TESTING AND EVALUATION OF MODIFYING REAGENTS IN POTASH FLOTATION

by

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ABSTRACT

Potash is the main source of Potassium which is one of the three basic plant nutrients along with nitrogen and phosphorus. Between 60 to 80% of the potash ore is processed by flotation, the other two important unit operations are hot or cold crystallization and electrostatic separation.

Almost all the published literature on potash flotation refers to the insoluble minerals as "collector robbers" and increased collector usage is mentioned as the consequence of the presence of slimes in potash flotation circuits. The real effect of the presence of slimes in potash flotation is far more complex.

The test program developed in the following chapters aimed to determine the most suitable modifying reagent(s) and the optimum dosage to optimize grade and recovery of KCl, and to evaluate various flocculants used in flotation desliming in order to determine the factors that affect flotation desliming efficiency.

Based on the results, it can be stated that guar gum is probably the most effective insoluble slimes depressant for the ores under the conditions tested. The depressing action of the guar gum is strong even at low dosages (50 g/t). Carboxymethyl cellulose shows poor depressing ability at low dosages, with increasing depression performance at high dosages (200 g/t). Synthetic polyacrylamides can hardly be considered insoluble slimes "depressants" as a considerable amount of the insoluble slimes present in the feed report to the concentrate.

Anionic flocculants increase insoluble slimes recovery when used instead of non-ionic flocculant at the same dosage. The recovery of KCl to the insolubles concentrate

increases with the use of anionic flocculants as well but in a much smaller proportion. The flocculation-flotation of insoluble minerals is a flocculant dependent process, and the addition of a "insolubles collector" or a frother, or nothing at all merely affects the kinetics of the process.

OBJECTIVES

Depressants play an important role in effecting selective sylvite flotation in potash ore processing. Long chain polysaccharide depressants have been used effectively in sylvite flotation from potash ores to depress insoluble slimes and thereby improve concentrate grades and KCl recoveries.

In this work, three different polymers – CMC and guar gum (polysaccharides) and a high molecular weight anionic polyacrylamide have been compared with respect to their depressing effect on insoluble slimes and the effect on KCl flotation. The objectives of this thesis include: determination of the most efficient modifying reagent(s) and the optimum dosage to optimize sylvite recovery.

Desliming by flocculation-flotation of the insoluble slimes is a process alternative to mechanical desliming, intended to control the harmful effect of insoluble slimes on the separation of sylvite from the gangue minerals. This thesis intends also to evaluate the various flocculants used in flocculation-flotation desliming in order to determine the factors that affect desliming flotation efficiency.

TABLE OF CONTENTS

LIST OF TABLES

2.1.   LIST OF FIGURES

ACKNOWLEDGMENT

1.0   INTRODUCTION

Potassium is one of the three basic plant nutrients along with nitrogen and phosphorus. There is no substitute for potassium compounds in agriculture; they are essential to maintain and expand food production.

Potash is a general term used for water-soluble, potassium-bearing salts formed from ancient seas, lakes and present-day brine lakes. The naturally occurring minerals with commercial value are potassium chloride (sylvite), potassium-magnesium chloride (carnallite), and potassium-magnesium sulphate (langbeinite or kainite).

Most of the potash produced is extracted from buried ancient evaporites by underground or solution mining. The balance is derived from evaporation of brines from landlocked water bodies in arid climates, such as the Dead Sea (Israel and Jordan), Salar de Atacama (Chile), or Great Salt Lake (USA).

Almost 70% of the potash ores are processed by flotation; the other two important unit operations are crystallization and electrostatic separation. Crystallization is also used in addition to flotation at many plants to increase recovery and increase particle size of the final product.

Because the potassium content varies between minerals, their product grade is commonly expressed in percentage of equivalent potassium oxide (K_2O), to standardize comparison. While pure sylvite is equivalent to 63.2% K_2O, langbeinite has an equivalence of 22.7% K_2O and potassium sulphate an equivalence of 54% K_2O. The

1

common agricultural minimum specifications for these products are 60%, 22% and 50%, respectively.

Potash products are marketed based on grain size, ranging from Fine, Standard, Coarse to Granular. The agricultural market for potash has moved toward coarser products to match the size of nitrogen and phosphate fertilizers used in batch blending. About 95% of potash produced worldwide is used in agriculture. The rest is used in several other industrial applications including glass manufacture, soaps, water softening, plastics and pharmaceuticals.

In most Canadian potash mines, sylvite (KCl) is separated from halite (NaCl) and from various other minerals by froth flotation in saturated brines.

It has long been observed that the presence of very fine particles (below 20 microns) can adversely affect the separation of coarser particles from mixed mineral suspensions. These fine particles are known as slimes in the mineral industry. Because of their relatively high specific surface area, slimes consume much of the reagents intended for the separation of the coarser particles in froth flotation. Slime particles also attach themselves to the coarser mineral particles. The consequence is a great loss of separation efficiency of the desired coarser particles, as their surfaces are coated with slimes and become inaccessible to flotation reagents.

The deleterious effects of the undesirable slimes can be controlled in most cases by a desliming step, which consists of separation of all fine particles below 20 microns from the slurry ahead of the potash separation by flotation. This practice, however, results in

excessive losses of the valuable minerals to tailings. Mechanical desliming is carried out by screening and classification including combination of hydroseparators and hydrocyclones. It can also be achieved by flocculation-flotation. Both desliming methods use modifying reagents, slime depressants and flocculants, to control the harmful effect of slimes on potash flotation.

2.0 SOLUBLE SALT FLOTATION

2.1 Particularities of Soluble Salts Flotation

Since potash ores contain water-soluble salts, flotation has to be carried out in saturated brine, a highly concentrated electrolyte system. The properties of an aqueous system at such a high electrolyte concentration are very different from dilute aqueous solutions employed in conventional flotation processes.

The high ionic strength of the media results in a compressed electrical double layer and reduces the range of the electrical forces operating between particles. As a result the particles can approach each other more closely to distances over which strong dispersive attractions dominate.

Flotation chemists have studied the selective flotation of alkali halide salts such as KCl and NaCl for many decades. During the past 60 years, numerous researchers have advanced their theories to explain the mechanism of collector adsorption in soluble-salt flotation systems. None of these hypotheses can be used alone to adequately explain KCl flotation and the collector adsorption phenomena observed in these systems, probably due to the fact that the collectors used are highly insoluble in saturated brine solutions, which makes detailed experimental work difficult.

Based on a combination of the various theories and some experimental results, it can be postulated that although surface charge plays an important role in KCl collector adsorption, a good coincidence between the lattices of the particle molecules and the

collector is required to get a strong attachment of the cationic collector to the KCl particle at the air/brine interface.

2.2   Review of Soluble Salts Flotation Theories

Haelblich (1933) initially proposed an early theory that assumed the formation of insoluble products by mutual neutralization of collector and alkali mineral ions with opposite charges.

Neunhoffer (1949) proposed that alkali metal salts should form "double salts" with the collector in order to float. As K^+ and NH_4^+ do form double salts much more easily than Na^+ and NH_4^+, the theory was able to explain the flotation of K^+ salts with ammonium compounds.

Gaudin (1951) assumed that KCl could be floated with amines because the order of magnitude of the radius of the NH_3-group is the same as the radius of the K^+ cation. According to Gaudin, an exchange $K^+ <—> {}^+NH_3$ - R takes place, which cannot happen with NaCl because the radius of the Na^+ cation is much smaller.

The Fuerstenaus (1956) extended this theory of ion exchange to the whole group of alkali and heavy earth metals and stated that only those minerals that have a cation similar in size to the RNH_3^+ will float with amines. However, certain exceptions had to be made from this rule in view of the fact that certain Na minerals did indeed float; these exceptions were then thought to be due to expanded lattices. Unfortunately, this

theory did not explain why an anionic reagent such as an alkyl sulphate could separate two salts with the same anion such as KCl and NaCl.

Bachmann (1951, 1954, 1958) explained the selective flotation of KCl from NaCl by the formation of continuous layers of the collector on the surface of the sylvite mineral, based on the similarity between the dimensions of the KCl lattice and the quasi-crystalline structure of the amine collector; there is no such a metric similarity with the NaCl lattice. Thus, a powerful collector adsorption or in other words, a good coincidence between the condensed collector film and the mineral counterions was an essential prerequisite for flotation.

Rogers and Schulmann (1957) proposed that ion hydration, instead of crystal geometric conditions, was the determining factor for differences in adsorption of different collectors. Weakly hydrated minerals can be floated with alkylamines and alkylsulphates, strongly hydrated minerals with fatty acids, but very powerfully hydrated minerals cannot be floated at all. A measure of hydration may be the heat of dissolution, which is the difference between lattice energy and heat of hydration. Table 2.1 shows the resulting correlation between heat of dissolution and floatability.

According to Singewald (1961), ion hydration affects the adsorption of a collector much more than the possible reaction between the mineral surface with the molecules of the solution. He postulated that coating of KCl or NaCl surfaces by fatty acid anions proceeds without hindrance. The collector film on the KCl is a little looser and for that reason perhaps a little more labile than the film on NaCl.

This theory can be regarded as the first approach to consider selective flotation of soluble salts as an adsorption phenomenon.

	Li$^+$	Na$^+$	K$^+$	Rb$^+$	Cs$^+$	NH$_4^+$
			No Flotation			
F$^-$	-4.2	-1.7	9.2	24.7	35.6	-6.3
Cl$^-$	20.5	-2.1	-15.9	-17.6	-16.3	-14.2
Br$^-$	32.2	-19.3	-17.2	-24.7	-27.6	-18.8
I$^-$	61.9	-16.3	-14.2	-26.4	-33.9	-15.1
	No Flotation		**Flotation**			

Table 2.1: Heats of solution (ΔH) of alkali halides at 18° C in kJ Mol^{-1} (After Singewald, 1961).

Schubert (1967) observed that the adsorption of n-alkyl ammonium cations on sylvite increases suddenly when the concentration of amine in the brine approaches its solubility limit. This seemed to indicate that the crystallization energy of the collector in the brine is of the same order of magnitude as the adsorption energy. Schubert realized that in sylvite flotation the collectors are added in quantities exceeding their solubility limits in the brine.

Considering the amount of energy available for the adsorption, the straight chain collectors were preferred. The more the non-polar chains deviate from linearity the less energy was supplied by the association. This idea was supported by the fact that sylvite and other alkali halides can best be floated with straight chain primary amines and with straight chain alkyl sulphates. The secondary, tertiary and quaternary amines and the

7

branched chain collectors are ineffective. These results indicated that the energy furnished by the association of the non-polar groups in saturated alkali salt solutions dominated the energy requirements for the adsorption.

Roman et al. (1968) confirmed that no adsorption of amine on KCl occurred until precipitation of the amine had occurred in solution. They noted that KCl is floated well only after precipitation of the collector salt has occurred, when higher molecular weight dodecyl and tetradecylamines are used.

The configuration of $RNH_3Cl_{(aq)}$ suggests asymmetry of electrical charge because of the lack of symmetry of the molecule. Further, it seemed likely that the negative end of the amine chloride dipole will be toward the chloride end, or:

$$RNH_3Cl_{(aq)}$$

$$+ \qquad -$$
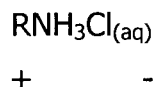
These correlations suggested to the authors that an electrical mechanism of adsorption may be involved, and that the surface of KCl might be positively charged, while NaCl might be negatively charged, especially since RSO_3^- is a collector for KCl. At the time of the publication of the Roman et al. report, there were no techniques available to determine surface charge of salt systems as soluble as NaCl and KCl. Their predicted surface charges were speculative and based on simplified lattice ion hydration theory.

Roman et al. added that surface charge phenomena might also explain some of the product contamination that is associated with KCl-NaCl separation. For many potash ores, the NaCl contamination of a flotation concentrate is due to the presence of very fine particles of NaCl in the concentrate. Part of this contamination may be due to mechanical occlusion in the froth or to actual flotation of NaCl, but the electrical attraction between KCl and NaCl may also be a factor.

According to Leja (1982), the ability of a surfactant to adhere to a given halide depended to a great extent on the degree to which the molecules are condensed, that is, on the structure of the surfactant film at the air-saturated salt solution interface.

Due to the fact that the surfactants used in soluble salt flotation are highly insoluble in concentrated brine, they cannot readily diffuse through the liquid to make the solid hydrophobic. However, they can spread readily at the air/liquid interface the moment a contact is made between the insoluble surfactant particulate and a generated air bubble. The spread monolayer at the air-bubble surface is compressed to a definite area per molecule depending on the quantity of surfactant added for a given rate of bubble generation. A contact between a hydrophilic salt particle colliding with an air bubble, which is covered with a spread surfactant, leads to an attachment if a sufficiently high degree of adhesion is developed on contact. This can also be accounted for by Bachmann's postulation.

The various theories previously proposed to explain selectivity in soluble salt flotation provided, according to Leja, an interesting example of the intricate balance that must be achieved between numerous factors likely to contribute to the overall

phenomenon of selective flotation. Crystal lattice, ionic size, hydration of polar groups and of salt surfaces, solubility of surfactants and their complexes, surface charge, temperature, etc. all contribute to the chemical aspect of flotation. In addition there are physical parameters due to hydrodynamics, agitation, etc. Each parameter whether physical or chemical becomes of critical importance in a particular set of conditions but otherwise may not play a major role.

Laskowski et al. (1986, 1988, and 1989) determined the electrophoretic mobility of the colloidal amine species as a function of pH. Their results show clearly that dodecylamine precipitate is positively charged up to pH 11 and assumes negative charge when the pH exceeds this value. This correlates extremely well with Schubert's findings that KCl flotation with dodecyl amine as collector ceases at around a pH of 11, while NaCl flotation displays a reverse trend. Laskowski et al. concluded that in saturated brine KCl crystals are negatively charged and NaCl crystals are charged positively.

The findings of Laskowski et al. were developed further when Miller et al. (1991, 1992, 1993, and 1994) determined experimentally the surface charge of KCl and NaCl particles by measuring their electrophoretic mobility. These measurements provided a direct indication of the sign of the surface charge of the salt particles in their saturated brine. Although small in magnitude, NaCl exhibits an electrophoretic mobility of $+0.171 \pm 0.148$ μm/(s V cm), which is distinctly positive, while the electrophoretic mobility for KCl, -0.47 ± 0.263 μm/(s V cm), is distinctly negative (Miller et al., 1992).

It can be concluded then, that although the electrical charge of colloidal amine particles and KCl particles are responsible for fast adsorption kinetics, a good fit between the KCl lattice and the lattice of the colloidal amine is also required in order to develop a strong adhesion at low solid hydration.

2.3. Potash Flotation in Saskatchewan

Sylvinite ores from Saskatchewan mines contain sylvite (20 - 30 % K_2O), halite, sometimes carnallite and 1 - 5 % insolubles. Liberation is substantial in some cases at 9.5 mm and virtually complete in all cases at 1.2 mm. The separation process includes dissolution of the carnallite and removal of the insolubles, followed by separation of sylvite from halite.

After a first stage of roll crushing underground intended to reduce the largest lumps to the 150 - 250 mm range to minimize transportation problems, liberation of the minerals is attained by both dry and wet crushing methods. Impact crushers are used, generally in closed circuit, to crush to 9.5 mm for processing or secondary grinding, or to crush to the final 2.4 - 1.7 mm for flotation.

After crushing, the ore is scrubbed in a series of small highly agitated cells, designed to liberate insoluble slimes from the potash mineral. During this stage, enough fresh water is added to the high carnallite ores to dissolve magnesium chloride. Desliming is usually accomplished in two or three stages. The primary separation may be done with cyclones, siphon sizers or wet screens, while the secondary separation is usually

11

accomplished with hydro-separators, cyclones, rake classifiers, or thickeners. The desliming by flotation of insoluble slimes from the ore in two stages is practiced at Agrium Vanscoy Potash Operations. This method has the advantage of reduced capital expenditures for desliming equipment but suffers the disadvantages of higher reagent costs.

Sizing and debrining, processes usually in combination with the previous stages, are accomplished with stationary wet screens, vibrating screens and cyclones, or combinations of all three. Separate coarse and fine material streams are each conditioned with both a depressant and a collector. An extender oil is added to the coarse conditioners. An alcohol is used as a frother. Some conditioning is required for tailings scavenger circuits where large particles require optimum reagentizing, or flotation is preceded by secondary grinding to liberate the middlings. The cleaning, re-cleaning and centrifuge scavenging circuit require no further reagentizing.

3.0 REAGENTS

3.1 Brines in Potash Flotation

The chemical composition of the brine is an important parameter in soluble salt flotation. Figure 3.1 shows a typical solubility-temperature equilibrium diagram for the KCl-NaCl-H_2O system (Seidell, 1958). The figure reveals that while KCl solubility decreases sharply with decreasing temperatures, the solubility of NaCl increases slightly.
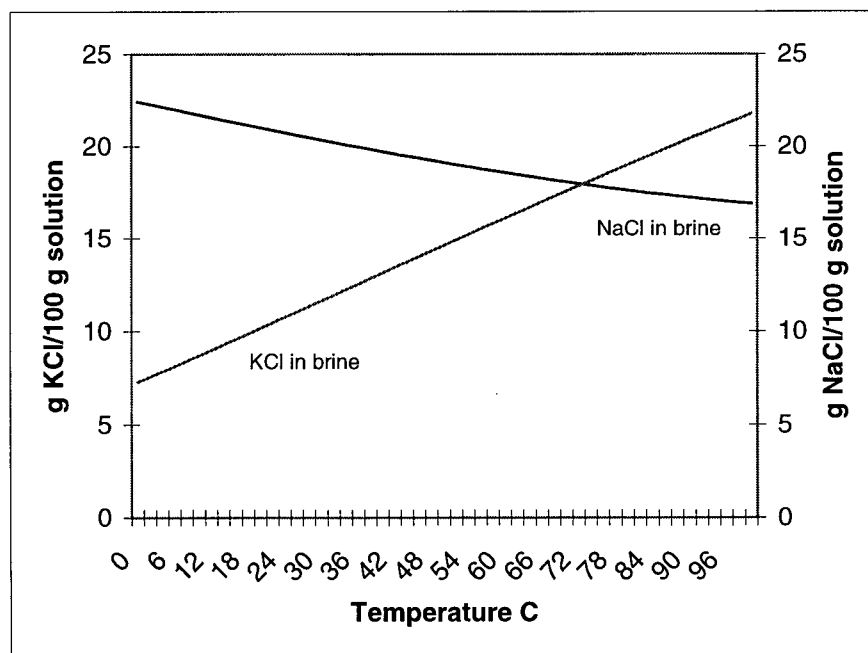
Figure 3.1: The equilibrium diagram of the system KCl-NaCl-H_2O (Data from Seidell, 1958).

The presence of other salts in the system affects the equilibrium and alters the solubility of KCl. In the competition for free water, less hydrated ions are salted out by more hydrated ones, and the position of the potassium ion, characterized by negative hydration, is especially vulnerable to any changes in the ionic composition of the brine. The composition of the brine influences not only brine density and viscosity, but also colloid chemistry of flotation reagents and consequently the mineralogical composition of the floating products.

The composition of the brine used as the medium in the flotation of potash ores is determined by the mineralogical composition of the ore and by the temperature. The presence of carnallite in sylvinite ores increases the Mg^{2+} content in the brine. Mg^{2+} values over 2 - 4 % increase brine viscosity, which may affect flotation efficiency. This effect seems to be more pronounced in the presence of slimes. SO_4^{2-} ions, when in excess of 2.5 % in KCl-NaCl brine also depress KCl flotation (Eatock, 1982).

3.2 Cationic Collectors

Primary long chain amines (RNH_2) are used in potash flotation as collectors. Cationic collectors are organic molecules that have a positive charge when in an aqueous environment. The common element shared by the cationic collectors is an amine group. The cationic collectors can be divided into five main classes: fatty amines, fatty diamines, ether amines, ether diamines and condensates (Table 3.1).

14

The extent of the compression and the structure of the collector film are determined by collector concentration and structure and length of its hydrocarbon chain (Table 3.2). The much lower degree of compression attainable by branched hydrocarbons explains the poor flotation performance of branched-chain amines.

Collector	Structure	Form
Fatty Amine	$R-NH_2$; $R = C_{12} - C_{24}$	Solid/Paste
Fatty Diamine	H | $R-N-C-C-C-NH_2$; $R = C_{12} - C_{24}$	Solid/Paste
Ether Amine	$R-O-C-C-C-NH_2$; $R = C_8 - C_{13}$	Liquid
Ether Diamine	H | $R-O-C-C-C-N-C-C-C-H2$; $R = C_8 - C_{13}$	Liquid
Condensate	H H | | $R-C-N-C-C-N-C-C-N-C-R$; $R = C18$ ‖ ‖ O O	Solid/Paste

Table 3.1: Cationic collectors (Gefvert, 1986).

Primary aliphatic amines are exclusively used in the differential flotation of sylvite from halite. Because long chain amine solubilities in water are extremely low, their salts, either acetates or hydrochlorides have been utilized by industry. In mining operations, especially where a local source of acid is present, the primary amine is still utilized due to cost factors.

Under standard flotation conditions, amine concentrations are well above their solubilities, and normal flotation temperatures (30 - 40° C) are well below the amine Krafft point. Under such conditions the amine precipitates.

	C_8	C_{10}	C_{12}	C_{14}	C_{16}	C_{18}	C_{20+}
Coconut	6	7	46	18	11	12	
Tallow				3	32	65	
Soya					10	90	
Tall						100	
Rapeseed					3	38	59
Fish				4	26	30	40

Table 3.2: Chain lengths of commonly used fatty amines (Gefvert, 1986).

In the presence of polar surfactants such as alcohols (frothers) in brine, the colloidal solubility of amine is altered by both the salting out effects of electrolyte and the formation of mixed micelles with the surfactant (Laskowski, 1994). While electrolytes and alcohols both reduce the critical micelle concentration (CMC) of amines, their effect on solubility is totally different. The presence of alcohol brings about the depression of the Krafft point and has a pronounced effect on the colloidal properties of long-chain amine solutions. Leja claims that the rate of spreading of amine films, which is very low in brine, increases dramatically in the presence of alcohols.

