fact that both profiles mirror each other would suggest that this was indeed the case. We note that even though the amine dosage in the column was reduced to 0.33 lb./T, the concentrates from the plant and DEC unit were comparable.



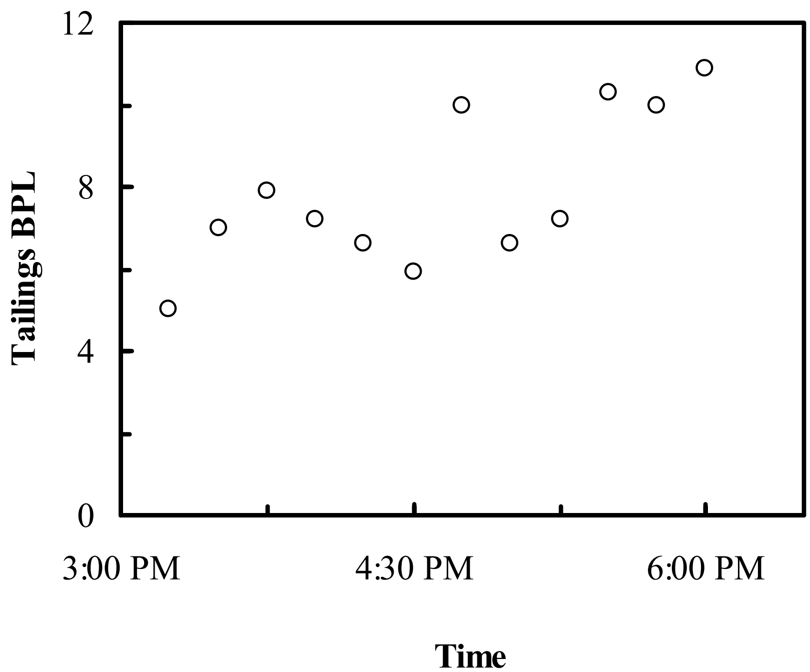


Figure 5. Real-Time Variation of Concentrate and Tailings Grades for Run 1.

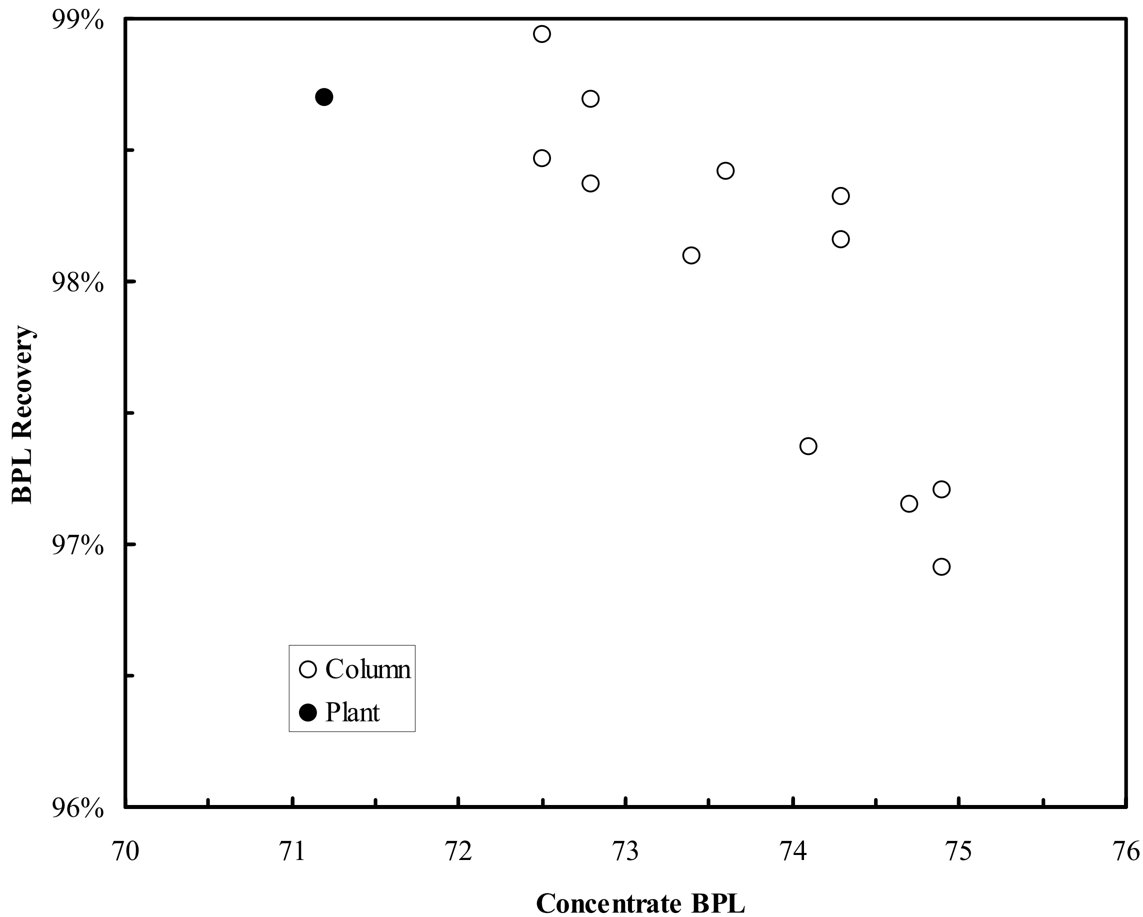


Figure 6. Grade-Recovery Profile for Run 1.

Our results show that the plant tailings were generally lower than those from the DEC unit. However, we attribute this observation to be the consequence of the shallow nature of the pilot DEC unit. Since a shallow froth depth was maintained in the unit, turbulence in the cell would result in phosphate losses. In a full scale unit, the use of a deeper cell is expected to lower the phosphate losses to the tailings stream, thus resulting in slightly higher phosphate recoveries.

A summary of the results from fine amine flotation of rougher concentrate at Plant A are shown in Table 2. These results clearly show that the DEC unit consistently produced concentrates with 72 BPL or higher even when it was operating at much lower amine dosages. It is evident from the foregoing that column flotation is an effective process for amine flotation of rougher concentrate.

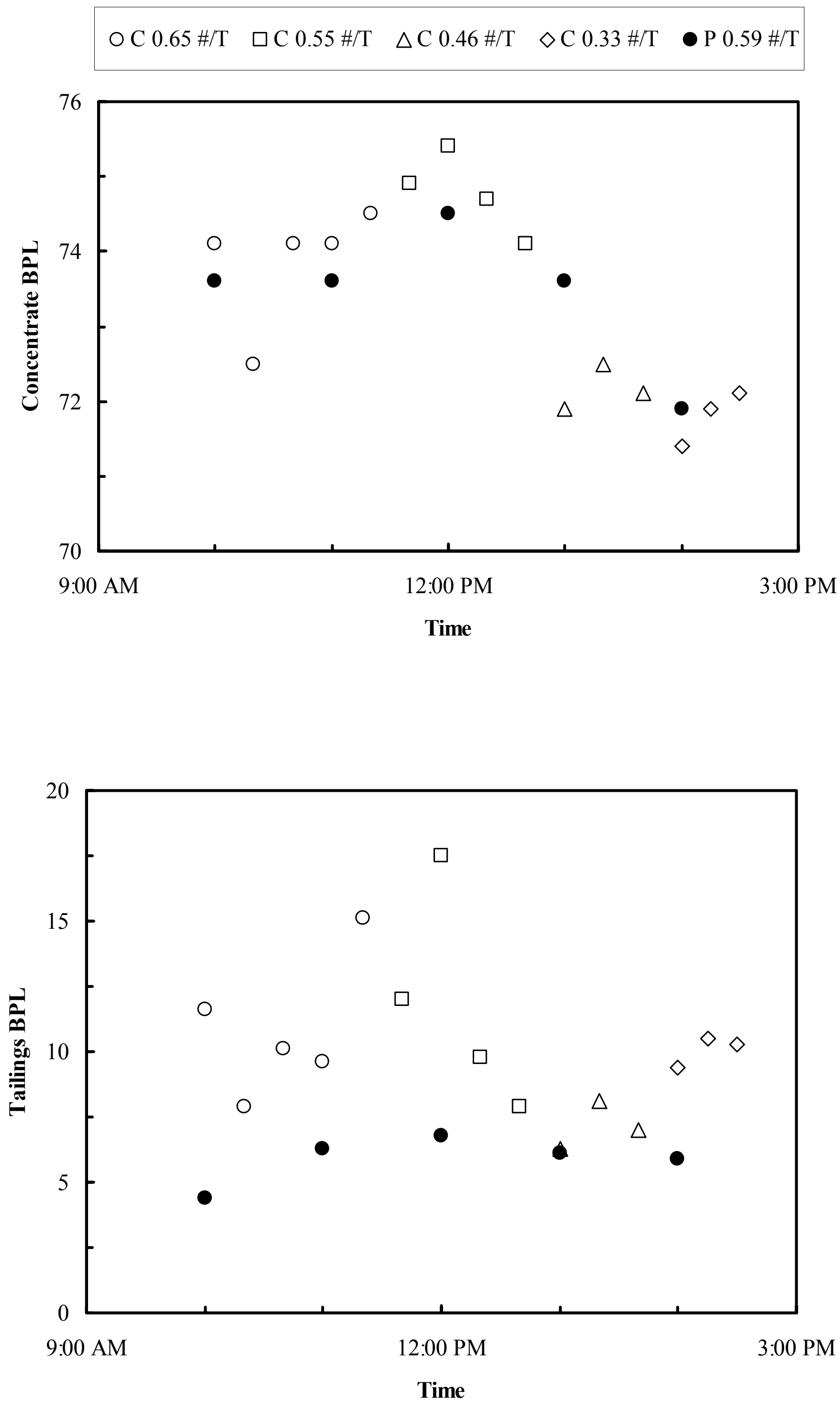


Figure 7. Real-Time Variation of Concentrate and Tailings Grades for Run 2.

Table 2. Summary of Test Results on Fine Rougher Concentrate Flotation at Plant A.

Run	Unit	Amine Dosage, #/T	Concentrate		Tailings BPL	Recovery, %
			BPL	Insol		
1	DEC	0.62	73.7 ± 0.92	2.9 ± 0.85	7.96 ± 1.93	95.5 ± 1.4
	Plant	0.60	71.2	N/A	6.8	96.5
2A	DEC	0.65	73.9 ± 0.78	2.4 ± 0.88	10.9 ± 2.71	95.9 ± 1.3
	Plant	0.59	73.6	2.8	5.40 ± 1.34	98.2
2B	DEC	0.55	74.8 ± 0.54	2.3 ± 0.40	11.8 ± 4.15	95.2 ± 2.2
	Plant	0.59	74.5	2.9	6.8	97.6
2C	DEC	0.46	72.2 ± 0.31	3.2 ± 0.17	7.10 ± 0.91	97.7 ± 0.37
	Plant	0.59	73.6	2.5	6.1	97.9
2D	DEC	0.33	71.8 ± 0.36	3.5 ± 0.12	10.1 ± 0.59	96.8 ± 0.29
	Plant	0.59	71.9	3.8	5.9	98.2

At this site the maximum solids feed rate was about 2 TPH, however, the results obtained at other locations suggest that the column can be operated at higher loading rates. We also note that the maximum gas velocity employed at this site was 1.05 cm/s, and the use of higher gas velocities should permit the operation of the column at higher loading rates (Ityokumbul 1993).

Another method for comparing the separation efficiencies is the use of the yield index (see Figure 8). While the data from the plant and DEC units lie on the same separation line, the highest yield indices were obtained with the latter. It appears that the optimum amine dosage for fine feed flotation at Plant A is in the range 0.3-0.6 lb./T. This value is consistent with that determined during the preliminary tests, even though the tests were carried out at different times. The yield index plot provides an excellent assessment of the operational efficiency of the plant. In the case of Plant A, the results show that the plant fine amine circuit was operating at near maximum separation efficiency. However, the yield index in the DEC unit was slightly higher than that of the plant. Since lower amine dosages are needed in the column, the replacement of the conventional cells with columns is recommended for Plant A.

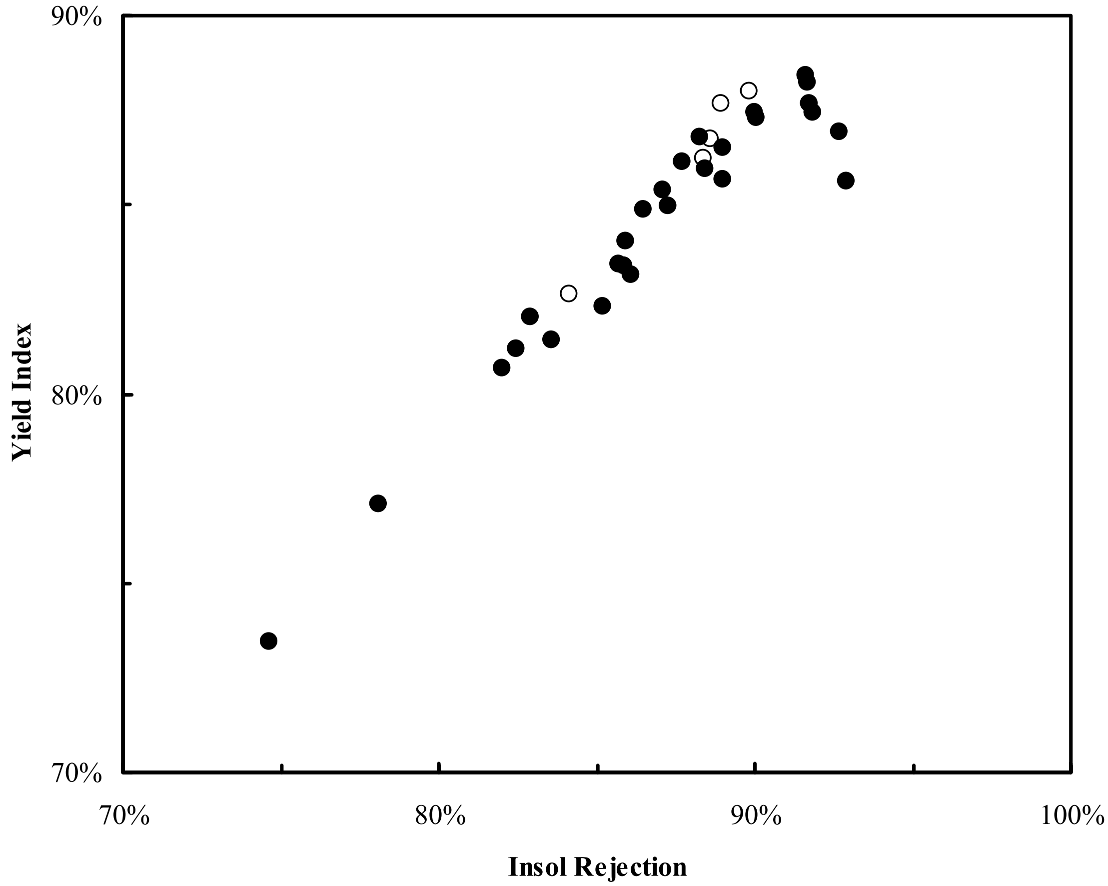
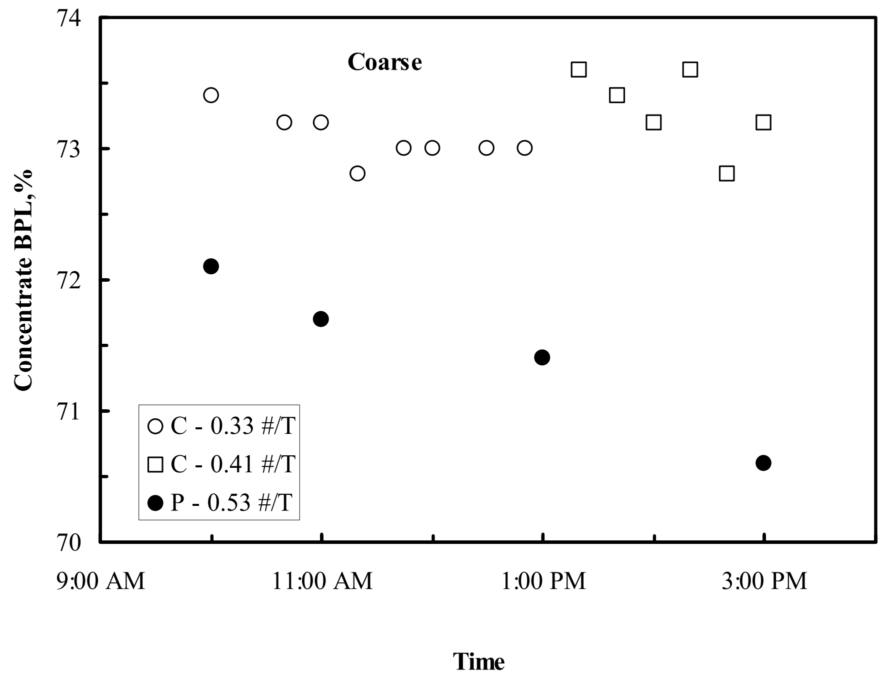


Figure 8. Loci of Separation for Fine Rougher Concentrate Flotation in the DEC and Plant (Closed and Open Symbols Are for Plant and DEC Unit, Respectively).

Coarse Amine Feed

As indicated earlier, the optimum amine dosage determined for the coarse rougher concentrate during the preliminary studies was quite high, most likely due to the dilute nature of the feed. With the implementation of changes to the feed arrangement, higher solids feed rate were introduced into the column. Figure 9 shows a real-time comparison of the concentrate and tailings grade from the DEC and plant operations during the test period. While the amine dosage in the plant was 0.53 lb./T, the values employed in the DEC unit were 0.33 lb./T and 0.41 lb./T. For this test, the feed rate to the column was maintained at approximately 3.8 TPH. The results show that the concentrate grade from the DEC unit was consistently higher than that in the plant even though the former was operating at lower amine dosages. For example, while the plant gave concentrates with 72 BPL or lower, the DEC consistently produced concentrates with 73 BPL or higher. While the concentrate grade in the DEC unit did not appear to change with amine dosage, the tailings grade increased with amine dosage, thus suggesting that the optimum amine dosage was exceeded. We note that at the lower amine dosage, the plant and DEC tailings grades were comparable. A comparison of the DEC and plant performance is shown in Figure 10. Our results show that the BPL recoveries were generally above 98.5%; however, better separation results were obtained in the DEC, especially when it was operating at the lower amine dosage.



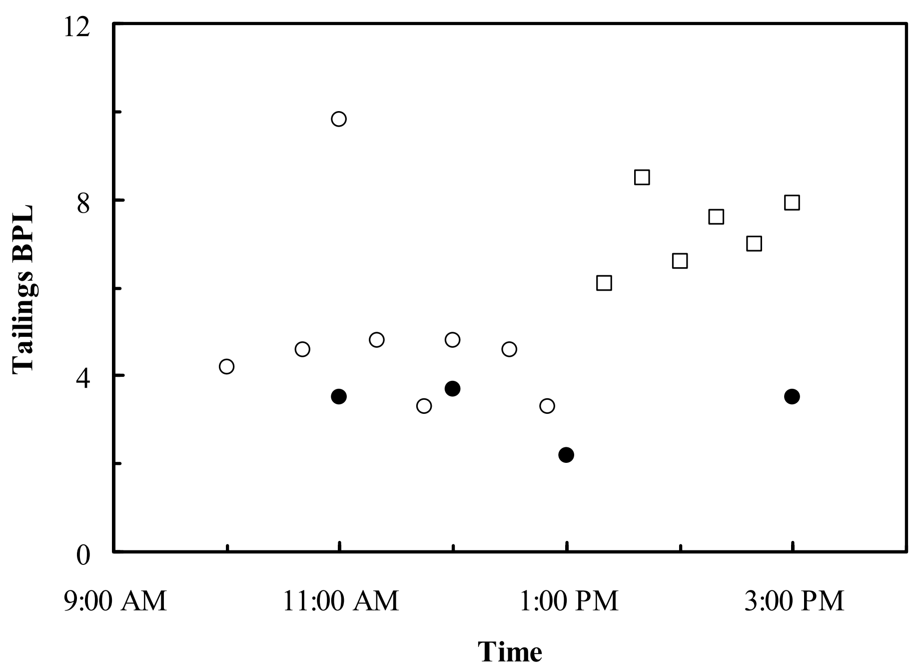


Figure 9. Effect of Amine Dosage on Coarse Rougher Concentrate Flotation for Run 1 (Open and Closed Symbols Are for DEC and Plant Grades, Respectively).

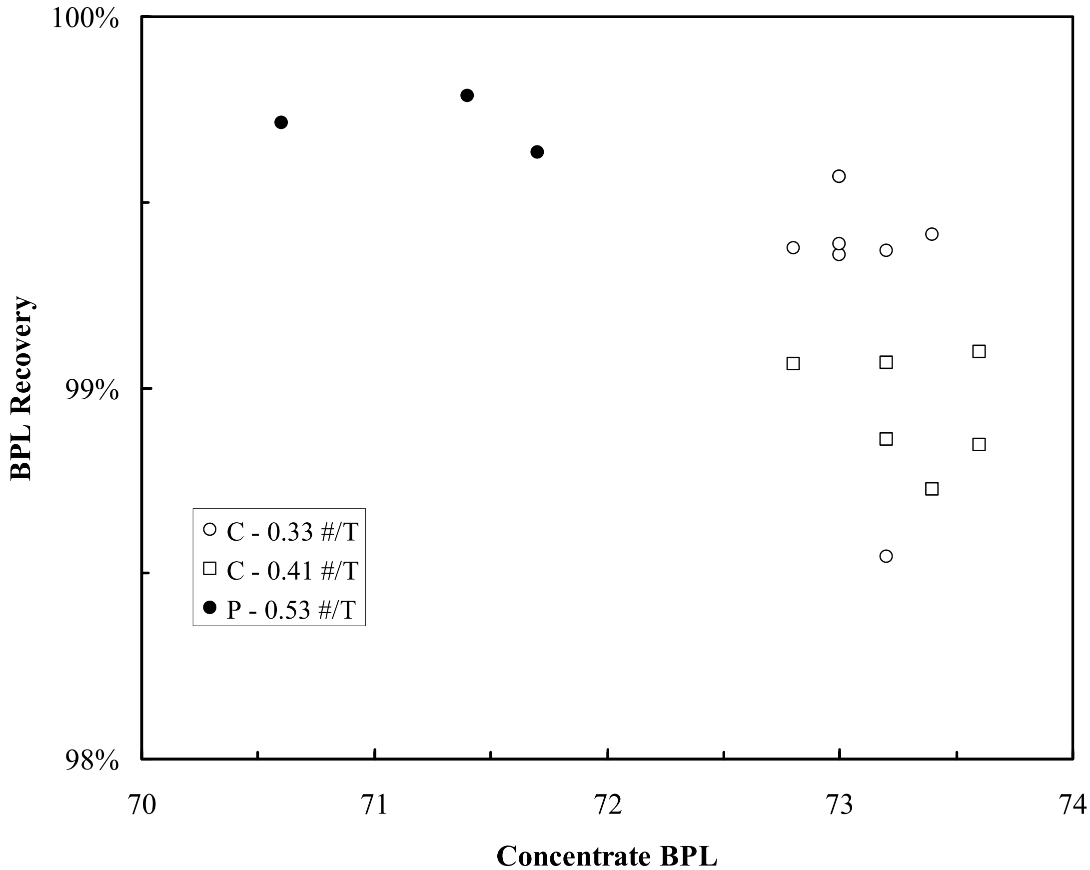


Figure 10. Comparison of DEC and Plant Grade-Recovery Profiles (Open and Closed Symbols Are for DEC and Plant Profiles, Respectively).

Another run was carried out on a different day to validate the results in Figures 9 and 10. As noted above, it appears that the optimum amine dosage was exceeded at 0.41 lb./T. Thus, in order to determine the optimum amine dosage for coarse rougher flotation, we lowered the amine feed rate during this test. For this test the solids feed rate to the DEC unit was approximately 3.4 TPH while the amine dosage ranged from 0.27-0.36 lb./T. By contrast, the plant amine dosage was approximately 0.69 lb./T. A real time comparison of the products from the DEC and plant is shown in Figure 11.

At the start of the test, the plant concentrate was below 70 BPL while the column consistently produced concentrates above 70 BPL. With time (and possibly increasing feed grade), the plant concentrate increased to the same levels found in the column. Figure 12 shows a comparison of the grade-recovery profile for the coarse rougher flotation. For the calculation of the BPL recoveries, we used the composite feed sample. The results show that the BPL recoveries in the DEC were in the same range as the plant.

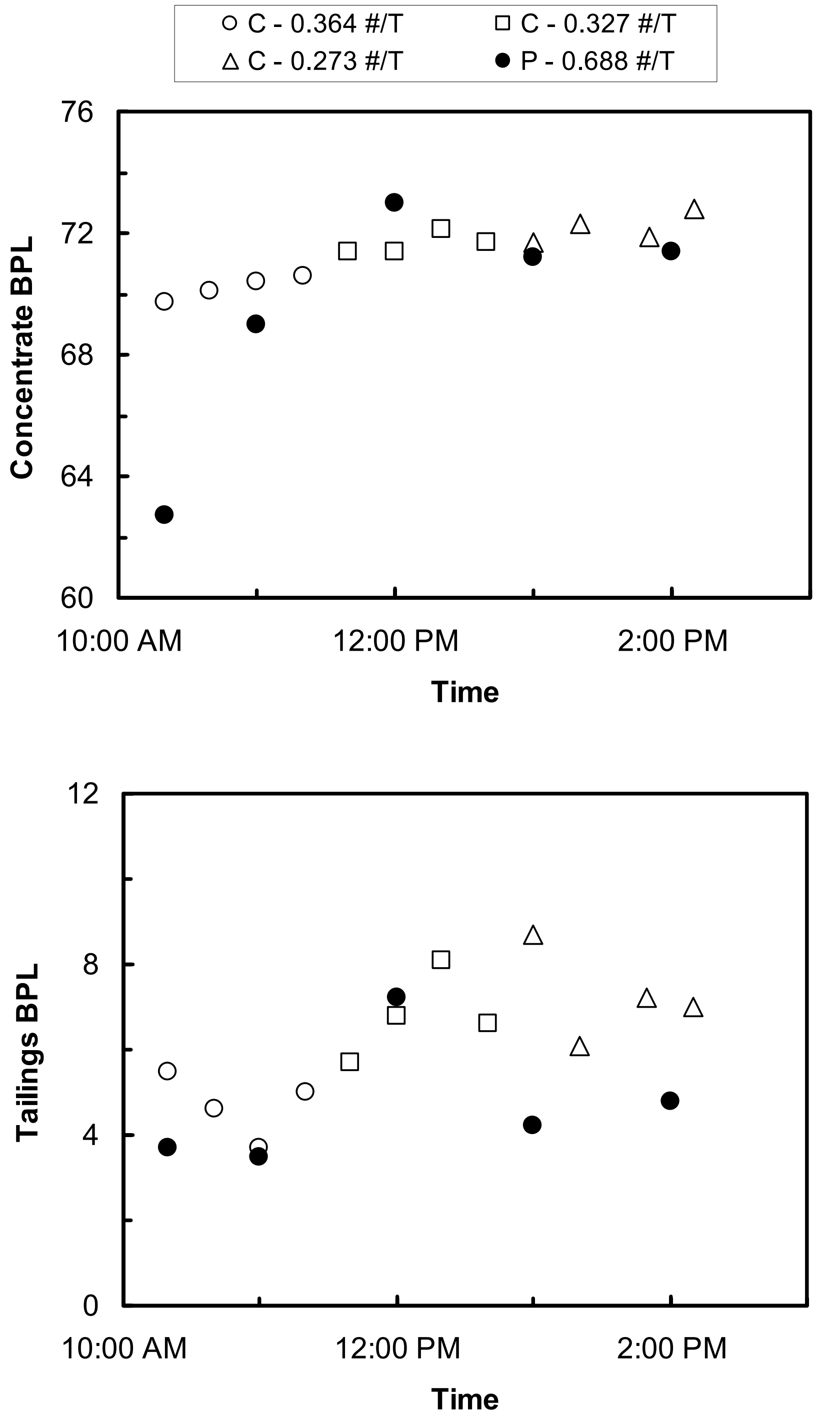


Figure 11. Effect of Amine Dosage on Coarse Rougher Concentrate Flotation (Run 2).

