

Chapter 16

Potash Flotation and Process Optimization at Great Salt Lake Minerals Corporation

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Abstract

Great Salt Lake Minerals (GSLM) uses flotation to utilize low grade harvest evaporites in the leaching/ crystallization process. A final potash product coloration problem was encountered that was attributed to AERO® 801s promoter - magnesium ion precipitation in flotation which carried into the dissolution/crystallization circuit. Bench studies resulted in changing 801s to 845 which provided acceptable product brightnesses and higher flotation potassium recoveries. This is a case history reviewing the unique GSLM processing scheme, the bench and plant work identifying the potash quality problem, the bench work and solution, and subsequent process study and circuit optimization efforts.

INTRODUCTION

The Great Salt Lake Minerals Company (GSLM) confronted a low brightness potash fertilizer product quality issue in 1989. GSLM's competition had higher potash brightnesses which were perceived as being superior, although the brightness characteristic was actually an aesthetic rather than a technical problem. The coloration was in evidence during previous campaigns and was overcome by not running flotation (to upgrade low grade harvest materials) when producing potash requiring high brightnesses. Changed economics during 1989 spurred efforts to recover much more of the low grade harvest necessitating running flotation full time. Therefore, identification and solution of the coloration problem was needed.

BACKGROUND

The Great Salt Lake Mineral Company, which is located on the Great Salt Lake just west of Ogden, Utah, began operations in 1970 and produces potash fertilizer (K_2SO_4), halite (NaCl), salt cake (Na_2SO_4), and MgCl₂ brine from Great Salt Lake brine. Based on 1991-92 data, the lake brine intake at GSLM had ion concentrations of 0.4% K, 8% Na, 0.7% Mg, 14% Cl, and 1.5% SO_4 . According to the Encyclopedia of Chemical Technology (1971), the Great Salt Lake has salt ion concentration gradients with the highest ion concentrations in the north arm of the Lake where the GSLM intake is located. The magnesium and sulfate levels and the manner in which the solar ponds operate determine the resulting evaporite mineralogies at GSLM (see Patent # 3,589,871).

During the summer months, evaporation in a series of 80 solar evaporation ponds results in the precipitation and pond deposition of soluble salt minerals including halite (NaCl), schoenite ($K_2Mg(SO_4)_2 \cdot 6H_2O$), kainite ($KClMgSO_4 \cdot 3H_2O$), epsomite ($MgSO_4 \cdot 7H_2O$), and carnallite ($KCl \cdot MgCl_2 \cdot 6H_2O$). By controlling the brine flows through the pond system, the salt concentrations are managed to obtain bulk halite deposition at the beginning of the pond system with the remaining salts precipitated in the following ponds.

Pond harvesting, usually begun in September, is accomplished through the use of special equipment to scrape the salt from the pond floors. The potash harvest is segregated into low, marginal, and high grade plant feeds depending on K, Mg, and SO_4 content and NaCl concentrations. The marginal and high grade salt harvest materials are fed directly to the potash production plant where they are treated in a water leaching and potash crystallization process to produce K_2SO_4 fertilizer in accordance with U.S. Patent 3,634,871. Flotation plant feed analyses during the 1991-92 campaign, for example, showed that the low grade harvest mineral composition consisted of 58% halite, 15% schoenite, 21% kainite, and 6% epsomite.

A flotation plant was built in 1975 to upgrade the low grade materials for direct utilization in the potash plant rather than recycling to the ponds. Figure 1 shows the flotation process flowsheet. Low grade feed is conveyed and sluiced into a ball mill with plant end liquor (PEL). PEL is a saturated solution recovered from the potash plant CCD wash circuit. Note that, although the PEL is saturated, it is not likely to be in complete chemical equilibrium with the highly variable fresh salts and salt entrainment compositions fed to grinding. Cyclone classification immediately follows grinding for size control. The classified product passes through three reactors, which provide several hours slurry residence time, giving the salt slurry time to equilibrate. Tippin and Huiatt (1977) reported that this five ion mineral system is unstable and considerable conversion of the kainite and carnallite to schoenite occurs. Following the conversion and equilibrium reactions, the slurry mineralogies are typically 58% halite, 38% schoenite, 3% kainite, and 1% epsomite -- a significant mineral conversion to schoenite.

Anionic and cationic collectors (AERO® 801s promoter and diamine, respectively) are conditioned in the slurry for several minutes

before flotation. Frother is added to the flotation cell feed box. The flotation circuit consists of 8-14.2 m³ cell rougher and scavenger banks with the froth concentrates from each stage combined to feed the water leach section of the potash plant. The flotation tails flow to a holding pond for reintroduction to the evaporation pond system and salt recovery.