As previously mentioned, sylvite particles are negatively charged in saturated NaCl-KCl brines, while NaCl particles are charged positively, although the electrical double layer is completely compressed at such a high ionic strength. Electrical charges of colloidal amine and KCl particles are then responsible for the interactions, although a good fit between the KCl lattice and the lattice of the solidified amine produces a stable adhesion (Laskowski, 1994).

Ambient temperature fluctuations between summer and winter seasons, which in some locations may be within 15 - 35° C, affect sylvite flotation results. Dependent on chain lengths, amines are temperature limited because sylvite flotation depends upon the formation of insoluble collector species and increased temperature promotes amine solubility (Gefvert, 1987). Generally, the shorter the chain length the lower the temperature at which they are effective. Thus, an amine with a C-18 chain length content in the 60% range is effective at pulp temperatures below 24° C. At higher temperatures; longer chain lengths are required. Since the effective flotation of KCl depends upon the formation of insoluble collector species, increased temperature, which promotes amine solubility, depresses flotation. Longer chain amine blends are employed in summer to counteract the effect of higher temperatures.

Straight-chain primary amines are commonly used in the flotation of sylvite. Attention is paid to the degree of amine unsaturation, characterized by the "iodine value" (IV). Laskowski reports that the more refined amines do not work as well as the industrial grade amines (Laskowski & Wang 1997). Koehler (1984) reports that heating amine for long periods of time before its use (for example when unloading tanker

trucks) reduces its flotation activity. Thermal treatment was found to be especially harmful for amines characterized by IV>20 (Koehler et al., 1982).

3.2.1 Cationic Collectors in the Flotation of Sylvite

Cationic collectors are generally used as aqueous dispersions (0.5 - 5.0 % in water). The use of a dispersion increases the accuracy of metering the collector and the kinetics of collector-pulp mixing.

Since cationic collectors are insoluble in water, some emulsification aid is needed in order to produce a stable dispersion. The salt of an amine can be used as an emulsifier for the amine-in-water system. The addition of acetic acid at a collector to acid ratio of 5 to 1 is sufficient to provide stable emulsion for most amine-based collectors. Since most amines are foamers, care should be taken not to introduce air into the dispersion mixing system.

Liquid ether amines can be used unmixed, as their rapid dispersion permits the addition to the flotation cell without extensive conditioning. The direct addition of the collector eliminates the need for dispersion tanks and the handling of acid for neutralization.

3.2.2 Collector Selection

There is no precise rule for selecting the proper collector for a given flotation operation. There are three factors that affect the response of the cationic collector and

must be taken into account when screening possible collectors. These factors are sylvite particle size, water chemistry and water temperature.

The particle size of the mineral to be floated influences the choice of collector. The coarser the particle size the more hydrophobic the particle surface must be for successful flotation and longer hydrocarbon chains are preferred. As particles become finer and easier to float, the emphasis in collector selection will swing towards selectivity: short-chain amines are the choice.

When ultra-fine (> 10 µm) slimes are present, cationic flotation suffers from high reagent consumption. A slime-stabilized froth can occur to the extent that completely immobilizes the surface of the flotation cell. In this case prior removal or depression of slimes is essential in a cationic flotation scheme.

Water chemistry depends basically on pH, and ions present in solution (hardness). Amines are dissociated up to pH ~ 10; since most amine flotation is done below this level, pH does not influence the amine choice. Increasing water hardness (Ca^{2+} and Mg^{2+}) decreases the effectiveness of cationic flotation due to increased competition for the negative surface sites on the particles. Water hardness affects all cationic collectors by increasing effective dosages when compared to those required for soft water. The presence of certain negative ions such as sulphate and carbonate reduces flotation response in some cases due to the formation of insoluble salts.

Water temperature affects the solubility and the dispersability of the cationic collectors. As the solubility of the amine increases in hot water, the effectiveness as a

flotation reagent decreases. If plant water temperature varies considerably due to seasonal changes of ambient temperature or changes of water supply, a change of collector may be needed to maintain optimum flotation response.

3.3   Long-Chain Neutral Molecules

3.3.1 Alcohols.

Methyl Isobutyl Carbinol (MIBC), a branched aliphatic alcohol has been used as frother in potash flotation, due to its ability to deliver fast kinetics, brittle froth and lower water retention resulting in more selectivity.

Most recently, polyglycol ethers have replaced MIBC in several potash plants in Canada, mainly because they are more powerful frothers, able to maintain tenacity of frothing and improving coarse KCl particle recoveries. Health concerns associated with the use of MIBC have also played a role in the replacement process.

3.3.2 Extender Oil

While fine sylvite particles can be floated with amines only, extender oil is required in addition to amine to float coarse KCl particles. Saskatchewan potash producers have been using Esso 904 as extender oil in the flotation of coarse potash particles for many years. Unfortunately, Esso 904 contains significant amounts (up to 80% by volume) of polycyclic aromatic compounds which are considered a health hazard.

Esso 904 oil is a mixture of a catalytic cracking scrubber bottom residue and a light gas oil composed mostly of aromatic hydrocarbons. It is considered that nitrogen containing compounds are the active ingredients of the oil (Laskowski and Day, 1993).

3.4 Modifying Agents Used in Potash Flotation

3.4.1 Organic Modifying Reagents

The organic modifying agents and their derivatives used as modifiers by the potash industry include different polysaccharides: starches, galactomannans (guars) and cellulose derivatives. These are water-soluble polymers and vary in structure, molecular weight, biodegradability, ease of dissolution, and temperature of preparation. Although the price per kilogram for natural products tends to be low relative to synthetic products, dosage requirements tend to be high. Furthermore, the composition of natural products tends to fluctuate, and they are more susceptible to microbiological attack, which creates storage problems. Figures 3.2 and 3.3 shows the structure and features of the natural polymers carboxymethyl cellulose and galactomannan.

Granules of starch are extracted from ground grains of cereals (corn, wheat, rice, millet, etc.) or from potatoes and roots (cassava, tapioca, etc.) with water and weak acid (SO_2) or alkali in order to separate them from the non-carbohydrate constituents (proteins, fats, and fibrous matter). Changes in the process variables lead to different degrees of granule disruption by hydrolysis (Whistler et al., 1953).

Galactomannans are branched polysaccharides composed of D-galactose and D-mannose commonly found in the endosperm of leguminosae where they serve as reserve foods. Ratios of galactose to mannose vary in different species, e.g. guar (1 to 2), locust bean gum (1 to 4), tara (1 to 3). Guar is the most important species of galactomannans. Guar gum is widely used as a flocculant in industrial processes. The major commercial source of this gum is the seed of a small legume, *Cyamopsis tetragonolobus*, which is indigenous to India and Pakistan. The gum consists of the ground endosperm of the seeds, and it is used as a flocculant by dispersing in water. Other commonly used derivatives of guar include hydroxylpropyl, hydroxylethyl, carboxymethyl, carboxymethylhydroxypropyl, and cationic guar (Levine, 1981).

Cellulose is the main structural element of the cell walls of trees and other higher plants. It is made up of a poly-disperse carbohydrate of high molecular weight comprised of long chains of D-glucose units joined together by beta 1-4 glycoside bonds. The anhydroglucose unit contains three hydroxyl groups, one primary and two secondary. Cellulose derivatives include cellulose nitrate, cellulose acetate, methyl cellulose, carboxymethyl cellulose, hydroxyethyl cellulose, cellulose xanthate, and hydroxypropyl cellulose. Of these, the derivative most widely used is carboxymethyl cellulose (CMC) (Levine, 1981).

On reacting cellulose with sodium acetyl chloride, the hydrogen of the branch-chain hydroxyl group is replaced by the sodium acetyl group, producing sodium carboxyl cellulose.
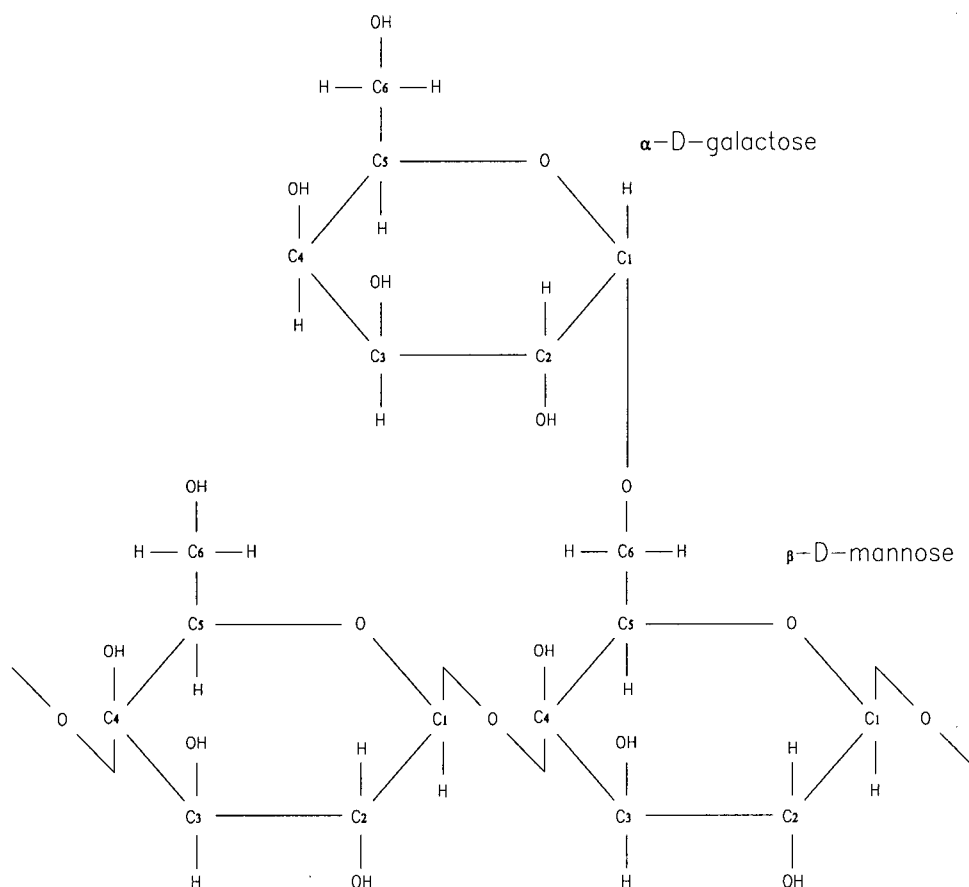
Figure 3.2: Structure of Galactomannan.

The sodium carboxymethyl groups (CH_2COONa) replace a portion of the hydrogen atoms associated with the hydroxyl groups of the glucose units, which form the cellulose molecule. Sodium chloride is formed as byproduct.

The Degree of Substitution (DS) is the average number of carboxymethyl groups introduced into one glucose unit. For commercial qualities of CMC, the DS varies between 0.4 and 1.5. The CMC content is the degree of purity of the product, and is measured as the percentage of sodium CMC present. The Degree of Polymerization (DP) expresses the average number of glucose units per cellulose ether molecule, and

is a function of chain length, and therefore molecular weight. Increasing the DP

increases the viscosity of an aqueous solution of sodium carboxymethyl cellulose, and

the solution viscosity is therefore used as an indication because it is more easily

measured than DP.
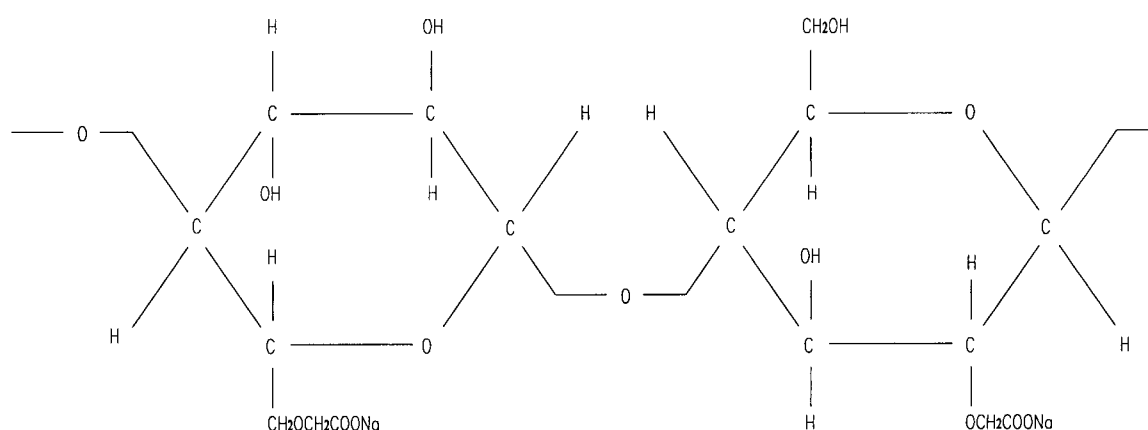
Figure 3.3: Sodium carboxymethyl cellulose, degree of substitution 1.0 (Rodhes, 1980).

The structure of the carboxymethyl cellulose molecule varies with the degree of

substitution and the degree of polymerization. However, for reagents with the same DS

and DP it is possible to introduce further variations by repositioning the substituted

radicals. For instance the DS would be 1.0 if two radicals were substituted on every

other unit but the structure would be different.

The viscosity of a carboxymethyl cellulose solution drops with increasing temperature, but the effect is reversible if the solution is not heated above 80 C or for lengthy periods.

Rath et al. (1997) have postulated that hydrogen bonding and chemical interaction were the determining mechanism in the adsorption of guar gum reagents on clay silicates. These mechanisms have also been suggested for the carboxymethyl cellulose molecule.

When the chain length of the polymer increases, more OH^- groups are contained in each molecule, and the hydrogen bond adsorption energy would be expected to increase. Reagents with a high DP might be expected to adsorb onto the valuable minerals and depress them.

3.4.2 Synthetic Modifying Agents

Synthetic polymers dominate as the chief type of polymeric flocculants used in the minerals industry. These polymers are almost invariably based on polyacrylamides (Figure 3.4), though a significant number of other chemical types also exist. Commercial polymeric flocculants can be anionic, cationic, or nonionic, and it is useful to classify them as such for the following discussion.

Anionic polymers containing carboxyl groups are by far the most important class of anionic flocculants, both in the high- and low- molecular weight ranges. The most common of these anionic polymers are based on polyacrylic acid, salts of polyacrylic

acid, or on acrylamides. The latter type is generally prepared by either polymerization/hydrolysis of acrylamide or by copolymerization of acrylamide with acrylic acid or acrylate salts.

Polyacrylamides contain a rather random distribution of a relatively few carboxylate groups along the predominantly polyacrylamide chain. Since only a fraction of the carboxylate groups are present and make a contribution to the molecular charge, these types of polymers are relatively weakly ionized. However, salts of the polyacrylic acids are highly ionized in alkaline medium.
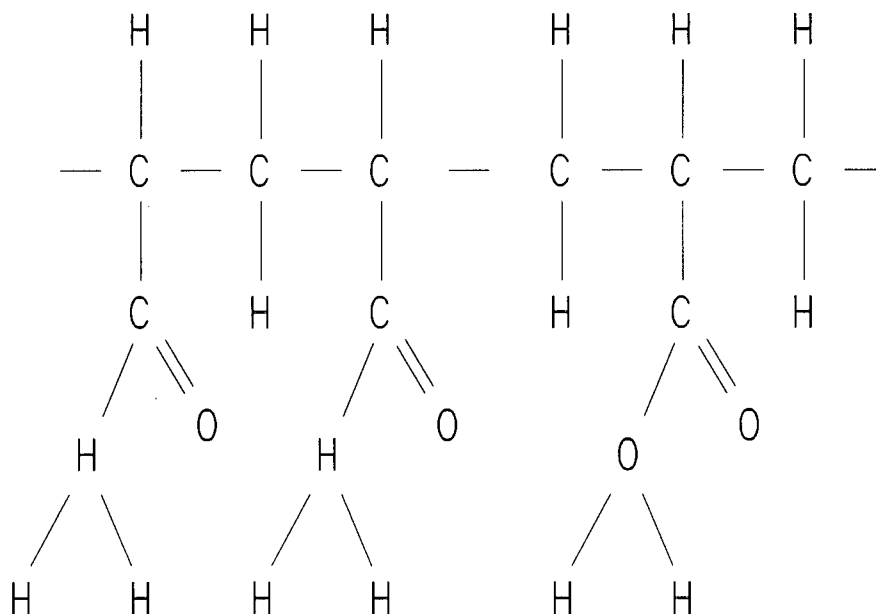
Figure 3.4: Structure of Polyacrylamides.

Cationic polymeric flocculants contain positively charged groups, such as amino ($-NH_3^+$), imino ($-CH_2-NH_3^+-CH_2$), or quaternary amino ($-^+NR_4$). Most of the high molecular weight cationic polymers available today are made by free-radical copolymerization of acrylamide with minor amounts (10 mol-% or less) of the cationic monomer. A unique feature of cationic polymeric flocculants is that low-molecular-weight polymers function as coagulants and high-molecular-weight polymers function as flocculants. This is primarily because the majority of particles found in nature bear negative charges. These cationic polymers usually function by a combination of charge neutralization and bridging mechanisms.

Nonionic polymeric flocculants are fewer in number than anionic or cationic polymers. Two of the most common nonionic polymeric flocculants used in the mineral industry are polyacrylamide and polyethylene oxide. A truly nonionic polyacrylamide flocculant does not exist, because the polymerization of acrylamide intended to produce high-molecular-weight polymers is conducted adiabatically in aqueous solution, conditions that favor hydrolysis. Most commercial "nonionic" polyacrylamides contain about 1% or 2% hydrolyzed amide groups, making them slightly anionic. The other nonionic polymer that has been of significant use in the mineral industry as a flocculant is polyethylene oxide (PEO). These polymers range in molecular weight from 20,000 to about 8 million.

27

4.0   EFFECT OF SLIMES ON FLOTATION

4.1   Effect of Slimes on the Flotation Process.

Two classes of mineral fines can be distinguished in conventional flotation circuits: (a) naturally occurring clay minerals, such as kaolinite, illite, montmorillonite, which are intrinsically composed of very small particles (mainly 2 μm downwards), mostly of platy form under the electron microscope; and (b) the fines inevitably produced during the crushing and grinding stages of the beneficiation operations.
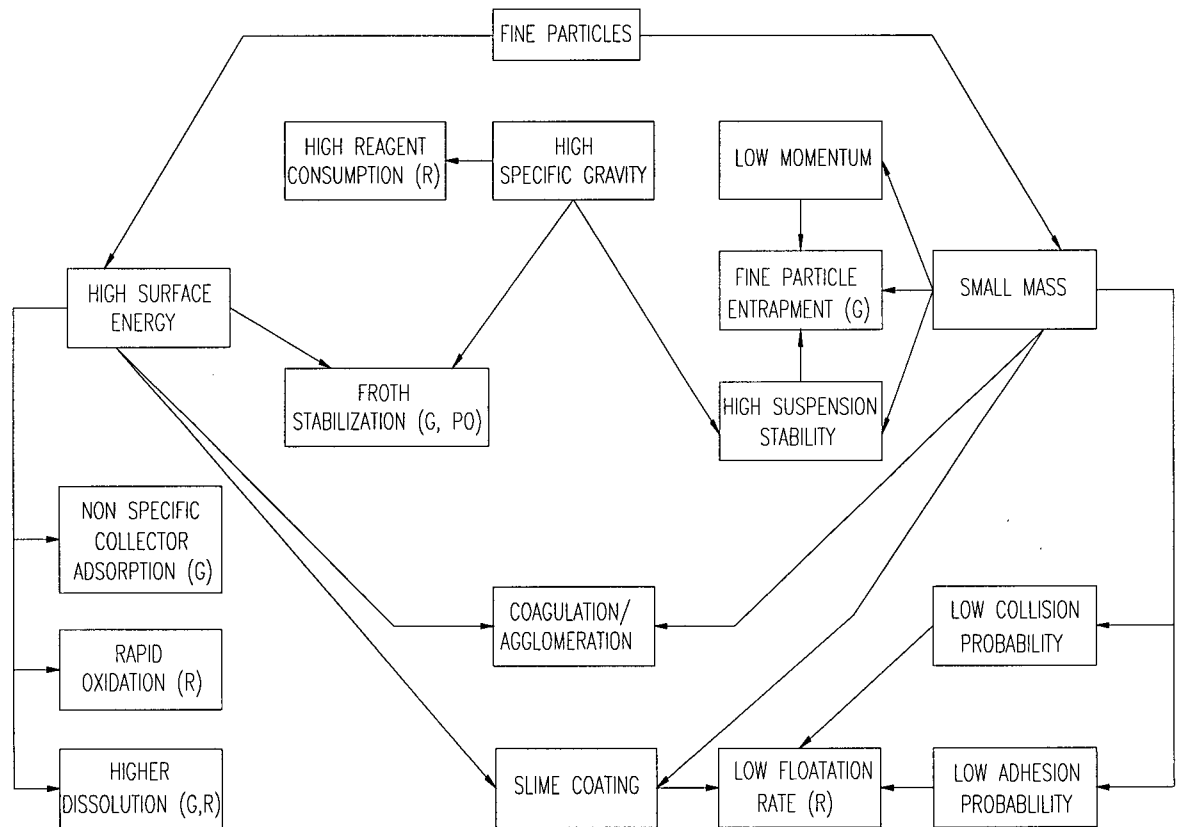
Figure 4.1: Schematic diagram showing the relationship between the physical and chemical properties of fine particles and their behavior in flotation. (G) and (R) refers to whether the phenomena affect grade and/or recovery. The arrows indicate the various factors contributing to a particular phenomena observed in flotation of fine particles (Fuerstenau, 1980).

Two characteristics begin to dominate as the particle size is reduced: the specific surface becomes large and the mass of the particle becomes very small. In Figure 4.1, a schematic diagram has been drawn to illustrate the relationship between the physical and chemical properties of fine particles and their behavior in flotation.

Because of the small mass and momentum of fine particles, they may be carried into the froth after getting either entrained in the liquid or mechanically entrapped with particles being floated.

The detrimental effect that slimes have on the selectivity and the recovery of flotation processes, can be attributed to the following phenomena:

- their adsorption of the cationic collector,

- their nonselective coagulation (or slime-coating),

- their nonselective entrainment into the froth as a result of the nearly homogeneous suspension of the fine particles in the pulp, and;

- their strong stabilizing action on the froth.

The particle size of the slimes has a pronounced effect on the flotation process; it can be said that the finer the slimes, the greater is the depression of flotation probably as a result of all the listed phenomena.