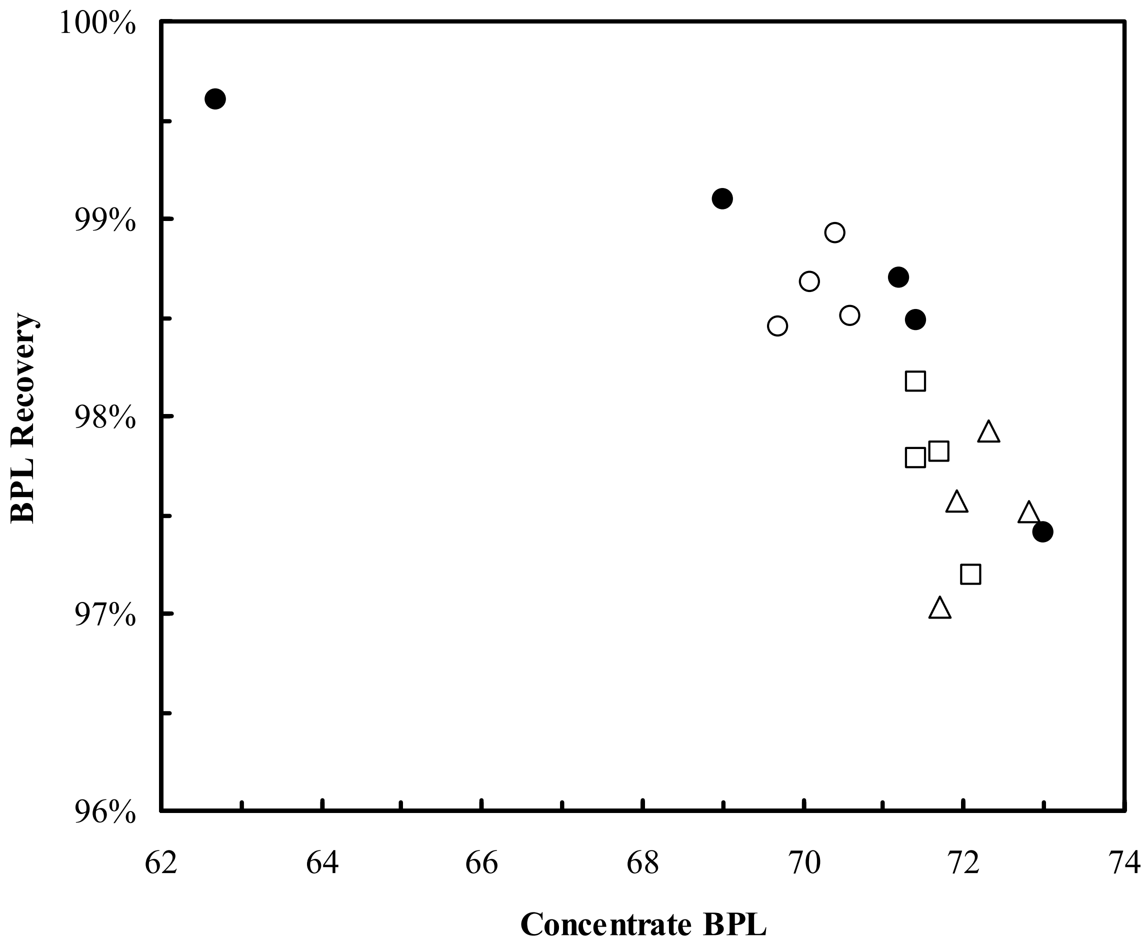


Figure 12. Grade-Recovery Profiles for Run 2 (Symbols Are the Same as in Figure 11).

Overall, the concentrate grades were higher in the DEC unit even though the amine dosages were only 40-53% of those employed in the plant. The tailings BPL were lowest at amine dosage of 0.36 lb./T and were comparable to those in the plant. These results as well as those in Figures 9 and 10 appear to confirm that the optimum amine dosage for coarse rougher flotation in the range of 0.33-0.42 lb/T. A summary of the coarse rougher flotation results in the column cell is given in Table 3.

Table 3. Summary of the Results on Coarse Rougher Concentrate Flotation at Plant A.

Run	Unit	Amine Dosage, #/T	Concentrate		Tailings BPL	Recovery, %
			BPL	Insol		
1A	DEC	0.33	73.1 ± 0.18	3.6 ± 0.23	4.9 ± 2.06	99.3 ± 0.33
	Plant	0.53	71.9 ± 0.28	6.2 ± 0.42	3.6 ± 0.14	99.6
1B	DEC	0.41	73.3 ± 0.30	3.0 ± 0.23	7.3 ± 0.88	98.9 ± 0.15
	Plant	0.53	71.0 ± 0.57	6.6 ± 0.14	2.9 ± 0.92	99.8
2A	DEC	0.36	70.2 ± 0.39	5.2 ± 0.26	4.7 ± 0.76	99.3 ± 0.10
	Plant	0.69	65.9 ± 4.45	11.9 ± 5.73	3.6 ± 0.14	99.9 ± 0.39
2B	DEC	0.33	71.7 ± 0.33	4.3 ± 0.25	6.8 ± 0.99	98.8 ± 0.24
	Plant	0.69	73.0	4.1	7.2	98.5
2C	DEC	0.27	72.2 ± 0.49	4.9 ± 0.21	7.3 ± 1.08	98.6 ± 0.19
	Plant	0.69	71.3 ± 0.14	5.3 ± 0.35	4.5 ± 0.42	99.3 ± 0.09

The results in Table 3 show that for coarse amine flotation in a DEC unit, lower collector dosages are needed. Our results show that in both plant and DEC flotation of coarse amine feed, low collector dosages are needed. A comparison of the amine dosages reveals that only 40-60% of the plant quantities are needed in the DEC unit. It is evident from the foregoing that the deployment of DEC units in amine flotation of rougher concentrates will produce significant savings in reagent and maintenance costs.

In order to compare the separation efficiencies of the plant and DEC unit in processing coarse rougher concentrate, we have plotted the yield index as a function of insol rejection and the results are shown in Figure 13. In general, the results show that the performance of the column was superior to that of the plant even though it was operating at 40-60% of the plant amine dosages. A comparison of the plant results shows that the separation efficiencies increased with amine dosage. For example in Run 2 where the amine dosage was 0.69 lb./T, the performance was comparable to that observed with the DEC. However, as noted earlier, the DEC performance was accomplished at much lower amine dosages.

Figure 14 shows a comparison of the separation results for amine flotation of fine and coarse rougher concentrates. While the loci of the separation generally lie on the same line, better separation efficiencies were observed with the fine feed. This observation is attributed to improved liberation of the silica at finer sizes.

For coarse rougher flotation, the maximum feed rate tested at Plant A was 3.7 TPH. If we assume that the capacity of the pilot unit is approximately 4 TPH, it would suggest that a full-scale DEC unit (10-ft diameter) could successfully process 100 TPH of coarse rougher concentrate. It is thus recommended that Plant A consider replacing their amine circuit with column cells. A retrofitting of the plant is expected to increase the plant capacity without expensive structural modifications.

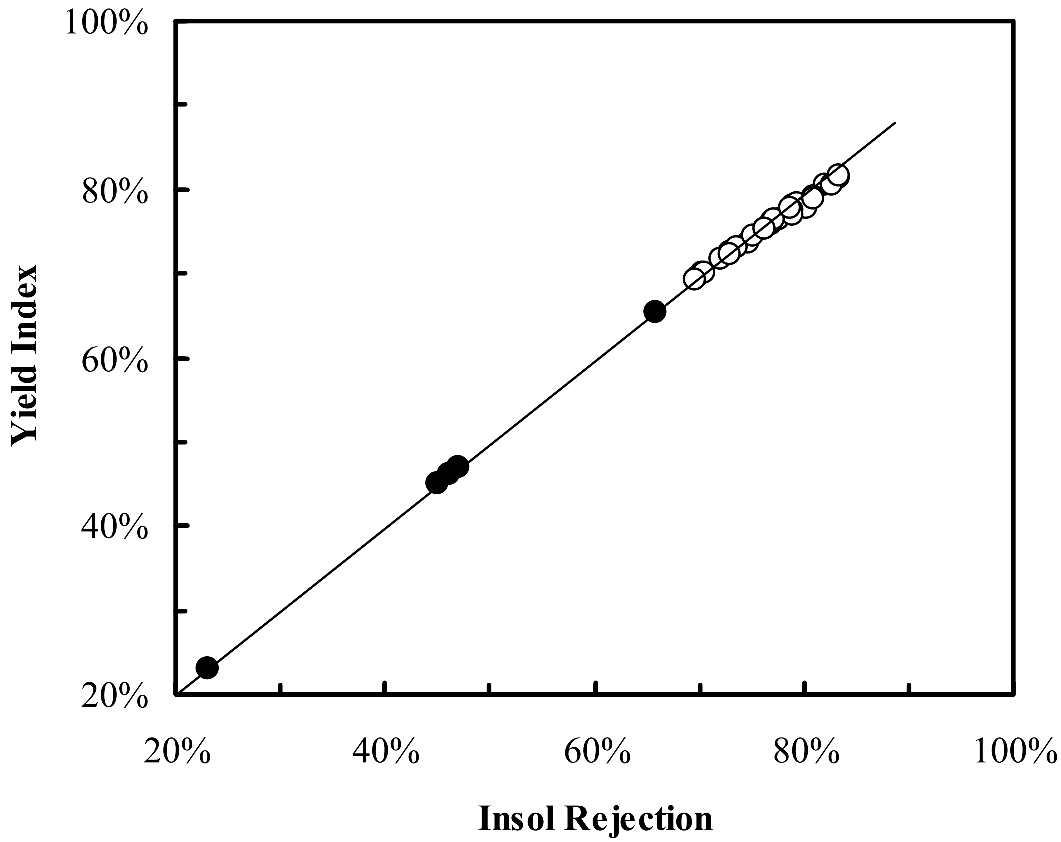


Figure 13. Loci of Separation for Coarse Rougher Concentrate Flotation in the DEC and Plant (Open and Closed Symbols Are for DEC and Plant Performances, Respectively).

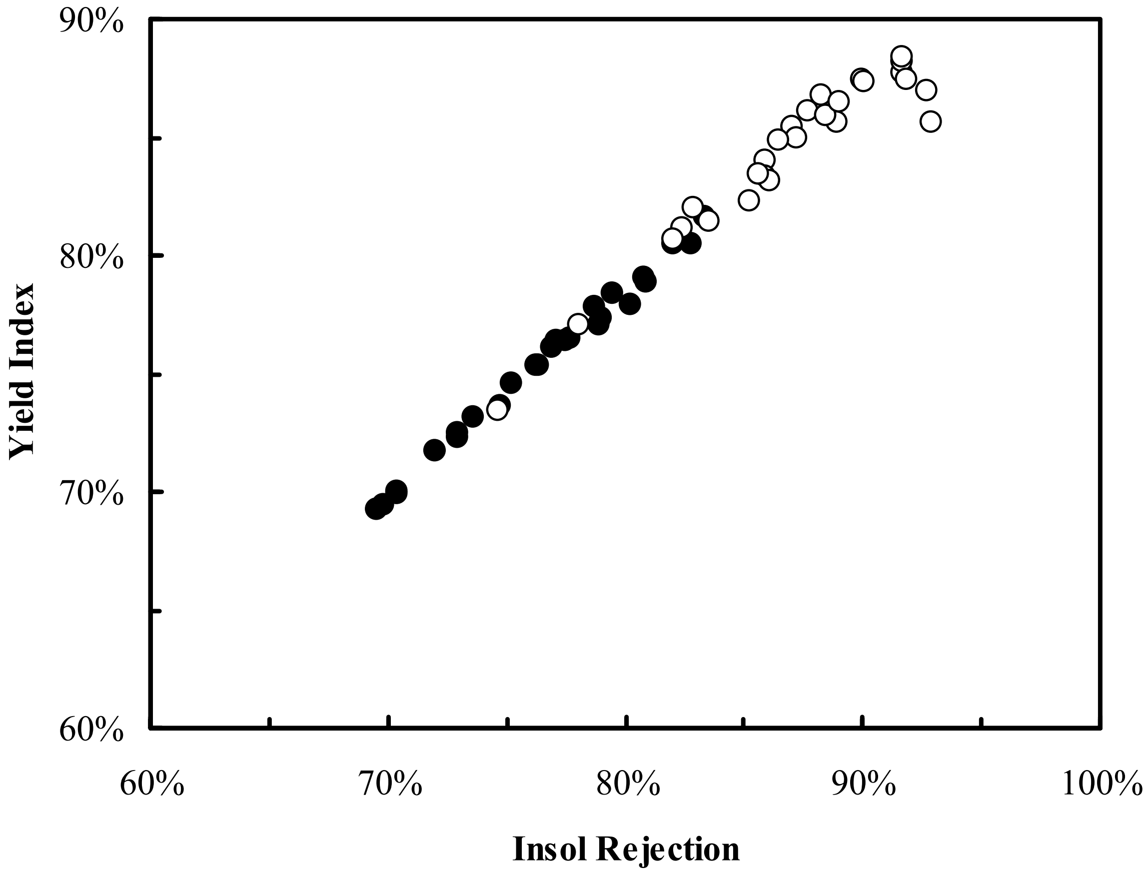


Figure 14. Loci of Separation for Fine and Coarse Rougher Concentrates Flotation in the DEC Unit (Open and Closed Symbols Are for Fine and Coarse Rougher Flotation, Respectively).

PLANT B

Preliminary Evaluation of Column Flotation

Preliminary experiments were carried out to determine the optimum amine dosage. We considered this to be important because of differences in mine location and plant operations including the type of amine in use. Figure 15 shows the variation of concentrate and tailings grade with amine dosage. We initiated the study at a higher amine dosage because of the high insol content of the feed. Initially, the concentrate grade increased with amine dosage. However, there was a sharp drop in concentrate grade when the amine dosage was increase from 0.83 lb./T to 0.92 lb./T. While it is possible that the drop may be due to changes in feed characteristics, we cannot be certain since the feed was not sampled. Since further increases in the amine dosage resulted in improved concentrate grade and the phosphate content of the tailings stream were very low, we can only surmise that this was indeed the case. From the preliminary studies, it was not possible to establish the optimum amine dosage at this site, thus we resorted to the steady state studies to determine the optimum amine dosage.

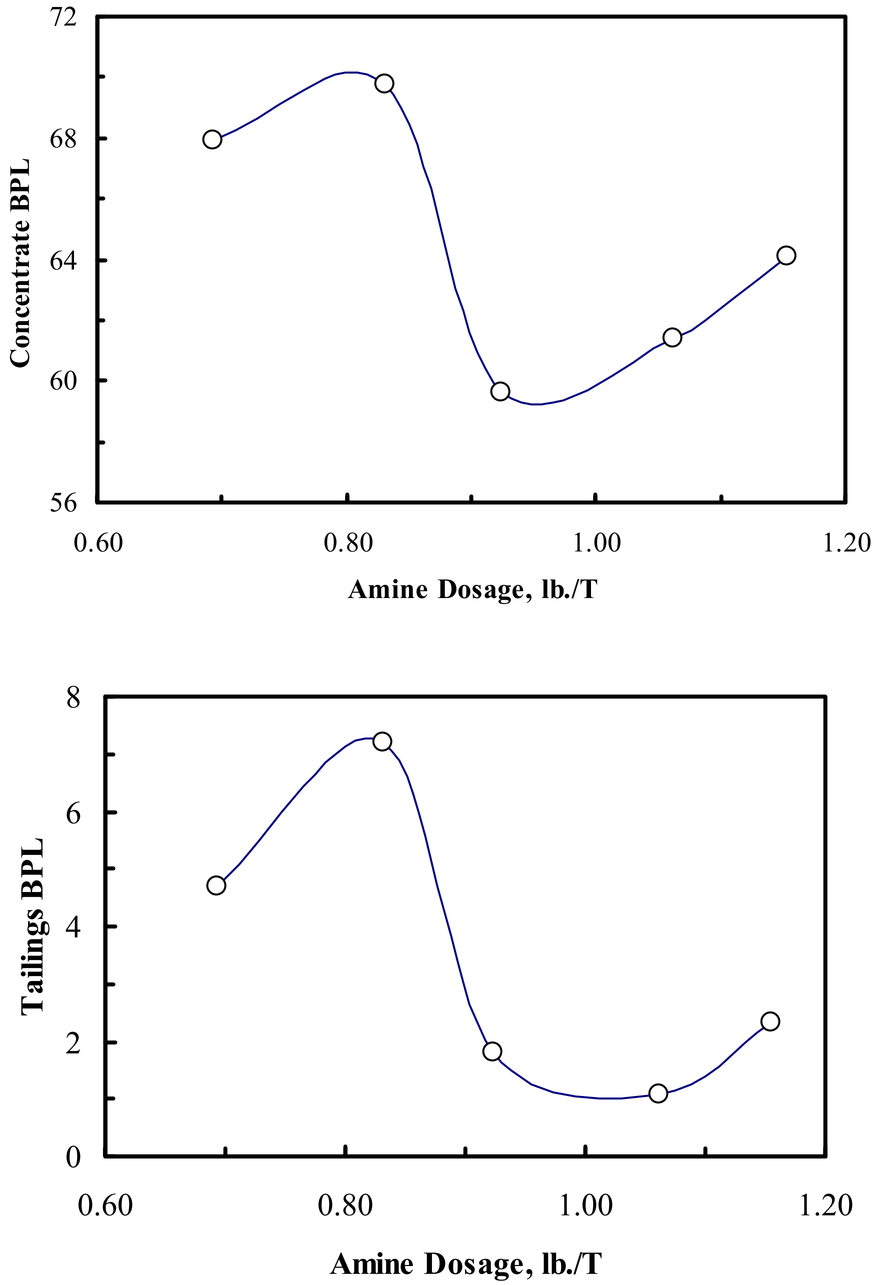


Figure 15. Effect of Amine Dosage on Fine Rougher Concentrate Flotation.

Steady State Studies

The test conditions at Plant B were also ideal (steady flow rate) and may be taken to be representative of amine flotation of rougher phosphate concentrate in a column cell. For the initial steady state test, we fixed the amine dosage at the mid-point (0.92 lb./T) of the dosage test presented above. Figure 16 shows a real-time comparison of the concentrate and tailings grades from the plant and DEC unit. Since we were able to sample the feed, its grade is also provided for comparison. During the test period lasting 2.67 hours, the measured feed characteristics varied from 41.4 BPL and Insol of 42.6 to 54.2 BPL and Insol of 26.9.

The results in Figure 16 show that while the feed grade varied throughout the test period, the DEC unit was able to maintain a fairly steady product grade. Furthermore, the performance of the DEC unit was maintained even when the feed grade dropped to about 40 BPL. By contrast the plant performance varied with the feed grade and was consistently below 70 BPL. Both the plant and DEC tailings grade were consistently below 5 BPL, with the values in the plant being slightly lower. The present results highlight one of the difficulties of in-plant test work where the feed grade changes with time. It is therefore important that any meaningful performance comparisons must be done on a real-time basis. While the BPL recoveries from the plant and the DEC units were comparable, the separation was more efficient in the latter (see Figure 17).

In general, for the same recovery, the concentrate grade was higher in the DEC unit. However, we note that the amine dosage employed in our column tests was 0.92 lb/T while that in the plant was only 0.54 lb/T. For this reason, it is not clear if the differences in the grade-recovery characteristics of the DEC and plant are due to the amine dosages employed.

In order to evaluate if amine dosage differences were responsible for the results observed in Figures 16 and 17, we carried another series of tests in which the amine dosage was varied. Figure 18 shows a real-time comparison of the DEC performance at different amine dosage rates with that of the plant. For these tests, solids feed rate to the DEC unit was approximately 2.32 TPH while the amine dosages were 0.60 lb./T and 0.72 lb./T for the DEC and 0.77 lb./T for the plant.

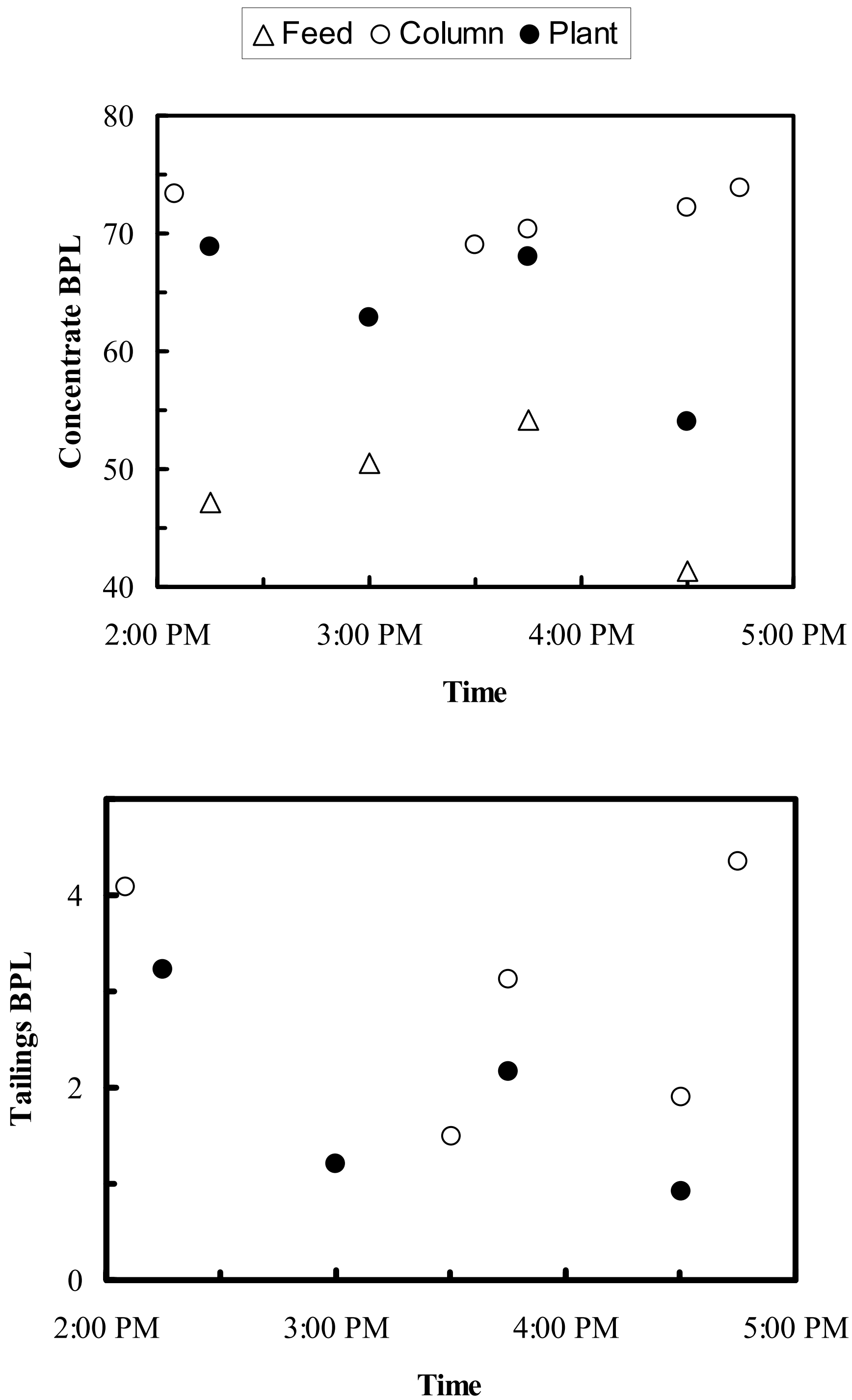


Figure 16. Real-Time Comparison of DEC and Plant Performance for Run 1.

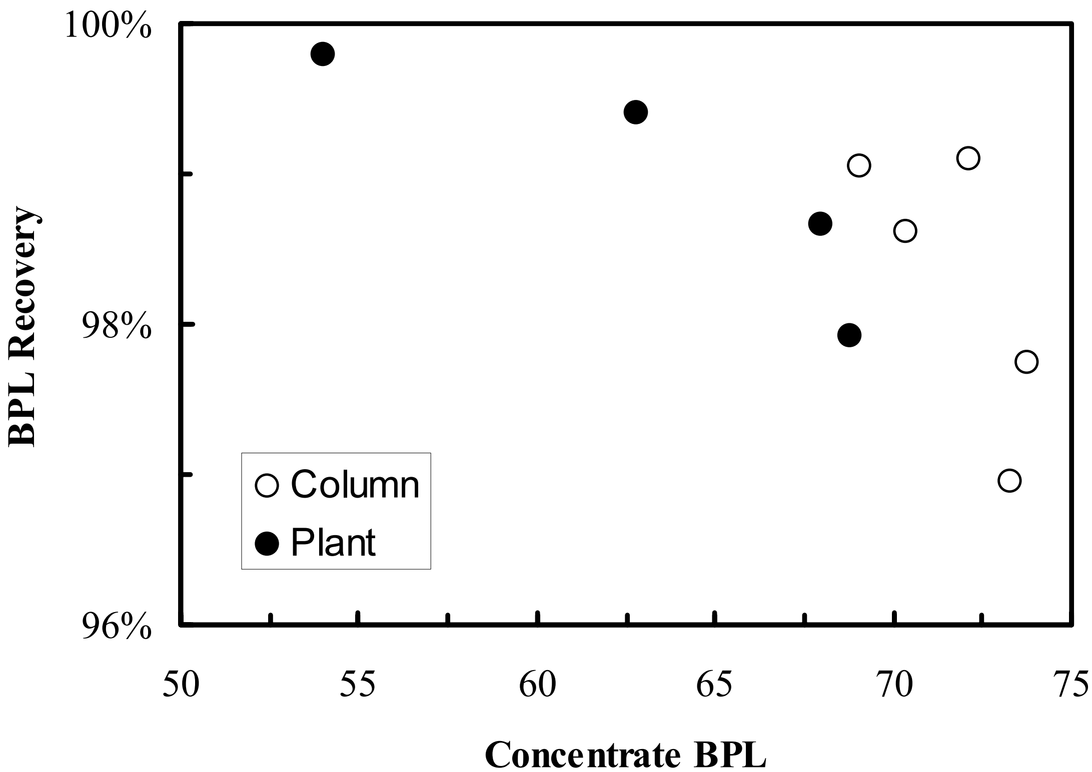


Figure 17. Comparison of Grade-Recovery Profile for Run 1.

The results show that the concentrate grade for both the column and plant mirrored that of the feed, however, the effect of decreasing feed grade was more pronounced with the plant results. Even though the DEC unit was operating at lower amine dosages, the concentrate from the unit was consistently above 70 BPL. By contrast, the plant was only capable of producing concentrates with 70 BPL or higher when the feed grade was approximately 60 BPL. The tailings grade for the DEC and plant were comparable and were generally in the range of 4-7 BPL.

Figure 19 shows a comparison of the grade-recovery profiles for the DEC and plant operations. In general, the performance of the column was consistently better than that of the plant. With feed containing 44-52 BPL and insol of 30-42%, only the DEC unit was able to produce a concentrate of 70 BPL or higher at BPL recoveries of 94-97%.

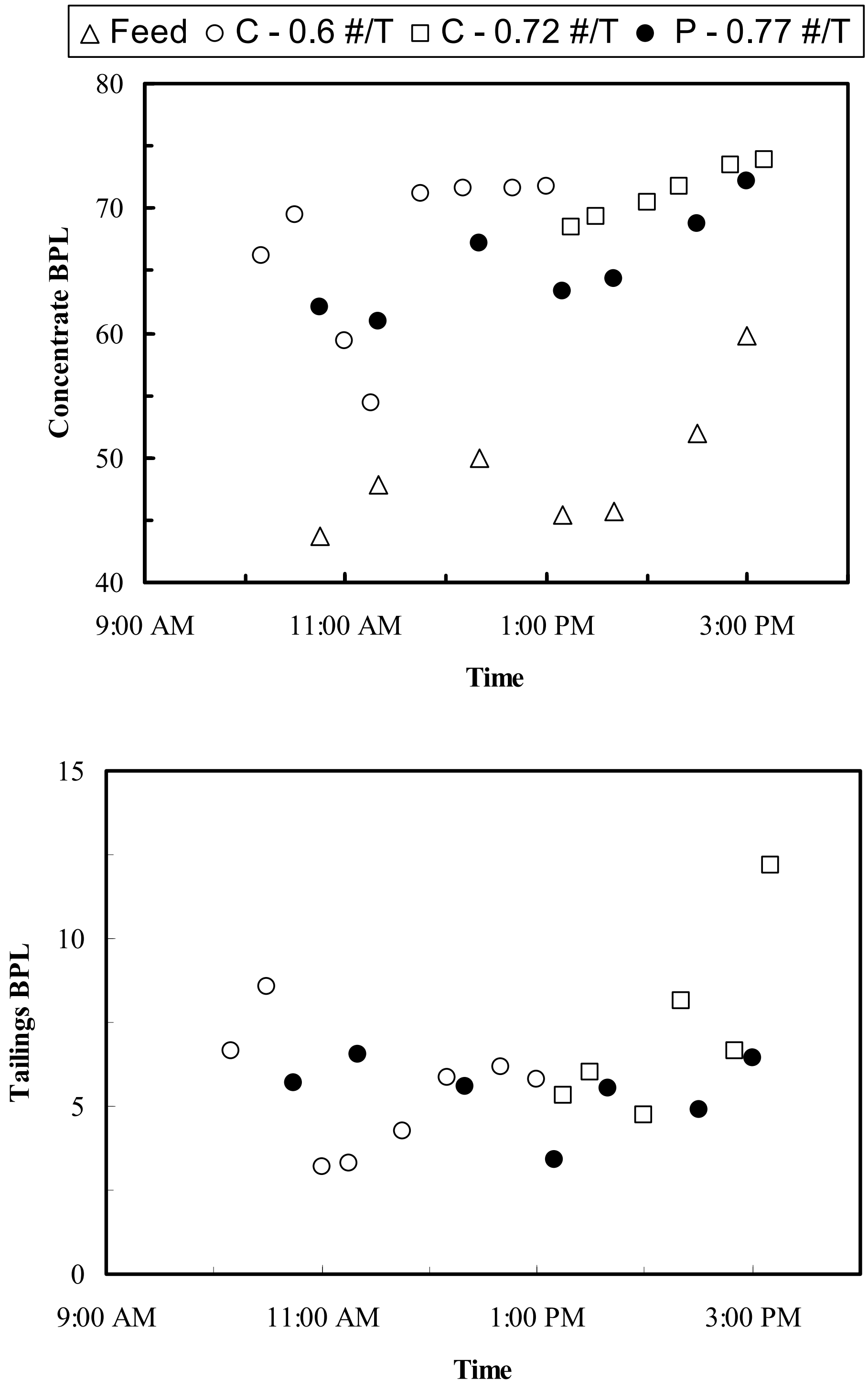


Figure 18. Effect of Amine Dosage on Fine Rougher Concentrate Flotation (Run 2).