During the early flotation development work, as Tippin (1977) explained, anionic reagents were found to be the most effective schoenite collectors as compared to amines used in sylvite flotation systems. Data from the 1976-77 production campaign showed that caprylic acid provided 11-14% K flotation concentrate grades (schoenite theoretically contains 19% K) with recoveries in the 75-90% range. The plant results confirmed the pilot plant findings of Huiatt, Tippin, and Potter (1975) that found that fatty acids easily floated schoenite but was ineffective for kainite collection.

The collector suite was changed from fatty acid to the petroleum sulfonate-diamine suite in the late 1970-early 1980 period, but the reason for the change is not fully understood. By happenstance, the abnormally high amounts of precipitation during the early 1980's raised the Great Salt Lake and flooded out the GSLM pond system resulting in potash operations cessation from 1984 to 1987. As a consequence, a certain amount of "institutional knowledge" was lost and limited organized records are available to explain why the two component collector system had been implemented. By 1988, the pond floors had been reestablished and sufficient harvest deposited to resume plant operations.

Problem-Opportunity

After the 1988 plant restart, the final potash product brightness levels, measured on a Photavolt Meter Model 670 with a green filter, ranged from about 39 to 67 (reference value of 100 was with white filter paper) with competitive products having values in the mid 80 range. In the pre-flood days, flotation was shutdown during those periods when high potash brightnesses were required for specific products and brightnesses comparable to the competition were obtained. It was known that adding flotation concentrates into the potash plant feed degraded final product brightness. After plant restart, economic factors made maximization of potash production necessary and resulted in full time flotation plant operation while providing little flexibility for high brightness potash production runs. Therefore, a metallurgical project was undertaken to improve the product color/brightness and %K recovery, if possible.

One of the primary sources of potash brightness deterioration was found to be related to the AERO® 801s promoter, a water soluble low molecular weight petroleum sulfonate collector which was apparently precipitating in the slurry. Diagnosis of the problem was accomplished in a simple lab qualitative test where each collector was added to controlled lab solutions made up of Na, K, and Mg salts of Cl and SO₄ ions. There was a definite precipitation reaction when 801s was added to the Mg salts of both Cl and SO₄, and there were no apparent reactions in the other solutions. The magnesium:801s precipitates were dark, oily masses and similar to that found on plant thickeners and tanks. Neither the diamine nor frother formed any precipitates.

Laboratory Investigations

Once it was established that Mg:801s

precipitation carryover to the water leaching-potash crystallization circuit was at least part of the coloration problem, the solution was broadly defined as changing the collector suite. Four bench flotation test series were conducted to identify candidate 801s collector replacements. Various collectors were evaluated and included petroleum sulfonate variants, sulfosuccinamates, sulfosuccimates, and guanidine based chemistries.

The loss of historical process information became a problem because a test procedure was not available and little was known concerning the rate of kainite to schoenite conversion. As a consequence, although some relevant information was obtained, the first 3 test series were, in essence, procedure development.

Fresh low grade harvest samples were collected for each test series. These feed samples had grades ranging from approximately 3.0 to 7.0% K. The samples were crushed to size with a small hammer mill before flotation. Bench flotation was conducted at 30% solids, after accounting for about 30% brine entrainment, using PEL for dilution. Due to the chemical disequilibrium and reactions that occur, conditioning and flotation times were held absolutely constant, as well as PEL flotation dilution additions, within each test series in order to make the mineralogies between test series as constant as possible.

Table 1 presents the fourth test series results where various anionic 801s substitute collectors were evaluated following the test procedure development work. The diamine dosages were held constant in this series with only the anionic collector dosage levels varied. AERO® 845 promoter, which is a sulfosuccinamate ester, provided about 80% K recovery at a 9-10% K concentrate grade as compared to the standard suite which provided 35-38% K recoveries and 13-14% K grades. AERO® 830 promoter, a sulfosuccinamate, provided 63-70% K recoveries at 12.5-13.0% K grades and was observed to produce less frothing than 845 but more than 801s. The relative frothing characteristics of the different collectors probably accounted for the concentrate-recovery differences obtained.

The 830 and 845 concentrate brightness levels were equivalent to or lower than that obtained for 801s, which was initially thought discouraging. However, the feed sample had a low 51 brightness value suggesting the existence of considerable foreign color bodies. Because the 845 and 830 recovered more weight than 801s, enhanced entrainment of these coloration materials into the froth concentrate may have resulted. This observation eventually led to an investigation for non-reagent caused coloration agents.