4.2   Adsorption of Collector

It has been demonstrated that fine particles are carried into the froth as mechanical carryover with water attached to air bubbles (Johnson et al., 1974). When such particles are of gangue minerals, the effect is a reduction in the grade of the concentrate. The large specific surface of fine particles increases their adsorption capacity (determined by the amount adsorbed per unit area of surface) when considered on a mass basis. Thus, a significant proportion of the reagent is consumed by small particles. Sufficient reagent may not be available for the flotation of larger particles, with a resultant decrease in recovery. Calculations using the Gaudin-Schuhmann equation indicate that most of the surface area of even a minus 13 mm jaw crusher product may be in the minus 2 μm fraction, even though this fraction accounts for only 0.13% of the total weight.

Surface properties of fine particles tend to be different from the properties of coarse particles of the same material. Fine mineral particles have higher specific surface energy because of increased number of edges, corners and crystallographic imperfections. High specific surface energy of fine particles may influence flotation in a number of ways. Increased dissolution from the surface of fine particles (as predicted by the Kelvin equation) may introduce undesirable impurities into solution, affecting collector-mineral interactions.

Fine particles are known to attach to liquid-vapor interfaces. When such particles are present at the interface, they make it difficult for "colliding" particles to adhere to the air bubbles leading to low recoveries. When the particles at the interface are

gangue minerals, the grade of the concentrate becomes poorer. In addition, fine particles at the interface may also stabilize froth, causing concentrate handling problems as the froth remains stable after removal from the flotation cell (Ross, 1997 and Somasundaran et al., 1988).

As particles become colloidal in size, long range intermolecular forces and electrical double layer forces dominate and control the interaction of a particle with other particles or bubbles in a suspension. If the double layer is thick and the potentials are large, the particles will remain dispersed whereas if the double layer is thin or absent, attractive forces dominate and the particles will coagulate and, if hydrophobic, the coagula will also adhere to the air bubbles. This tendency of fine particles to aggregate has been utilized to some extent for flotation of fine particles. Dominance of surface electrical effects is mainly responsible for the lack of selectivity in fine particle flotation.

4.3   Slimes Coating

The process of slime coating refers to the attachment of fine particles to larger particles, and is controlled by long-range intermolecular forces similar to those that determine the interactions between a bubble and a particle. Slime coatings can be detrimental to flotation in several ways. If the fine particles are the valuable mineral and the coarse particles are the gangue minerals, the grade of the concentrate becomes poor. When the fine particles consist of gangue minerals that coat the coarse particles of the valuable mineral, these particles prevent the attachment of air bubbles

and the recovery of valuable mineral may then decrease significantly. In addition, the slimes contained in the pulp produce an alteration of the hydrodynamics of the multiphase flow (Weiß et al., 1988).

4.4   Froth Stabilization by Slimes

Historically slimes have been observed to impart excess stability to froth. The mechanism by which this is thought to occur is that they physically retard drainage of the bubble (Sivamohan, 1990). Air bubble surfaces become coated with slime particles which produce a "froth regulating" effect that reduces average particle size in the concentrate and increases entrainment of fine salt particles.

4.5   Slimes in Flotation of Potash Ores

Potassium is found throughout the world in both soluble and insoluble forms. Only the soluble forms are economically attractive to process primarily as chlorides and sulphates, with potassium chloride being by far the most important potash salt. Sylvinite ore, comprising of sylvite [KCl] and halite [NaCl], is the most important source of potassium. This ore is found in commercial quantities in Saskatchewan, New Mexico, Russia, Belarus, the Ukraine, Germany, Spain, and the United Kingdom.

The mines in the Saskatoon area extract the ore from the Patience Lake member. The potash mines in the southeast corner of Saskatchewan produce from the Esterhazy member; while a solution mining operation near Belle Plaine, extracts potassium

chloride from all three of the potash bearing members. The ore from the Esterhazy member is coarse-grained with insolubles less than 2%, and can contain some carnallite. Ore from the Patience Lake member is medium-grained, with insolubles averaging up to 5.5%, but with little or no carnallite (Cormode et al., 1985).

The insolubles occur as seams and disseminations within the ore zone. They consist of a variety of compounds, some of which were carried as a suspension of solids and other which may have been formed in the evaporation process (Mossman et al., 1982).

4.5.1   Mineralogical Composition of Slimes

Three main halides occur in the potassium-rich members of the Prairie Evaporites: halite [NaCl], sylvite [KCl], and carnallite [$KMgCl3.6H2O$]. Scattered small patches and "wedges" of clay are ubiquitous throughout the ore. Additionally, clay occurs dispersed among crystals of the various salts. Interspersed between beds of salts are numerous bands of red-brown to grey fine-grained, water insoluble material, a large portion of which falls into the particle-size category of clay.

On the average, the clay seams incidentally mined are about 5 cm thick, and even in the extreme case of PCS Lanigan, the thickness of individual seams rarely exceeds 10 cm.

With the proportion of 20-cm thickness of clay to a total of 340-cm thick mining zone the total water-insoluble mineral content is about 6 %. This value of 6 %

compares closely with the values reported by the various mills in the Saskatoon area: 4-5 % Agrium and IMC-Colonsay, 8 % PCS Lanigan.

Clay minerals make up nearly one-third of the total water-insoluble material mined. Anhydrite and dolomite are the other main phases. Also present are small amounts of quartz, K-feldspar, calcite, mica, and hematite.

In normal mining operations the clay seams are used as markers to control the mining elevation. This would be a straightforward matter but for the widespread occurrence of salt-solution collapse features throughout the potash beds.

The clay minerals are troublesome as they contribute to roof instability during mining operations, are responsible for ore dilution, underground airborne pollution, and affect ore recovery during milling and subsequent beneficiation (Banks, 1979).

4.5.2 Clay Minerals

The three main clay minerals reported in the Saskatoon-area ores are illite, chlorite and septechlorite. In addition, smaller amounts of smectite, mixed-layer clays, vermiculite, and sepiolite are present in some samples. Other clay minerals usually present in potash ores are halloysite (hydrated kaolinite), sometimes with impurities of montmorillonite (Mossman et al., 1982).

As in many other major potash deposits, illite is the most prevalent species. It accounts for about 40 % of the total volume of clay present, with chlorite being the next most abundant phase (Mossman et al., 1982).

4.5.3 Other Water-Insoluble Minerals

Water insoluble (WI) minerals are usually represented by anhydrite $[CaSO_4]$, dolomite $[(Ca,Mg)CO_3]$, calcite, $[CaCO_3]$, iron oxides, $[Fe_xO_y]$, and silica $[SiO_2]$.

Anhydrite and dolomite are in most cases intimately intergrown in irregularly sized lumps. Generally those "clay seams" richest in anhydrite are light colored, and contain relatively small amounts of clay minerals.

Small amounts of K-feldspar, and quartz occur to the extent of at least several percent. Minor amounts of a ferromagnetic mineral (presumably magnetite), ferrous (hydro)oxides and amorphous material are also present (Mossman et al., 1982).

4.6   Effect of Slimes on Potash Ore Flotation

In most potash ores there are free insoluble minerals outside the KCl crystals that can be released by scrubbing the ore with saturated brine. These are normally colored from gray to gray-brownish and are typically a mixture of non-clay minerals (-150 µm; +2µm) such as dolomite, anhydrite, hematite and silica, and clay minerals (-2 µm) principally illite, chlorite, and septechlorite. The insolubles had been compressed under pressure and are cemented together by dried soluble salts and possibly anhydrite (Mossman et al., 1982).

Red clay locked inside the crystals imparts a red color to the concentrate, which is released only when the crystals are dissolved. This clay consists essentially of magnesium aluminum silicate plus hematite as its principal components.

Canadian sylvinite ores contain 2 to 8% of insoluble gangue minerals. There is very little published data on the metallurgical characterization of the insoluble gangue minerals, and it is not clear which fraction of those insolubles is attached to the surface of KCl crystals and can be released by attrition scrubbing, and which fraction of insolubles is locked inside the crystals. Based on testing on the Agrium ore, it is estimated that near-perfectly-scrubbed potash particles still contain 0.3 - 0.5% insolubles locked inside the crystals.

Clays are soft particles that tend to disintegrate into individual platelet crystallites which are minute in size and become highly dispersed, increasing the surface area available for reagent attachment by several orders of magnitude. This decreases the effective concentration of available collector for sylvite flotation.

In the tetrahedral sheet of alumino-silicates, tetravalent Si may be partly substituted by trivalent Al (isomorphous substitution) resulting in a deficit of positive charge (or net negative charge). This excess of negative lattice charge is compensated by the adsorption of K^+ and Na^+. The exposed edges produced when a platelet is broken contain AlOH groups that are amphoteric. When ammonium ions, RNH_3^+, are added to a clay-in-water suspension, the organic cations replace the cations that were present on the clay surface and "exchange adsorption" takes place. Organic anions are adsorbed at the edges of clay particles and because of the large inner areas which may become available upon clay swelling in aqueous solution, clay's eagerness for cationic surfactants is very large. It has been reported (Arsentiev and Leja, 1977a) that 0.2% of slimes increases amine consumption by 1.5 - 2 times.

The ion exchange process that takes place on the surface of clay in the presence of amines follows the reaction (Cowan and White, 1958):

$$AZ + RNH_3^+ <---> A^+ + RNH_3Z$$
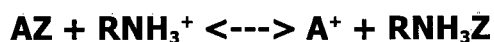
where **A** stands not only for sodium but also for any other exchangeable ion on the clay surface, and Z stands for clay.

The harmful effect of clays is even more pronounced at higher temperatures, because of the increased ability of the clay minerals to adsorb short-chain amines at higher temperatures (Laskowski, 1994).

A two-step process can be used to ameliorate the harmful influence of water insoluble slimes on the flotation process:

a) removal of the bulk of the slimes

b) chemical neutralization (blinding) of the remaining slimes

4.7   Scrubbing and Desliming

Liberation of the potash values is attained by both dry and wet crushing methods, often combined with secondary wet grinding in cage or rod mills. The sizing of the ore is a compromise between maintaining the potassium chloride in as large a size as

possible, while giving liberation from the sodium chloride and ease of scrubbing to remove the insolubles released during crushing-screening (Strathdee et al., 1982).

Scrubbing is usually accomplished in a series of highly agitated cells, normally at high percent solids (60 to 70% solids in a KCl-NaCl saturated brine), designed to liberate the insolubles attached to the potash particles. Two-stage scrubbing is common in the high insoluble areas. Normal design retention times for the Saskatoon-area mills are around 10 to 15 minutes.

The desliming stage is considered critical, as a virtually slime-free product is necessary to minimize reagent costs and to ensure good potash recovery.

Desliming is accomplished in two or three stages. The primary separation is usually done with hydrocyclones, while the secondary separation is done with a combination of cyclones, hydroseparators and thickeners.

After the desliming stages, the potash flotation feed is treated with modifying reagents in order to prevent the subsequent collector adsorption on clay particles.

Polyelectrolytes, usually referred as slime depressants, are applied to reduce the harmful effect of clays on sylvite flotation. The main mechanism involves, however, blocking off the clay surface for amine adsorption. Therefore, conditioning with the polyelectrolyte modifier precedes conditioning with the collector.

Natural polymers such as starches, carboxymethylcellulose and guar are commonly used as modifiers as they proved to help achieve better separation of sylvite from halite and slimes. These natural products have many drawbacks: poor selectivity, low

performance/dosage ratio, instability and sensitivity to aging (and rotting in the case of starches) and batch variability in composition. So far no synthetic polymer has proven to be a cost-effective alternative to natural products.

The adsorption of cationic polyelectrolytes by clay minerals largely occurs through electrostatic interactions between the polyelectrolyte cationic groups and the negatively charged sites at the mineral surface. Anionic polymers tend to be repelled from the clay surface and they do not enter the interlayer space. Clays also adsorb non-ionic polymers, such as polysaccharides. Unlike cationic polymers, anionic polymers do not enter the interlayer space (Leja, 1983).

4.8   Flocculation-Flotation of Slimes

The disadvantages of the conventional desliming by scrubbing and hydrocyclone classification are potash losses as fine sylvite particles in the insoluble slimes product and the significant amount of process brine required for the desliming operation. In the flocculation-flotation technology, the insoluble slimes are flocculated prior to collector addition in order to reduce their surface area and facilitate their flotation. This method was successfully implemented for processing potash ores containing 4-8 % insolubles at Agrium operations at Vanscoy. This method has the advantage of reduced capital expenditures for desliming at almost the same efficiency as the conventional method but is still able to treat a fairly coarse ore. The major drawback of the method is an increase in reagent usage (Banks, 1979; Chan et al., 1983).

In conventional diluted systems, selective flocculation of slimes is done with a high molecular weight polymeric flocculant that binds the slime particles by means of a bridging mechanism. Such aggregation is primarily controlled by the interaction between the polymer molecules and the different slimes particles. In the case of brine this phenomenon is not understood at all, as the behavior of polymer macromolecules in brine is not known.

In the case of Agrium at Vanscoy, Saskatchewan, a nonionic high molecular weight polymer (polyacrylamide) is used to selectively flocculate the insolubles and an ether amine is used to float the flocculated material.

5.0   EXPERIMENTAL

Part I: Depression of Slimes

Laboratory batch flotation tests were chosen as the method to evaluate the efficiency of various modifying reagents. The test program was inspired by the work of Arsentiev et al. (1988).

The efficiency of a modifier was determined based on the recovery of KCl and water-insoluble slimes to the KCl concentrate. A highly efficient modifier would simultaneously produce a high KCl recovery and a low insoluble slimes recovery at the lowest reagent consumption possible.

5.1   Materials

The sample used in the flotation experiments was minus 850 μm potash ore from Agrium Vanscoy Potash Operations. A fraction of the sample was also used for brine preparation.

The ore used during the entire test program was assayed and its chemical composition is shown on Table 5.1.

Size	% KCl	% NaCl	% W.I.
- 850 μm	40.2	55.2	4.6

Table 5.1: Test program feed ore.

In order to assure an unbiased comparison among the various reagents tested, a large amount of minus 850 µm sample was prepared, and then divided into 800-g batches. A large amount of brine was also made by dissolving the coarser fractions of the same sample. The flotation brine was prepared by dissolving an excess of the potash ore in tap water. The dissolution process involves adding the potash ore into an agitated container filled with hot water. Under continuous agitation the mixture was allowed to cool down overnight. Agitation was then shut off, to allow the slimes to settle and the clear brine was transferred by siphoning into the storage container. Brine density (1.24 g/cm^3 when saturated) was checked regularly to control the saturation level.

All flotation tests were carried out at room temperature, i.e. 20 ± 2 C. The flotation unit used for the testing program was a Denver D12 laboratory batch cell, with either a 4.6 L or a 2.4 L tank running at 1,500 rpm. A Denver D12 laboratory cell with an attrition-scrubbing impeller running at 900 rpm in a 0.5 L attrition tank was used for the slime scrubbing.

5.2   Reagents

Potash Collector

At laboratory temperature, KCl flotation is usually done with a fatty amine having a C18 chain length as the collector. For that reason, Witco Adogen MG-102 W a saturated

tallow amine, was used at a 1.0 % dispersion in water. The dispersion was prepared by adding the solid amine to water at 60 C. The liquid was under continuous agitation and acetic acid was added to neutralize the mixture.

Frother

A polyglycol ether, Dowfroth 319, which produces a strong froth was used. A constant frother dosage of 50 g/t was used in all the tests.

Modifiers

During this stage various reagents with potential slime-blinding capabilities were examined. All flotation modifiers were tried at concentrations ranging from 50 to 350 g/t. The following slime modifiers were tested:

Carboxymethyl Celluloses: Commercial CMC's are usually available in three grades: technical (about 75% pure), purified (94 - 96% pure) and food grade (99.5% pure). The other two parameters usually used to specify CMC's are degree of substitution (DS) and viscosity. Molecular weights of CMC products are usually not specified (Table 5.2).

The most widely used CMC's at Saskatchewan potash mills are the brand names Finnfix 300 (Rantec) and Depramin 96 (AKZO). For purposes of the present screening process, Finnfix 300 (FF300) was utilized as the baseline for comparison. Other CMC's of known molecular weight were tested and compared against FF300.

Finnfix is the denomination for a broad range of carboxymethylcelluloses produced by Rantec-Serla Chemicals AB of Finland. Finnfix 300 is a variety of sodium carboxymethylcellulose purified for industrial applications. It has a molecular weight around 300,000 and usually has a minimum Na-CMC content of 98% with the rest being mainly NaCl. It is supplied in palletized 20-kg paper bags. It is a low-toxicity product, dissolves relatively quickly in warm water and is soluble in saturated NaCl-KCl brine. It is usually utilized as a 0.5 - 1.0 % solution in brine.

A fresh Finnfix 300 sample was obtained from Rantec Corporation and was used for the entire testing program. Standard practice was to prepare a fresh 1 % solution of FF300 in brine to be used in the tests of the day.

Name	Supplier	Active Content	MW [g/g-mol]	DS
ACZ	S&G Resources	65%	90,000	0.55
ACZ HVSG	S&G Resources	65%	100-200,000	0.65
ACZ HVG	S&G Resources	65%	200-300,000	0.65
PE 23EX	S&G Resources	96%	700-800,000	0.75
Finnfix 300	Rantec Corp.	98%	200-300,000	0.82
Depramin 96	Hollimex	65%	200-300,000	0.85
P 30 Super	Amtex S.A.	65%	250,000	0.50

Table 5.2: Summary of the tested CMC's.

Depramin is the trade name for a series of water-soluble polymers specially developed as depressants for froth flotation by AKZO Salt and Basic Chemicals. Besides

potash flotation, the various types of Depramin are also used in the depression of talc in hard rock flotation.

Guar Gum: The Guar Gum sample, Rantec KP 4000 used during the testing program was obtained from Agrium. Guar Gums used by industry are usually classified according to their viscosity as a 1 % solution measured in a Brookfield Viscometer with spindle No. 3 at 20 rpm at 25 C and 24 hours after preparation. The measured viscosity is usually stable for 40 hours. Guar Gums are available in different viscosities, from 3.2 to 6.5 Pa-s, depending on their molecular weight. Viscosities normally used range from 3.5 Pa-s to 4.0 Pa-s; which are still pumpable under plant conditions. Rantec KP 4000 has an 82.5 % Galactomannan Gum minimum content.

Guar Gum is usually dissolved in warm water under strong agitation and then diluted to 0.5 % with plant brine. It is a natural product, with a low toxicity and is produced exclusively in Pakistan and India.

Carboxymethyl Hydroxypropyl Guar: Galactasol 650 polymer is an anionic carboxymethyl hydroxypropyl guar manufactured by Aqualon Oil Field Chemicals, a division of Hercules Inc. It has a molecular weight near 2,000,000 and a carboxymethyl DS of 0.12 - 0.15 and a hydroxypropyl MS of 0.28 - 0.31. Galactasol was selected for testing because it represents a chemical combination between Guar and CMC; the objective of these particular tests was to find out if there was a synergistic effect between the modifiers.

Carboxymethyl Starch: Glucostar 65 is an anionic carboxymethyl starch, a non-fermentable, highly soluble polymer with good bonding properties. It can be used in water based systems with no-biodegradation. It has a molecular weight of 100,000 and a degree of substitution of 0.55.

Polyacrylamides

Polyacrylamide flocculants have gained wide usage in the potash industry mainly as aids to thickening and clarification. Also at Agrium and at PCS Rocanville, polyacrylamides are used to flocculate insoluble slimes prior to their flotation separately from the conventional sylvinite flotation.

A series of Polyacrylamides were obtained from various mining reagent manufacturers (Table 5.3). As was the case when testing CMC's, the idea was to investigate the effect of various molecular weights and degrees of ionicity on KCl recovery and the selectivity of the flotation.

Sample	Supplier	Ionicity	MW [g/g-mol]	Charge density
Percol E-10	CIBA	anionic	15 - 20 M	5 %
Percol 351	CIBA	non-ionic	10 - 15 M	0 %
Percol 156	CIBA	anionic	10 - 15 M	40 %
Superfloc 362	Cytec	cationic	10 M	40 %
Acco-Phos 950	Cytec	anionic	1 M	5 %

Table 5.3: Characteristics of the polyacrylamides tested.

CIBA Specialty Chemicals manufactures polyacrylamides under the common name of Percol. Percol E10 is a very high molecular weight Polyacrylamide (MW of 15,000,000 to 20,000,000), slightly anionic (5 % anionicity) and widely used as silicate slimes flocculant. Percol 351 is a non-ionic, high molecular weight Polyacrylamide (MW of 10,000,000 to 15,000,000) used as silicate flocculant. Percol 156 is a highly anionic Polyacrylamide with a molecular weight similar to that of Percol 351.

A sample of Superfloc 362 was obtained from Cytec Canada. This is a highly cationic (40 % cationic) Polyacrylamide that, according to the supplier, was successfully tested to facilitate slimes depression in coarse potash flotation (May and Lambert, 1983).

A low molecular weight (MW ≈ 1,000,000), slightly anionic Polyacrylamide from Cytec was also tested. It is called Acco-Phos 950 and was developed as a modifier (silicate depressant) for phosphate flotation.

5.3 Development of the Testing Procedures

A preliminary set of familiarization tests were run, using the Denver D12 laboratory flotation cell using minus 3.37 mm ore. During the preliminary tests it was observed that the large amount of slimes present in the feed ore caused a severe turbulence dampening in the suspension, producing the settling of the coarser KCl and NaCl particles to the bottom of the cell tank as soon as the air was turned on. Increasing the power input (i.e., increasing the cell rotational velocity) did not help much. The

reduction of the sample size from 1,600 g to 800 g only slightly alleviated the problem. This phenomenon is consistent with the observations of Weiß et al. (1988).

Based on the experience gained from the preliminary tests, several changes were introduced to the test procedure. Feed ore was changed to minus 0.8 mm potash ore to be used for all future tests, to ensure that all available KCl particles will eventually float under the right conditions.

Laboratory flotation cells are not particularly efficient in floating particles with sizes over 2 mm; besides these particles need an addition of extender oil which may affect the modifier behavior during flotation. Because of this, changing the ore feed size to a finer fraction was used as a way to eliminate the flotation cell efficiency as a determining factor in the flotation performance and to eliminate the need for adding an extender oil.