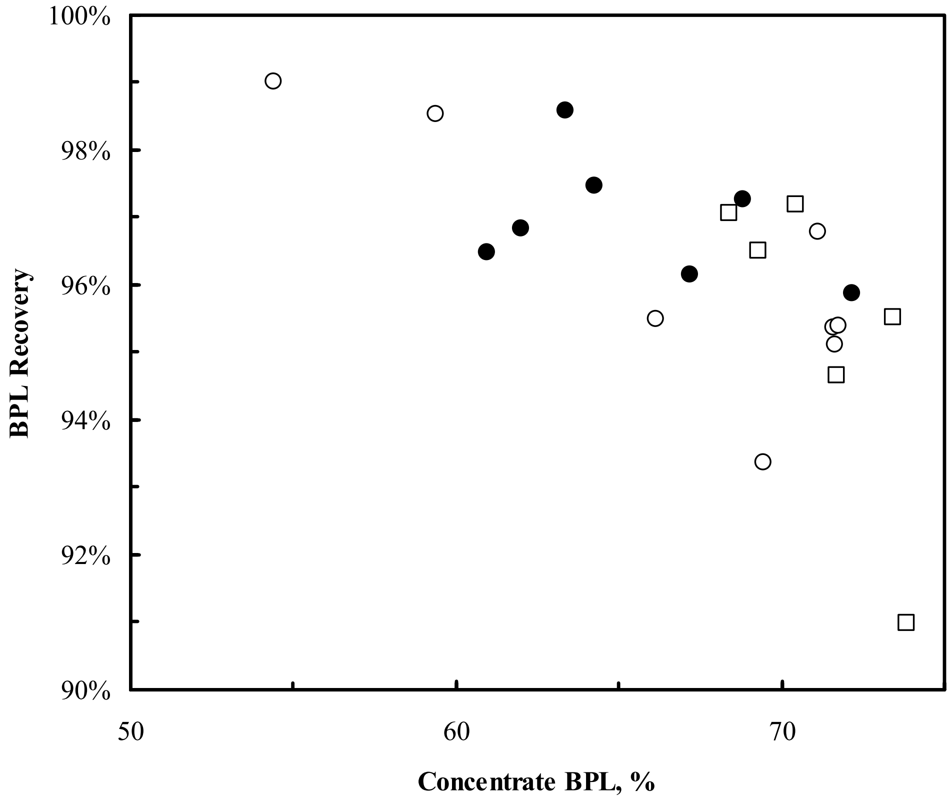


Figure 19. Grade-Recovery Profiles for Run 2 (Symbols Same as in Figure 18).

Given the excellent performance observed with the DEC unit, the management at Plant B requested that we evaluate the performance of the DEC unit using recycled process water instead of the deep well water that is currently used in the cleaner circuit. While the Florida phosphate industry has made substantial reductions in water usage, the on-going draught has necessitated the evaluation of alternatives that reduce fresh water requirements. Since the water characteristics can influence flotation performance, we carried out preliminary analysis on the process and deep well water samples at Plant B and the results are shown in Table 4.

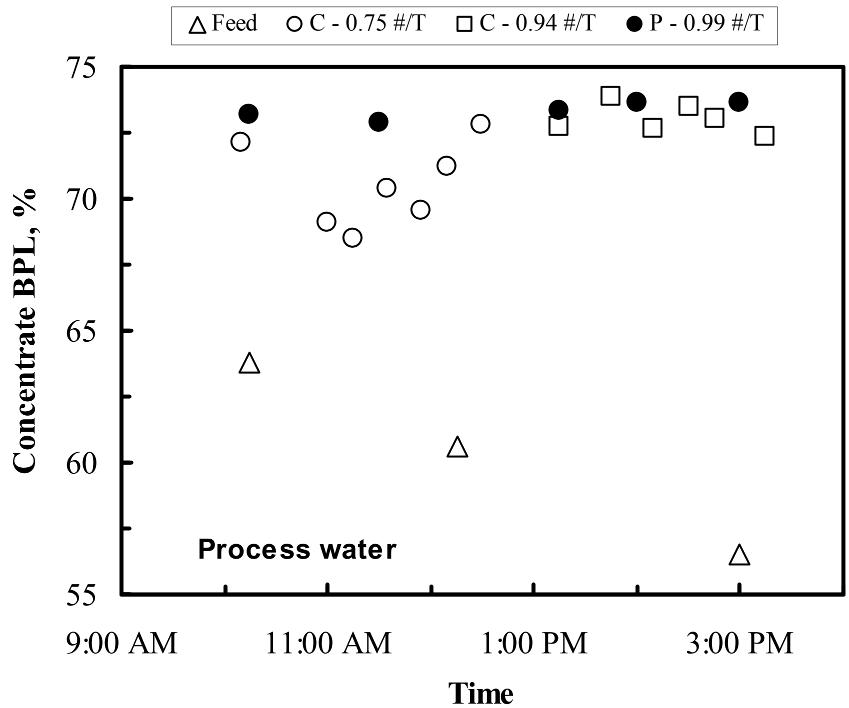
Table 4. Characteristics of Deep Well and Process Water at Plant B.

Parameter	Water Source	
	Deep Well	Process
pH	7.8	9.2
Turbidity, NTU	0.48	19.2
Temperature, ^oF	73.9	82.0

The parameters selected were those that can be easily measured by a plant operator without recourse to extensive laboratory equipment and focuses on those parameters that can influence the separation. For example, the turbidity provides a measure of the suspended solids content of the water (the slimes content of the water) while the pH and temperature can influence the interaction of the collector with the surface. A comparison of the water characteristics shows that the process water was greenish in appearance, had more suspended particles (slimes), higher pH and temperature. It was thus expected that the slimes would negatively impact the results of the separation. Figure 20 shows a real-time comparison of the product and tailings grades from the plant and column tests. For this test, solids feed rate was 1.99 TPH with amine dosage rates of 0.75 lb./T and 0.94 lb./T. By comparison, the plant amine dosage was 0.99 lb./T.

The results obtained show that when the amine dosage was 0.75 lb./T, the product grade in the DEC unit was lower than that in the plant. However, when the amine dosage was comparable the use of process water in the DEC unit had no effect on the concentrate grade. We note that the plant was able to produce concentrate with high BPL but this was only possible at the expense of high phosphate losses to the tailings stream. For example, when the amine dosages were comparable, the DEC unit produced tailings that were consistently lower than those from the plant. In general, the flotation results with the process water were more sensitive to the amine dosage. A comparison of the grade-recovery profile shows that for the same concentrate grade, the BPL recoveries were about 4-5% higher in the DEC unit (see Figure 21). Since the plant was operating with deep well water in the cleaner circuit, it shows that the performance of the DEC unit is superior to that of the plant. The results show that the DEC unit is very robust and its performance is not significantly impacted by the presence of slimes. This effect is attributed to operation of the DEC. For example, we note that in its operation, the process water comes in contact with an already conditioned feed, which is not the case in a conventional cell. By contrast, if process water is used in a conventional cell as dilution water, the slimes contained in it would have a greater opportunity to interact with the collector, resulting in high consumption rates, unless the amine is added ahead of the first cell. In the light of this, it is suggested that the DEC is an ideal unit for amine flotation, especially where slimes may be present. These results clearly show the advantage of DEC unit over the conventional cells in use in the plant.

In order to further confirm the efficacy of the DEC unit in amine flotation of rougher concentrate, the amine dosage was fixed while the water source was changed. The initial tests were carried out using process water which was later switched to deep well water. For these tests, the amine dosage in the DEC and plant were 0.60 lb./T and 0.78 lb./T respectively while the solids feed rate and grade to the column were 3.09 TPH and 63.3 BPL respectively. Figure 22 shows a real-time comparison of the feed, concentrate and tailing grades from the DEC unit and plant. As expected, the use of process gave a concentrate with a lower BPL relative to the plant. However, high concentrate grade in the plant was accomplished at the expense of increased phosphate losses.



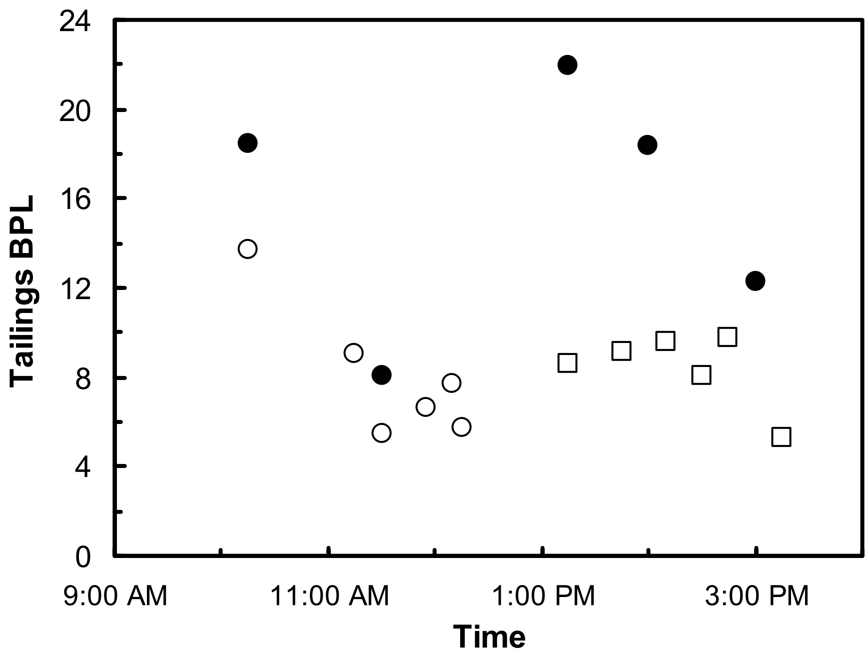


Figure 20. Effect of Process Water on Fine Rougher Concentrate Flotation (Run 3).

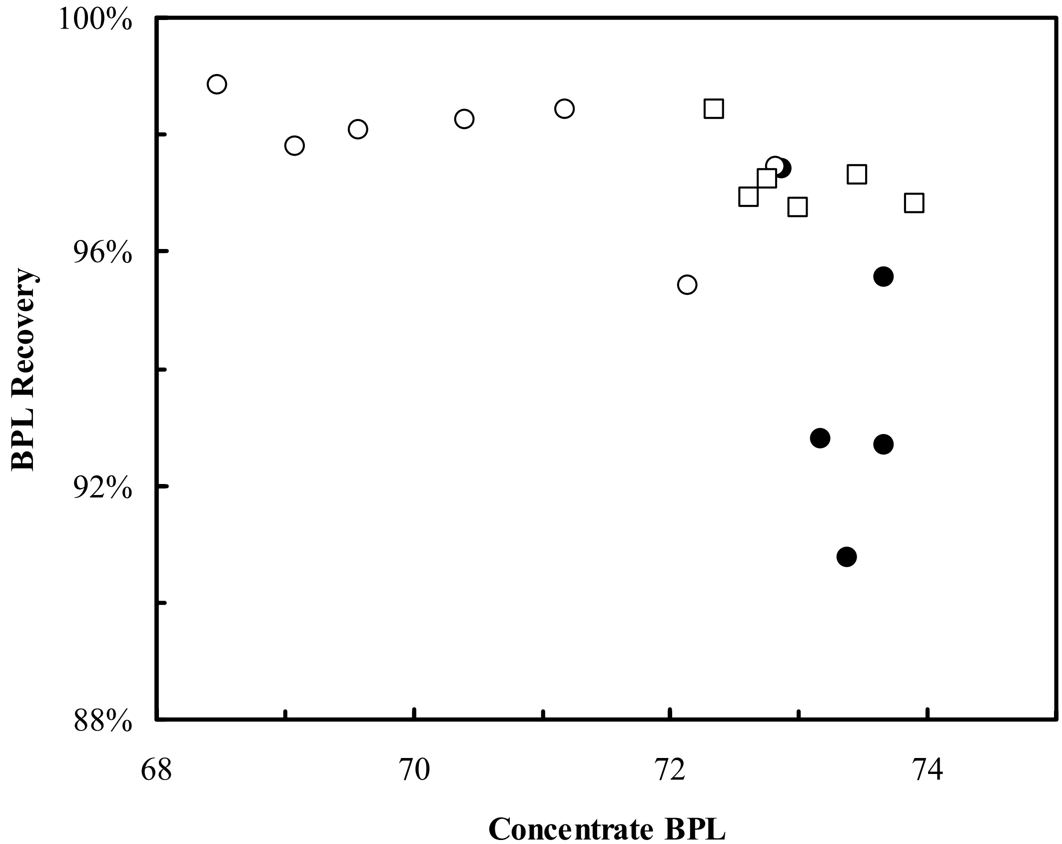


Figure 21. Grade-Recovery Profiles for Run 3.

We note that while the DEC produced a concentrate with over 70 BPL with tailings assaying less than 4 BPL, the plant had tailings of over 13 BPL. However, when the same deep well water source was used, the DEC produced concentrates with higher BPL even though it was operating at a lower amine dosage. For the same concentrate grade, the DEC unit gave slightly higher BPL recoveries (see Figure 23). A summary of the results obtained at Plant B is shown in Table 5.

Our results indicate that for this particular mine, amine dosages of 0.70-1.0 lb/T are required to produce a high BPL product. When the amine dosage was in the range of 0.9-1 lb/T, both the DEC unit and plant produced high grade concentrates (> 71 BPL). The amine dosage determined for this mine is different from that determined in the preliminary studies which were carried out at a different mine. The reason for this observation may be due to either difference in amine type, nature of the feed, and/or process conditions. Our results confirm that process water may be substituted for deep well water in the cleaner circuit.

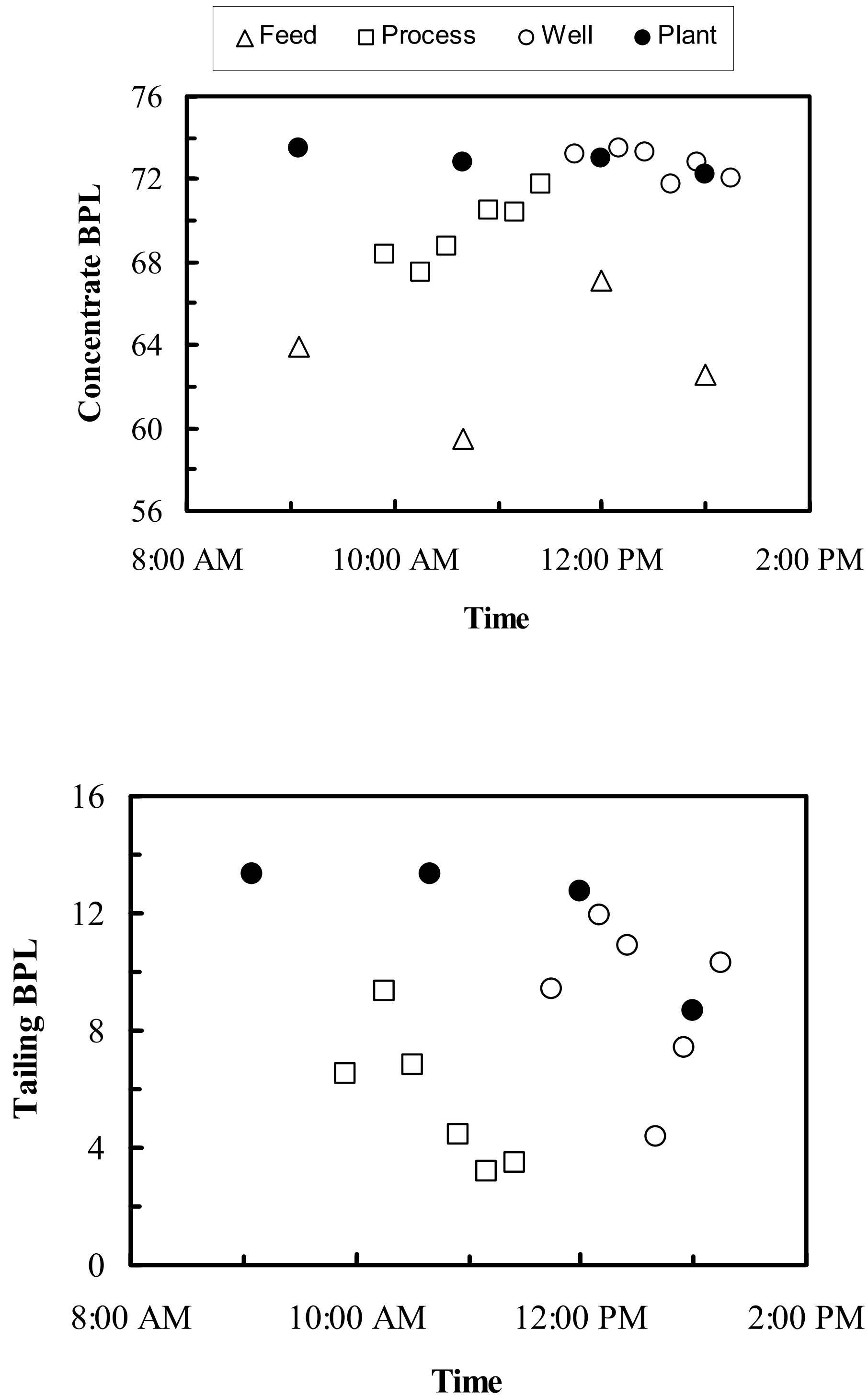


Figure 22. Effect of Water Source on Fine Rougher Concentrate Flotation in DEC Unit (Run 4).

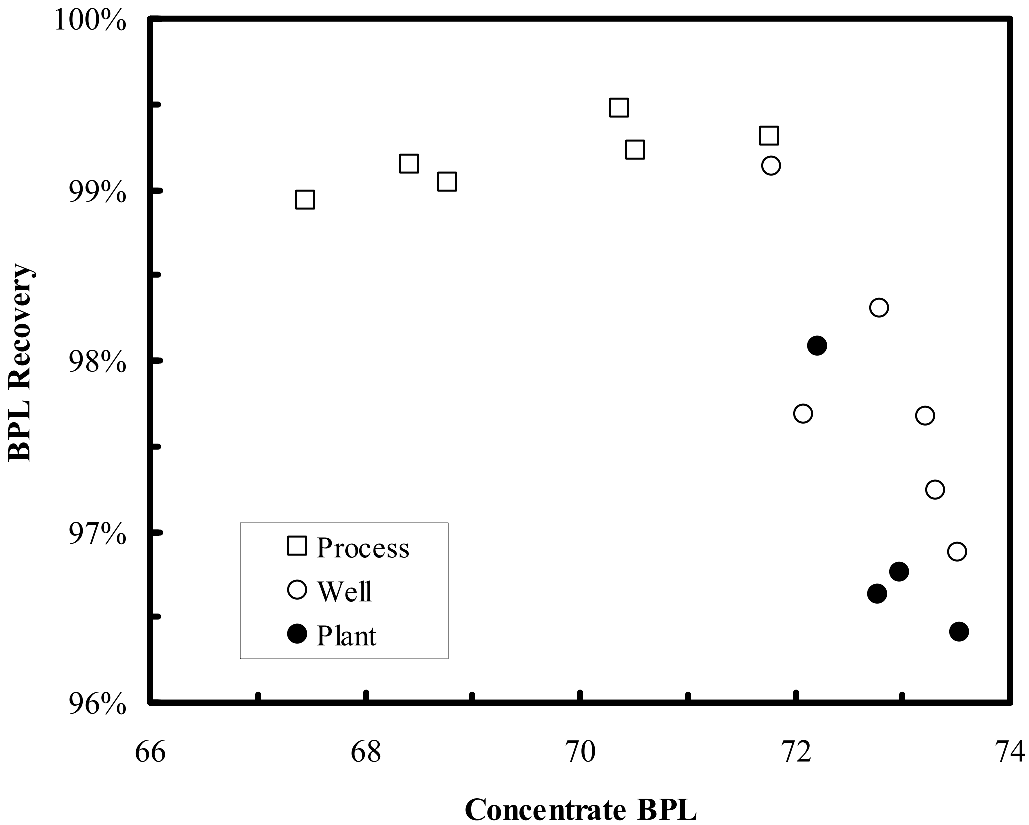


Figure 23. Comparison of Grade-Recovery Profiles for Run 4.

The feed solids concentration to the DEC unit at this site ranged from 30-48 wt. % with an average value of 39 wt. % while the solids feed rate at Plant B ranged from 1.99-3.09 TPH. If we assume most of the infuser water leaves with the concentrate, it would suggest that the solids concentration in the lower cell was of the order of 2 wt. %. The water recovered from the concentrate stream can be a good candidate for recycle to the amine circuit as it is unlikely to contain any slimes.

The results obtained in this study show that column cells may be successfully applied in amine flotation of fine rougher concentrate at Plant B. For comparable concentrate grade, the DEC unit gave higher BPL recoveries. Figure 24 shows a comparison of the separation efficiencies from the DEC unit and plant operations. In general, the plant operations were slightly lower than those of the DEC unit. However, when the rougher concentrate was over 50 BPL, the plant operations approached the optimum separation (Insol rejection of 93.5-95%, BPL recovery of 95.5-97%). By contrast, when the rougher concentrate was below 50 BPL, only the DEC unit produced good separation results. The maximum feed solids rate tested at Plant B was approximately 3.1 TPH. Using this value, it is estimated that a full-scale DEC unit will conveniently process about 80 TPH of rougher concentrate at this plant with BPL recoveries of 94-97%.

Table 5. Summary of Test Results at Plant B.

Run	Unit	Amine Dosage, #/T	Concentrate		Tailings BPL	Recovery, %
			BPL	Insol		
1	DEC	0.92	70.5 ± 2.1	7.1 ± 2.4	2.7 ± 1.3	98.3 ± 0.9
	Plant	0.54	63.4 ± 6.8	14.8 ± 9.0	1.9 ± 1.0	98.9 ± 0.8
2A	DEC	0.60	66.9 ± 6.6	10.7 ± 7.3	5.5 ± 1.8	96.3 ± 1.9
	Plant	0.77	63.4 ± 3.4	16.0 ± 4.8	5.9 ± 0.5	96.5 ± 0.3
2B	DEC	0.72	71.2 ± 2.2	5.2 ± 2.8	7.2 ± 2.7	95.3 ± 2.3
	Plant	0.77	67.2 ± 4.1	10.8 ± 5.3	5.1 ± 1.3	97.3 ± 1.1
3A	DEC*	0.75	70.5 ± 1.6	6.6 ± 1.8	8.0 ± 2.8	97.4 ± 1.3
	Plant	0.99	73.0 ± 0.2	3.9 ± 0.5	13.3 ± 7.3	94.4 ± 3.7
3B	DEC*	0.94	73.0 ± 0.6	3.7 ± 0.6	8.4 ± 1.7	96.9 ± 0.7
	Plant	0.99	73.6 ± 0.2	2. 9 ± 0.1	17.5 ± 4.9	92.1 ± 2.7
4A	DEC*	0.60	69.6 ± 1.6	7.8 ± 2.7	5.6 ± 2.4	98.8 ± 0.4
	Plant	0.78	73.2 ± 0.5	3.4 ± 0.4	13.1	95.5 ± 0.2
4B	DEC	0.60	72.8 ± 0.8	3.4 ± 1.0	9.0 ± 3.1	97.2 ± 1.1
	Plant	0.78	72.6 ± 0.5	3.4 ± 0.8	10.7 ± 2.9	96.7 ± 1.2

* DEC unit was operating with process water instead of deep well water.

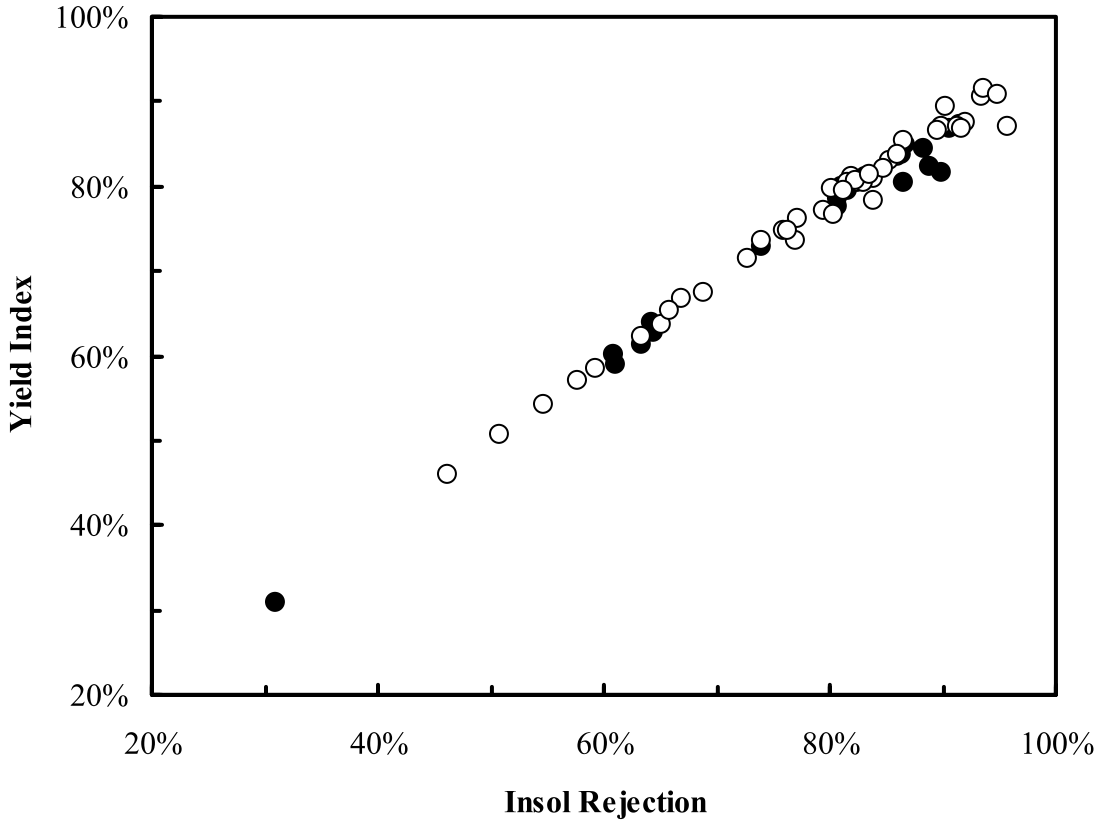


Figure 24. Loci of Fine Rougher Concentrate Separation in the DEC and Plant (Open and Closed Symbols Are for DEC and Plant Data, Respectively).

PLANT C

At this mine location, the turn-around time for samples at this site was quite long. In order to assess the performance of the column, duplicate samples were taken to PhosLab, a private laboratory in Lakeland, FL for analysis. Following the analysis at the private laboratory, the remaining samples were handed to the plant laboratory for analysis. Figure 25 shows a comparison of the analysis from PhosLab with those of the plant laboratory. In general, there is an excellent agreement between the laboratories on both concentrate and tailings samples. Even though there was excellent agreement between the laboratory analyses we decided for the sake of consistency, to use the results provided by the plant.

The steady state studies were initiated at this site. However, the feed extraction point at this location was after amine addition and there was no opportunity to vary the amine dosage. For the entire duration of the test, we witnessed inclement weather conditions and the plant operations were not at steady state. Since the PI was yet to complete the mine safety course, we could not remain at this site for more than four days.

Figure 26 shows the real-time comparison of the DEC unit and plant performance on the fine amine feed. While the test lasted for only an hour (due to inclement weather conditions), it is noted that the DEC unit already attained steady state operating conditions. For example, by the time the second sample was taken after about 30 minutes, the concentrate and tailings grades were already at steady state. In general, the DEC unit produced a higher grade concentrate relative to the plant with lower phosphate losses.