Since neither 845 nor 830 precipitated in the earlier discussed precipitation test procedure and because of the encouraging metallurgy obtained, these two collectors were recommended for plant evaluation as 801s replacements. There was some concern about the high frothing capability of 845, so a 50:50 combination of 830 and 845 was recommended for plant evaluation with the thought that the collector combination would provide a more well behaved froth in the plant.

Given the importance of resolving the coloration problem, plant testing was immediately begun at this point, although further refinement of bench test procedure, evaluation of the collector interactions, and determination which mineralogies were collected by each collector probably would have been helpful. Application of metallurgical experience and intuition plays a

role in any flotation investigation but these played a particularly large role in this program.

Plant Testwork

A two shift "runnability" plant test was conducted on October 25 and 26, 1990 with the recommended test reagents. GSLM has a single flotation line so it was not possible to compare the test programs directly to the standard suite. On the first day, a 50:50 830:845 combination was used with the diamine and on the second day an attempt was made to run the circuit with 845 alone, without diamine.

The first days results showed that the 830:845 combination would acceptably replace 801s in the reagent suite. Operator assays showed that the 801s:diamine suite before and after test provided 84-92%K recoveries with concentrate grades ranging from 12.9 to 16.5% K compared to test reagent recoveries of 84-91% K with concentrate grades ranging from 10.2 to 15.5% K. The test on the second day with 845 alone was a failure and confirmed that the diamine was a necessary component in the reagent suite. The standard 801s:diamine suite %K recoveries were in the 84-91% K recovery range while 845 alone gave only 58-67% K recoveries.

A follow-up 7 1/2 day test with the 845:diamine combination was conducted to better define whether final potash product brightnesses would be increased and to evaluate possible downstream operational effects. This test was run from November 7 to 26, 1990, the results of which are given in Table 2. The potash brightnesses were increased from the mid 60's to mid 70's using 845. This improvement was considered significant. Additionally, it was observed that the oily floating masses in the potash plant were significantly decreased during the test. Metallurgically, the average concentrate %K grades achieved with the standard and test reagent suites were similar with the 845:diamine combination giving at least a 2% K higher recovery compared to the standard 801s:diamine combination.

These potash product brightness and metallurgical results lead to substitution of 845 for 801s in the plant reagent suite. GSLM began using 845 on a regular basis establishing its performance and adjusting to the performance differences to 801s. A considerable amount of effort was made to collect and analyze the following campaign data (1991-92) and begin studying the 845 process response --some of the data is summarized in Table 3.

Follow-up Work

An interesting conclusion derived from analysis of the harvest and cell product mineralogy data collected during the second trial phase was that the conversion of kainite to schoenite was not as complete as earlier thought. The second plant test mineralogy information provided in Table 4 shows that schoenite accounts for about 80% of the potassium based minerals reporting to flotation (after grinding and conditioning several hours). Cause for the less than complete conversion is obviously chemical equilibrium based but may account for the late 1970's-early 1980's caprylic acid to 801s:diamine suite change.

Although the potash fertilizer product brightness levels were meaningfully and significantly increased to the mid 70 range, there obviously was still room for further improvement. Indications from the bench and plant reagent investigations that other

factors also accounted for part of the potash brightness degradation problem lead to the finding that insols were the color bodies being introduced into the system. The insols may have been originating from the pond dikes, lake bottom sediments introduced during the pond flooding period, and from an old road bed that had been installed in the ponds.

The insol problem contributor became obvious on the crystallized potash product filter where the insols migrated to the edges of the filter cake. Efforts during the 1991-92 harvest period emphasized minimizing the introduction of insols during harvesting. These actions made a visually positive impact, although the improvement has not been quantified.

A follow-up bench investigation was conducted to assess the effect of varying 845 and diamine dosages on metallurgy. The test goal was determination of the optimum 845 and diamine dosage ratio. The 845, diamine, and Aerofroth® 70 dosages were set based on a standard three factor experimental test design with the %K recovery and grade data subsequently least squares fitted to exponential equations.

Incorporation of a 60 minute slurry reaction conditioning stage apparently overcame the prior difficulty of achieving reproducible and meaningful bench results and resulted in near duplication of plant metallurgy. Several collector dosage ratios provided %K recoveries in the high 80% to low 90% range with %K concentrate grades in the 14-17% range. Other tests gave low recoveries which should be expected from a robust test series using a wide range of collector dosages and ratios. Response surface contour plots of %K recovery and grade against 845 and diamine dosages are presented in Figure 2.