5.4   Test Procedure for the Depression of Slimes.

The test procedure developed for examining reagents used as water-insoluble slime depressants included a mechanical desliming step, as illustrated in Figure 5.1.

Factors:
Collector and modifier dosages

Constants:
Brine temperature, room temperature
Feed density, 23% solids
pH, natural

Conditioning time, 15 seconds per reagent addition

Feed grade (45.4% KCl; 52.9% NaCl; 1.7% Slimes after mechanical desliming)

Feed size distribution (minus 20-mesh Tyler, see Table 5.4)

Potash Collector (Witco Adogen 102 H)

Frother (Dowfroth 319)

Mesh Tyler	microns	% retained
20	850	3.4
28	589	15.7
35	416	48.9
48	294	76.9
65	208	89.3
100	147	92.1

Table 5.4: Test ore size distribution.
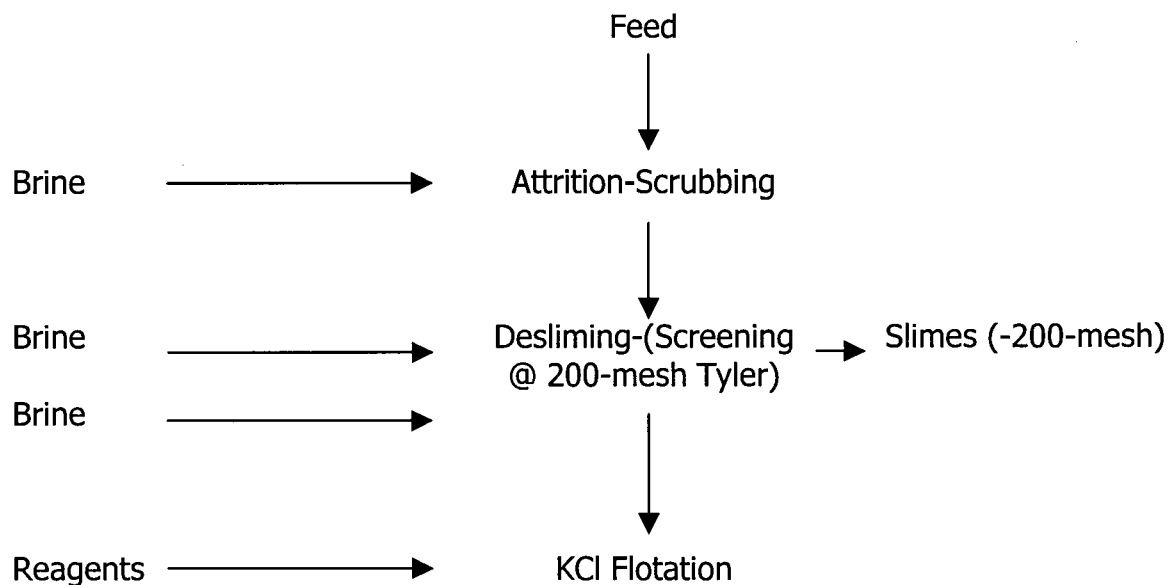
Figure 5.1: Test procedure for depression of insoluble slimes.

Scrubbing:

The attrition-scrubbing stage was intended to liberate the insoluble slimes. It was done by strong agitation of the sample, in two stages due to the capacity of the Denver laboratory attrition-scrubbing cell. Each stage consisted of a 800-g fraction mixed with 350 ml of brine ($\approx$ 70 % solids) under agitation for 2 minutes.

This scrubbing stage was developed after a series of attrition tests intended to determine the degree of insoluble slimes separation obtained at a certain energy input. Table 5.4 shows the KCl recovery and the final insoluble slimes grade on the fraction over 200 mesh, at various attrition retention times. A 2-minute scrubbing time produced good liberation of the insoluble slimes, yet avoided excessive KCl attritioning. A scrubbing time of 2 minutes was thus selected for all the subsequent tests.

T [min]	W.I. grade + 75 µm	R_{KCl} + 75 µm
1	3.2 %	93.3 %
2	2.4 %	70.1 %
5	0.7 %	50.5 %
10	0.4 %	33.3 %
15	0.4 %	29.9 %

Table 5.5: Attrition time vs. insoluble grade and KCl recovery for the + 75 µm fraction.

Desliming

After scrubbing the sample was wet screened at 75 µm during 4 minutes using 3 litres of brine to remove the fines. This stage reduced the amount of slimes down to about 2.5 %, rendering the sample amenable for flotation. This residual slimes concentration is considered a conservative value for purposes of reagent testing, considering that a conventional rougher circuit in the Saskatoon area mills usually deals with a value between 0.8-1.5% W.I. after desliming (Eatock, 1982).

Conditioning

Conditioning was performed in the same flotation cell, at a pulp density of 50% solids; the reagent addition sequence was as follows:

a. slimes depressant, as a 0.1% solution in brine, conditioning time: 15 seconds

b. amine collector, as a 1.0-% suspension of Witco Adogen MG-102 W, conditioning time: 15 seconds

c. frother, Dowfroth 319 added directly without prior preparation of a stock solution, conditioning time: 15 seconds.

Flotation

After conditioning for a total time of 45 seconds, brine was added to fill up the tank and airflow was turned on. Flotation was then carried out at 23% solids, flotation time was kept constant at 4 minutes. The airflow rate was kept constant at 7.5 l/m.

The flotation products, namely concentrate, tailings, together with the slimes from the desliming step, were filtered and weighed prior to drying for at least 24 h at 105 °C. Once dried, the products were re-weighed, then crushed, quartered and a fraction sample was pulverized to be sent to the analytical laboratory where they were assayed for K_2O using a Philips X-Ray analyzer and assayed for insoluble slimes by wet gravimetry.

Assays values were input to a spreadsheet (see Appendix B), that calculates the metallurgical balance for each flotation test. The spreadsheet also includes the brine-corrected assays and calculated recoveries.

Modifier	Collector	KCl Recovery		W.I. Recovery	
[g/t]	[g/t]	Average	S.D.	Average	S.D.
Guar					
50	50	89.0	0.25	6.5	0.54
200	100	97.1	0.24	7.1	0.63
CMC ACZ					
200	50	88.0	0.38	24.0	3.46
200	100	99.1	0.41	20.2	2.03
Depramin 96					
50	50	86.6	0.21	30.0	3.66
200	100	96.2	0.88	23.4	1.70
Finnfix 300					
200	50	84.9	0.50	23.9	2.29
200	100	98.6	0.49	23.9	1.44

Table 5.6: Average values and std deviation for the tests on depression of slimes.

The parameters reported throughout the test program were percent KCl recovery (measured as the KCl units in the concentrate divided by the total KCl units originally in the feed times 100), and insoluble slimes (W.I.) recovery (measured as the W.I. units in the concentrate divided by the total W.I. units originally in the feed times 100). Concentrate grade in % K_2O was observed to be determined by the recoveries of KCl and insoluble slimes, so it was not used to evaluate the reagent performance.

Reproducibility of the results was assessed by randomly repeating four times at least two sets of tests for each reagent under evaluation. In each case, the average of KCl and slimes recoveries were used as the final value for evaluation purposes. A summary of the reproducibility tests is shown on Table 5.5.

Part II: Flocculation-Flotation Desliming.

The performance of the various reagents under investigation was evaluated in laboratory batch flotation tests. An efficient flotation desliming results in a high recovery of insoluble slimes in the concentrate and a low KCl recovery (low KCl losses).

5.5.   Reagents

Collector for Insolubles Flotation

Procol CK 910, an isodecyloxypropylamine 30 % acetate was used as a collector for the flotation of insoluble slimes during the reagent screening. The sample utilized in the testing program was provided by CIBA Specialty Chemicals.

Flocculants

A series of polyacrylamide flocculants with the same molecular weight but varying in charge density was obtained from CIBA Specialty Chemicals. The flocculants used as thickening and dewatering aids are known under the commercial name of Percol followed by a number. In order to avoid confusion, the flocculants used here for flotation of insoluble minerals will be referred to as Flocculants A, B and C.

Name	MW [g/g-mol]	Charge Density
Flocculant A	17-21 M	0 %
Flocculant B	17-21 M	3 %
Flocculant C	17-21 M	10 %

Table 5.7: Characteristics of the polyacrylamide flocculants tested.

The reason for choosing this particular series of flocculants was to investigate the relationship between flocculating effect and the ionic character of the reagent. All flocculants were tolerant to saturated brines. They were prepared by dissolving in a small amount of tepid water and then diluting to 0.5 % solutions using saturated brine. Table 5.6 shows some characteristics of the abovementioned flocculants.

54

5.6. Test Procedure for Flocculation-flotation Desliming

The test procedure developed for examining modifying reagents in desliming flotation is illustrated in Figure 5.2.

Factors:

Collector and flocculant dosages.

Constants:

Brine temperature: room temperature

Feed density; 23 % solids

pH, natural brine pH

Conditioning time, 15 seconds per reagent addtiton

Feed grade (40.2% KCl; 55.2% NaCl; 4.6% Slimes)

Feed size distribution (minus 20-mesh Tyler)
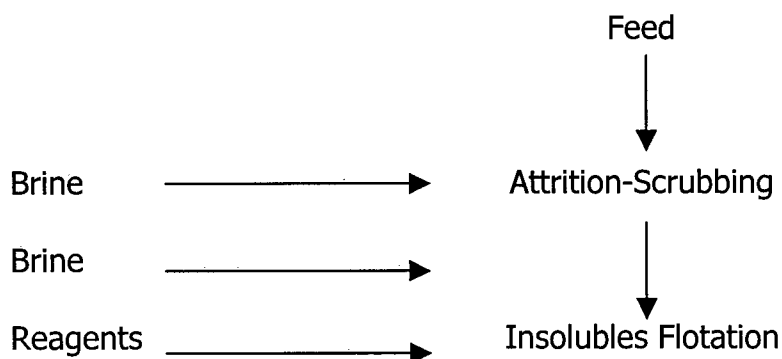
Slimes Collector (CIBA Procol CK910)

Feed

↓

Brine ⟶ Attrition-Scrubbing

Brine ⟶ ↓

Reagents ⟶ Insolubles Flotation

Figure 5.2: Test procedure for the flocculation-flotation desliming tests.

Slimes Conditioning

Conditioning was performed in the Denver D12 flotation cell, at a pulp density of 50% solids, with the following reagent addition sequence:

a) slimes flocculant, as a 0.1% solution in brine, conditioning time: 15 seconds

b) slimes collector, as a 1.0-% aqueous dispersion, conditioning time: 15 seconds

Slimes Flotation:

After conditioning, total volume of the cell is filled with brine and air is turned on. Slimes flotation at 23% solids is carried out during a total time of 2 minutes, at a constant airflow rate of 7.5 l/m.

The slime concentrates are filtered and weighed before drying for at least 24 h at 105 °C. Once dried, they are weighed again, then crushed, quartered and a fraction sample is pulverized to be sent to the analytical laboratory. A Philips X-Ray analyzer was used to assay for K_2O and wet gravimetry for insoluble slimes.

The parameters reported are W.I. recovery and the percentage KCl loss into the slimes concentrate.

Again, selected tests were duplicated four times in order to determine the reproducibility of the results (Table 5.7). It was observed that the laboratory scale flotation test shows good reproducibility. The weak point of the test program usually is the assaying. By using X-Ray techniques for K_2O and wet gravimetric analysis for

insoluble slimes, the standard deviations are usually half of those obtained when using atomic adsorption or flame photometry for K_2O and X-Ray techniques for insoluble slimes.

Flocculant	Collector	KCl Recovery		W.I. Recovery	
[g/t]	[g/t]	Average	S.D.	Average	S.D.
Flocculant A					
30	3	3.9	0.45	70.1	2.52
20	2	3.6	0.44	67.5	3.36
Flocculant C					
30	1	2.7	0.52	62.1	2.49
10	1	2.0	0.39	57.1	4.14

Table 5.8: Average values and standard deviation for the tests on flocculation-flotation desliming.

6.0   DEPRESSION OF SLIMES.

6.1   Baseline Tests

A series of flotation tests at increasing collector dosages but without modifier were carried out in order to determine the collector concentration required to obtain a sylvinite recovery exceeding 90%. This was considered a satisfactory recovery under the conditions of the testing program.

Five different collector dosages were used, from 100 to 3,000 g/t. Frother dosage was kept constant at 50 g/t. Table 6.1 shows the KCl and WI recoveries obtained at each collector dosage.

Collector g/t	KCl recovery	%K$_2$O	WI recovery
100	20.1	41.4	27.3
300	47.0	42.2	53.1
500	64.5	42.1	62.3
1,000	66.6	46.3	64.6
3,000	84.4	50.7	68.4

Table 6.1: Baseline tests without modifier added.

It was observed that the floating slimes stabilized the froth at any collector dosage. At amine dosages of 500 g/t and higher, the froth became very persistent and difficult to handle, due to the slime-stabilizing effect. With this type of froth it was impossible to obtain a good recovery of sylvite.

It was concluded then, that without the prior addition of a modifying agent it is impossible to obtain an acceptable KCl recovery under the conditions of the present testing program. Indiscriminate increase in collector dosage only produces a thick, stable and difficult to handle froth that does not contribute to the achievement of good KCl recoveries.

6.2 Screening Tests for Modifying Reagent Evaluation

6.2.1 Tests with Carboxymethyl Cellulose Finnfix 300

Figure 6.1 shows the effect of Finnfix 300, the carboxymethyl cellulose utilized as a reference, on KCl recovery at two different collector dosages.
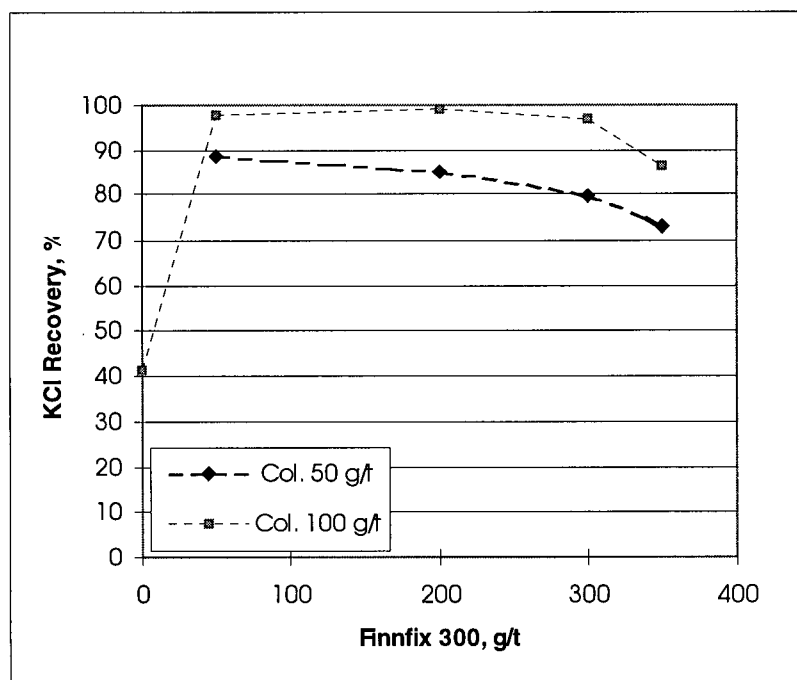
Figure 6.1: The effect of Finnfix 300 dosage on KCl recovery, at various collector dosages.

At a collector dosage between 50 and 100 g/t (typical for the Saskatchewan potash industry) the flotation was very selective, with both high KCl recovery and relatively low insoluble slimes recovery. The higher collector dosage (100 g/t) increased KCl recovery, without affecting much the insoluble slimes recovery (Figure 6.2).
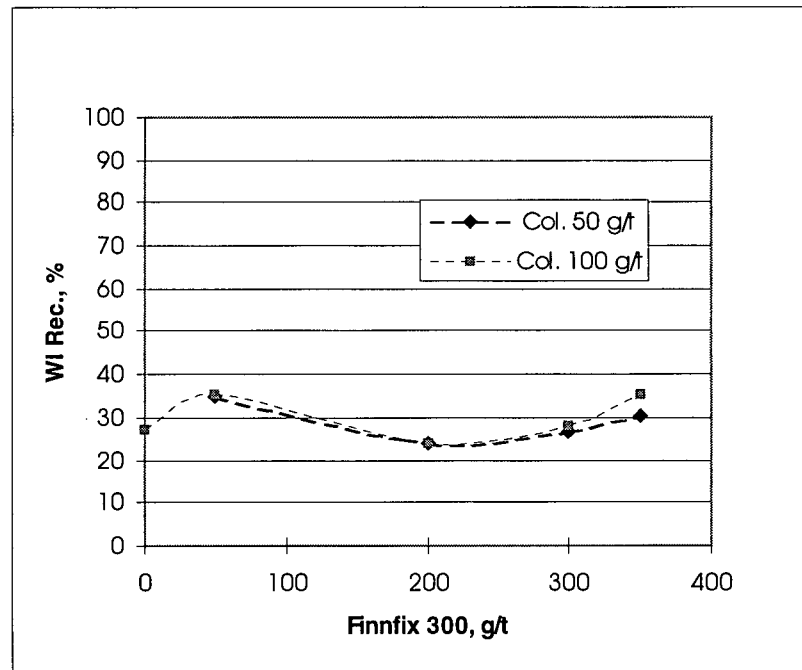
Figure 6.2: The effect of Finnfix 300 dosage on insolubles recovery at various collector dosages.

At a collector dosage of 50 g/t and at a FF300 dosage of 200 g/t, KCl recovery was slightly over 85 %. An increase in collector dosage to 100 g/t improved recovery to 98 %, but raised W.I. recovery from 22 % to almost 25 % and increased concentrate grade by about 1 % K_2O.

Figures 6.1 and 6.2 show that although the collector and the modifier act independently, both are needed in order to produce an efficient potash flotation. A certain minimum collector dosage is required to produce a satisfactory KCl recovery. At the same time, a minimum Finnfix 300 dosage is required to depress the insoluble slimes.

Figures 6.3 and 6.4 show the KCl recovery for different CMC's compared at a fixed collector dosage of 100 g/t. As it can be seen, all the tested carboxymethyl celluloses produced similar KCl recoveries and also similar insoluble slimes recoveries.
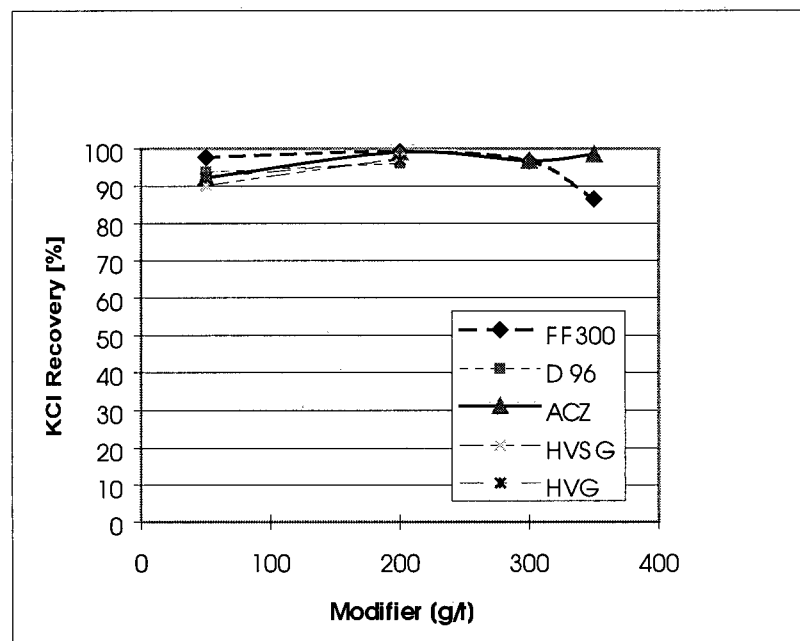
Figure 6.3: The effect of various CMC's on KCl recovery at a collector dosage of 100 g/t.
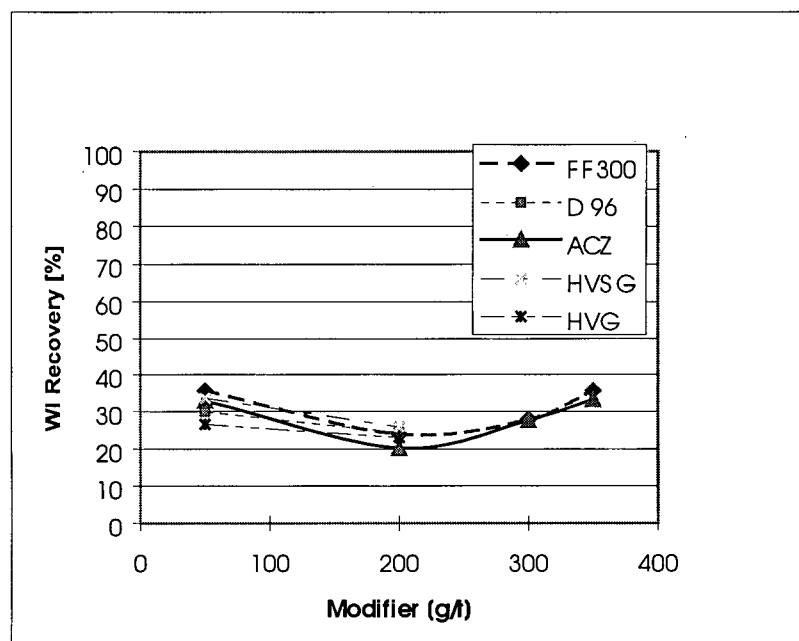
Figure 6.4: The effect of various CMC's on WI recovery at a collector dosage of 100 g/t.

ACZ, FF300 and HVG carboxymethyl celluloses all produced highly selective results, i.e. with a relatively low amount of insolubles reporting to the concentrate. It is interesting to note that although the carboxymethyl celluloses differ in both their MW and their DS, the flotation results are very similar.

The effects of the addition of collector and Finnfix 300 as modifier were analyzed using a polynomial regression model (Yuan et al., 1999). The general form of the polynomial model is as follows:

$$y = a_0 + a_1 x_1 + a_2 x_2 + a_{11} x_1^2 + a_{22} x_2^2 + a_{12} x_1 x_2 \quad (6.1)$$

where **y** is the recovery of KCl or insoluble slimes, **x₁** is the dosage of the modifier, **x₂** is the collector dosage, and **a₀, a₁, a₂, a₁₁, a₂₂,** and **a₁₂** are the model regression coefficients. A least squares algorithm is used to calculate the regression coefficients based on the experimental results; then the contour map is obtained from the model. The test data are also shown on the contour map to allow comparison between the experimental results and the expected values. This procedure, used by Lekki and Laskowski (1975), permits one to determine the optimal reagent dosage. The software STATGRAPHICS (Manugistics, 1998) was used in the statistical analysis.