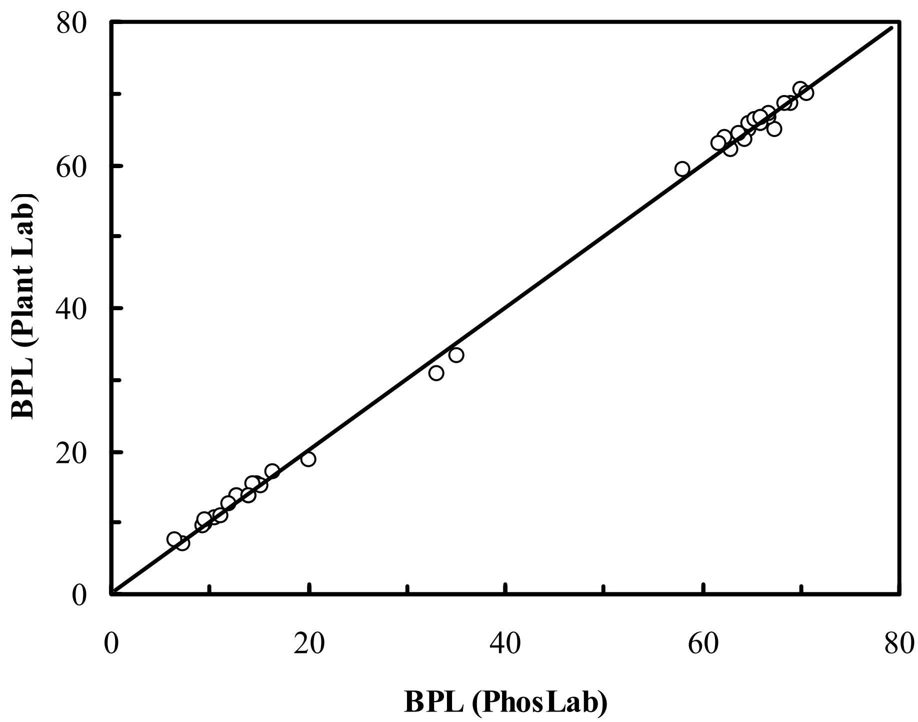


Figure 25. Comparison of Plant and External Laboratory Analyses of Phosphate Samples.

Following the encouraging results from the first test, another run was initiated on the second day and the results are shown in Figure 27. This test started at about 11:00 am, but after forty minutes of operation, we had to shut down because of an approaching lightening storm. While the product from both DEC unit and plant were of acceptable grade, the tailings grades were high. When the plant was restarted after the storm, the concentrate grade from both the DEC and plant decreased over time while the tailings grades were in the range of 9-15%. With the approach of another storm system, the DEC unit had to be shut down. This is evident from the sharp increase in the DEC tailings grade to over 30 BPL.

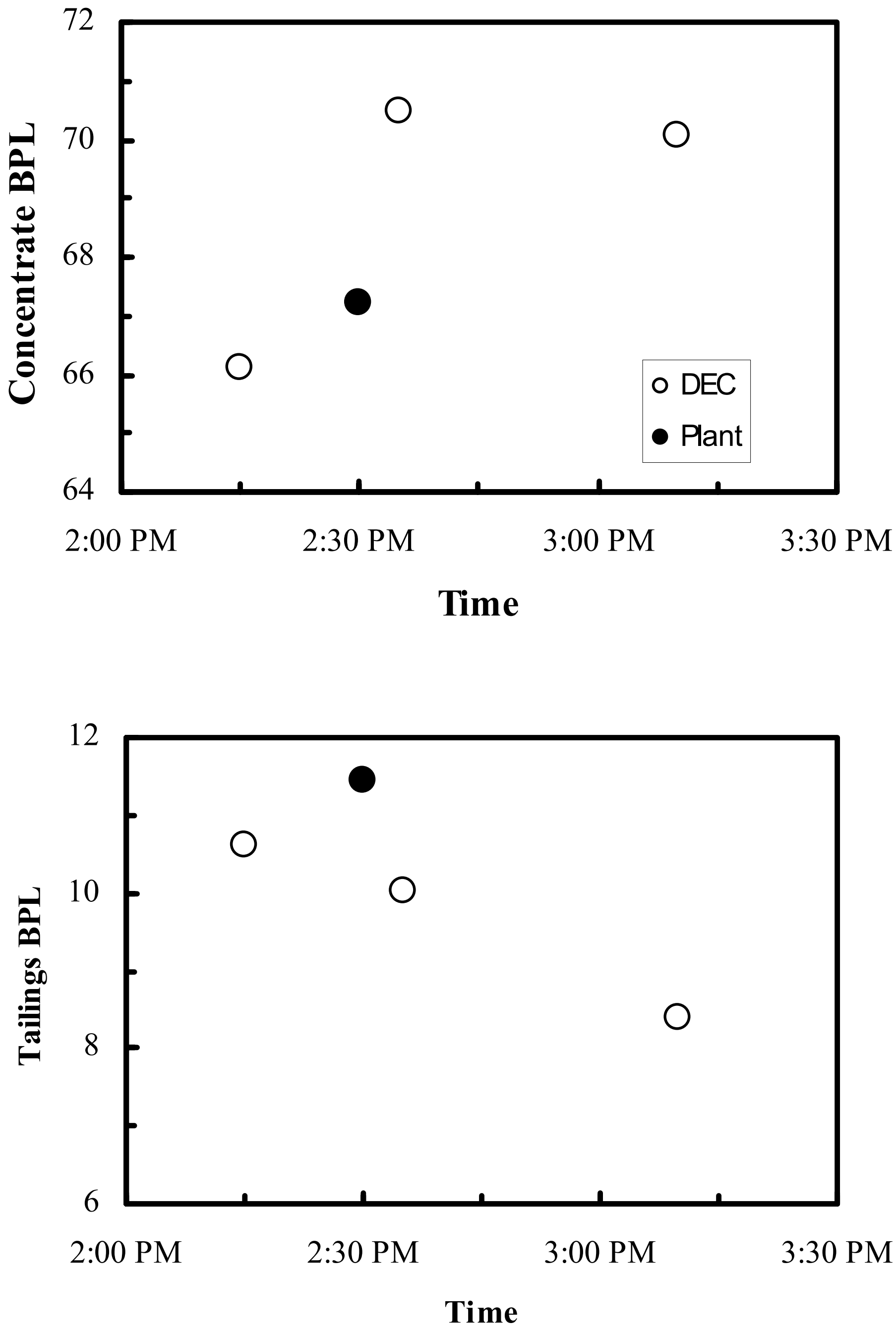


Figure 26. Real-Time Comparison of DEC and Plant Performances on Conditioned Feed (Run 1).

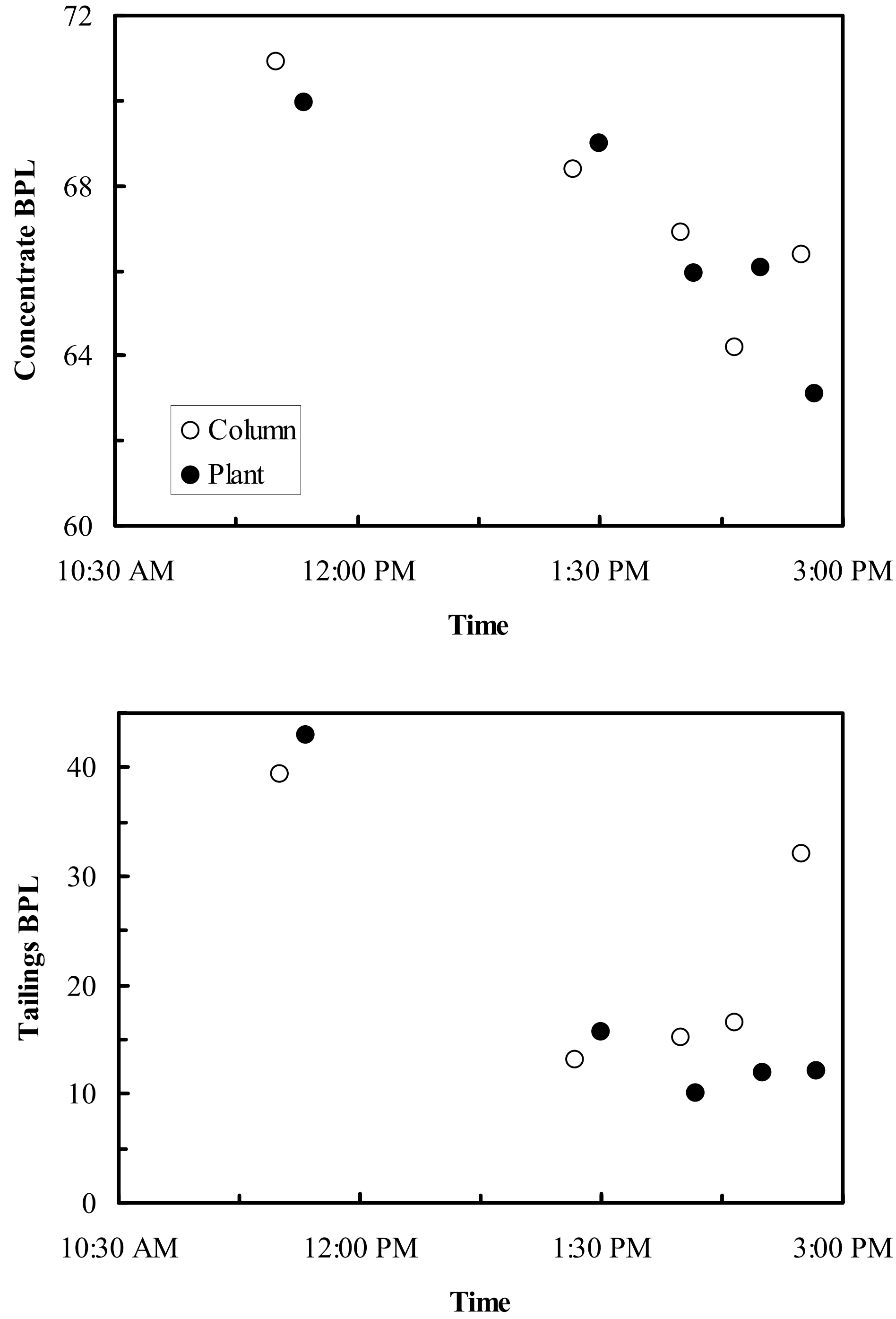


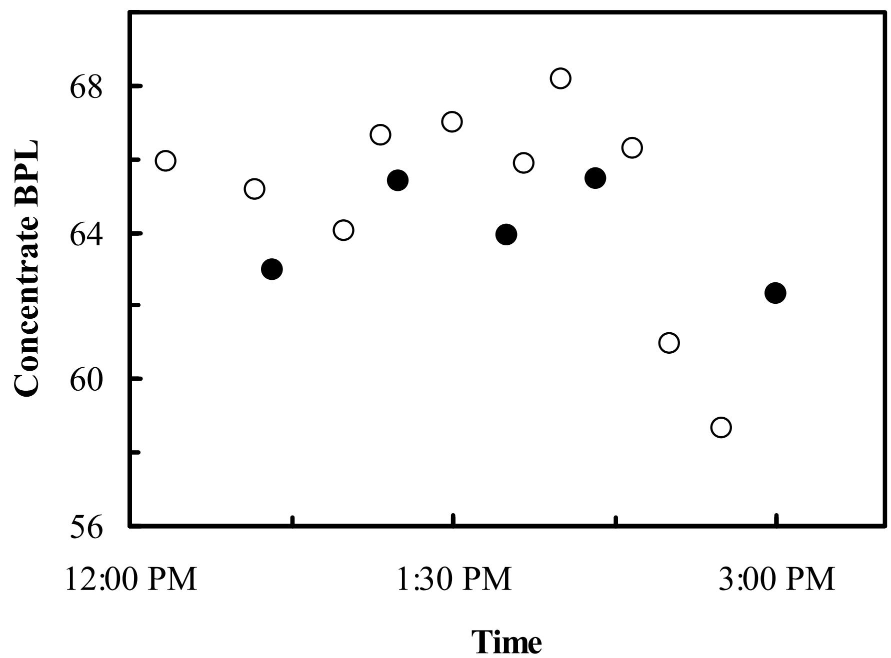
Figure 27. Real-Time Comparison of DEC and Plant Performances on Conditioned Feed (Run 2).

Since none of our steady state studies lasted for up to two hours, we initiated another test on a Saturday before moving the unit to the next site. Owing to inclement weather conditions in the morning, the test was started around 12:00 noon and lasted for about three hours. Figure 28 shows a real-time comparison of the DEC and plant results during the test period. In general, the results show that the DEC unit gave final products with slightly higher BPL while the tailings grades were comparable. However, as with the first test, the unit had to be shut down with the approach of another storm as evidenced by a sharp rise in the tailings grade in the DEC unit. In general, the performances of the DEC and plant mirrored each other as the results clearly illustrate, thus confirming that columns can be deployed for amine flotation of rougher concentrate at Plant C. A summary of the results obtained at this mine site is given in Table 6.

Table 6. Summary of Test Results at Plant C.

Run	Unit	Concentrate BPL	Concentrate Insol	Tailings BPL
1	DEC	68.9 ± 2.4	8.2 ± 3.1	9.7 ± 1.2
	Plant	67.2	9.1	11.5
2A	DEC	70.9	5.3	39.4
	Plant	69.9	5.6	43.0
2B	DEC	66.5 ± 2.1	11.5 ± 1.9	14.9 ± 1.7
	Plant	67.0 ± 1.7	11.1 ± 1.4	12.6 ± 2.9
3	DEC	66.1 ± 1.2	12.0 ± 1.6	12.6 ± 2.2
	Plant	64.4 ± 1.2	14.4 ± 1.8	12.8 ± 5.0

As indicated earlier, the performance of the DEC unit was comparable with that of the plant. Since we did not have the opportunity of independently varying the amine dosage at this site, we cannot be certain if the amine dosages employed during the test were optimal for the DEC unit. However, our experiences at Plants A and B would suggest that lower amine dosages are needed in column flotation of rougher concentrates.



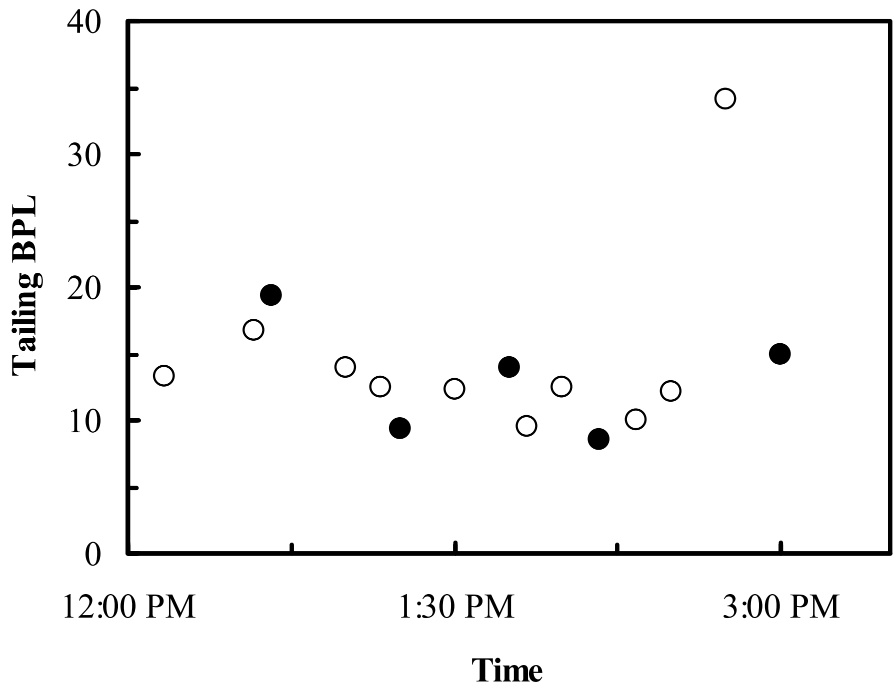


Figure 28. Real-Time Comparison of DEC and Plant Performances on Conditioned Feed (Run 3).

PLANT D

Determination of Optimum Amine Dosage

At this location, preliminary experiments were carried out to determine an optimum amine dosage for the rougher phosphate concentrate. Figure 29 shows the variation of concentrate and tailings grades with amine dosage and air flow rate. In general, the concentrate grade increased with decreasing air flow rate. Similarly, cleaner tailings were also obtained at the lower air flow rate, thus indicating that the separation efficiency was higher at the lower air flow rate. While the reason for this observation is not entirely clear at the present time, it may be related to changes in the feed grade (which was not monitored during the test).

At low collector dosages, the concentrate grade increases with amine dosage. However, when the optimum collector dosage was exceeded, the concentrate grade decreased with increasing amine dosage, with the decrease being more pronounced at the higher air flow rate. For both air flow rates, the optimum amine dosage appears to be in the range of 0.5-0.6 lb./T. The tailings grade at the optimum amine dosage was generally in the range 6-8 BPL.

Steady State Tests

Limited steady state runs were carried out at this location owing to transfer line problems. In addition, some samples were not labeled so matching the results after the analysis was not possible. The only steady state run that lasted over three hours is shown in Figure 30. The results also show a real-time comparison of the plant and DEC performances. It is noted that the plant amine dosage was 1.24 lb./T while the values employed in the DEC unit were 0.49 lb./T and 0.65 lb./T. In general, the concentrate grade from the DEC unit was either higher or equal to that of the plant. Except for the start-up period, the tailings from the DEC unit were consistently lower than those of the plant, thus showing that the column cell was more efficient in upgrading the rougher concentrate. It is noted that the feed solids concentration to the column was consistently low (less than 20 wt. %). Since conditioning is best carried out at high percent solids, it is likely that the column performance was negatively impacted. Even with this limitation, its performance compared favorably with that of the plant while operating at 39-52% of plant amine dosage. Our results confirm that lower amine dosages are needed for the flotation of rougher concentrate in a column cell. This finding is consistent with our earlier observations at Plants A and B where unconditioned feed was supplied to the column.

Figure 31 shows a comparison of the grade-recovery curve from the DEC unit and plant. For the duration of the test at this site, the measured solids feed rate was 2.16 ± 0.02 TPH. The results show that for the same concentrate grade, the BPL recovery from the DEC unit was 2-3% higher, thus confirming the benefits of employing column cells in amine flotation of rougher concentrate. As indicated earlier, other steady state studies were initiated at this site but due to transfer line problems, the duration of the tests was rather short.

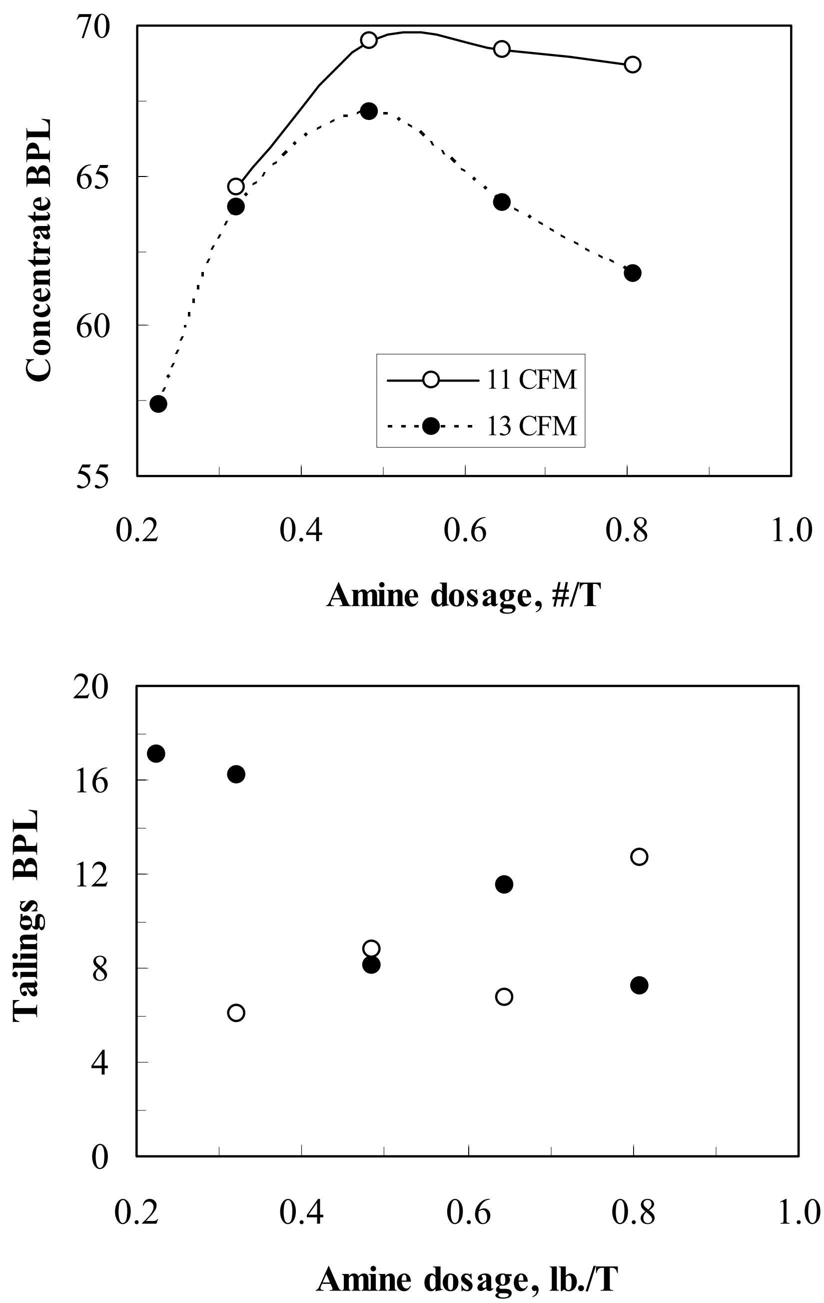


Figure 29. Effect of Amine Dosage and Air Flow Rate on Rougher Concentrate Flotation.

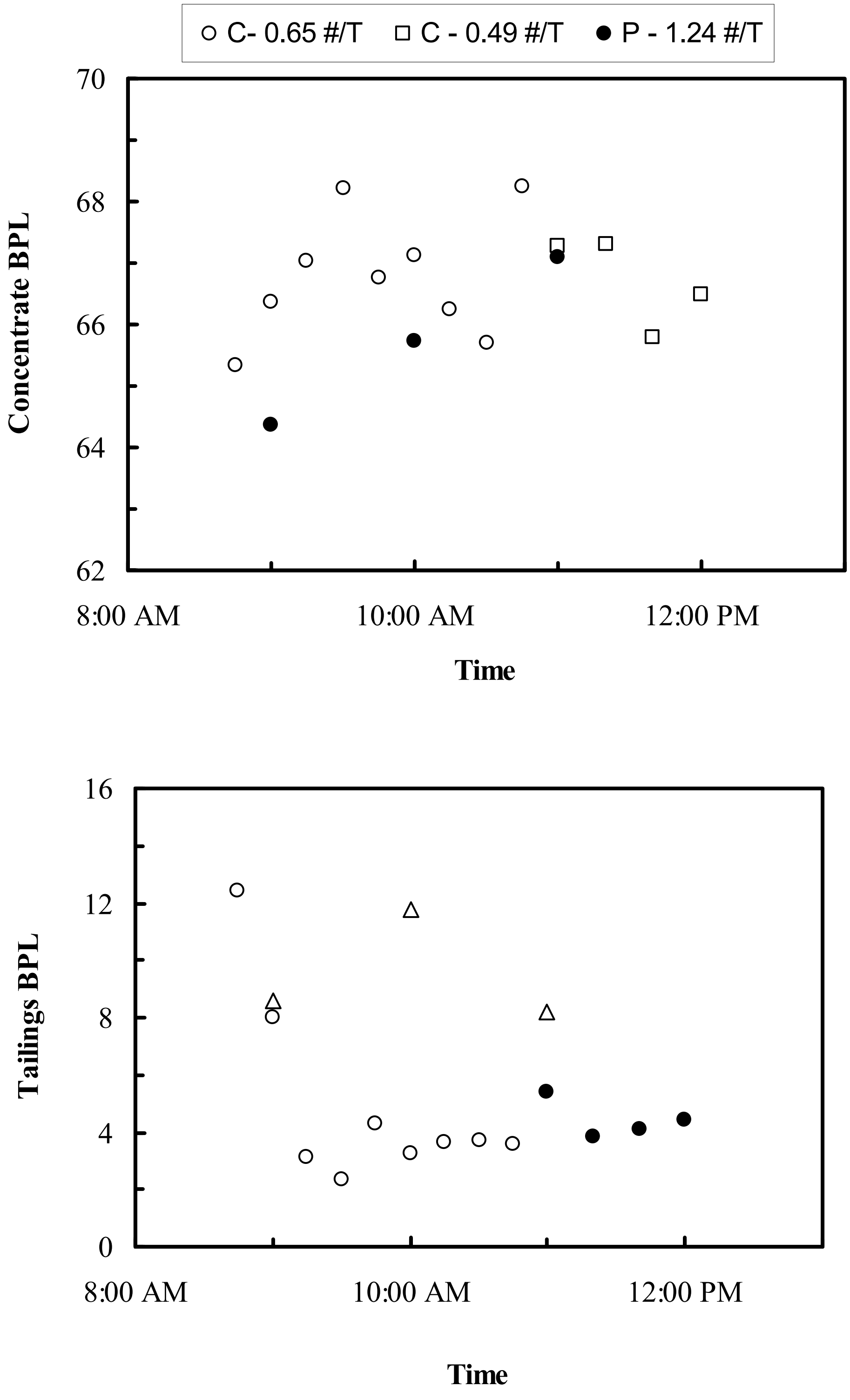


Figure 30. Effect of Amine Dosage on Flotation Performance for Run 2.

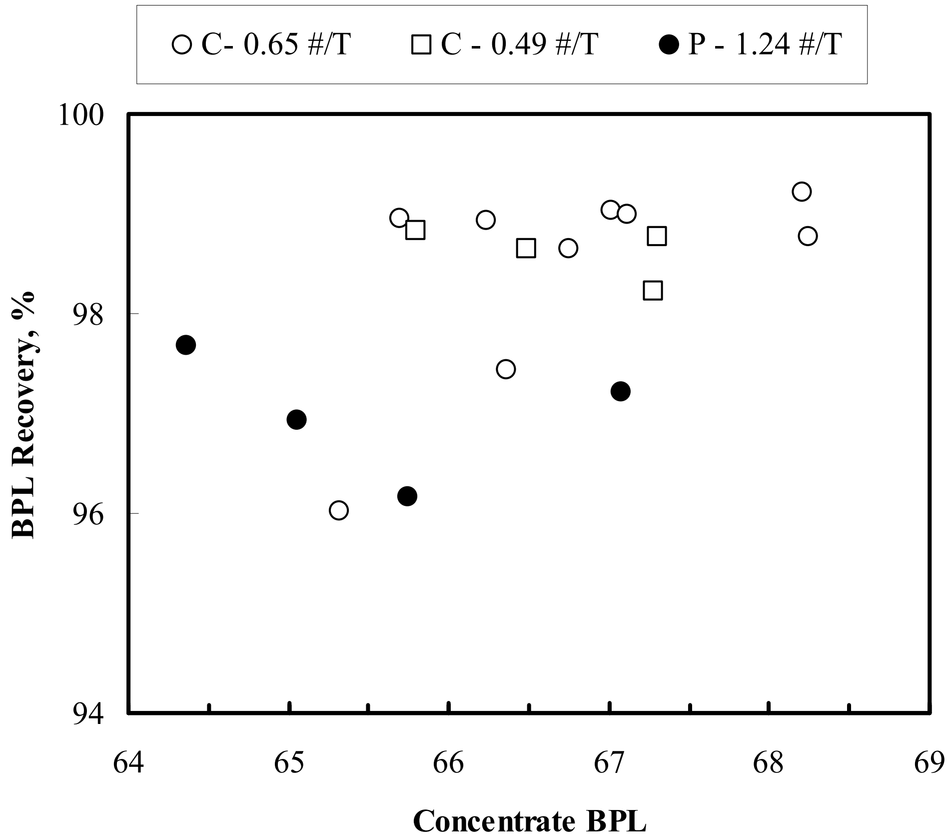


Figure 31. Grade Recovery Curve for Rougher Concentrate Flotation at Plant D.

A comparison of the separation efficiency in the DEC unit and plant is shown in Figure 32. Overall, the results show that the DEC gave higher separation efficiencies. A summary of the results obtained at this site is shown in Table 7. The results shown in Table 7 show that lower amine dosages are needed in column flotation of rougher phosphate concentrate. It is apparent from the foregoing that column cells offer significant advantages over conventional cells in amine flotation of rougher concentrates in the phosphate industry.