Two conclusions were obtained from this investigation: a) confirmation of plant findings that both collectors are required to maximize recoveries, which suggests significant interactions between the collectors and b) the optimum bench 845:diamine dosage ratio, given that 845 is less expensive than the diamine, was in the 3:1 to 5:1 range. As shown in Table 3, the average 1991-92 campaign dosage ratio was 1.2:1 and it is not known whether the trend discrepancy between the bench results with the plant experience may indicate that the bench procedure still required further modification, that the mineralogies in the bench feed sample were unrepresentative to typical plant feed and/or the dosage ratio employed by the plant operators is due to past experience and preferences rather than requirements.

A study has been undertaken, as yet uncompleted, analyzing operating data for the entire 1991-92 campaign in an attempt to define the most important operating variables affecting the metallurgical process and response trends. While there were episodes of 90%+ %K recoveries for periods during the campaign where the reagent addition rates were relatively stable, these periods were demarcated with shorter periods where the %K recoveries were in the mid 80% range. The plant operators varied reagent dosages in an attempt to improve recoveries which was usually attained at different reagent dosage levels and ratios than before the low recovery episodes developed. After the fact data analysis thus far has not found a reason for these periodic metallurgy fluctuations but more detailed mineralogy and process variable studies are planned during the 1992-93 campaign in an attempt to explain these response variations.

CONCLUSIONS

As explained in this case review, GSLM encountered a product brightness problem, part of which was caused by magnesium ion precipitation of 801s. Changing the anionic collector from 801s to 845 significantly increased the brightness values from the mid 60 to mid 70 ranges. The reagent change also increased %K recoveries from the mid to upper 80% K range to the low 90% K range. Further study found that insols were an additional coloration agent and salt harvesting procedure changes were made in an attempt to minimize insols in the plant feed.

As suspected, the testwork and investigations into the brightness problem uncovered unexplained process response aspects that require further assessment. These studies have continued in order to develop greater process understanding to continue the process optimization efforts.

Acknowledgements

The authors are grateful for the permission and support provided by Great Salt Lake Minerals Company and American Cyanamid Company to write this paper.

References

1. Encyclopedia of Chemical Technology, Supplemental Volume, 2nd ed., John Wiley & Sons: New York, 1971, pp 438-466.
2. Huiatt, J.L., Tippin, R.B., and Potter, G.M., "Potassium Salt Flotation From Great Salt Lake Evaporites," AIME Trans, Dec, 1975, pp 303-310.
3. Tippin, R.B., "Potash Flotation Process Handles Variable Feed," Chemical Engineering, July 18, 1977, pp 73-75.
4. Tippin, R.B., Huiatt, J.L., "A New Potash Flotation Process from Laboratory Testing Through Pilot Plant Studies and on to Full Scale Operation," presented at the 106th AIME Meeting, March 6-10, 1977, Atlanta, GA.

Table 2. Second Plant Test Results.

Dates	Dosage -- kg/tonne		Feed %K	Concentrate	
	801s or 845	Diamine		%K	%k Rec
11/7 - 12: Control	0.42	0.36	6.60	15.55	89.8
11/12-20: Test	0.56	0.56	6.54	14.61	94.1
11/20-26: Control	0.43	0.43	6.83	13.81	92.3

Table 3. 1991-92 Campaign Average Flotation Metallurgy Results.

	Average	Standard Deviation	95% Conf. Range
Feed Grade (%K)	7.2	1.4	4.4-10.0
Conc. Grade (%K)	15.6	1.5	14.1-17.1
Recovery (%K)	91.4	4.6	86.8-96.0
Reagent Dosages (kg/tonne):			
845	.51	.12	
Diamine	.42	.05	
MIBC	.02	.015	

Table 4. Comparison of Harvest and Flotation Feed Mineralogies During the Second Plant Test