Figure 6.5 shows the contour map generated by fitting the experimental points obtained using Finnfix 300 as modifier at dosages ranging from 50 g/t to 350 g/t, for various collector dosages. Equation (6.2) describes the recovery of KCl for the experiments.

$$y = 41.9869 + 0.0874x_1 + 1.0317x_2 - 0.0003x_1^2 - 0.0049x_2^2 - 7.66E\text{-}5x_1x_2 \quad (6.2)$$

The response surfaces of KCl recoveries versus consumption of collector and Finnfix 300 show that the optimum consumptions reach a plateau at 134 g/t of Finnfix and 103 g/t of collector. The accuracy of the model was examined by carrying out an additional test under the conditions specified in Table 6.2 and by comparing the experimentally determined KCl recovery with that predicted by the model. The last column in Table 6.2 shows the difference between the calculated and the measured KCl recovery at this particular dosage combination, expressed as a percentage as follows:

$$Difference(\%) = \frac{R_{KCl(calculated)} - R_{KCl(exp\,erimental)}}{R_{KCl(calculated)}} * 100$$

The significance of the coefficients of equation (6.2) was not checked as the interaction between the collector and the depressant was assumed to be of no statistical significance (Yuan et al., 1999).

Figure 6.5: Response contour of KCl recovery as a function of collector and Finnfix 300 dosages. The dots in the graph represent the experimental points.

Finnfix 300 g/t	Collector g/t	calculated KCl recovery	measured KCl recovery	Difference %
200	100	99.5%	99.2%	0.3%

Table 6.2: The difference between calculated and measured recovery of KCl.

Figure 6.6 shows a plot of the regression between KCl recovery values measured and predicted using equation (6.2). The regression has a R^2 = 0.9577, which implies the model fits the experimental data very accurately.
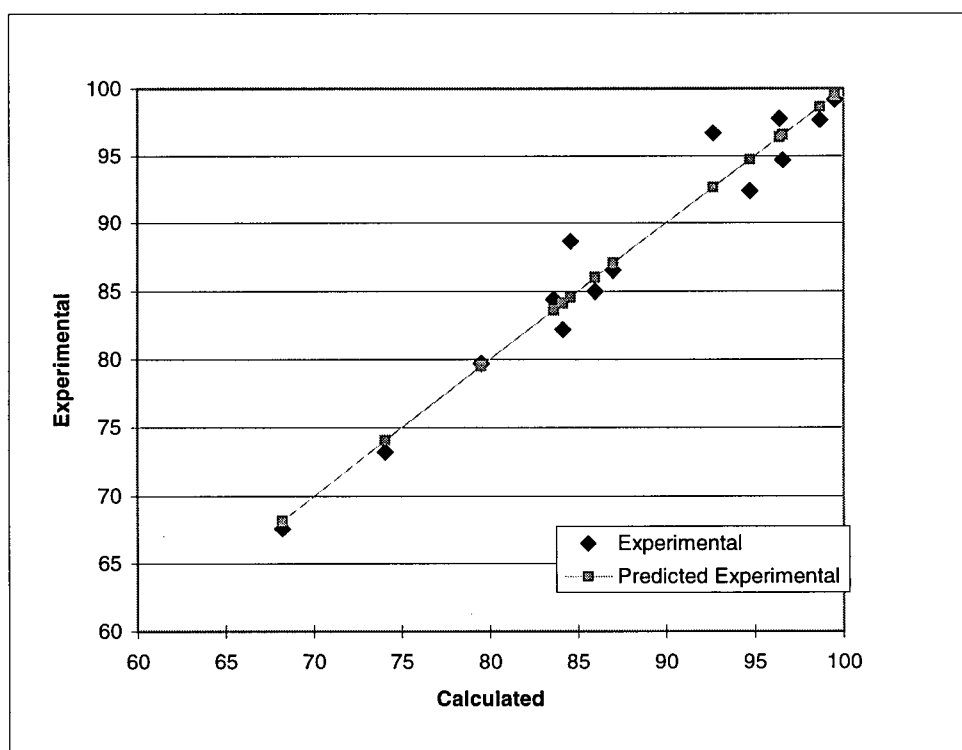
Figure 6.6: Calculated line fit plot for the regression between measured and predicted KCl recovery values.
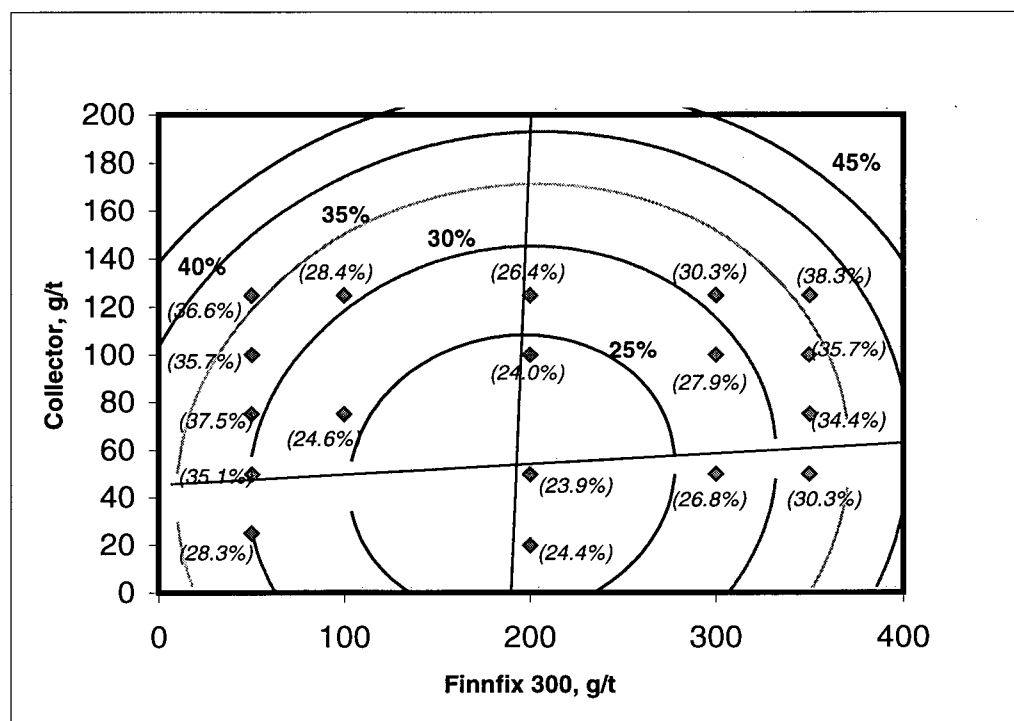
Figure 6.7: Response contour of insoluble slimes recovery as a function of collector and Finnfix 300 dosages. The dots in the graph represent the experimental points.

Finnfix 300	Collector	calculated	measured	Difference
g/t	g/t	WI recovery	WI recovery	%
200	100	24.2%	24.0%	0.8%

Table 6.3: The difference between calculated and measured recovery of insoluble slimes.

The same statistical procedure was applied for the recovery of insoluble slimes to the potash concentrate. Figure 6.7 shows a contour map for the insoluble slimes recovery

66

using Finnfix 300 as modifier at dosages ranging from 50 g/t to 350 g/t, for various collector dosages. The polynomial model for the recovery of insoluble slimes was:

$$y = 37.7529 - 0.1485x_1 - 0.0693x_2 + 0.0004x_1^2 + 0.0009x_2^2 - 8.92E\text{-}5x_1x_2 \quad (6.3)$$

The response surfaces show that the optimum consumption reaches a plateau at 190 g/t of Finnfix and 50 g/t of collector. The accuracy of the model is shown in Table 6.3.
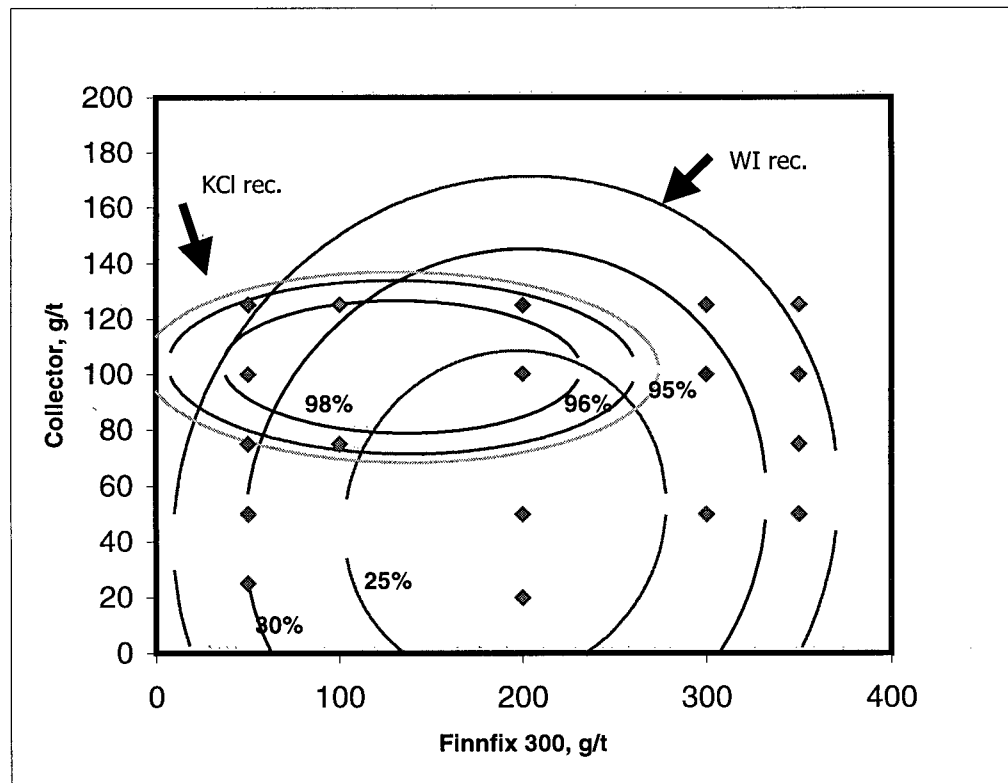
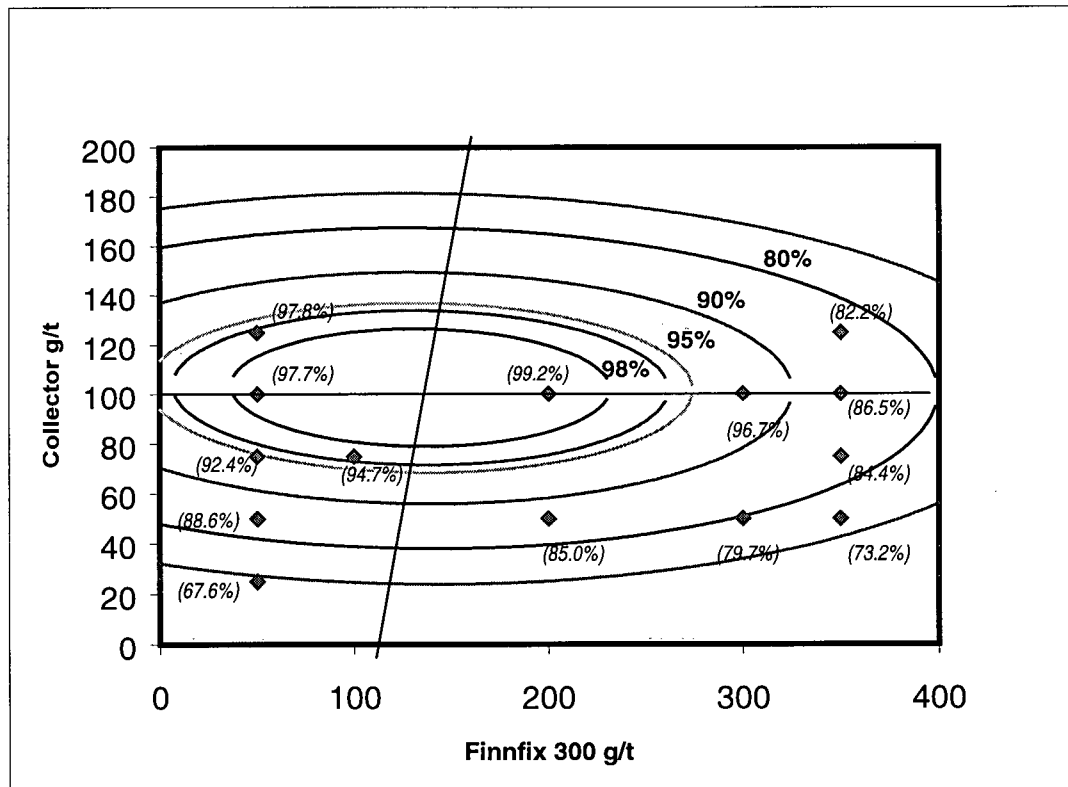**Figure 6.8:** Superimposed response contour maps of KCl and insoluble slimes recoveries as a function of collector and Finnfix 300 dosages. The dots in the graph represent the experimental points.

67

Figure 6.8 shows the response contour map for the KCl recovery superimposed on the response contour map for the insoluble slimes. As can be seen, it is not possible to optimize both recoveries at the same reagent dosages. Obviously, the optimization of KCl recovery should take precedence.

6.2.2 Tests with Carboxymethyl Cellulose ACZ

Equation (6.4) describes the recovery of KCl for the series of experiments using the low molecular weight and low degree of substitution CMC ACZ.

$$y = 59.2228 + 0.0495x_1 + 0.6678x_2 - 0.0002x_1{}^2 - 0.0036x_2{}^2 + 0.0003x_1x_2 \quad (6.4)$$

Figure 6.9 shows the response surfaces of KCl recoveries versus consumption of collector and CMC ACZ obtained from Equation (6.4). The map shows that the optimum consumptions reach a plateau at 247 g/t of CMC ACZ and 105 g/t of collector. The accuracy of the model was examined by carrying out an additional test under the conditions specified in Table 6.4.

CMC ACZ g/t	Collector g/t	calculated KCl recovery	measured KCl recovery	Difference %
200	100	100.0%	99.1%	0.9%

Table 6.4: The difference between calculated and measured recovery of KCl.

Figure 6.9: Response contour of KCl recovery as a function of collector and CMC ACZ dosages. The dots in the graph represent the experimental points.

The same statistical procedure was utilized to find the optimum dosages of ACZ and collector to minimize insoluble slimes recovery. The polynomial model for insoluble slimes recovery for this series of tests was:

$$y = 67.8656 - 0.2573x_1 - 0.5033x_2 + 0.0006x_1^2 + 0.0027x_2^2 + 0.0002x_1x_2 \quad (6.5)$$

Figure 6.10 shows a contour map obtained from Equation (6.5) for the insoluble slimes recovery using the CMC ACZ as modifier. The response surfaces show that the optimum consumption reaches a plateau at around 207 g/t ACZ and 84 g/t collector,

values that are better that those obtained with Finnfix 300 (Figure 6.7). The accuracy of

the model as estimated in Table 6.5 is quite satisfactory.
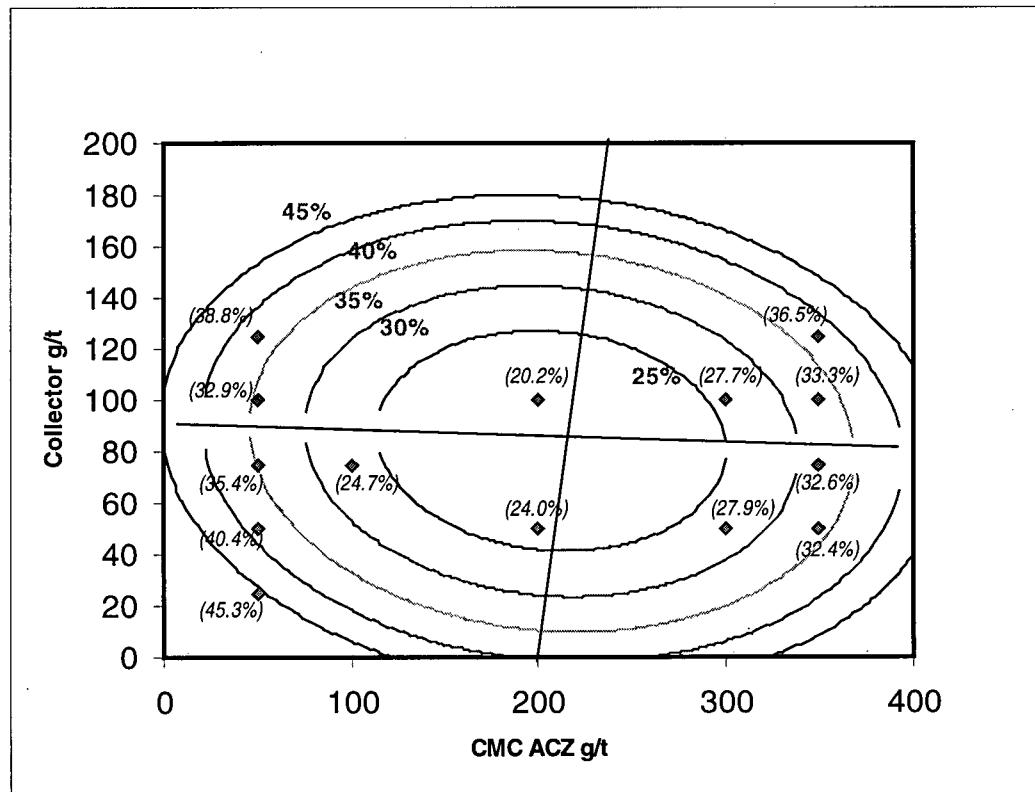
Figure 6.10: Response contour for recovery of insoluble slimes as a function of collector and ACZ dosages. The dots in the graph represent the experimental points.

ACZ g/t	Collector g/t	calculated WI recovery	measured WI recovery	Difference %
200	100	20.2%	20.7%	2.6%

Table 6.5: Difference between calculated and measured recovery of insoluble slimes.

Figure 6.11 shows the response contour map for the KCl recovery superimposed on the response contour map for the insoluble slimes. As it was the case with Finnfix, it is not possible to optimize both recoveries at the same reagents dosages. Again, the optimization of KCl recovery should take precedence.

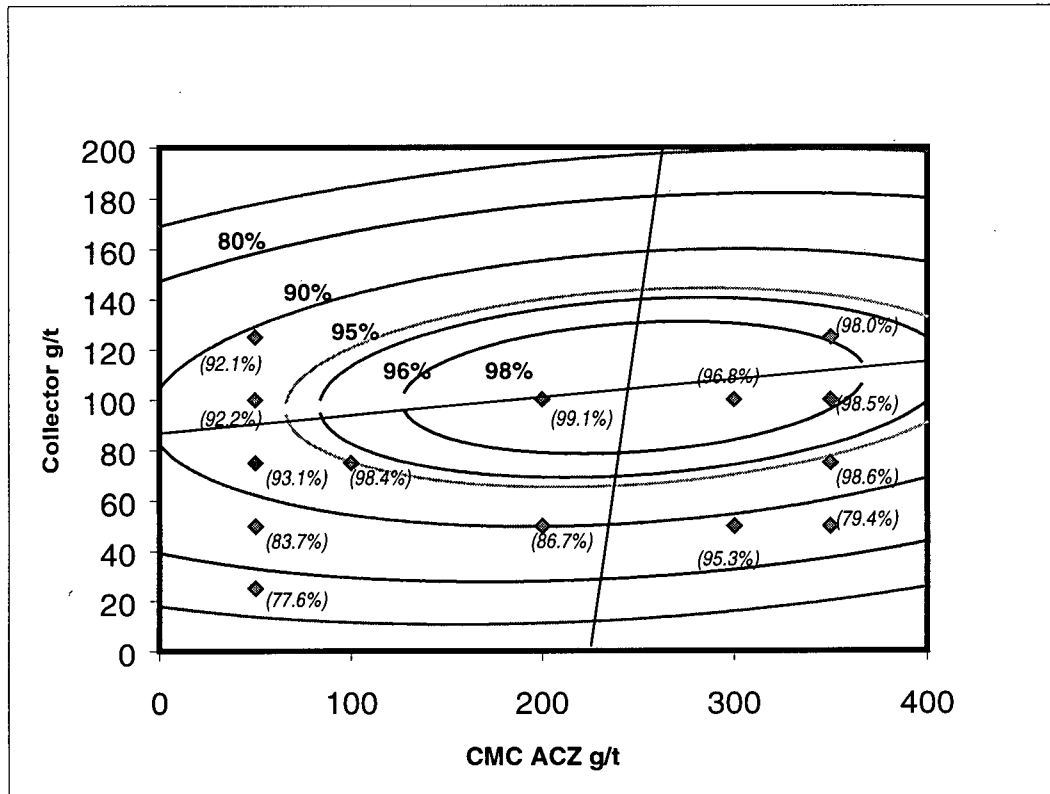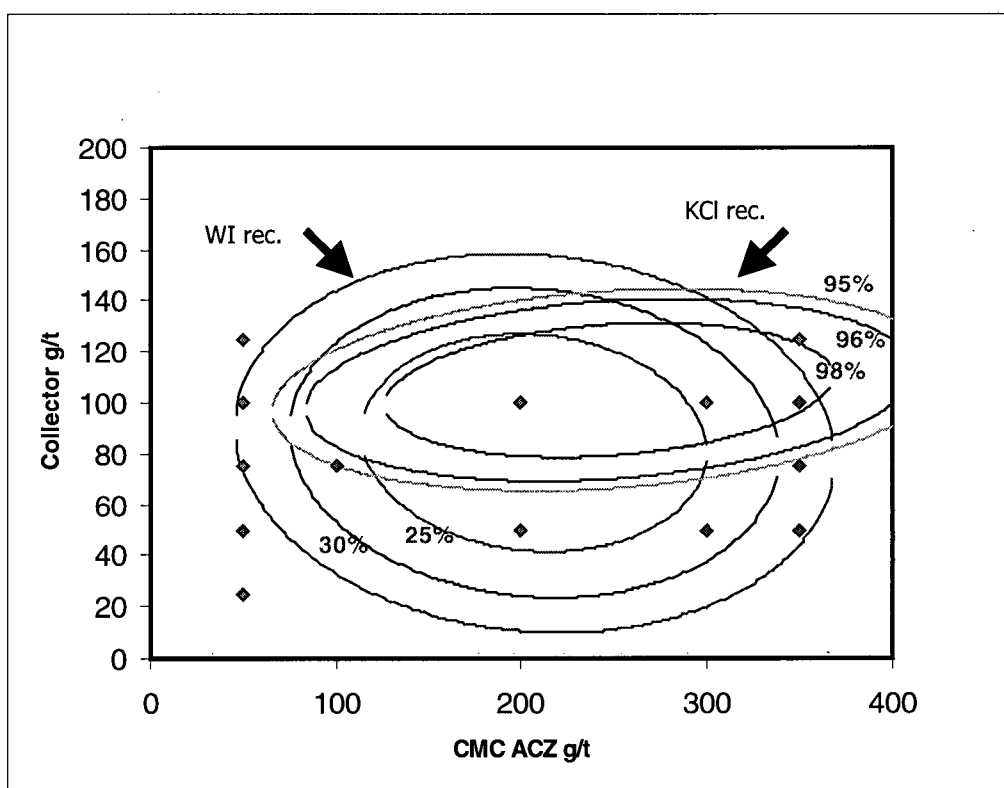**Figure 6.11:** Superimposed response contour maps of KCl and insoluble slimes recoveries as a function of collector and CMC ACZ dosages. The dots in the graph represent the experimental points.