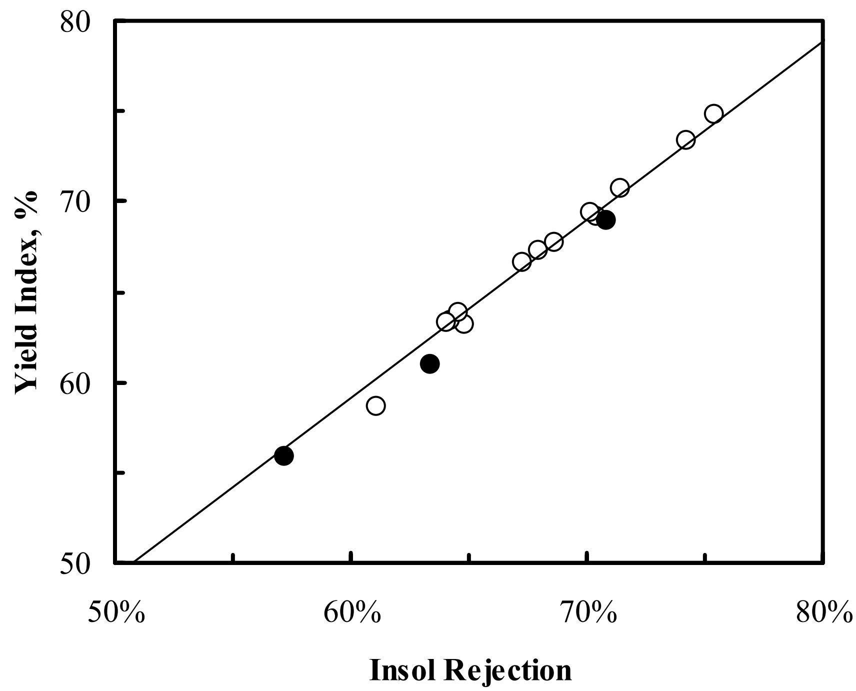


Figure 32. Loci of Separation in the DEC and Plant.

Table 7. Summary of Test Results at Plant D.

Run	Unit	Amine Dosage, lb./T	Concentrate BPL	Tailings BPL	BPL Recovery, %
1	DEC	0.66	59.3 ± 5.3	2.7 ± 1.1	97.2 ± 1.4
	Plant	1.07	54.5 ± 7.2	10.6 ± 6.2	89.9 ± 7.6
2A	DEC	0.65	67.0 ± 0.9	3.41 ± 0.6	98.9 ± 0.2
	Plant	1.24	65.1 ± 1.0	10.2 ± 2.2	96.9 ± 1.1
2B	DEC	0.49	66.7 ± 0.7	4.44 ± 0.7	98.6 ± 0.3
	Plant	1.24	67.1	8.22	96.7

PLANT E

Effect of Amine Dosage and Frother

The in-plant tests were started and concluded at Plant E. Since the PI had yet to undertake the plant safety course, the initial stay at Plant E was limited to one week. During the first phase of the study our focus was on understanding the process issues in amine flotation of rougher phosphate concentrate in a column cell. Since columns are not traditionally employed in amine flotation in the phosphate industry, such factors as collector dosage, air flow rate, frother use, etc. needed to be established. It was also at this site that we evaluated the efficacy of frother in the cleaner circuit. The frother used in the test was F-579 (a polyglycol). Figure 33 shows the effect of frother addition on silica flotation from the rougher concentrate. For these tests, the solids feed rate was estimated to be 2 TPH. Our results show that while the recoveries were comparable, the product grade increased with frother use. The results obtained can be explained on the basis of increased bubble surface area with frother use. It is noted that the use of frother is expected to reduce the bubble size. Thus for the same air flow rate, higher bubble surface area will be available for the flotation of the silica present in the rougher concentrate. Thus, the subsequent tests were carried out with frother addition.

Figure 34 shows the effect of air flow rate and amine dosage on silica separation from the rougher concentrate. In general, the results at an air flow rate of 13 CFM were less variable and show that at amine dosages of 600 ml/min (3.39 lb/T) or higher, the performance of the DEC unit remains fairly constant. Unfortunately, no tests were carried out at lower amine flow rates so it is not possible to determine if lower amine dosages are needed in the DEC unit. The improved results at the higher air flow rate is attributed the increased availability of bubble surface area for flotation of the silica. The amine dosage determined in these preliminary studies is much higher than that typically employed in the phosphate industry. Unfortunately, the amine dosage at this site is reported per ton of feed so a direct comparison is not possible.

Steady State Tests

Given the experience that we garnered from steady state evaluations at the other sites, our steady state tests at Plant E were intended to explore the limits of column flotation of rougher concentrate. As our previous results have shown, columns are effective in amine flotation of rougher concentrate and generally require less collector than the conventional cells in use in the plant. Figure 35 shows a typical comparison of the DEC unit and plant performance during the steady state studies. For this test, the solids feed rate was 7.45 TPH while the amine dosage was fixed at 0.5 lb/T. In general the performance of the DEC unit was similar to that of the plant even though slightly lower concentrate grades were obtained in the former. However, the higher concentrate grades in the plant were accomplished at the expense of higher BPL losses to the tailings stream. For example, while the DEC unit gave tailings with 5.6-8.1 BPL, the plant tailings assayed 9.1-12.8 BPL. Why the reasons for the lower concentrate grade in the column are not entirely clear, they may be due to the following factors:

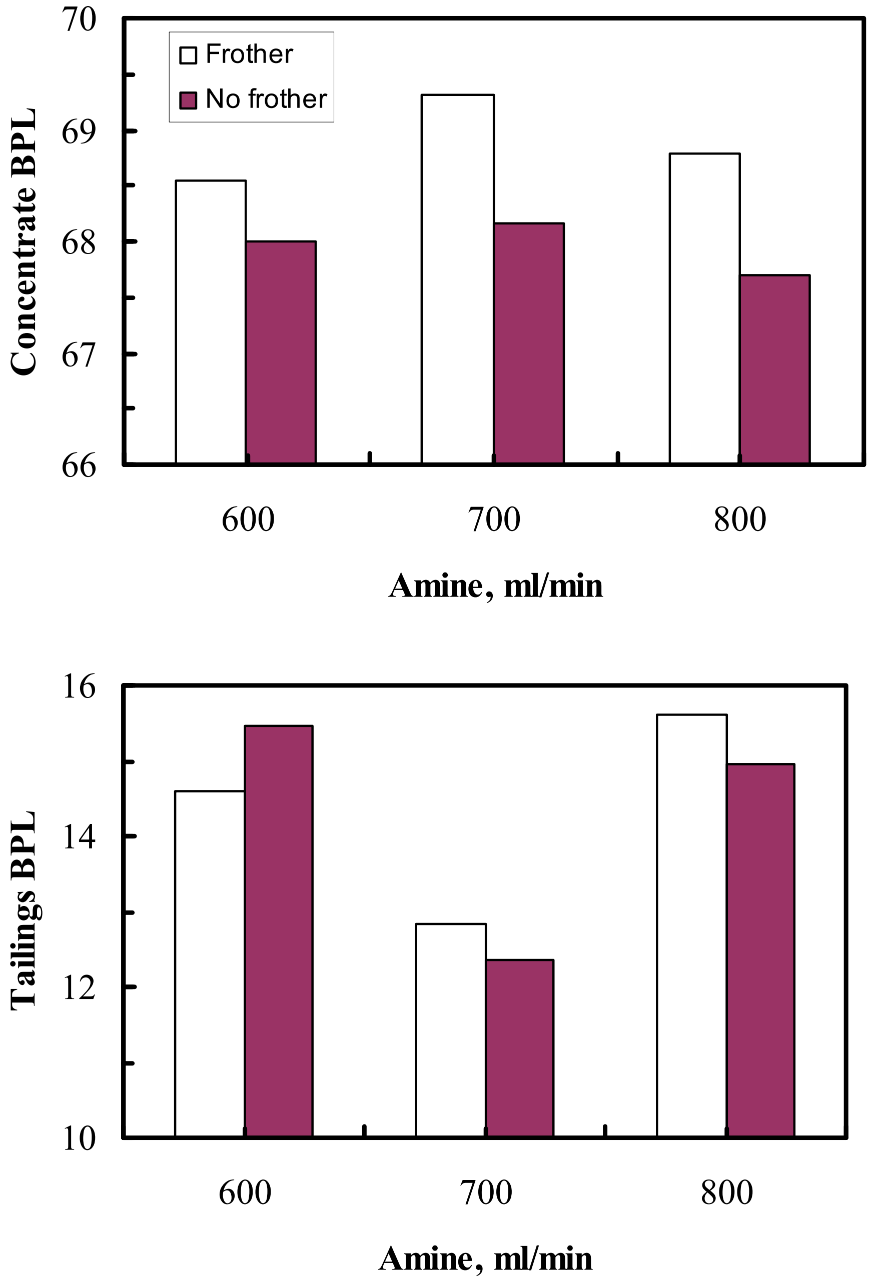


Figure 33. Effect of Amine Dosage and Frother on Fine Rougher Concentrate Flotation.

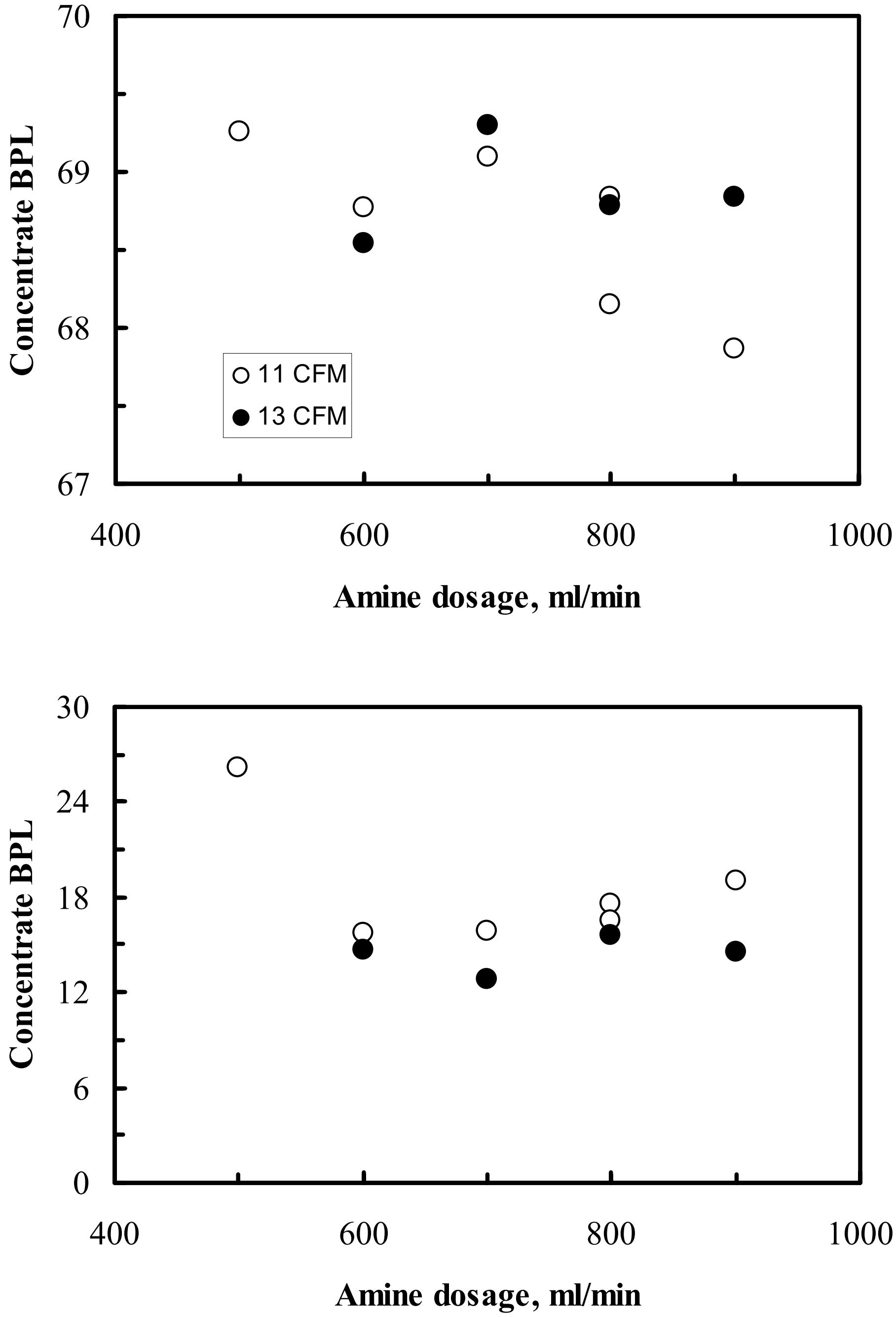


Figure 34. Effect of Amine Dosage and Air Flow Rate on Rougher Concentrate Flotation.

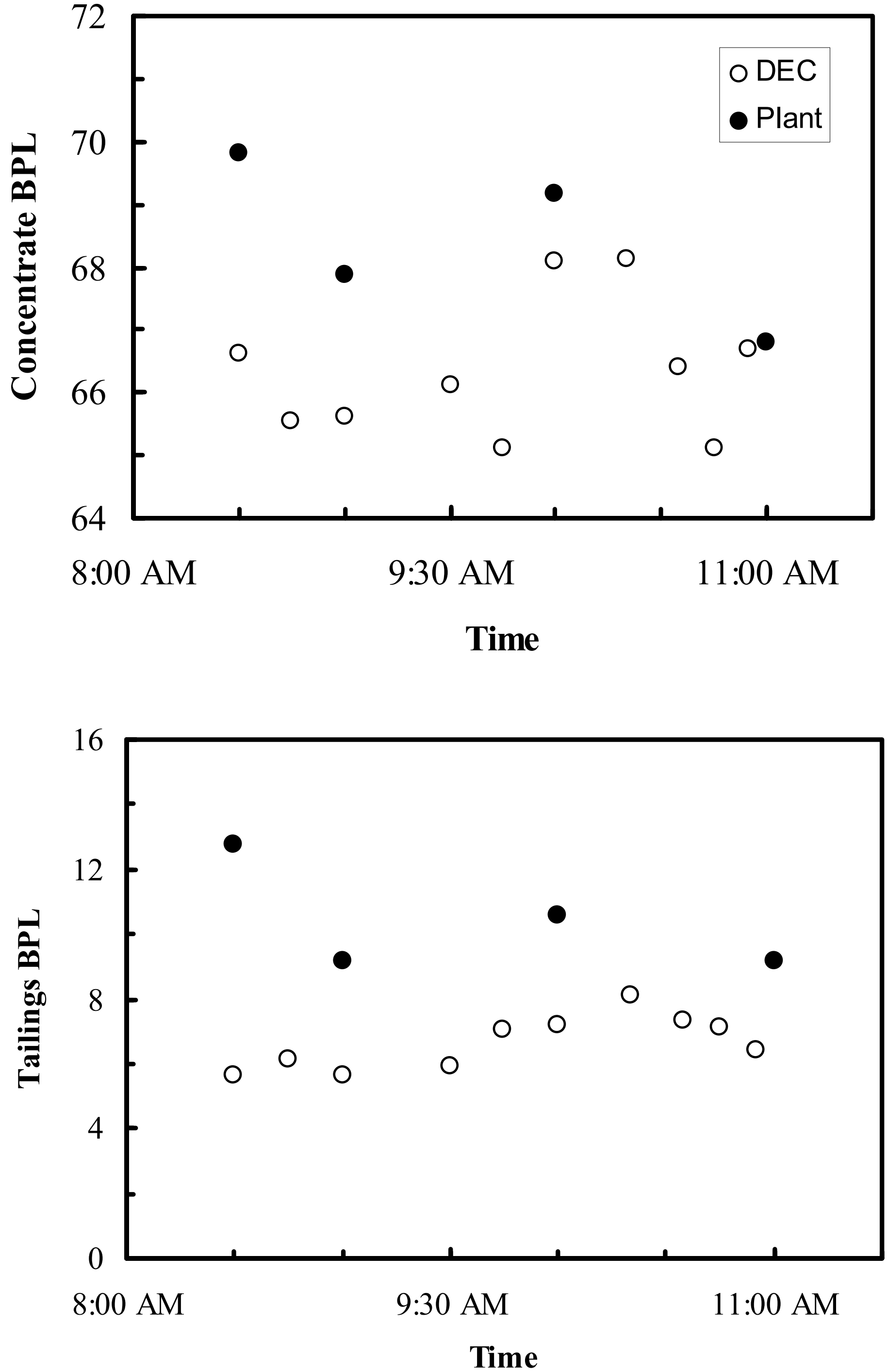


Figure 35. Real-Time Comparison of DEC and Plant Grades for Run 1.

1. Processing capacity limitation: The concentrate feed rate was approximately 6.8 TPH, which is much higher than the rated capacity of 2 TPH. Since the feed rate employed here is much higher than those employed at the other sites (max. 4 TPH), it is likely that the capacity of the unit was exceeded.

2. Amine dosage: The amine dosage in the plant was 1.64 lb/T while that used in the DEC unit was only 0.5 lb/T. Since the preliminary tests suggested higher amine dosages, it is possible that the value employed during the test was not optimal. Our results from the other sites suggest that amine dosages of 40-50% of the plant values are needed for optimum separation in the DEC unit. In the present case, the DEC dosage value is only 31% of the plant value.

A comparison of the grade-recovery from the DEC and plant is shown in Figure 36. The results clearly show that the performance of the DEC unit was similar to that of the column. If high product grades are required, the column can be operated under negative bias conditions. In general, for the same concentrate grade, the column gave higher BPL recoveries.

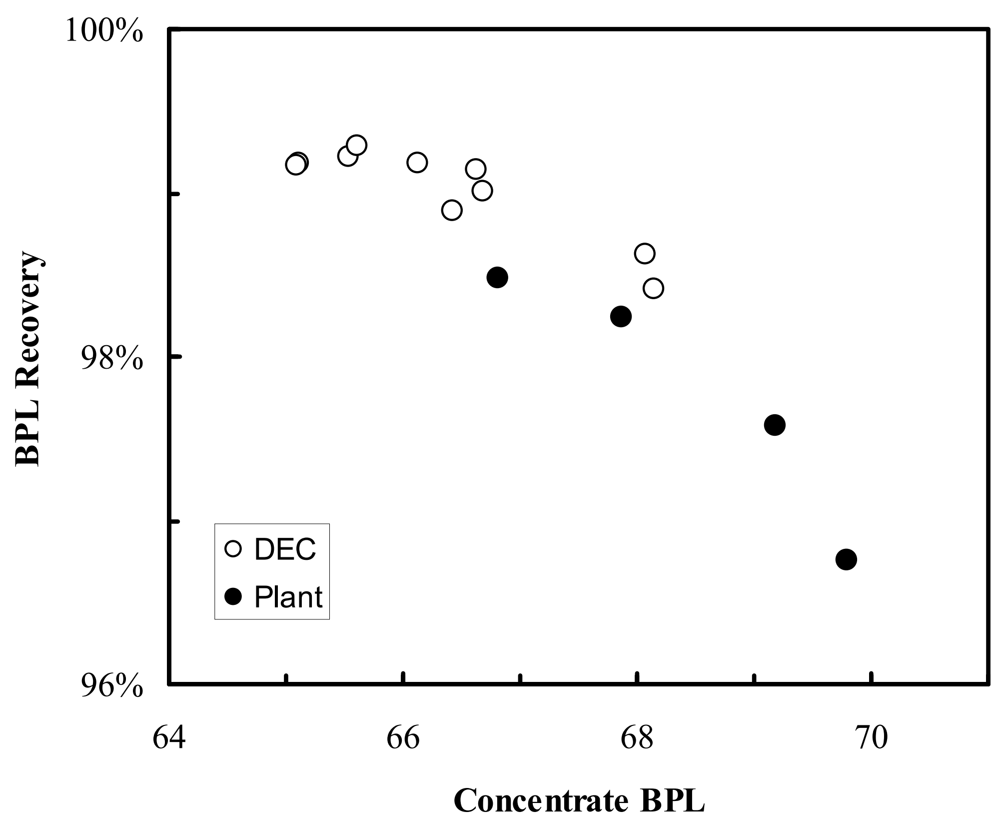


Figure 36. Comparison of Grade Recovery Profiles for Run 1.

During the test period, the plant switched the fatty acid normally employed in rougher flotation. We evaluated the performance of the column for the duration of the switch and the results are shown in Figure 37. The solids feed rate to the DEC unit was approximately 4.7 TPH. For comparison, the plant amine dosage was reported to be approximately 1.25 lb/T while that in the DEC unit varied from 0.72-0.85 lb/T. For the duration of the fatty acid switch, the concentrate from the DEC unit was lower than that of the plant. However, when the normal plant fatty acid was reintroduced, there was a steady increase in the final concentrate grade from the DEC unit. While the plant grade did not change substantially during the switch, the recoveries were 2% lower. Unfortunately, the plant shut down shortly after 11:00 am and the steady state test had to be terminated. However, we note that the DEC unit responded favorably when the switch to the normal plant amine was made. The results obtained in the DEC unit confirm that the normal plant fatty acid gave better separation results.

Another set of tests were carried out in which the amine dosage was varied from 0.56-0.7 lb/T while the corresponding plant amine dosage was reported to be 1.13 lb/T. For this test, the solids feed rate to the DEC was fixed at 4.9 TPH. A real-time comparison of the plant and DEC performances is shown in Figure 38. For this test as well, the plant performed better than the DEC unit. A summary of the steady state results obtained at this site is shown in Table 8.

As noted earlier, the plant performance at this site was superior to that of the DEC unit. While it appears that the amine dosage rates in the column were low and may have impacted on its performance, we cannot rule out the possibility that the capacity of the pilot unit was exceeded. Unfortunately, attempts to lower the feed rate to the values observed at the other sites were not successful and resulted in choking of the feed line. In the light of the foregoing, it is probable that both factors (amine dosage and column capacity) impacted the performance of the DEC unit.

The overall grade-recovery profile and separation efficiencies are shown in Figures 39 and 40, respectively. We note that the shape of the grade-recovery curve (Figure 39) suggests that with appropriate test conditions, the performance of the DEC unit would at least match the plant performance. This observation is supported by the loci of the DEC and plant separations (see Figure 40). However, judging from the conclusions at the other sites, it is probable that the amine requirement in the DEC unit would be lower.

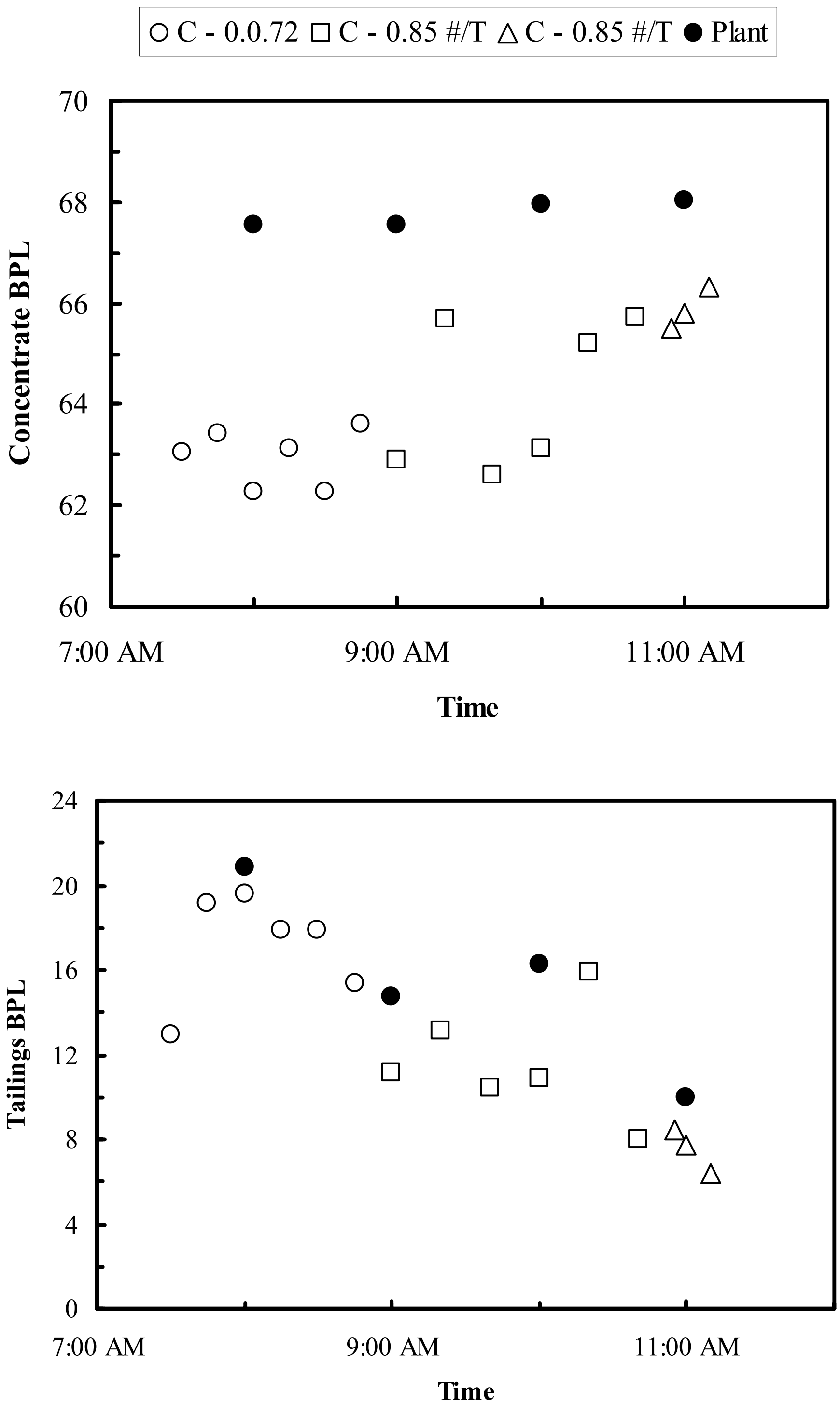


Figure 37. Real-Time Comparison of DEC and Plant Grades for Run 2.

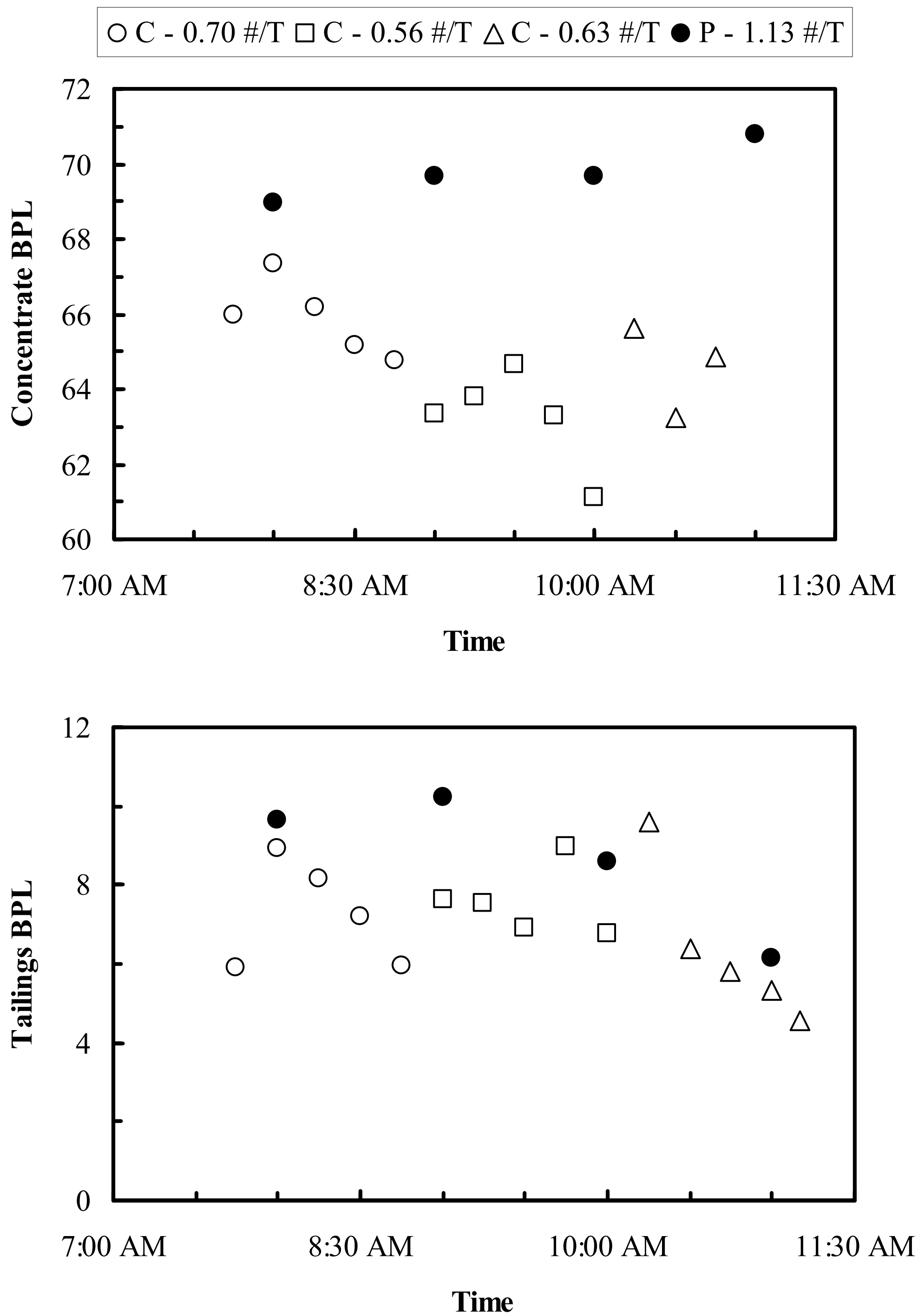


Figure 38. Real-Time Comparison of DEC and Plant Grades for Run 3.