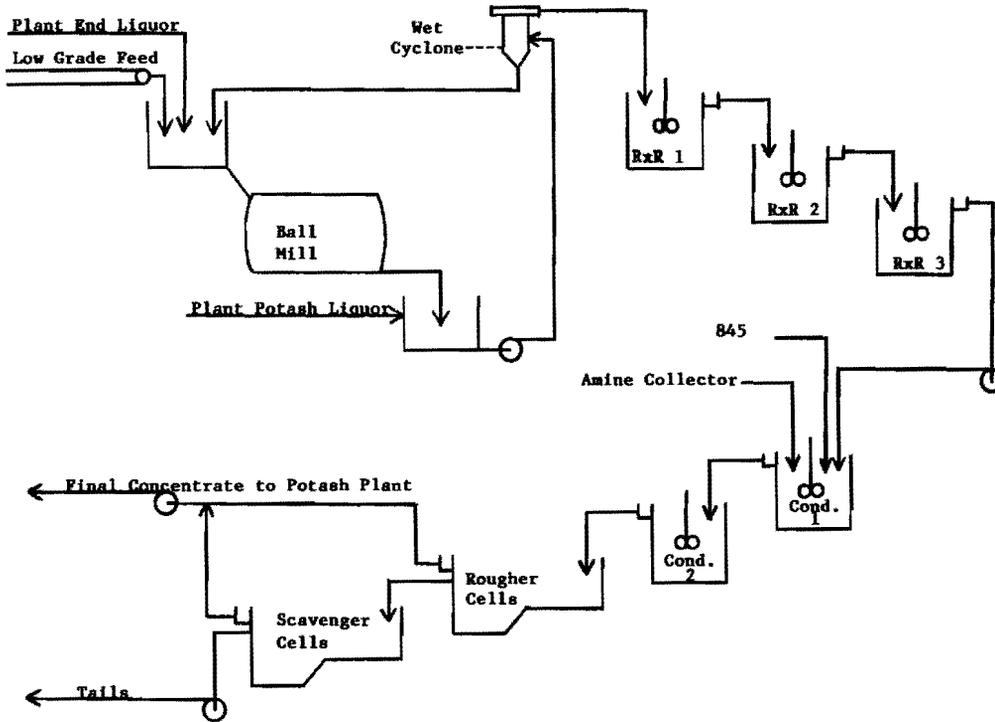
	Before Test		Test		After Test	
	Harvest	Float Feed	Harvest	Float Feed	Harvest	Float Feed
% Schoenite	16.1	30.6	10.8	28.5	14.2	28.5
% Kainite	23.6	6.5	27.8	8.9	20.0	7.8
% Epsomite	11.8	7.6	18.7	11.1	23.8	14.6
% Halite	48.3	55.4	42.7	51.3	41.9	49.1

Table 1. Bench Flotation Test Results.

Test	Anionic Collector	Dosage kg/tonne	Concentrate Analyses				Bright Index
			%wt	%K	%Na	%K Rec.	
1	801s	0.60	12.8	15.42	5.63	29.7	34
2	801s	1.00	18.5	14.86	5.04	38.7	42
3	801s	1.50	16.4	14.36	5.37	34.3	38
4	801s	2.00	18.1	13.71	5.38	35.4	44
5	845	1.00	43.7	12.65	8.95	78.4	35
6	845	1.50	45.1	12.38	10.11	80.2	30
7	830	1.00	34.3	12.72	9.17	62.9	43
8	830	1.50	40.3	12.53	9.35	70.4	42

The diamine dosage was held at a constant at 0.23 kg/tonne.

Fig. 1: GSL Minerals Flotation Flowsheet



K₂SO₄ RECOVERY AT 0.0125 KG/TONNE FROTHER 845 (KG/TONNE)

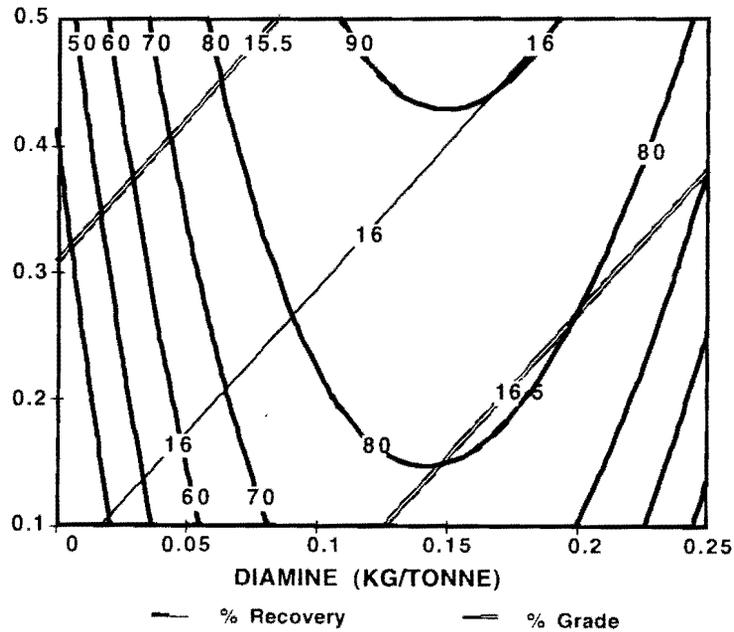


Fig. 2. K₂SO₄ Recovery at 0.0125 g/tonne Frother