6.2.3 Tests with Guar Gum

When compared with CMC modifiers, guar gum proved to be a very effective insoluble slimes depressant.
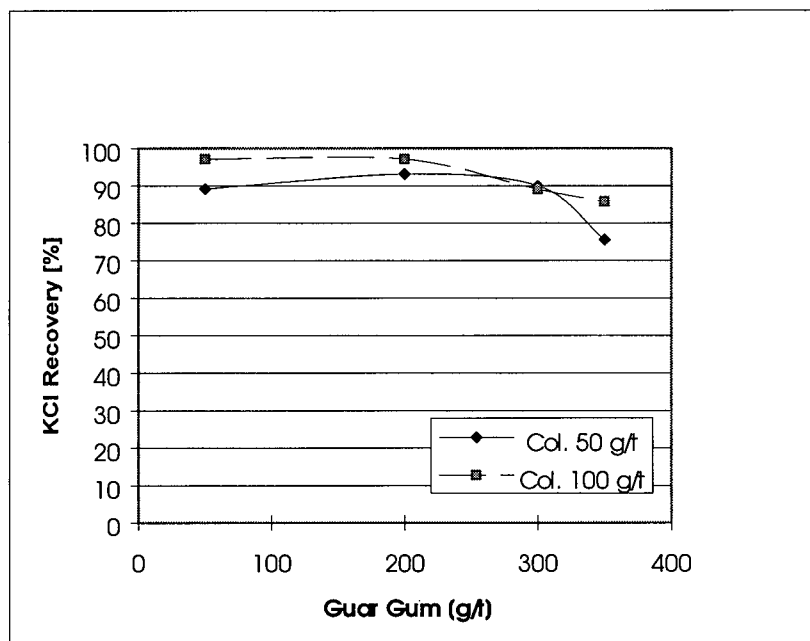
Figure 6.12: The effect of Guar Gum dosage on KCl recovery at collector dosages of 50 and 100 g/t.

Guar gum produced acceptable KCl recoveries over 97 % at dosages as low as 100 g/t (Figure 6.12), with collector dosages around 100 g/t. At the same time, the recovery of insoluble slimes was below 10% at a collector dosage of 100 g/t (Figure 6.13). This was one of the lowest insoluble slimes recovery obtained at such a low reagent dosage. This recovery is only about one-third of that usually obtained when using CMC modifiers. KCl recoveries, on the other hand, were slightly lower than those obtained when using Finnfix 300 and ACZ.

72
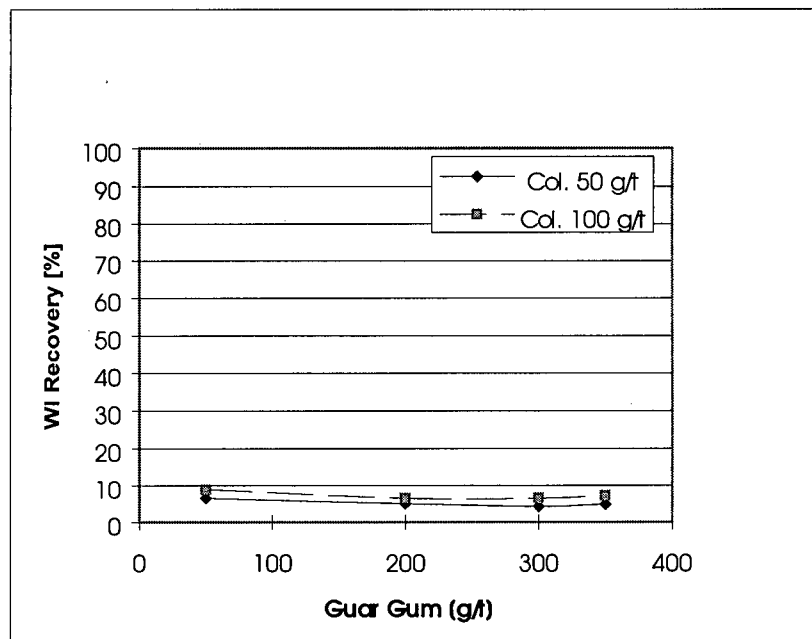
Figure 6.13: The effect of Guar Gum dosage on insoluble slimes recovery at collector dosages of 50 and 100 g/t.

As in the tests with CMC modifiers, higher collector dosages did improve KCl recoveries, but a higher percentage of the insolubles reported to the concentrate. Concentrate grades also improved at higher collector dosages as a result of improved flotation of coarser KCl particles.

At a collector dosage of 100 g/t, the increase in guar gum dosage up to 350 g/t leads to decreased KCl recovery, which seems to imply that the modifying effect of the guar extends into depressing KCl flotation.

The previously described statistical procedure was applied to find the optimum dosages of guar gum and collector. The following polynomial model for the recovery of KCl was found:

$$y = 51.5951 + 0.1427x_1 + 0.7394x_2 - 0.0004x_1^2 - 0.0035x_2^2 - 0.0002x_1x_2 \quad (6.6)$$
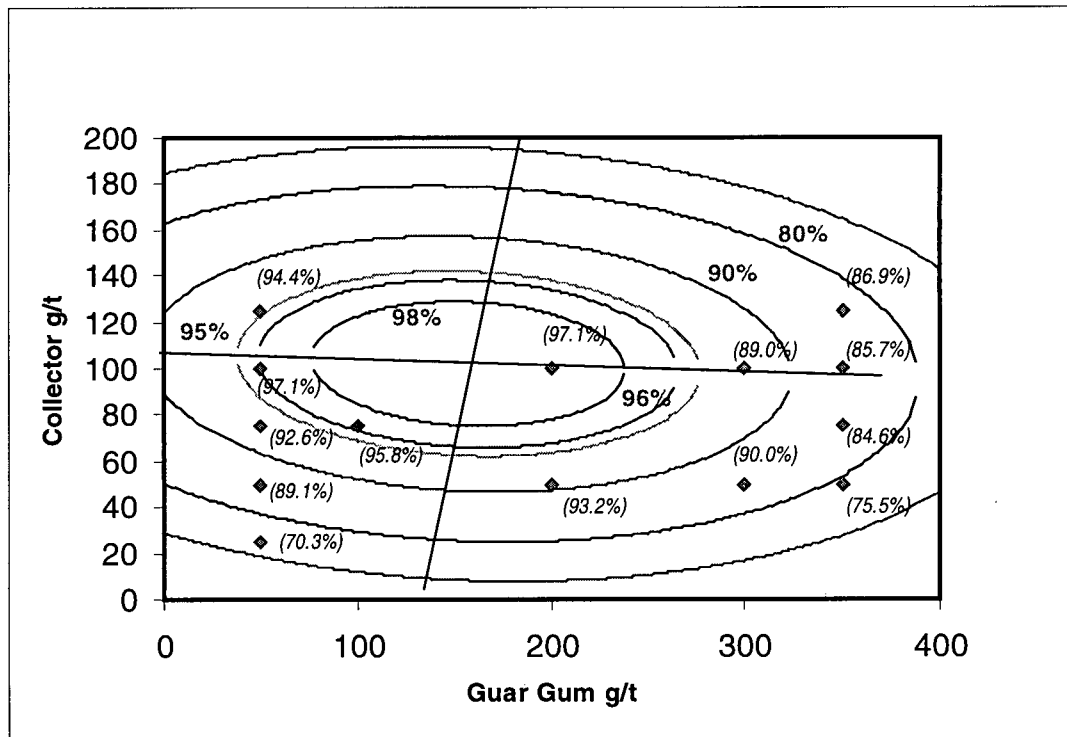
Figure 6.14: Response contour of KCl recovery as a function of collector and guar gum dosages. The dots in the graph represent the experimental points.

Figure 6.14 shows the contour map generated by fitting the experimental points obtained using guar gum as modifier at dosages from 50 g/t to 350 g/t, for various collector dosages to equation 6.6. The response surfaces for KCl recoveries versus

consumption of collector and guar gum show that the optimum consumption plateau is located around 157 g/t of guar gum and 102 g/t of collector.

Guar gum g/t	Collector g/t	calculated KCl recovery	measured KCl recovery	Difference %
200	100	99.8%	97.1%	2.7%

Table 6.6: The difference between calculated and measured recovery of KCl.
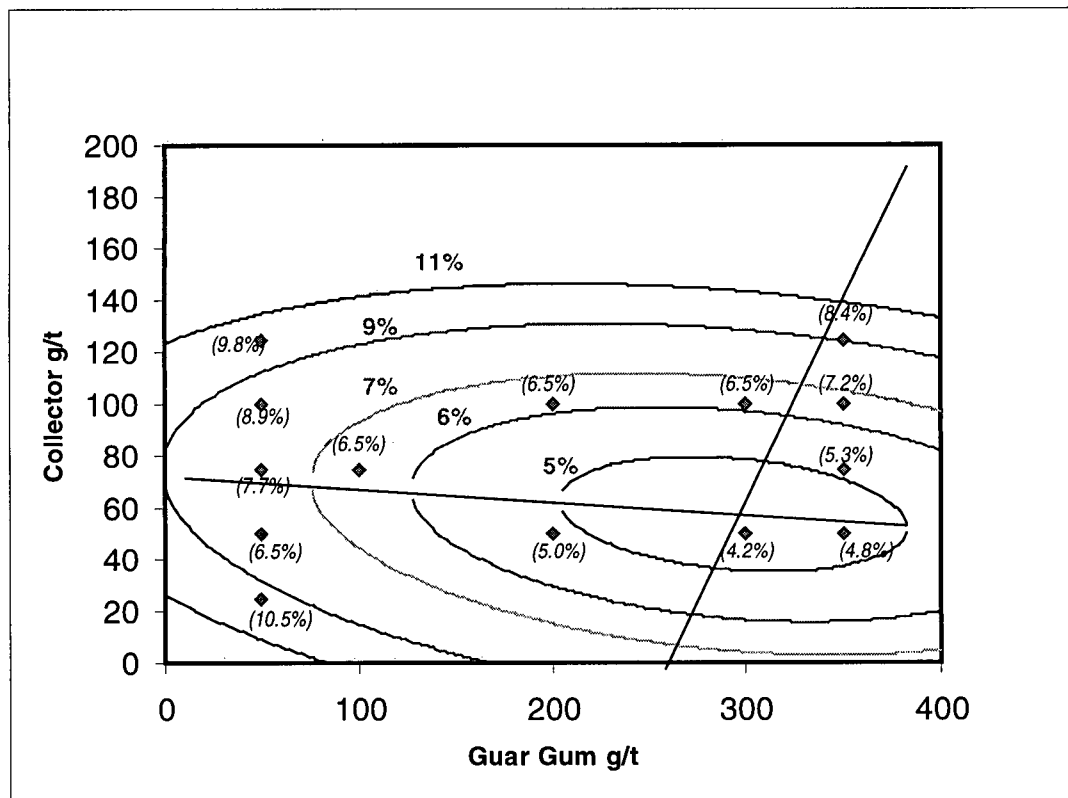
Figure 6.15: Response contour of insoluble slimes recovery as a function of collector and guar gum dosages. The dots in the graph represent the experimental points.

Figure 6.15 shows the contour map for the insoluble slimes recovery using guar gum as modifier at dosages ranging from 50 g/t to 350 g/t, for various collector dosages. The response surfaces show that the optimum consumption plateau is located around 293 g/t of guar and 57 g/t of collector. The accuracy of the model is evaluated in Table 6.7. The polynomial model for the recovery of insoluble slimes was found to be:

$$y = 13.7754 - 0.0374x_1 - 0.1288x_2 + 0.0001x_1^2 + 0.0009x_2^2 - 0.0001x_1x_2 \quad (6.7)$$

Guar gum g/t	Collector g/t	calculated WI recovery	measured WI recovery	Difference %
200	100	6.2%	6.5%	-4.4%

Table 6.7: The difference between calculated and measured recovery of insoluble slimes.

Figure 6.16 shows the response contour map for the KCl recovery superimposed with the response contour map for the insoluble slimes recovery. As it can be seen, it is impossible to optimize both recoveries at the same reagents dosages. Again, the optimization of KCl recovery takes precedence.
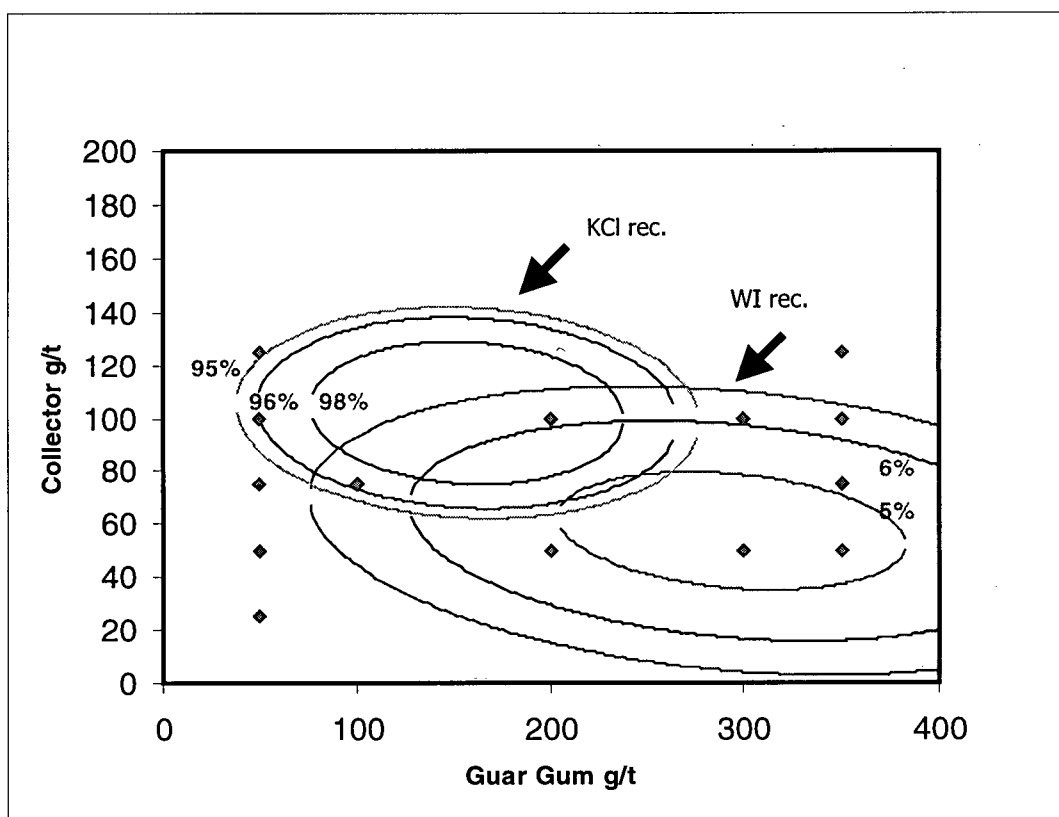
Figure 6.16: Superimposed response contour maps for recoveries of KCl and insoluble slimes as a function of collector and guar gum dosages. The dots in the graph represent the experimental points.

6.2.4 Tests with Carboxymethyl Isopropyl Guar

Several tests were conducted using Galactasol 650 as modifier at different collector dosages. KCl recoveries ranged from 80 to 90 %, with concentrate grades between 56 - 58 % K_2O and insolubles recoveries between 30 and 35 %. These results, although not bad, did not show any improvement over the use of either CMC or guar gum, and no further testing was done with this reagent.

6.2.5 Tests with Carboxyl Methyl Starch

Tests using Glucostar 65 as modifier at collector dosages ranging from 50 to 100 g/t produced KCl recoveries between 80 to 90 %, with concentrate grades between 53 - 57 % K_2O and insolubles recoveries between 30 and 35 %. These results, similar to those obtained using Galactasol did not justify the use of this modifier.

6.2.6 Tests Using Polyacrylamides

Polyacrylamides are good blinders of insoluble slimes (Yuan et al., 1999), since consumption of collector can be reduced when using PAM as modifier.
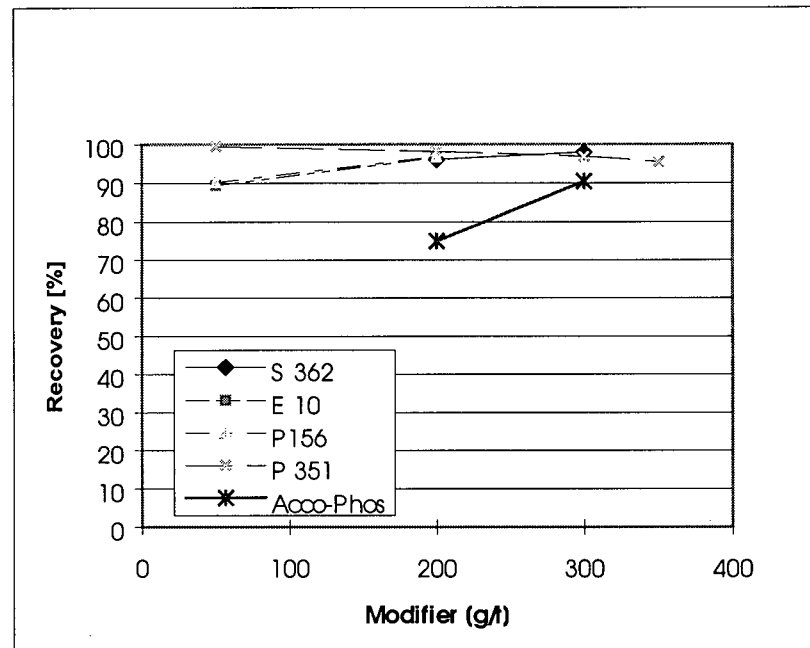
Figure 6.17: The effect of polyacrylamides on KCl recovery at a collector dosage of 100 g/t.

The high molecular weight polyacrylamides at dosages of 100 g/t and higher, produced in general fairly high KCl recoveries, ranging from 93 to 97 %. Flotation selectivity was poor however, with insoluble recoveries between 30 to 60 % (Figures 6.17 and 6.18).
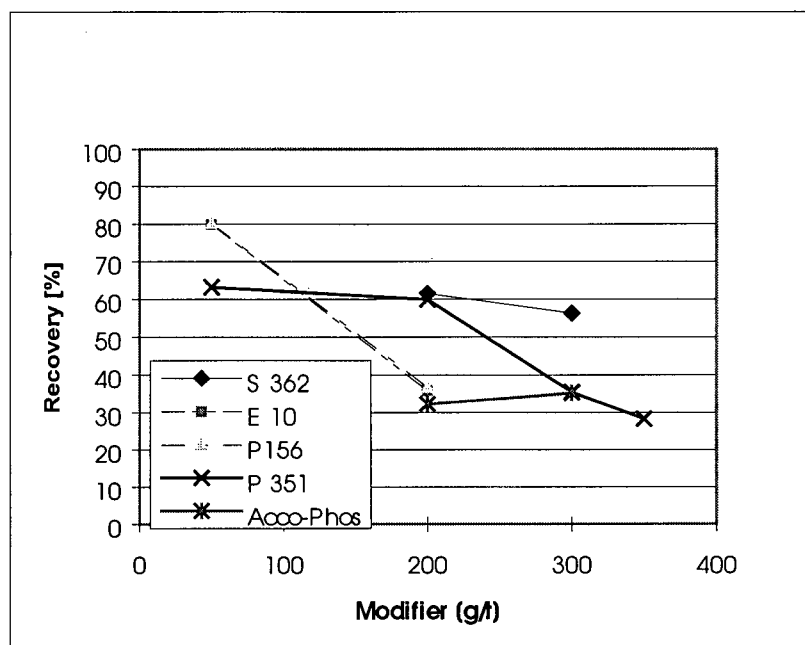
Figure 6.18: The effect of polyacrylamides on insoluble slimes recovery at a collector dosage of 100 g/t.

KCl recoveries reached a peak of 97 % at PAM dosages over 200 g/t when insoluble recoveries were at their minimum (Figure 6.17). The maximum concentrate grades and the minimum insoluble recoveries are usually almost coincidental, showing how interrelated these parameters are. Further increase in the polyacrylamide dosage from 200 g/t to 300 g/t increases flotation selectivity, as less insoluble slimes floats with the

79

KCl concentrate. This effect can be seen with all the tested polyacrylamides (Figure 6.18).

Percol E10 gave KCl recoveries around 95 %, and insoluble recoveries exceeding 40 %. A strong flocculation of the insoluble slimes seems to be the predominant effect produced by Percol E10.