Table 8. Summary of Test Results at Plant E.

Run	Unit	Amine Dosage, #/T	Concentrate		Tailings BPL	Recovery, %
			BPL	Insol		
1	DEC	0.50	66.3 ± 1.1	9.0 ± 1.1	6.7 ± 0.8	99.0 ± 0.3
	Plant	1.64	68.4 ± 1.3	4.8 ± 1.0	10.8 ± 1.8	97.6 ± 0.8
2A	DEC*	0.72	63.0 ± 0.6	11.4	17.1 ± 2.5	98.4 ± 0.4
	Plant*	1.25	67.6	5.6	20.9	94.7
2B	DEC*	0.85	71.2 ± 2.2	10.2 ± 1.5	11.6 ± 2.7	98.6 ± 0.8
	Plant*	1.25	67.7 ± 0.2	7.0 ± 1.3	15.5 ± 1.1	96.4 ± 0.5
2C	DEC	0.85	65.9 ± 0.4	7.4 ± 1.3	7.5 ± 1.0	98.8 ± 0.1
	Plant	1.25	68.0	N/A	10.0	97.8
3A	DEC	0.70	65.9 ± 1.0	9.6 ± 1.4	7.2 ± 1.3	99.1 ± 0.4
	Plant	1.13	69.0	4.9	9.6	98.0
3B	DEC	0.56	63.2 ± 1.3	12.9 ± 1.9	7.6 ± 0.9	99.6 ± 0.3
	Plant	1.13	69.7	4.3 ± 0.8	9.4 ± 1.1	97.9 ± 0.3
3C	DEC	0.63	64.6 ± 1.2	12.0 ± 1.5	7.3 ± 2.1	99.3 ± 0.3
	Plant	1.13	70.8	3.7	6.2	98.5

* Plant was operating with a different fatty acid in rougher flotation.

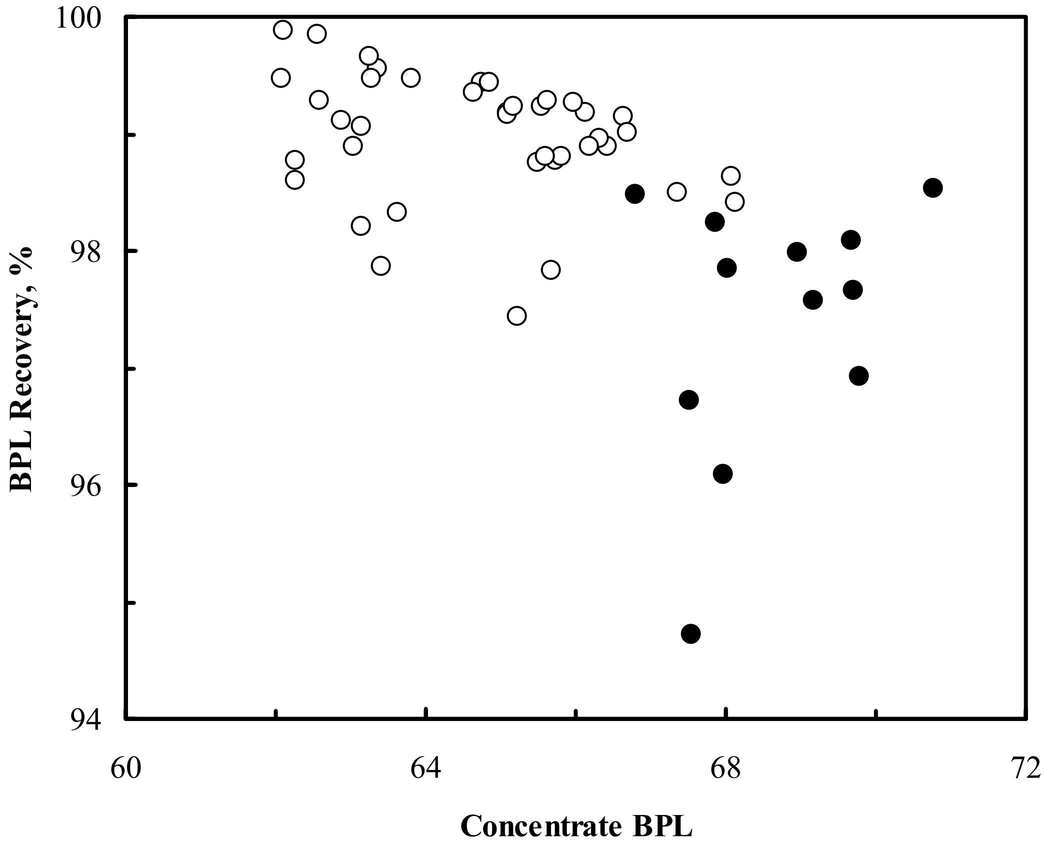


Figure 39. Overall Comparison of DEC and Plant Grade-Recovery Profiles (Open and Closed Symbols Are for DEC and Plant Data, Respectively).

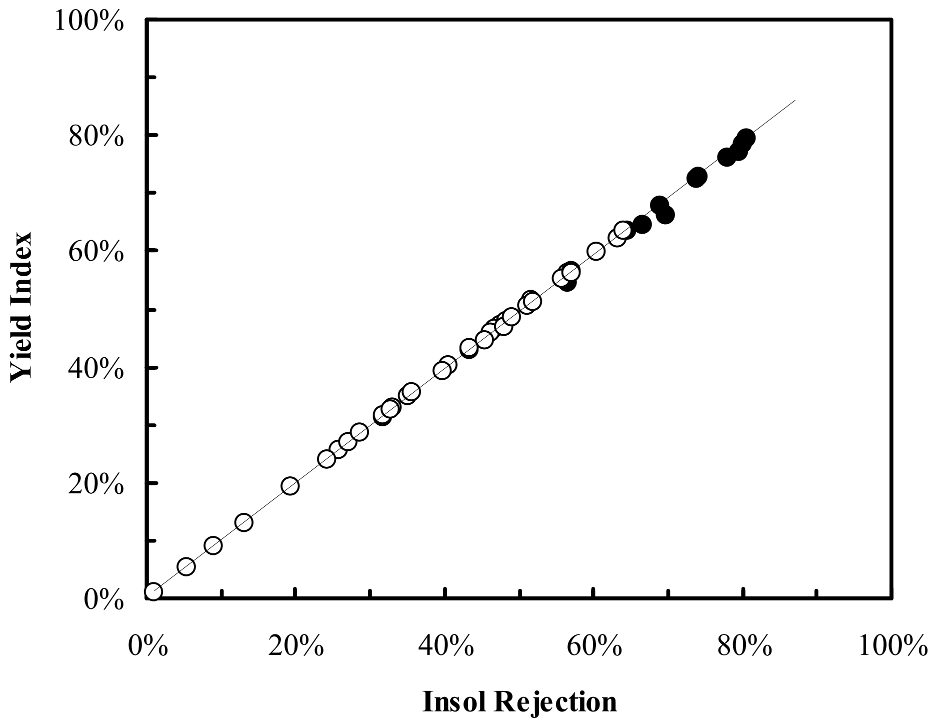


Figure 40. Comparison of Loci of Separation at Plant E (Symbols Same as in Figure 39).

CONCLUSIONS

The results obtained in the present study show that column cells can be deployed for cleaner duty in the phosphate industry. Our results show that lower amine dosages are required if column cells are employed. In addition to the normal advantages inherent in the use of column cells (low operating and maintenance costs, small footprint, etc.), process water did not appear to have a significant effect on the performance of the column. With continued draught in Florida, the use of process water in the cleaner circuit may further reduce the demand for fresh water intake. The 0.61 m pilot plant DEC unit was determined to have capacities of about 3 TPH and 4 TPH when operating on fine and coarse rougher concentrates respectively. Because of the shallow nature of the pilot plant unit, high air flow rates could not be employed. This means that a full-scale unit with a diameter of 3-m should have capacities of at least 75 TPH and 100 TPH for fine and coarse rougher concentrates respectively. The results of the study clearly show that the kinetics of the silica flotation is quite fast and long residence times are not required. In the light of this, column cells are ideal for such separation. The deployment of column cells in cleaner duty is expected to reduce the amine cost by about 30-40%. Since the capital, operating and maintenance costs for column cells are low, this will result in a lowering of the production cost for flotation concentrate. Our results show that the column cell was effective in both fine and coarse silica flotation from rougher concentrates.

PART II: INTERMEDIATE PEBBLE (IP) FLOTATION

INTRODUCTION

In the Florida phosphate industry, conventional flotation is extensively used in the upgrading of mined phosphate matrix. In Part I of the report, it was shown that column flotation can be successfully deployed to upgrade both fine and coarse amine rougher concentrates. In the light of the excellent results obtained with the DEC cell, it was decided to extend the study to cover intermediate pebble (IP) product that is currently generated in some plants. The IP is generated by separating the flotation feed (16x150 Tyler mesh) at 35 Tyler mesh. In general, Derrick screens are used to separate the IP product while the underflow is combined with the flotation feed (minus 35 mesh) recovered from the sizer operation. While the IP is blended with the pebble, the quality of the resulting product is lowered.

Table 9 shows a typical composition of IP from an operating phosphate mine in Central Florida. It is apparent from this that if the coarse silica in the IP product can be removed, the product grade can be considerably enhanced. The advantages of upgrading the IP include:

1. Reduction in build-up of the gypsum piles.
2. Reduction in grinding cost at the chemical plant.
3. Reduction in transportation costs to the chemical plant.
4. Simplification of the process flow sheet with the elimination of the fatty acid flotation step.

As the mining in Central Florida moves south, the phosphate grade decreases and separation becomes more difficult. With the depressed fertilizer market, it is important to reduce the operational costs. With decreasing ore grade, cost reductions, such as those to be derived from the use of column cells will ensure that Florida phosphate mineral value and products derived from it will remain competitive on the international market.

Table 9. Typical Characteristics of IP Rock from a Florida Phosphate Mine.

Size Fraction, mesh	Weight Fraction, %	BPL, %		Insol, %	
		Grade	Distribution	Grade	Distribution
+ 10	4.4	63.9	4.9	7.7	1.7
- 10 + 16	35.3	65.6	40.5	8.2	14.4
- 16 + 28	41.5	56.0	40.6	22.5	46.6
- 28	18.8	42.6	14.0	39.7	37.3
Head	100	57.2	100	20.0	100

EXPERIMENTAL METHODS

The study was carried out at three operating mines in Florida. The experimental set-up was similar to that described in Part I of the report. However, the amine used in the present study was a special formulation by Arr-Maz for IP flotation. While the collector used was specifically developed for coarse silica flotation, the same frother (F-507) was used in the current phase of the study.

At each location, a suitable point for tapping into the IP stream was identified by plant personnel. Since the feed solids concentration was generally low at most locations, we used the 0.15-m hydrocyclone which is part of the test rig for feed dewatering. Where this was done, the hydrocyclone was mounted on a beam and its underflow discharged either directly or into a 0.10-m PVC pipe which drained into the top section of the DEC unit. The required amine dosage was metered with the aid of a peristaltic pump (Masterflex Digital Console Drive Model 7523-20) and introduced inside the PVC pipe or as the underflow drained into the cell. For these tests, we used a 5-7% (v/v) solution of the amine supplied by Arr-Maz. Higher concentrations of the amine could not be used because of increased viscosity of the solution. With this arrangement, the amine conditioning time was limited to the time it took for the feed to travel the length of the pipe (typically less than 3 s). This arrangement was selected to mirror plant practice where the amine is added either directly to the first cell or in the feed box.

At each site, the air flow rate and amine dosage was fixed while froth and tailing samples were collected over an extended period to ascertain the attainment of steady state results. In addition, we selected random samples of the feed, concentrate and tailings streams for size analyses. The size analyses data was used to determine the fractional recoveries needed for mass balance calculations. At the end of the test period the samples were handed over to the respective plant laboratories for drying and chemical analysis. Since the turnaround time for analyses at two of the sites was rather long, some samples were sent to an external laboratory to provide preliminary results on the effectiveness of the conditions employed during the test-work. This allowed us to change the operating parameters so as to cover a broad spectrum of test conditions.

RESULTS AND DISCUSSION

PLANT A

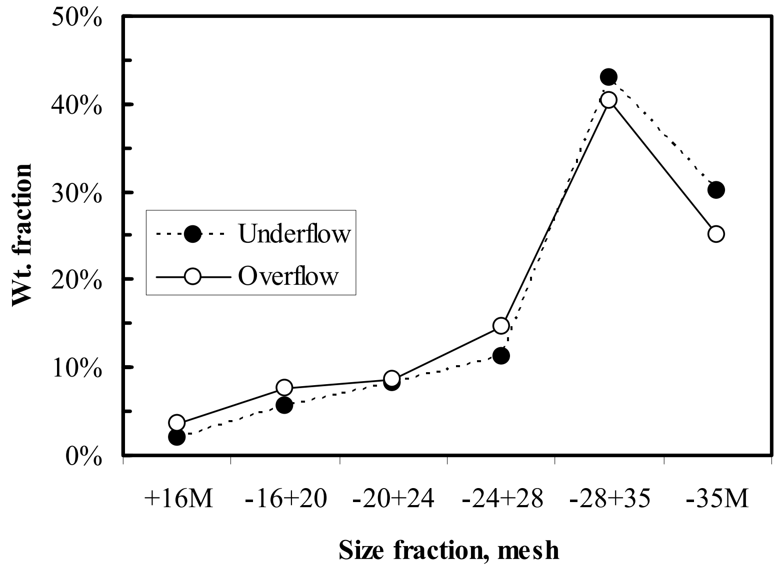
Testing conditions were most favorable at this location and we believe the results obtained here are representative of column performance on IP flotation. For example, when the plant was operating the solids flow rate was steady and the feed solids concentration after hydrocycloning was typically in the range of 68-73%. By contrast, at the other sites, the solids concentrations after dewatering were typically below 25%. To ensure that we had sufficient feed for the tests, the plant management would shut down the IP pump to allow build-up of solids in the IP tank.

In order to determine if additional concentration of the feed was taking place during dewatering underflow and overflow samples were taken for analyses. This test was carried out on the feed to the IP screens because it had a broader particle size distribution. A comparison of the underflow and overflow streams is shown in Figure 41. The results show that within the limits of experimental error, the two streams were identical and the hydrocyclone was only acting as a splitter. Our results show that the insol was concentrated in the -28 mesh fractions.

Flotation of Feed to IP Screens

At this plant, the management requested that we carry out some tests on the feed to the IP screens. This was done to evaluate if the IP screens can be replaced with an amine column cell. Our analyses of the IP at this site indicated that its phosphate value was consistently in the range of 36-45 BPL. Thus, if the feed to the IP screens can be upgraded to 55-60 BPL in a single step, the screens may be eliminated. Figure 42 shows the variation of feed, concentrate and tailings grades with time. From the feed, tailings and concentrate grades, the BPL recoveries were calculated. The average solids feed rate during the test was 3.4 TPH while the amine dosage ranged from 0.58-1.16 lb/T. Our results show that with a 25-34 BPL feed, the DEC unit consistently produced a concentrate with 56-64 BPL at recoveries of 50-70% (see Figure 43). While the DEC operations were not optimized, the results are promising and suggest the need for more detailed evaluation. The advantages of replacing the screens with a column cell include:

1. Elimination of the fatty acid flotation and rougher concentrate deoiling steps, resulting in lower operating and maintenance costs.
2. Elimination of capital equipment costs and hence simplification of the process flowsheet.



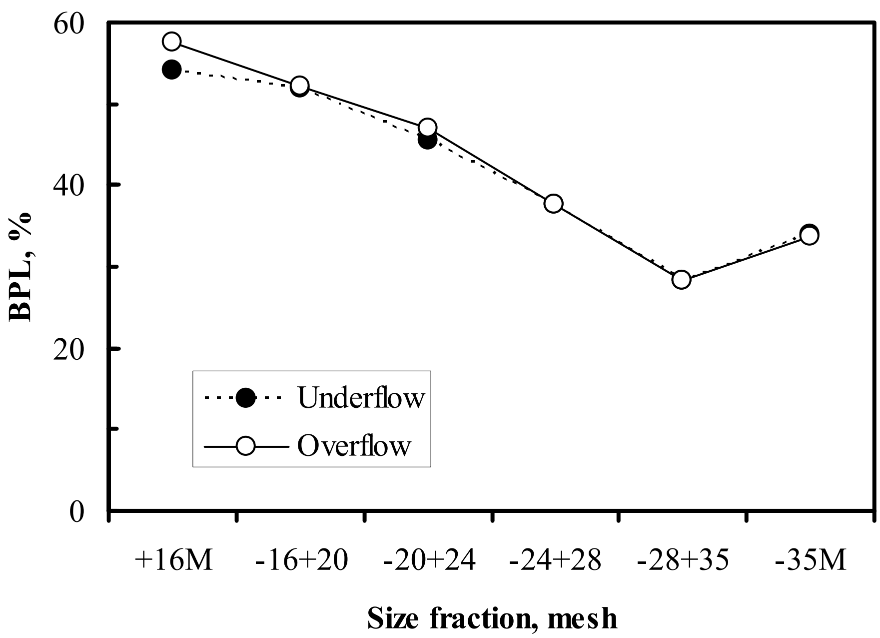


Figure 41. Comparison of Underflow and Overflow Streams from the Hydrocyclone.

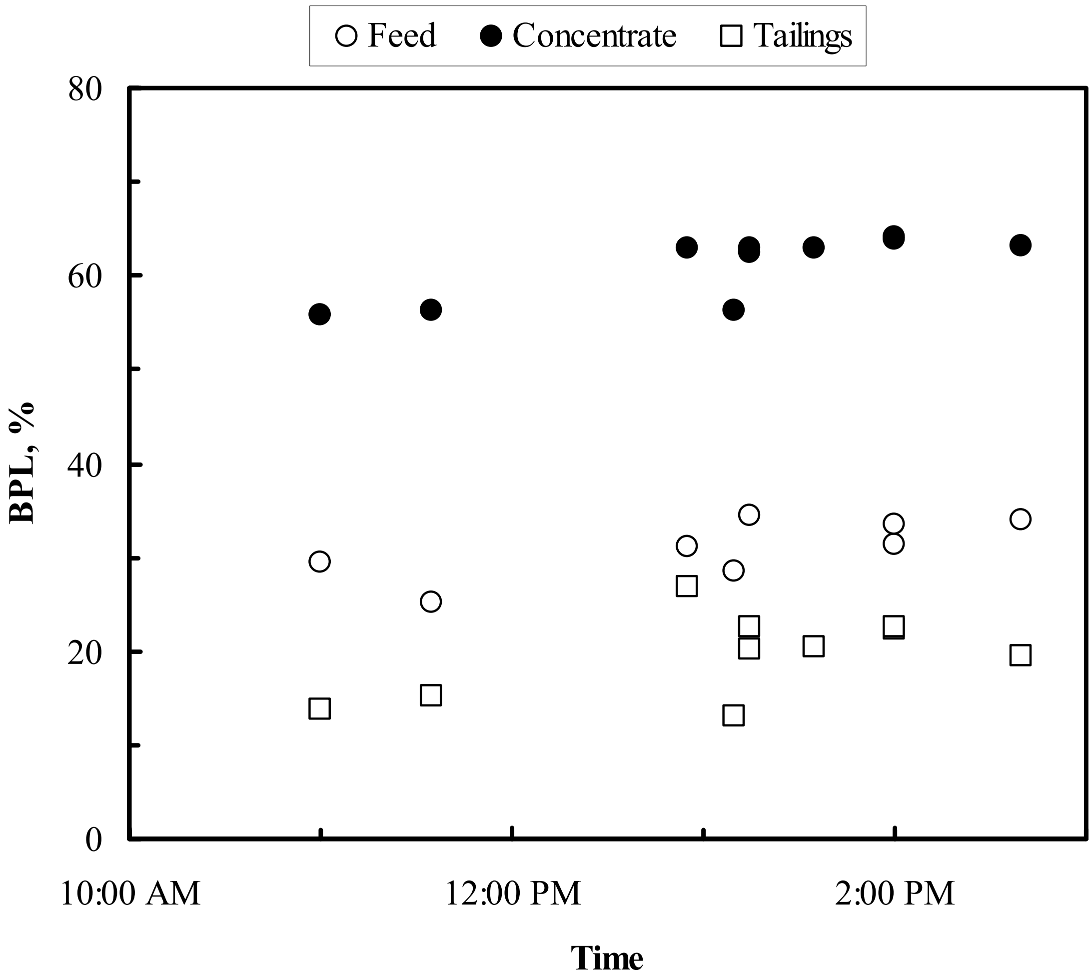


Figure 42. Real-Time Variation in the Feed, Concentrate and Tailings Grades.

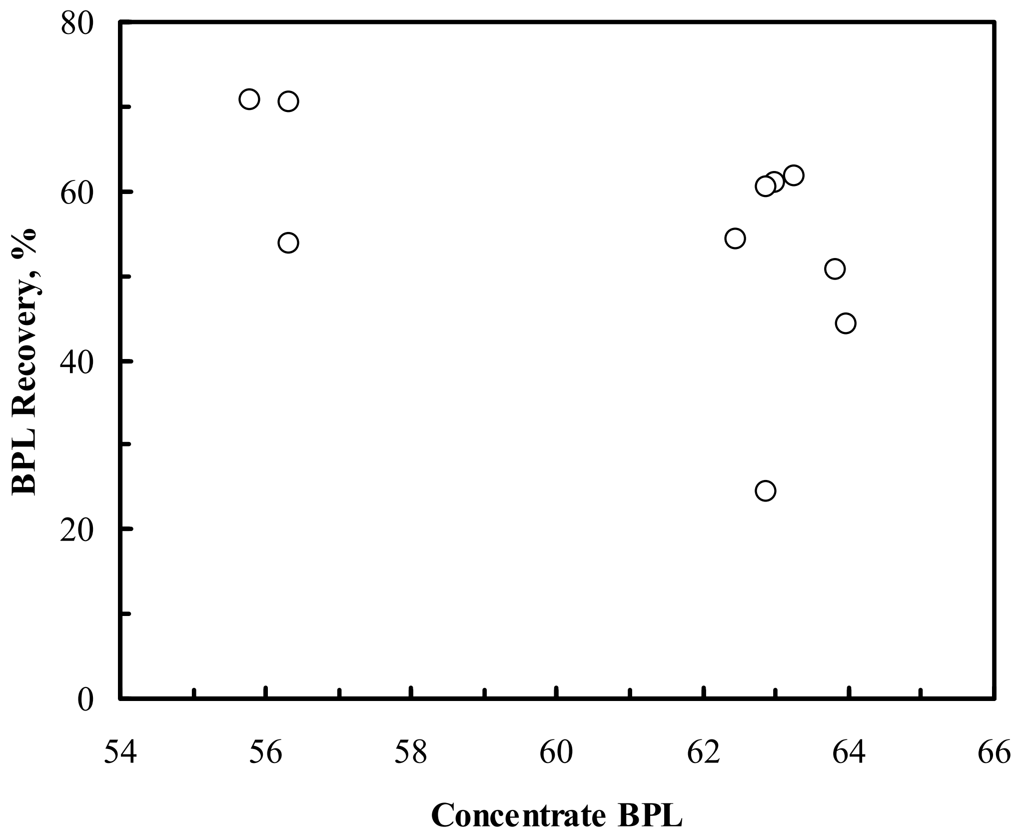


Figure 43. Grade-Recovery Profile for Flotation of Feed to IP Screens.

A sample of the feed, concentrate and tailings stream were taken for detailed size analyses and the results are shown in Figure 44. As expected, the insol content of the feed increased with decreasing size and was concentrated in the minus 28 mesh (i.e. -28 + 35 mesh and -35 mesh) fractions. For example, while 72.5% of the feed was found in the minus 28 mesh fraction, it contained 60.1% and 81.1% of the phosphate value and insol respectively. By contrast, the insol content of all concentrate fractions were reduced; however, the greatest reductions were observed in the finer fractions. For the coarser (+28 mesh) fractions, the BPL and insol contents of the product remained fairly constant. This finding suggests that for this particular feed liberation of the insol is only accomplished at 28 mesh.

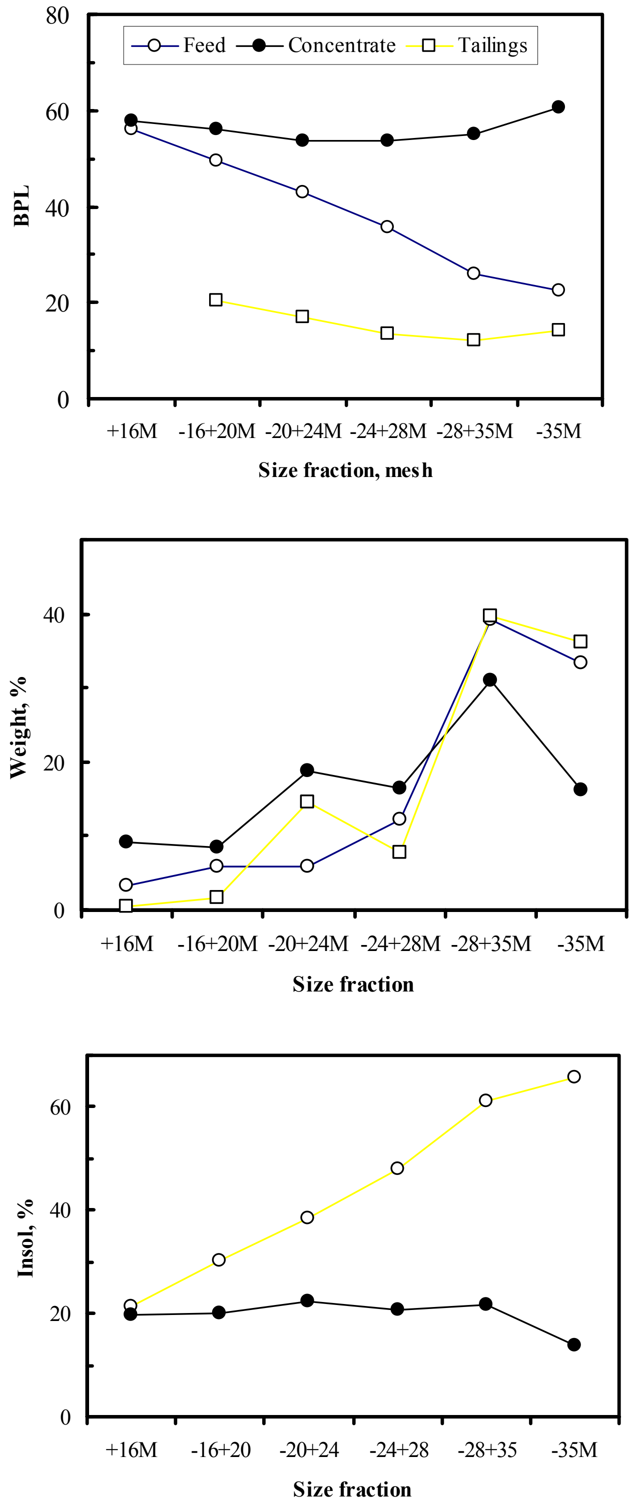


Figure 44. Size Fraction Analysis of Feed, Concentrate and Tailings Samples.

The BPL content of the tailings decreased with size; however, phosphate losses were concentrated in the minus 28 mesh fraction. As noted previously, the phosphate losses in the pilot-scale DEC unit is the combined effects of turbulence and shallow nature of the cell. Thus, we expect phosphate losses to be lower in a full-size industrial unit.

The effect of amine dosage on the flotation of feed to the IP screens is shown in Figure 45. Over the range studied, the results show that performance was not a strong function of dosage. In the light of the foregoing, it is suggested that an amine dosage in the range of 0.5-1 lb/T may be used in economic evaluation of column flotation of the feed to the IP screen. It is noted that for this plant, we determined the optimum amine dosage for the Phase I of this study to be in the same range. The results show that the Arr-Maz amine employed in this study was effective in the flotation of both fine and coarse silica from the feed to the IP screens.