Tests with Superfloc 362, the cationic polyacrylamide, also produced high KCl recoveries, (over 95 %), at low selectivity, due to a high recovery of insolubles (nearly 55 %).
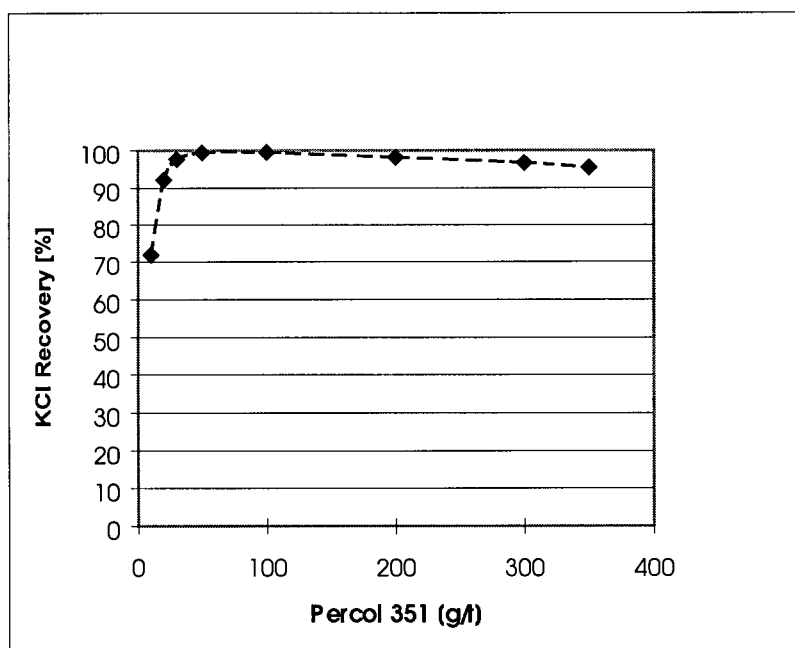
Figure 6.19: The effect of Percol 351 dosage on KCl recovery at a collector dosage of 100 g/t.

Acco-Phos 950 produced poor KCl recoveries, averaging 70 %; but fairly high insoluble slimes recoveries of near 30 %.

Tests using Percol 351 show that at low polyacrylamide dosages (10-30 g/t), the KCl recovery is relatively low, starting at 70% and increasing to 90% (Figure 6.19). At the same low Percol 351 dosages, insoluble slimes recovery increases to values as high as 65% (Figure 6.20).
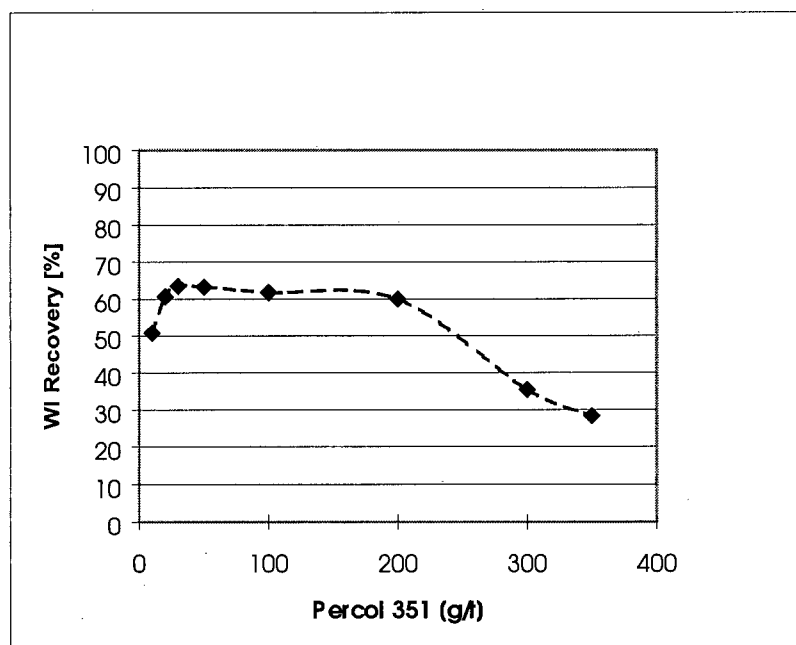
Figure 6.20: The effect of Percol 351 dosage on insolubles recovery at a collector dosage of 100 g/t.

When the Percol 351 dosage is increased up to 300 g/t the KCl recovery increases rapidly up to 90% and the insoluble slimes recovery to the concentrate decreases to just below 30%. This effect is even more pronounced when Percol 156 of a very high molecular weight and ionic charge is used (Figure 6.17).

It seems to be a transition where the polyacrylamide works as a flocculant aiding the insoluble slimes flotation up to a certain threshold dosage. Once this threshold dosage is exceeded, the polyacrylamide starts to act as a slimes depressant.

Applying the statistical model for the recovery of KCl using Percol 351 as modifier, the polynomial model was found to be:

$$y = 48.2528 + 0.2435x_1 + 0.4658x_2 - 0.0004x_1^2 - 0.0010x_2^2 - 0.0006x_1x_2 \quad (6.8)$$

Figure 6.21 shows the contour map generated by fitting the experimental points obtained using Percol 351 as a modifier at dosages ranging from 10 g/t to 350 g/t, for various collector dosages. The response surfaces of KCl recoveries versus consumption of collector and guar gum show that the optimum consumption plateau is located around 146 g/t of Percol 351 and 179 g/t of collector. These are fairly high dosages, when compared with those for carboxylmethyl celluloses and guar gum, (even more considering that in general polyacrylamides are very expensive reagents).
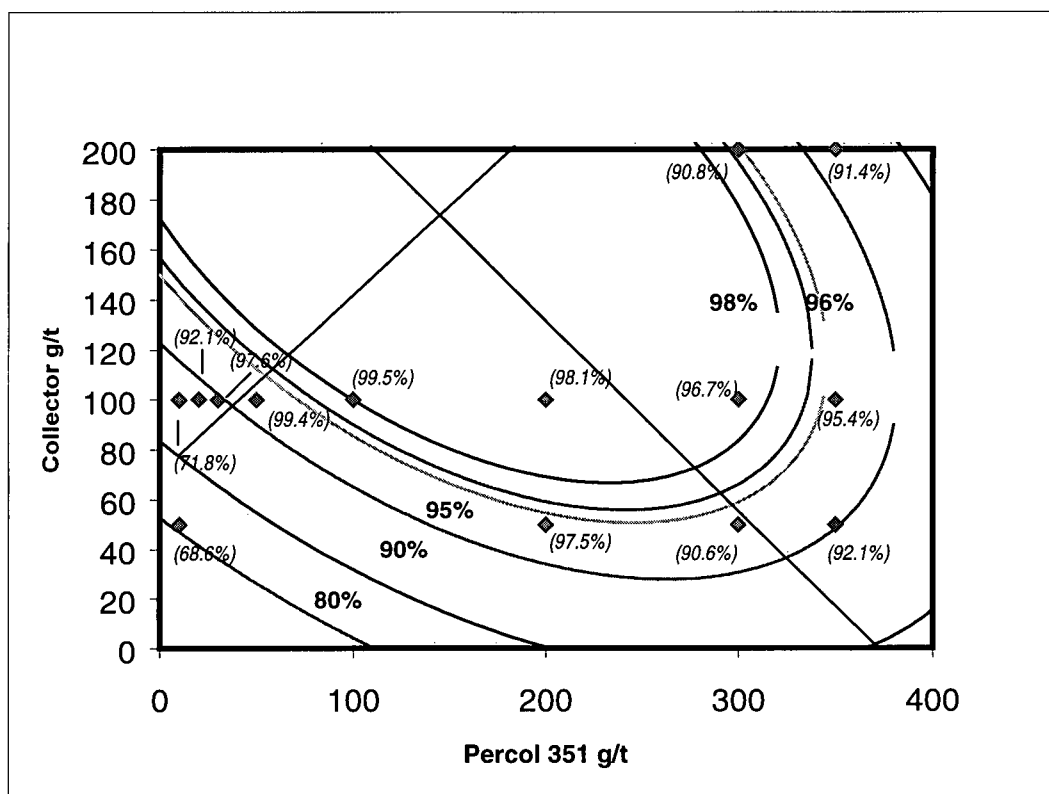
Figure 6.21: Response contour of KCl recovery as a function of collector and Percol 351 dosages. The dots in the graph represent the experimental points.

| Percol 351 | Collector | calculated | measured | Difference |
g/t	g/t	KCl recovery	KCl recovery	%
200	100	102.9%	98.1%	4.7%

Table 6.8: The difference between calculated and measured recovery of KCl.

The accuracy of the model is presented in Table 6.8. The table shows the percent difference between the calculated and the measured KCl recovery at a particular dosage combination.

Applying the statistical model for the recovery of insoluble slimes using Percol 351 as modifier, the polynomial model was found to be:

$$y = 52.3047 + 0.0893x_1 + 0.1066x_2 - 0.0005x_1^2 - 0.0006x_2^2 + 6.44\text{E-}5x_1x_2 \quad (6.9)$$
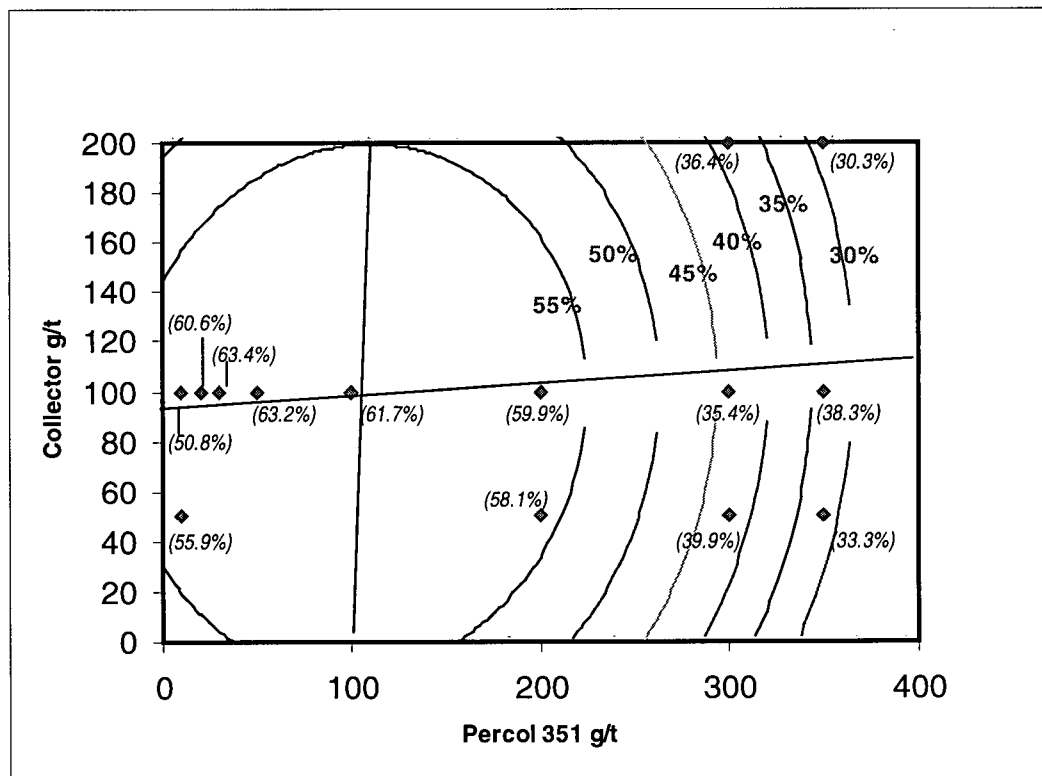
Figure 6.22: Response contour of insoluble slimes recovery as a function of collector and Percol 351 dosages. The dots in the graph represent the experimental points.

Figure 6.22 shows a similar contour map for the recovery of insoluble slimes using Percol 351 as a modifier at dosages from 10 g/t to 350 g/t, for various collector dosages. The model shows a maximum insoluble slimes recovery at a dosage of about 103 g/t of PAM and 93 g/t of collector. The response surfaces indicates that even at low Percol 351 and collector dosages, it is possible to achieve high insoluble recoveries.

Percol 351 g/t	Collector g/t	calculated WI recovery	measured WI recovery	Difference %
200	100	57.5%	59.9%	-4.1%

Table 6.9: The difference between calculated and measured recovery of insoluble slimes.

This phenomenon is used in the flocculation-flotation desliming process, where starvation dosages of a flocculant and a weak cationic collector are used to remove the insoluble slimes from the potash ore. The accuracy of the model is shown on Table 6.9.

Figure 6.23 shows the response contour map for KCl recovery superimposed with the response contour map for the insoluble slimes recovery. As can be seen from the map, the region of high KCl recovery almost coincides with the region of high recovery of insoluble slimes. This means that in general, Percol 351 and polyacrylamides are very poor depressants of slimes.
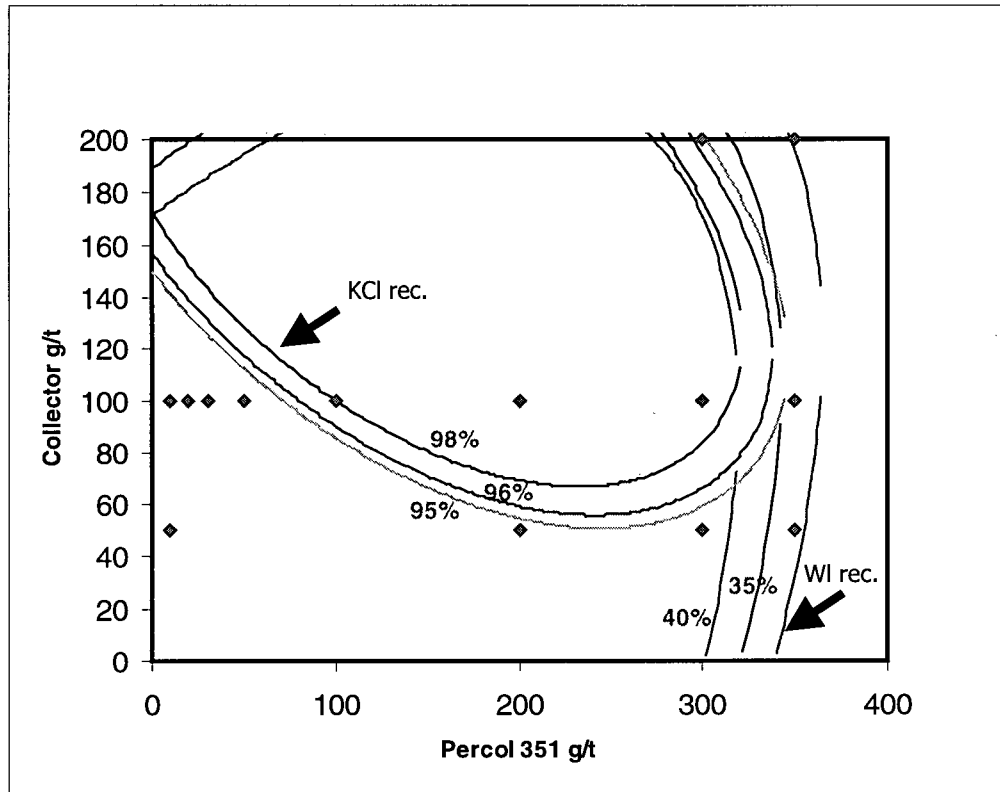
Figure 6.23: Superimposed response contour maps of KCl and insoluble slimes recoveries as a function of collector and guar gum dosages. The dots in the graph represent the experimental points.

6.3   Plant Scale Test Using Carboxyl Methyl Cellulose

A full scale plant test was run at Agrium Potash Operations on Friday May 22nd 1998, using a low molecular weight, low DS Carboxyl Methyl Cellulose supplied by S & G Resources instead of guar gum. This was originally planned to be a three-day plant test in which 6 tonnes of CMC ACZ were to be used at an average dosage of 100 g/t. The plant test failed and was terminated after approximately twelve hours.

The full-scale plant test using CMC ACZ as depressant failed mainly because of a very strong and persistent froth created by slimes floating with the potash concentrate. The persistent froth originated in the fines rougher bank and soon expanded to the cleaner and re-cleaner flotation banks until, after a few hours, it created a froth that extended almost to all the flotation circuit.

It was concluded from the test that the CMC failed to depress the slimes as efficiently as Guar. As a result of this, a large amount of slimes reported to the KCl concentrate producing a stable froth that could not be destroyed along the flotation circuit.

The amount of slimes present in the brine prior to potash flotation could be the reason for the difference between the performance of Guar Gum and CMC as slimes depressant at Agrium Potash. At IMC-Colonsay, mechanical desliming removes insoluble slimes very efficiently and the potash flotation feed usually averages 0.8 % slimes. Under these conditions, CMC works efficiently as a slimes depressant. At Agrium Potash, the rougher feed averages 2.0 % slimes and CMC fails to depress enough under such conditions. The mineralogical composition of the insoluble slimes at both IMC-Colonsay and Agrium is identical (Mossman et al., 1982).

7.0   FLOCCULATION - FLOTATION DESLIMING

7.1.   Reagent Screening

The most common commercial practice for removal of insolubles is mechanical desliming; however, this process results in excessive brine usage and high potash losses when the concentration of insolubles either exceeds 4 to 5% or fluctuates due to ore variability.

The flocculation/flotation of insolubles, the process developed by Cominco Fertilizers and Cyanamid is currently utilized at Agrium. In this process the scrubbed ore is subjected to a two-stage insoluble conditioning and flotation, prior to the conventional sylvite flotation. A very high molecular weight non-ionic polyacrylamide flocculant is used to selectively flocculate the insolubles, and an isodecyloxypropylamine cationic collector is used to float the flocculated slimes. Dosages used are around 25 g/t and 2 g/t respectively (Brogoitti & Howald, 1973; Chan et al., 1983; Cormode, 1985).

Although some KCl is lost by being either floated together with the insolubles or entrained in the insolubles concentrate froth, the flocculation/flotation is a more efficient desliming method than conventional mechanical desliming for the potash ores with a high content of insoluble slimes.

It has always been believed that in the flocculation-flotation method, the proper selection of the collector dosages is very critical in preventing the KCl from floating. Generally, the dosage of the collector in this process is controlled to a starvation level to prevent the flotation of KCl along with the insoluble slimes.

The test program consisted of a flocculant reagent screening, and included three different flocculants of the same molecular weight but different charge density (see page 65). The reason for choosing that particular series of flocculants was to investigate the relationship between flocculating effect and ionic character of the reagent.

The flocculants used for insoluble minerals flotation were denominated as Flocculant A, B and C. Flocculant A is Percol 351, a non-ionic polyacrylamide that is used at Agrium. Flocculant B is an anionic flocculant with a charge density of 3%. Flocculant C is a highly anionic flocculant with a charge density of 10%.
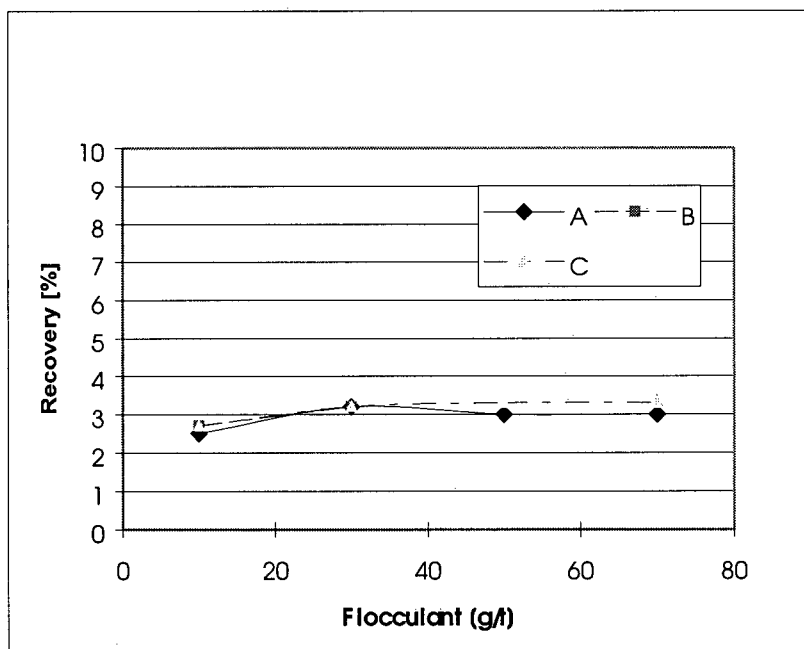
Figure 7.1: KCl recovery using various flocculants at a collector dosage of 3 g/t.

Figure 7.1 seems to indicate that the anionic polyacrylamides do float a little more KCl than the non-ionic, although this amount is very small. This fact was later confirmed at Agrium, after a three-month test using an anionic polyacrylamide.
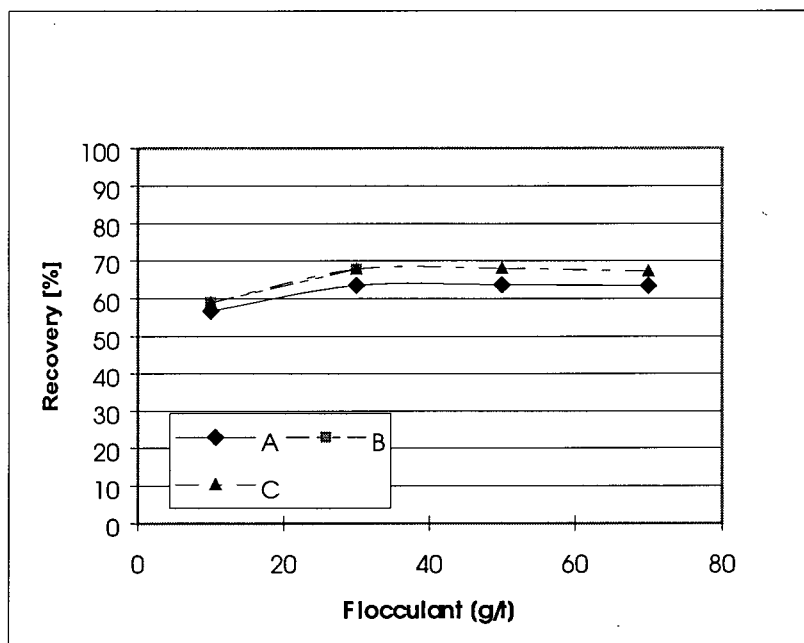
Figure 7.2: WI recovery using various flocculants at a collector dosage of 3 g/t.

Figure 7.2 indicates that the anionic polyacrylamides tend to provide higher recoveries of insoluble slimes than the non-ionic polyacrylamide. Nevertheless this difference is very small and was also proved after a full scale plant test at Agrium.