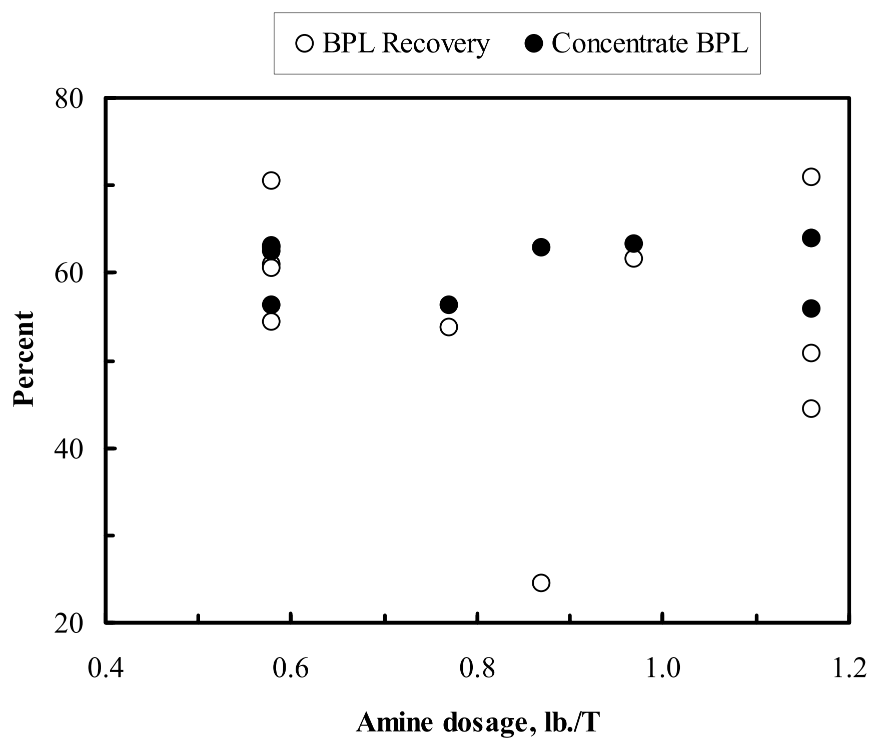


Figure 45. Variation of Concentrate BPL and Recoveries with Amine Dosage.

In order to carry out an economic evaluation of the process, it is necessary to optimize the recovery process. Figure 46 shows the locus of the separation for the feed to the IP screens. Also shown in Figure 46 is the size-by-size separation data. The advantage of this method is that the recovery profile can be reliably constructed with limited experimental work. It is interesting to note that the size-by-size separation data obtained agrees with the overall sample results. The results suggest that the optimum separation corresponds to an insol rejection of 80-83% and a yield index of 63%. From these values, the optimum BPL recovery is estimated to be in the range of 75-80%. With a 30 BPL feed with insol of 56%, we predict that the DEC unit will produce a 55-57 BPL concentrate with insol of 18-20%.

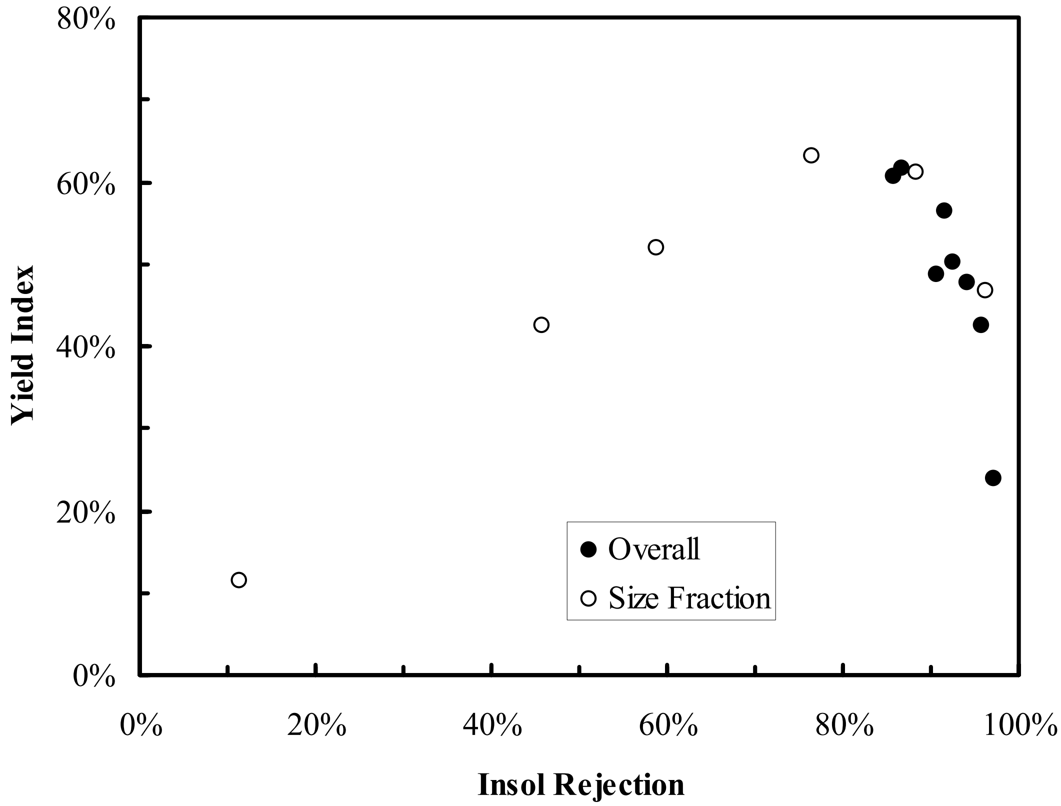


Figure 46. Locus of Separation for Flotation of Feed to IP Screens.

For the flotation of the feed to the IP screens, steady state runs were not carried out. While it may be argued that the results may not describe steady state data, the size-by-size data does provide an indication of whether steady state was achieved. For example, from the feed composition and fractional BPL recoveries, we computed the size and BPL distribution of the concentrate. A comparison of the experimental and calculated quantities is shown in Table 10.

In general, there was excellent agreement between the experimental and calculated values. The slight deviations observed with the intermediate (-16+20 and -20 +24 mesh) fractions is attributed to misplacement of solids. In addition, the calculated concentrate grade (55.9 BPL) and BPL recovery (72%) agree closely with the experimental values of 55.8 BPL and 71% BPL recovery. The mass balance data suggests that the process was indeed operating at steady state. While the flotation tests were not optimized, the results obtained show considerable promise and may warrant further study. Considering the economic benefits accruing from the elimination of the IP screens, such a study at this site could seek to determine the optimum amine dosage and the treatment of the froth products which contain considerable phosphate value. However, due to the limited time available for this study, these factors were not evaluated.

Table 10. Comparison of Experimental and Predicted Concentrate Characteristics from Flotation of IP Feed.

Size Fraction, mesh	Weight Fraction, %		BPL Distribution, %	
	Experimental	Predicted*	Experimental	Predicted*
+ 16	9.2	8.7	9.6	9.0
-16 + 20	8.4	12.9	8.4	13.0
-20 + 24	18.8	11.1	18.1	10.7
-24 + 28	16.5	17.5	15.9	16.8
-28 + 35	31.0	33.9	30.6	33.4
- 35	16.1	15.8	17.5	17.1

* Predicted using size-by-size mass balance.

Flotation of IP

Table 11 shows the characteristics of the IP product at this mine. The results show that the phosphate and insol content of the feed was widely distributed. Depending on the plant operating conditions, the fine fraction (-28 mesh fraction) of the IP accounted for 30-50% of the weight, while contributing 22-38% and 43-62% of the BPL and insol, respectively. In general, the insol content of the feed was concentrated in the -28 mesh fractions. As noted earlier, the close agreement between the calculated and head sample analyses does provide a good indication of the veracity of the analytical techniques employed. The results show that the IP grade is quite low and can benefit from additional upgrading.

Amine flotation is generally carried out with deep well water to avoid the introduction of slimes with their attendant high collector consumption. As a result of this, we initiated the tests with deep well water. However, we observed that the water pressure to the infusers was only in the range of 15-20 psi. Since the proper operation of the DEC unit requires water pressure of 30-35 psi, we switched to process water. Even with this switch the water pressure to the infusers increased marginally to 20-25 psi.

Table 11. Characteristics of IP at Mine A.

Size Fraction, Mesh	Weight Fraction, %	BPL Content, %	Insol, %
+16	10.9 ± 3.3	55.9 ± 1.1	23.4 ± 1.3
-16 + 20	16.6 ± 3.5	51.9 ± 1.7	28.8 ± 2.0
-20 + 24	16.1 ± 4.0	48.1 ± 2.0	33.6 ± 1.9
-24 + 28	15.1 ± 3.0	41.1 ± 2.3	42.9 ± 3.0
-28 + 35	26.8 ± 4.6	30.9 ± 3.0	56.3 ± 3.6
-35	14.7 ± 4.3	27.8 ± 2.7	60.3 ± 3.0
Calculated Head	100	40.8 ± 4.0	43.2 ± 4.7
Head*		41.2 ± 2.9	43.3 ± 3.1

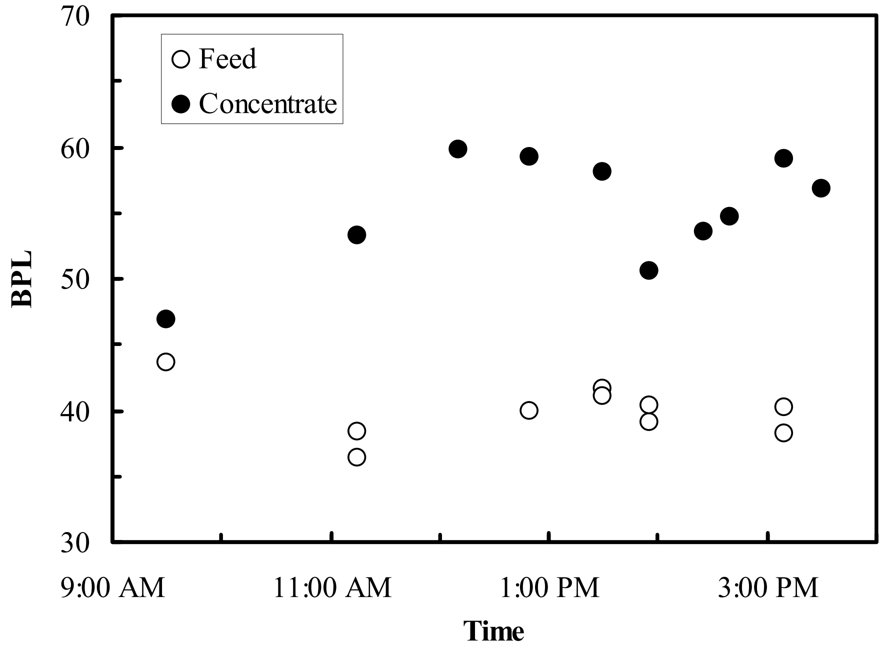
* Independent analysis of head sample.

A comparison of the column performance when using deep well and process water is shown in Table 12. Even though the increased water pressure was not in the optimal range for the operation of the infusers, there was a marked improvement in the performance of the column. For example, while the concentrate grades were comparable, the tailings grade was reduced from 21.6 BPL to 16.5 BPL with the use of process water. It would appear that the effect of slimes in the process water did not have a detrimental effect on the performance of the DEC unit. Since the amine dosage was the same in both cases, the reduction in the tailings BPL is attributed to increased bubble surface area in the column, resulting in approximately 10% higher BPL recoveries. It is therefore likely that with a high pressure water source, even higher BPL recoveries may be possible. The effect of process water observed here agrees with our earlier findings at Mine B in Part I of this study. In the light of these findings, the remaining tests were carried out using process water.

Table 12. Effect of Water Source on IP Flotation in a Column Cell.

Water Source	Concentrate BPL	Tailings BPL	BPL Recovery, %
Deep well	59.8	21.6	71.9
Recycled process	59.2	16.5	81.4

Figure 47 shows a real-time comparison of feed, concentrate and tailings grades with time. The discontinuities observed between 9:30 am and 11:15 am and between 12:10 pm and 12:50 pm were to enable us effect changes to the column set-up. In the first instance, we shut the system down to change the diameter of the hydrocyclone underflow from 1.5” to 0.75”, while in the second instance it was to switch the water source from deep well to process water.



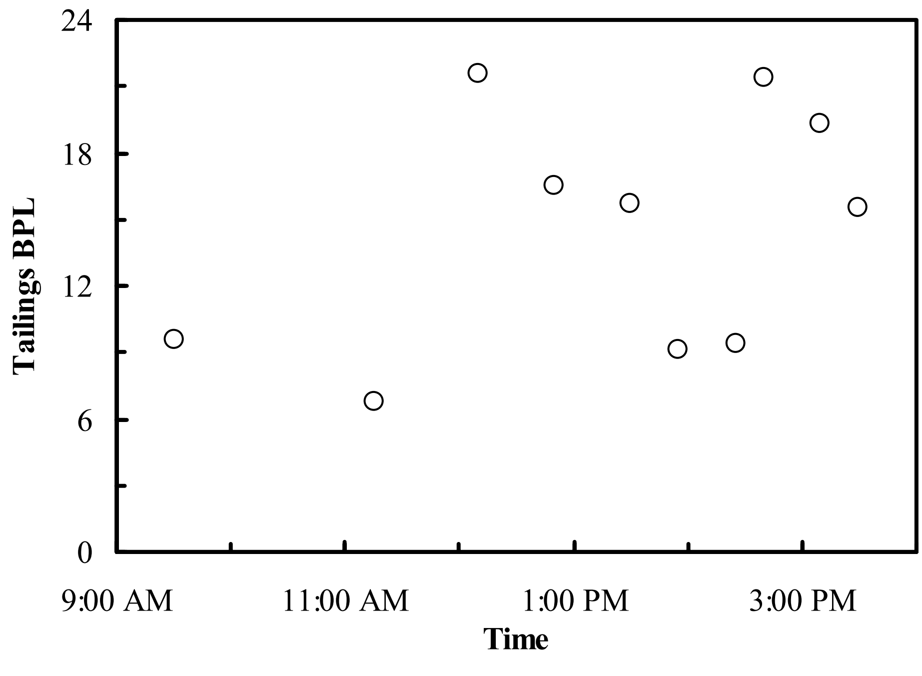


Figure 47. Real-Time Variation in Feed, Concentrate and Tailings Grades for IP Flotation.

For the tests reported here, the IP feed rate to the DEC unit was approximately 5.3 TPH while the amine dosages employed were 0.49 lb/T and 0.73 lb/T. The results clearly show that the DEC unit was successful in upgrading the IP feed from 40 BPL and 45% insol to a 56-60 BPL and insol of 18-22% at BPL recoveries of 72-96% (see Figure 48). While the tailings grade varied from 9-21 BPL, the highest concentrate grades were obtained when the tailings grade were in the range of 15.5-20 BPL. It is evident from the foregoing that the combination of the DEC and the collector from Arr-Maz were effective in reducing the insol content of the IP product.

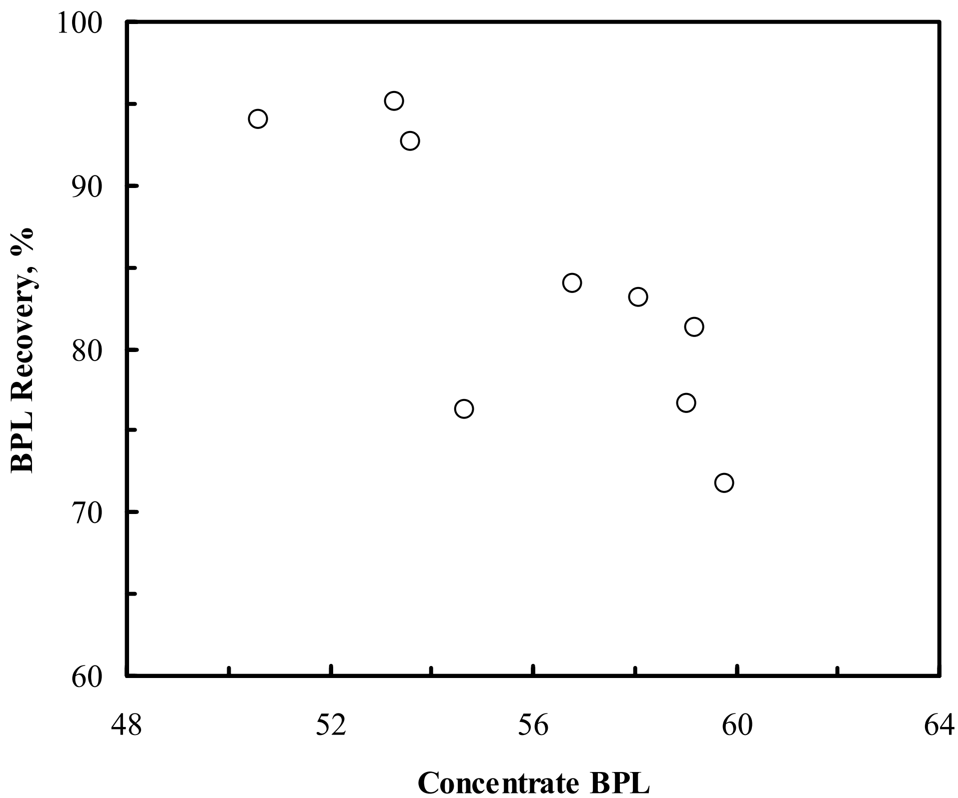


Figure 48. Grade-Recovery Profile for Run 1.

Given the performance of the DEC unit on processing IP, additional tests were carried out to evaluate its separation characteristics. Another test was run with a solids feed rate of 4.16 TPH and amine dosages in the range 0.32-0.63 lb./T. Figure 49 shows a real-time comparison of the feed, concentrate and tailings grades during the test period. For this test, most of the samples were collected at an amine dosage of 0.63 lb./T. The results show that with an amine dosage of 0.63 lb./T, the DEC unit operating with process water can produce a 56-58 BPL concentrate at 80-90% BPL recoveries (see Figure 50). At this amine dosage, the average concentrate and tailings grades were 57.4 ± 1.1 BPL and 14.1 ± 3.0 BPL respectively, while the average BPL recovery was 87.0 ± 4.2%. The result in Figure 50 suggests that amine dosages lower than 0.6 lb./T may not be optimal for the flotation of IP.

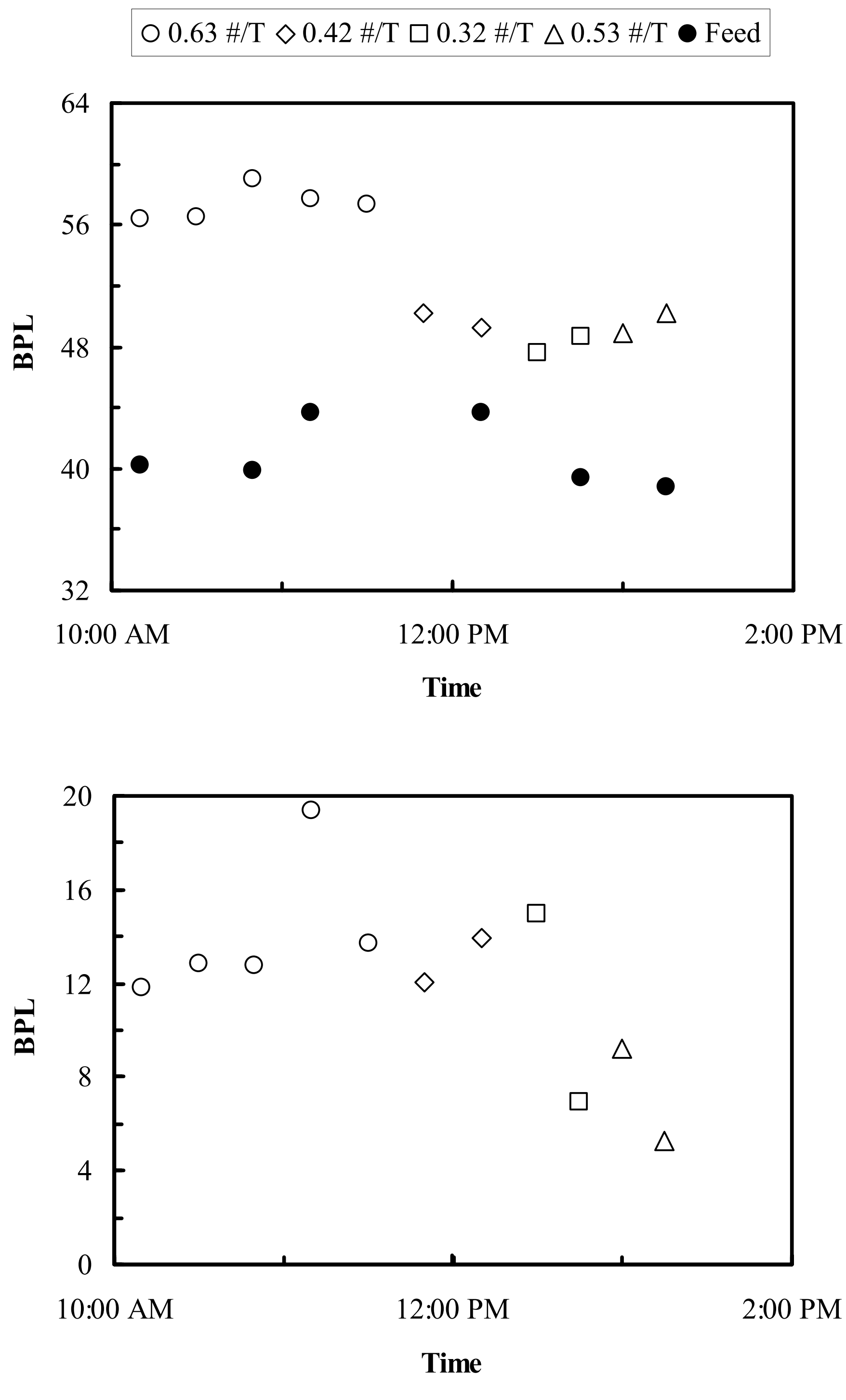


Figure 49. Effect of Amine Dosage on IP Flotation (Run 2).

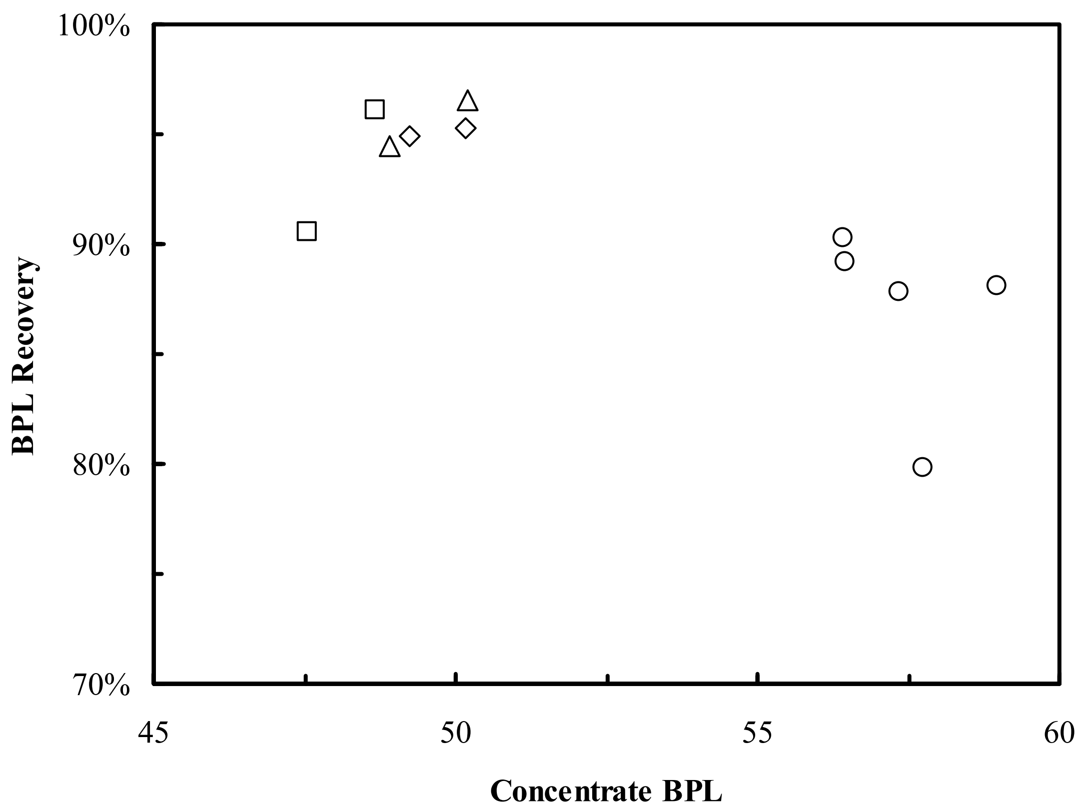
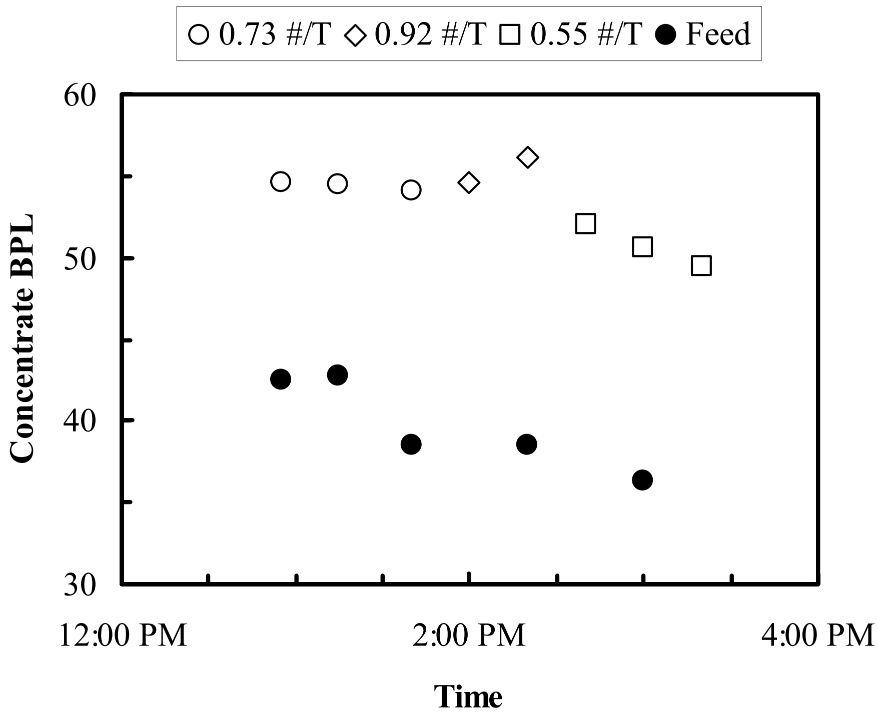


Figure 50. Grade-Recovery Profile for Run 2.

Another test was carried out with a solids feed rate of 4.74 TPH while the amine dosage was varied from 0.55-0.92 lb./T. The purpose of this test was to establish an optimum range of amine dosages for IP flotation. Figure 51 shows a real time comparison of the feed, concentrate and tailings grade during the test with the operational amine dosage. During the test, the feed grade decreased with time. While the concentrate grade exhibited the same trend, the effect was less pronounced when the amine dosage was increased to 0.92 lb/T. Figure 52 shows a grade-recovery profile for this test. In general the results show that with a 38-42 BPL feed, the column can produce a 54-57 BPL concentrate at 87-93% BPL recovery.



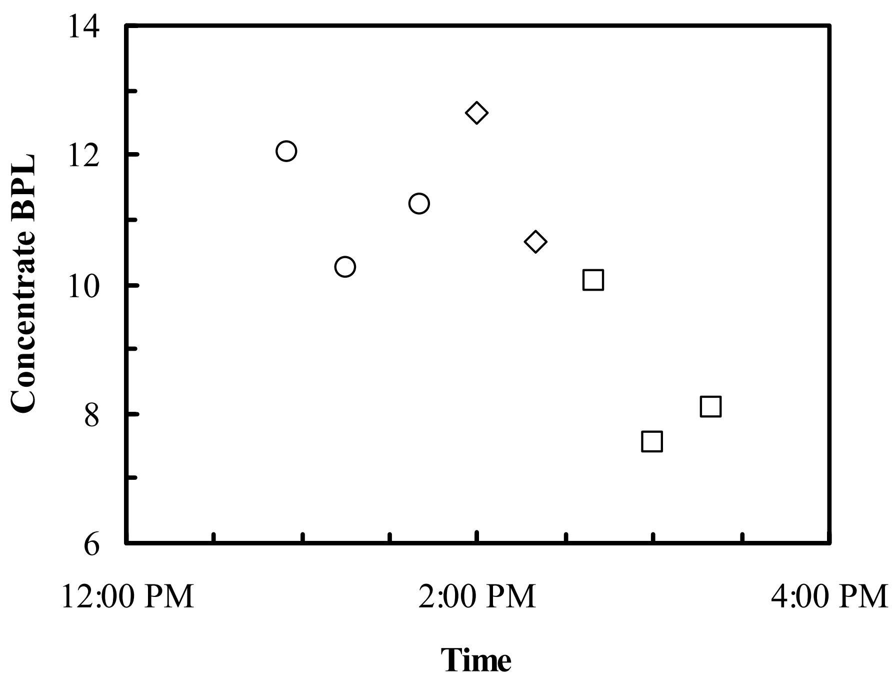


Figure 51. Effect of Amine Dosage on IP Flotation (Run 3).

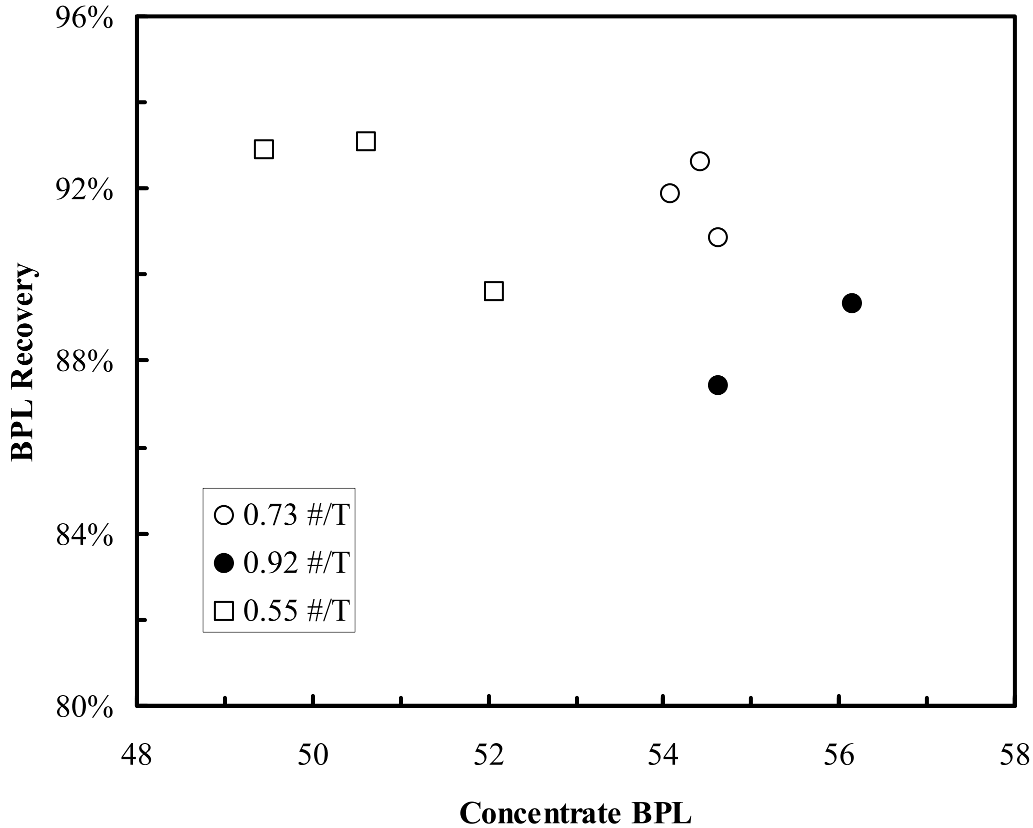


Figure 52. Grade-Recovery Profile for Run 3.

Our results show that the pilot column can process up to 5.3 TPH of IP, however, for scale-up we suggest that the maximum feed rate be based on 5 TPH in the pilot plant unit. Using this value, it is estimated that a full-scale unit with a diameter of 10 ft will successfully handle 125 TPH of IP. The tonnage suggested here is slightly higher than that recommended for coarse rougher amine feed at Plant A in Part I of the report. Since feed rates higher than 4 TPH were not evaluated in Part I of the report, we suggest that the values determined here be applied to the coarse rougher feed at Plant A.

In order to determine the optimum separation results from the flotation of IP, we plotted the yield index as a function of insol rejection (see Figure 53). Also shown in Figure 53 is the size-by-size data. The results show that the optimum separation corresponds to a BPL recovery of about 87% and an insol rejection of about 77%. Using these values and feed grade of 40-45 BPL with insol of 38-45%, the corresponding product grade is estimated to be in the range of 58-62 BPL with insol of 16-20%. The tailings grade is estimated to be in the range of 12-15 BPL. It is evident from the foregoing that amine flotation of IP in a column cell represents a viable process option for upgrading the feed to the chemical plants.

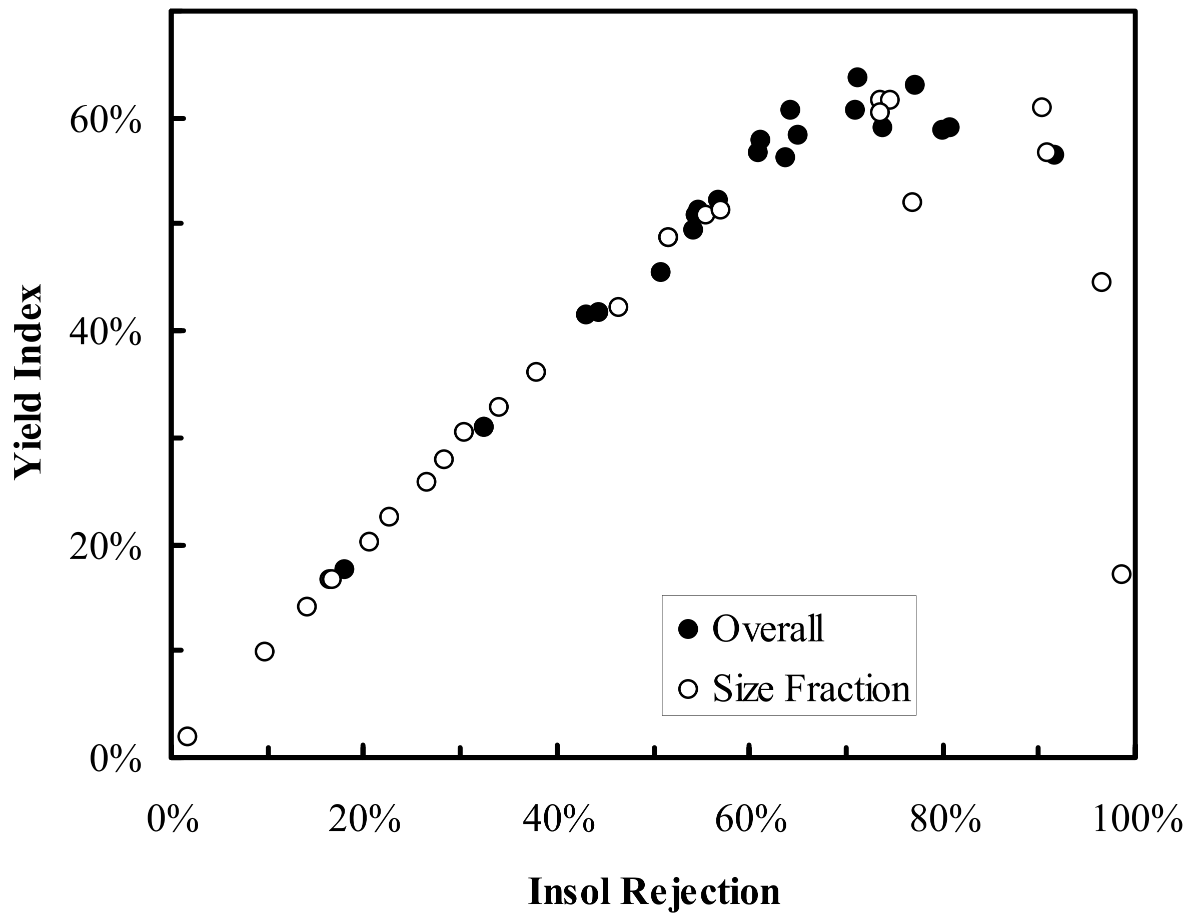


Figure 53. Locus of Separation for IP Flotation at Plant A.

PLANT B

Our IP tests were initiated at this site and as such the results were used to provide insights into amine flotation of IP in a column cell. The feed solids concentration at this site was low and the use of the hydrocyclone to dewater it produced only marginal improvements in the solids concentration. Table 13 shows a typical composition of the feed. The results in Table 13 show that the insol content was concentrated in the -20 mesh fraction. Since the +14 mesh fraction is of sufficient purity, attempts should be made to separate this fraction with the pebble product.

Table 13. Characteristics of IP at Mine B.

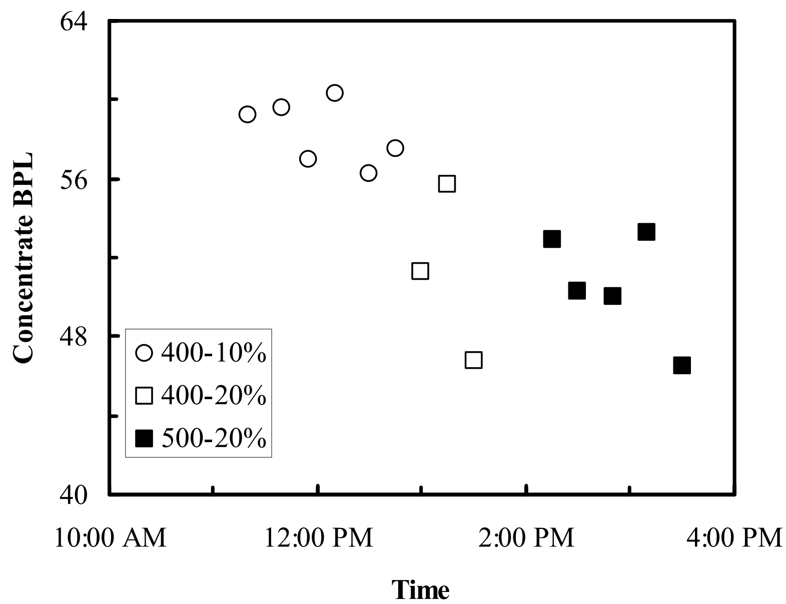
Size Fraction, Mesh	Weight Fraction, %	BPL Content, %	Insol, %
+14	13.9	64.4	9.2
-14 + 20	26.7	59.8	15.5
-20	59.4	34.6	51.0
Calculated Head	100	45.5	35.7
Head*		45.0	36.4

* Independent analysis of head sample.

Our initial tests focused on determining the concentration of amine solution to use in our test work. We initiated our tests with a 10% amine solution which was subsequently raised to 20%. Figure 54 shows the real time variation of the concentrate and tailings grades. In Figure 54, the open symbols represent a pump setting of 400 ml/min while the closed symbol was for a setting of 500 ml/min. Our results show that the highest concentrate grades were observed when the amine concentration was 10%. When the amine concentration was increased, the concentrate grade dropped. This observation is attributed to the difficulty in pumping the solution when the amine concentration exceeded 10%.

Another test was repeated in which the amine concentration was reduced to 5%. During this test, the solids feed rate through the column was 1.96 TPH. Figure 55 shows a real-time variation of the concentrate and tailings samples. Owing to the dilute nature of the feed, the amine dosage was quite high and ranged from 1.59-2.53 lb./T. In general, the concentrate grade increases with amine dosage. While the quantities employed in the present study are quite high, with increased solids concentration, we expect that the amine dosage can be lowered to the levels experienced in Plant A above. The amine tailings were quite low, thus suggesting that the column was operating under free flotation conditions. The BPL recoveries during this test were consistently higher than 96.5%.

A size analyses of concentrate and tailings samples is shown in Table 14. The results show that the concentrate grade of the intermediate fraction (-14+20 mesh) was lower than that of the -20 mesh and +14 mesh fractions. This would suggest that insol liberation occurs at finer sizes (-20 mesh).



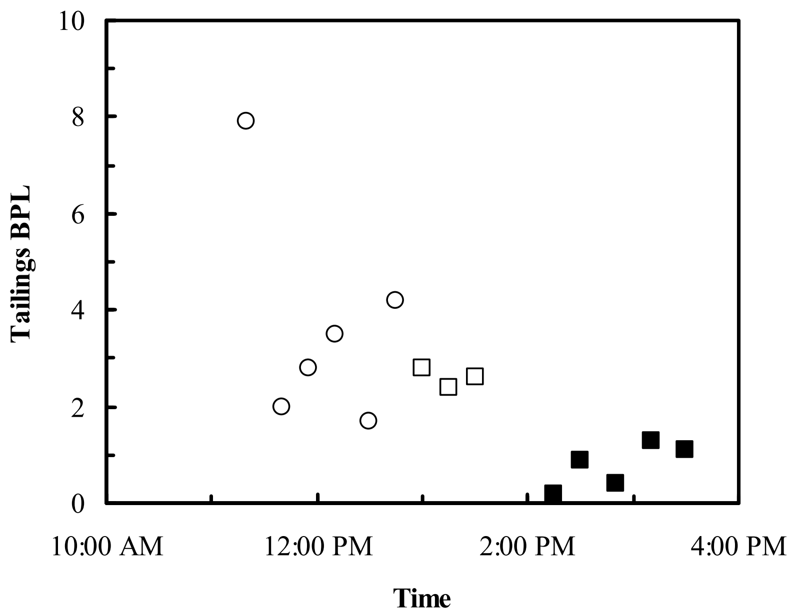


Figure 54. Effect of Amine Concentration and Dosage on Flotation Performance.

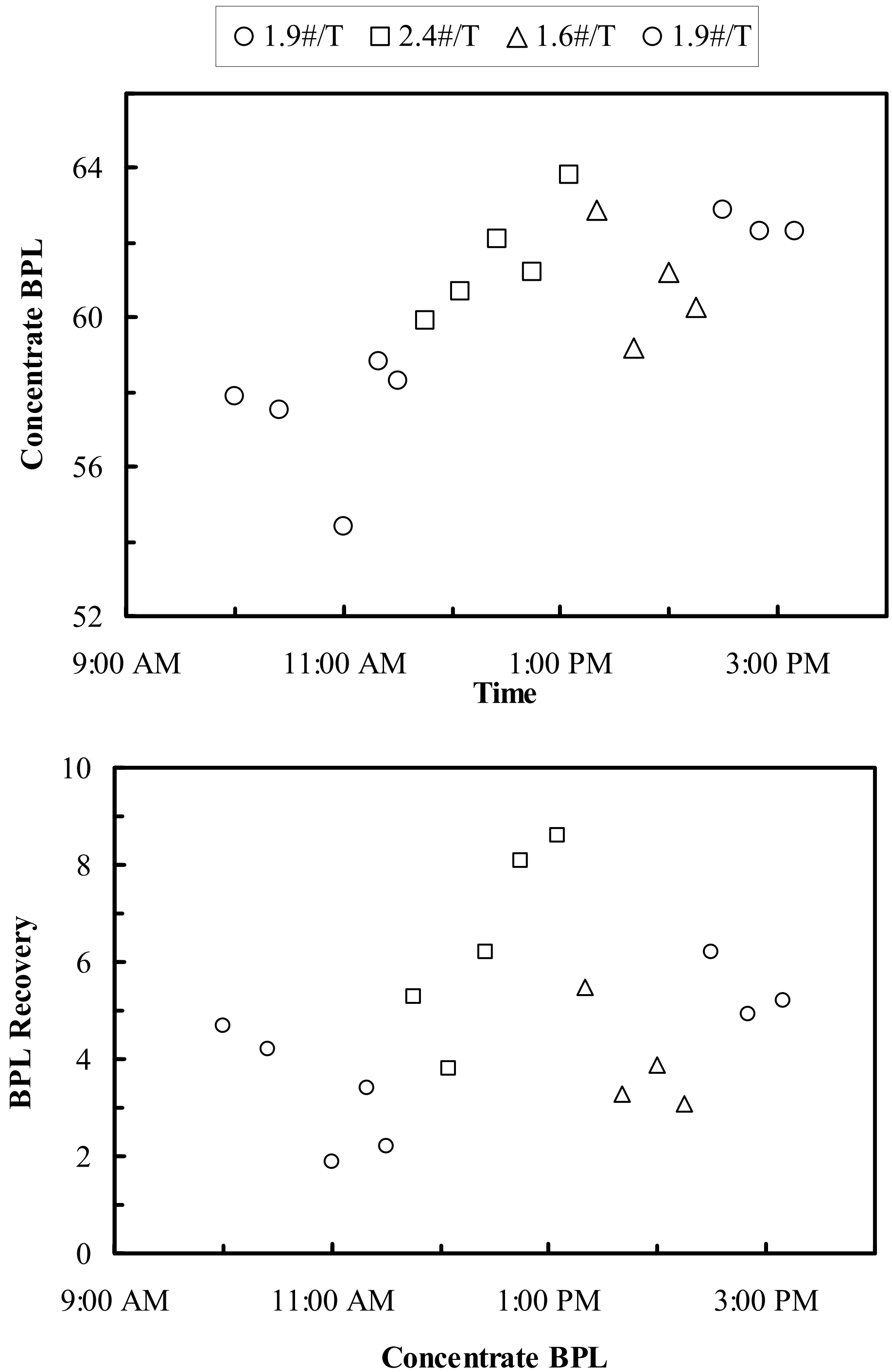


Figure 55. Effect of Amine Dosage on IP Flotation for Run 2.

Table 14. Typical Composition of Concentrate and Tailings Samples at Plant B.

Size Fraction, Mesh	Concentrate			Tailings		
	Wt., %	BPL	Insol	Wt. %	BPL	Insol
+14	34.4	65.0	8.5	0.2	14.1	80.5
-14 + 20	29.1	54.6	23.9	1.5	57.4	19.1
-20	36.5	62.6	12.0	98.3	3.8	93.8
Head		61.1	14.3		4.62	92.7
Head*		60.9	14.2		4.3	93.9

* Independent analysis of head sample.

A major difficulty with in-plant test work in the variability in the feed characteristics, however, the results provide a better representation of performance under normal plant operating conditions. Another test was carried in which feed samples were also taken for analysis. Figure 56 shows the real-time variations in the feed, concentrate and tailings grades. For this test, the solids feed rate was approximately 1.7 TPH while the amine dosage was varied from 2.2-2.9 lb./T. In general, the concentrate grade mirrors that of the feed. The tailings grades were considerably high and in the range of 12-18 BPL.

In order to determine the separation characteristics for IP flotation at this site, we plotted the yield index as a function of insol rejection (see Figure 57). In general, the results for all three runs lie on the same line. Since a maximum was not observed, it appears that the optimum separation for IP was not attained at this site. It is suggested that additional test work at this site may be needed to establish the separation conditions for IP flotation.

In general, the BPL recoveries at this site were generally greater than 95%. If IP flotation can be accomplished, it will result in substantial savings to the plant operators. For example, this site generates both fine and coarse flotation feeds which are initially subjected to fatty acid flotation to generate a rougher concentrate. Thus, if the feed is sent directly to an amine flotation, the rougher flotation stage and sulfuric acid deoiling stages will be avoided. It is expected that thus process change will result in considerable equipment, reagent, operating and maintenance costs.

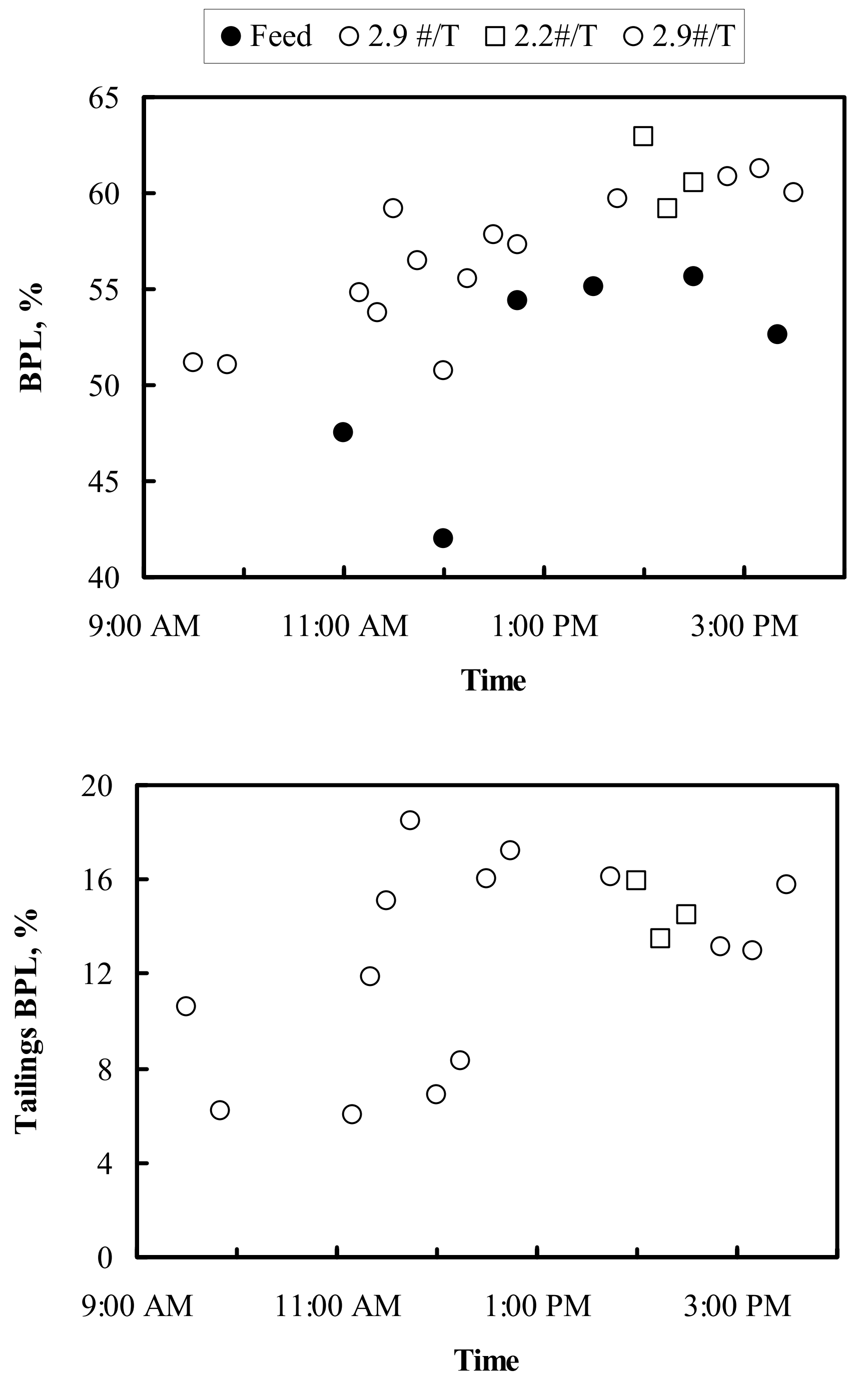


Figure 56. Effect of Amine Dosage on IP Flotation for Run 3.

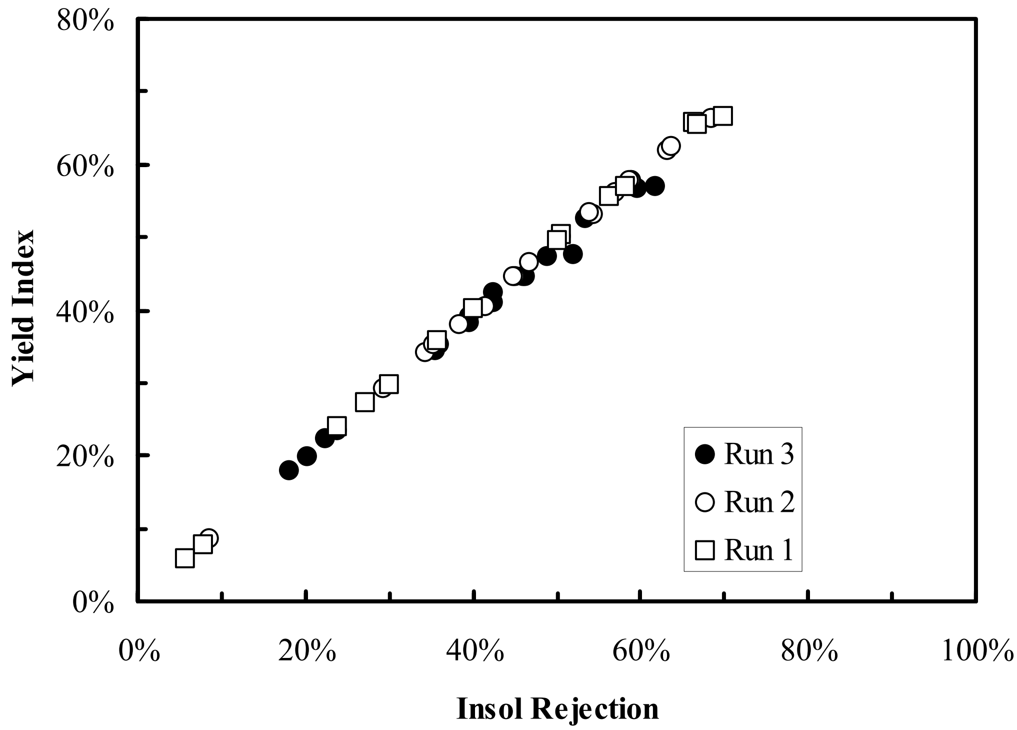


Figure 57. Locus of Separation for IP Flotation at Plant B.

PLANT C

The feed at this site was very dilute so we decided to use the hydrocyclone to dewater it. Initially the underflow diameter was set to 1.5" but was later reduced to 0.75". Even with this modification, the highest per cent solids encountered was 25%. The amine dosage was varied as indicated at the other sites and the samples turned over for analysis. However, the chemical analyses indicated that the concentrate grades were lower than those of the feed, especially for the coarse (+ 20 mesh) fractions. The reason for this anomaly are not clear at the present time, but may be related to sampling issues. Since the phosphate value was concentrated in these fractions (see Table 15), we did not see any benefit in further analysis of the results as computed size-recoveries were over 100% for the coarse fractions. For example, the predicted concentrate grade for the fractional recovery data is 76.0 BPL while the actual value calculated is only 61.2 BPL. Since this was the only site where this anomaly was encountered, our recommendation is to repeat the test at site. In the case of a repeat evaluation, it would be desirable to identify an appropriate sampling location which will either deliver feed at greater than 50% solids or with sufficient head to be dewatered in the hydrocyclone to a solids concentration in the range of 60-75%. It may also be desirable to evaluate the processing of the feed to the IP screens.

Table 15. Typical Mass Balance on Feed, Concentrate and Tailings Samples from Plant C.

Size Fraction, Mesh	Feed		Concentrate BPL	Tailings BPL	Fractional BPL Recovery, %
	Wt. %	BPL			
+16	67.0	65.3	63.4	57.7	130.5
-16+20	17.4	61.4	60.6	42.2	103.0
-20+28	9.9	58.7	56.8	16.7	101.4
-28+35	3.2	46.2	56.1	5.8	97.5
-35	2.4	28.8	58.3	6.2	87.8
Head		62.5	61.4	9.2	100.3
Head*			76.0		

* Calculated from fractional recovery data.

CONCLUSIONS

Amine flotation of IP product has been carried out in a column (DEC) cell operated at feed rates of 1.7-5.3 TPH. The amine employed in these tests was provided by Arr-Maz and was of the amine acetate family. Analyses of the results show that the optimum separating conditions correspond to a BPL recovery of 88% and an insol rejection of 75%. With a feed containing 40-45 BPL and 38-45% insol, the results show that the IP can be upgraded to 58-61 BPL and insol of 15-18%. Limited amine flotation tests were also carried out on the feed to the IP screens. However, our results show that the DEC unit can upgrade a feed containing 30-33 BPL and insol of 50-56% to a 56-59 BPL and insol of 16-20% product at BPL recoveries of 60-70%. Upgrading IP in a column cell offers significant benefits to plant operators including elimination of capital equipment, lowering of operating and maintenance costs.

The separation of silica from IP rock will increase the grade of the phosphate product. It will reduce the costs associated with the transportation of the product and its subsequent processing at the chemical plant. These reductions in production cost will ensure that Florida phosphate concentrates and products derived from it will remain competitive on the international market. The adoption of this process will reduce the quantities of waste generated in upstream operations.

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APPENDIX A

CALIBRATION CURVE FOR THE AIR BLOWER

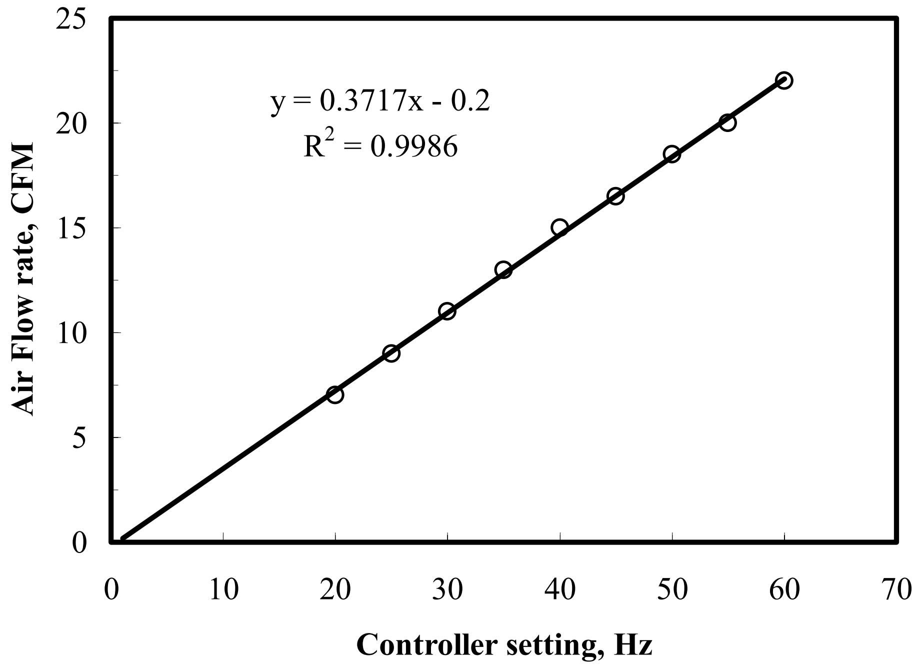

Figure A-1. Calibration Curve for the Air Blower.