7.2 Non ionic flocculant- Optimization

Figure 7.3 shows a contour map for the KCl recovery when using a cationic isodecyloxypropylamine as insoluble slimes collector at dosages from 1 g/t to 7 g/t, and a non-ionic polyacrylamide (Flocculant A) as insoluble slimes flocculant at dosages ranging from 10 to 70 g/t. The relationship between KCl recovery and the dosages of collector and flocculant obtained by the regression analysis fits the following polynomial model:

$$y = 1.5518 + 0.0003x_1 + 0.4662x_2 - 1.67\text{E-}05x_1^2 - 0.0092x_2^2 + 0.0012x_1x_2 \quad (7.1)$$

Figure 7.3: Response contour of KCl recovery as a function of collector and non-ionic flocculant dosages. The dots in the graph represent the experimental points.

The response surfaces do not show a clear plateau for an optimum dosage. Actually the model produced an intersection of the axis of the ellipse in the domain of the negative numbers. The accuracy of the model that generated the contour map is shown in Table 7.1. This would indicate that is not true flotation which causes the KCl to float. It is not entrainment alone either, but probably a combination of both mechanisms. By comparing the NaCl/KCl ratio in the feed and in the slimes concentrate, it can be seen that while the feed to flocculation/flotation has a ratio of 1.4; the insoluble concentrate has a NaCl/KCl ratio of 0.9. This seems to indicate that KCl particles are being floated preferentially over NaCl particles, which leads to the conclusion that some sort of combination of entrainment/flotation occurs.

Flocculant A g/t	Collector g/t	calculated KCl recovery	measured KCl recovery	Difference %
10	1	2.0%	2.0%	1.2%

Table 7.1: Difference between calculated and measured KCl recovery.

Figure 7.4 shows a contour map for the insoluble slimes recovery when using a cationic isodecyloxypropylamine as insoluble slimes collector at dosages ranging from 1 g/t to 7 g/t, and a non-ionic polyacrylamide as insoluble slimes flocculants at dosages ranging from 10 to 70 g/t. Equation (7.2) describes insoluble slimes recovery for the series of experiments using the collector and flocculant A combination.

$$y = 51.0314 + 0.3492x_1 + 1.6769x_2 - 0.0027x_1^2 - 0.1341x_2^2 - 0.0089x_1x_2 \quad (7.2)$$

Figure 7.4: Response contour for insoluble slimes recovery as a function of collector and non-ionic flocculant dosages. The dots in the graph represent the experimental points.

Flocculant A g/t	Collector g/t	calculated WI recovery	measured WI recovery	Difference %
10	1	55.7%	55.5%	0.4%

Table 7.2: Difference between calculated and measured insoluble slimes recovery.

The response surfaces show that the optimum consumption reaches a plateau at a collector dosage of 4.4 g/t and a flocculant dosage of 56 g/t. For these values the model predicts the insoluble slimes recovery of 64.5%. The accuracy of the model was examined by carrying out an additional test at a specified reagent dosages (Table 7.2) and by comparing the experimental recovery value with that predicted by the model. As seen, the percentage difference between these two values is 0.4%.

7.3   Anionic flocculant- Optimization

Figure 7.5 shows a contour map for the KCl recovery when using a cationic isodecyloxypropylamine as insoluble slimes collector at dosages ranging from 1 g/t to 7 g/t, and an anionic polyacrylamide with a density charge of 10% (Flocculant C) as insoluble slimes flocculants at dosages ranging from 10 to 70 g/t.

The polynomial model for KCl recovery for this series of tests was found to be :

$$y = 2.1040 - 0.0043x_1 + 0.1318x_2 + 0.0001x_1^2 + 0.0439x_2^2 + 0.0017x_1x_2 \quad (7.3)$$

The response surfaces do not show a clear plateau. As in Figure 7.3, the model produced an intersection of the axis of the ellipse in the domain of the negative numbers. The accuracy of the model that generated the contour map is shown in Table 7.3.

As in Figure 7.3, probably a combination of entrainment and true flotation is causing the KCl particles to float. A NaCl/KCl ratio of 0.9 in the slimes concentrate leads one to believe that some sort of combination of entrainment/flotation occurs.
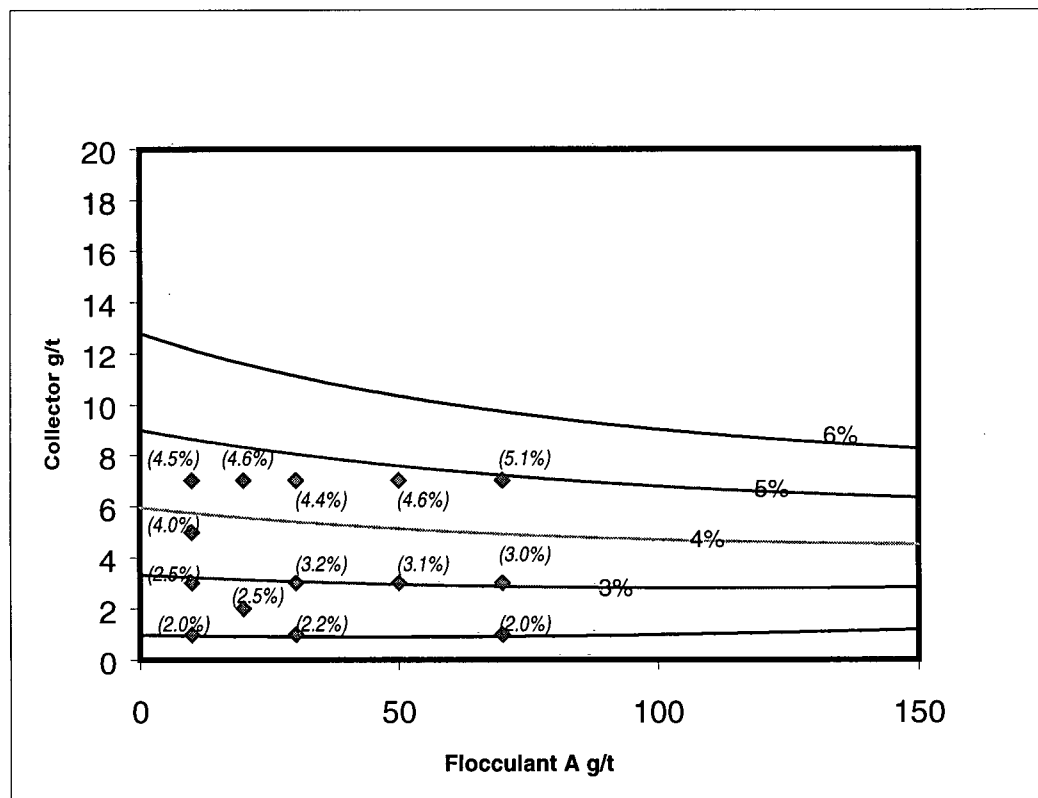

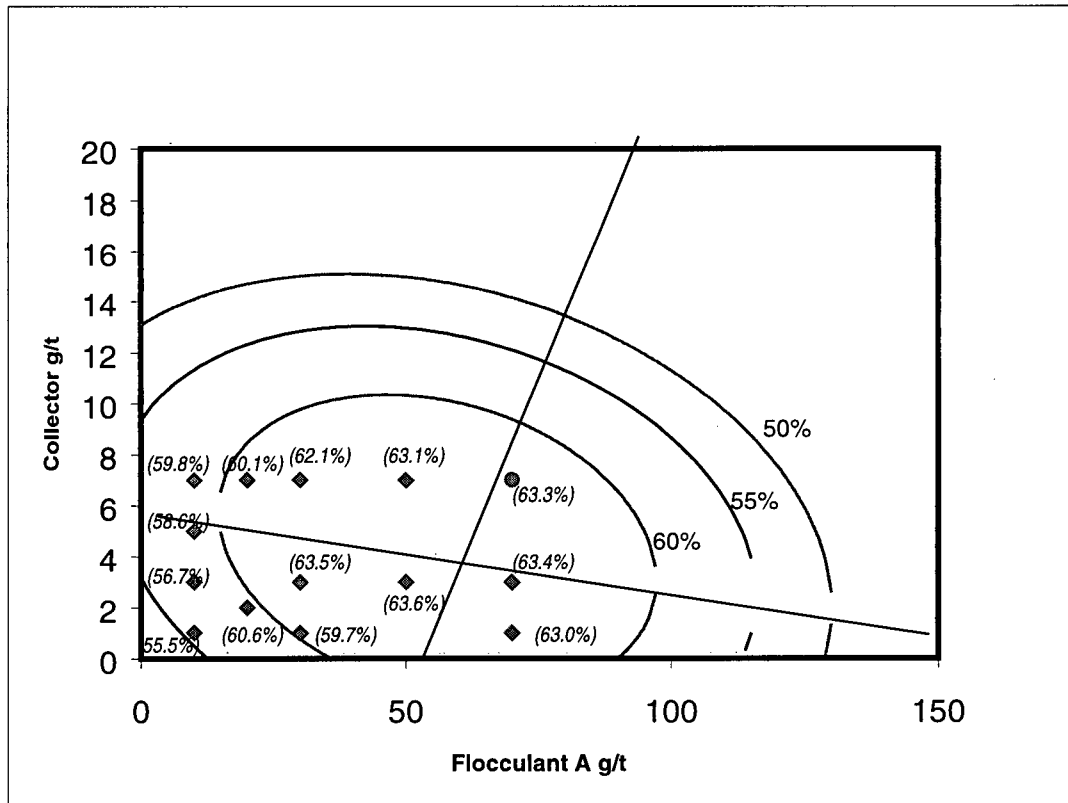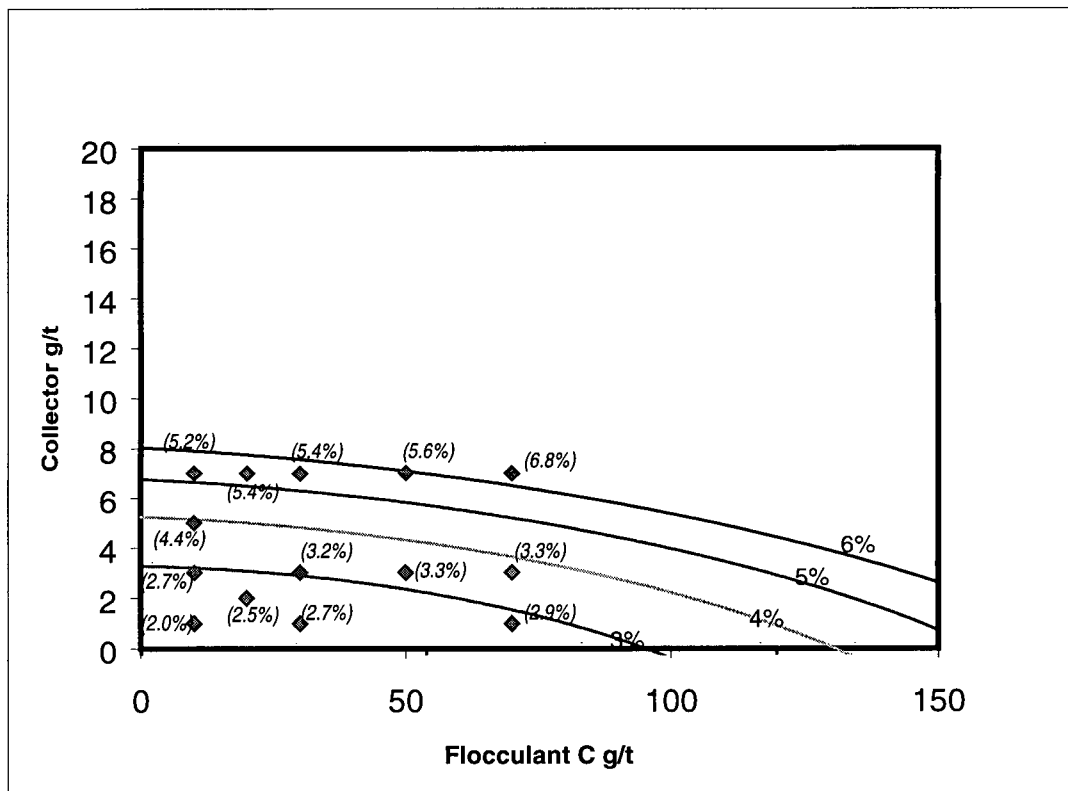**Figure 7.5:** Response contour of KCl recovery as a function of collector and anionic flocculant dosages. The dots in the graph represent the experimental points.

Flocculant C g/t	Collector g/t	calculated KCl recovery	measured KCl recovery	Difference %
10	1	2.3%	2.0%	11.8%

Table 7.3: Difference between calculated and measured KCl recovery.

Figure 7.6 shows a contour map for the insoluble slimes recovery when using a cationic isodecyloxypropylamine as insoluble slimes collector at dosages ranging from 1 g/t to 7 g/t, and a anionic polyacrylamide with a charge density of 10% (Flocculant C) as insoluble slimes flocculants at dosages from 10 to 70 g/t. The polynomial model for insoluble slimes recovery for this series of tests was found to be :

$$y = 51.3718 + 0.4139x_1 + 2.4466x_2 - 0.0032x_1{}^2 - 0.1857x_2{}^2 - 0.0086x_1x_2 \quad (7.4)$$

The response surfaces show that the optimum consumption reaches a plateau at a collector dosage of 5.2 g/t and a flocculant dosage of 57 g/t. For these values the model predicts a peak insoluble slimes recovery of 69.6%, which is higher that the value obtained for the non-ionic flocculant (Flocculant A). The accuracy of the model used to generate the contour map was evaluated by carrying out additional tests at a specified reagent dosage (Table 7.4). As can be seen, the agreement between the experimental and predicted values is fairly good.
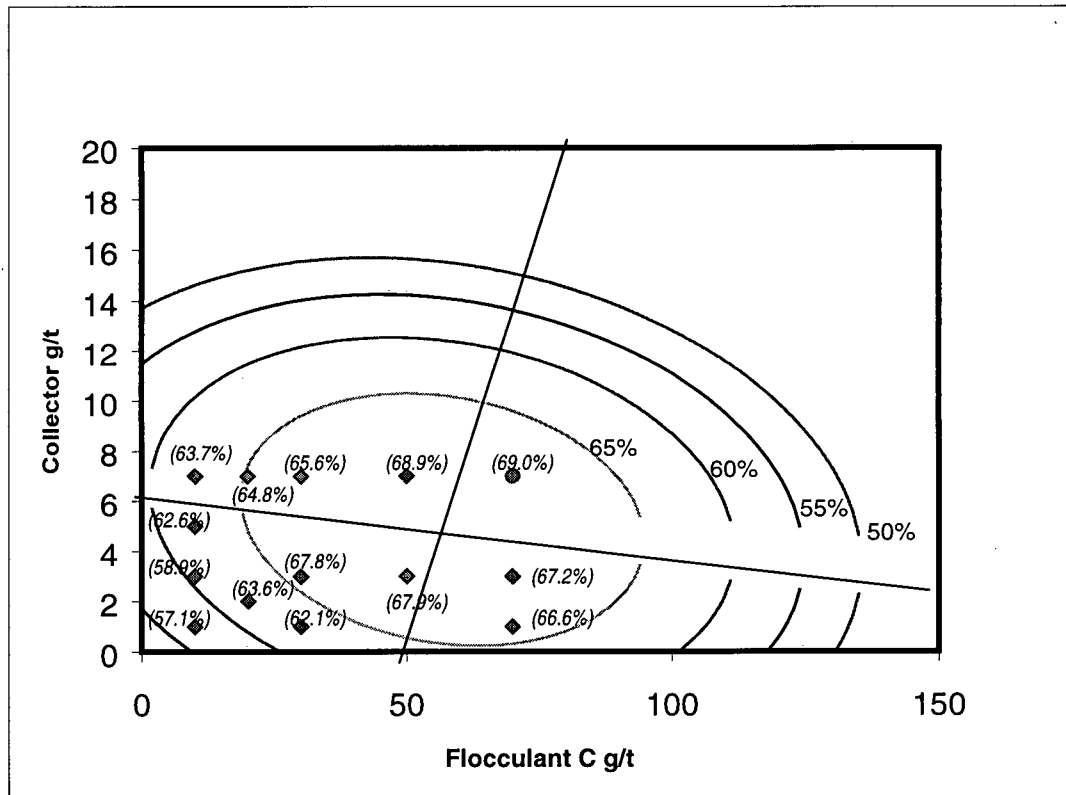
Figure 7.6: Response contour for recover of insoluble slimes as a function of the dosages of collector and flocculant C. The dots in the graph represent the experimental points.

Flocculant C g/t	Collector g/t	calculated WI recovery	measured WI recovery	Difference %
10	1	57.4%	57.1%	0.5%

Table 7.4: Difference between calculated and measured insoluble slimes recovery.

When assayed, the concentrates of insoluble slimes showed a NaCl/KCl ratio of 0.8 to 1.0. When compared with the NaCl/KCl ratio of 1.4 in the feed, these results suggest that the KCl reported to the insolubles concentrate by a combination of true flotation and entrainment mechanisms.

7.4 Collectorless Flotation of Slimes

Finally, a few tests were conducted using a constant flocculant dosage of 12.5 g/t without collector, and in another series 10 g/t of frother Dowfroth P along with 10 g/t flotation oil Esso 904.
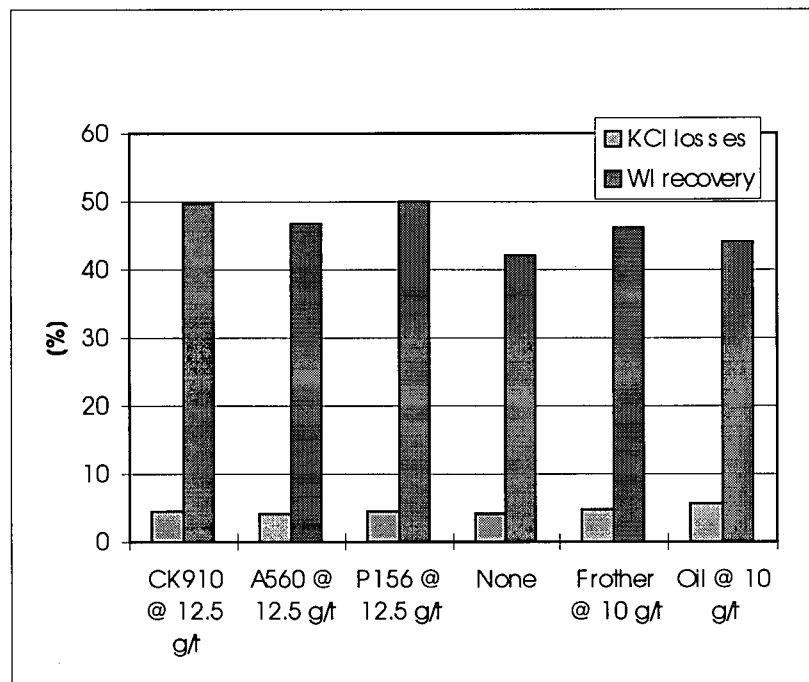
Figure 7.7: Tests of collectorless flotation of insolubles at a fixed Percol 351 dosage of 12.5 g/t.

All tests were performed at a pulp density of 25 % solids. The results of these tests are shown on Figure 7.6 and seem to indicate that there is no apparent need for the use of a collector - flocculant combination in order to float the insolubles out of the sylvite ore. Whether a collector is used, or a frother, or nothing at all, the flocculant seems to be able to achieve an acceptable insoluble recovery while the KCl losses remain almost constant.

8.0 DISCUSSION OF THE RESULTS

8.1 Depression of Slimes

Almost all the published literature on potash flotation refers to the insoluble minerals as "collector robbers", and increased collector usage is mentioned as the consequence of the presence of slimes in potash flotation circuits. The real effect of the presence of slimes in potash flotation is far more complex than this, affecting not only the collector dosage, but also affecting directly and dramatically the frothing process.

The baseline tests at increasing collector dosages but without any modifier show that it is impossible to obtain a good KCl recovery or a good concentrate grade at any dosage (See Table 6.1). Once the collector dosage exceeds 500 g/t the froth becomes stabilized by the presence of slimes and excess collector. Such a froth is not only very difficult to handle, but also loses all its ability for any secondary upgrading that is characteristic of a "healthy" flotation froth.

Mill operators and researchers have long been aware that the presence of fine solids in the froth greatly alters its characteristics. Fine particles in particular have been found to produce overstable froths, for example in flotation of phosphates, leading to severe operating difficulties.

Based on the test program developed here, it is apparent that the control of insoluble minerals in potash flotation should be focused on how to avoid stabilization of the froth by slimy particles.

Modifiers like carboxymethyl celluloses and guar gum exhibit good performance as slimes depressants. The amount of insoluble minerals reporting to the concentrate was kept almost always below 30 % in their presence. Modifier dosages of 200 - 300 g/t, although seemingly high, produced very selective sylvite separations.

Natural modifiers are a good choice because of their fast adsorption on clay particles (measured in terms of conditioning time), their biodegradability and their relatively low price.

The carboxymethyl celluloses most commonly used in Saskatchewan potash plants are Finnfix 300 supplied by Rantec Corp., and Depramin 96, supplied by Hollimex. Depramin 96 is a carboxymethyl cellulose with similar molecular weight and degree of substitution as Finnfix 300.

The flotation tests with the use of carboxymethyl celluloses of various molecular weights revealed that they are very effective insoluble slimes depressants for the type of potash ore used in the testing program (Figures 6.3 and 6.4); the obtained KCl recoveries were well above 90% and insoluble slimes recoveries between 20% and 30%.

The contour map for the recovery of KCl when using Finnfix 300 as insoluble slimes depressant (Figure 6.5) shows an optimum reagent dosage of about 134 g/t of Finnfix 300 and 103 g/t of collector. The KCl recovery for the optimum dosage is about 98%. Figure 6.8 shows the superimposed response contour maps of KCl and insoluble slimes recoveries as a function of collector and Finnfix 300 dosages. It can be seen that in the

domain of optimum KCl recovery, the expected insoluble slimes recoveries is about 25-30%, which is a fairly high value.

ACZ carboxymethyl cellulose characterized by a low molecular weight and a low degree of substitution, was slightly more selective than Finnfix 300, as it produced insoluble slimes recovery two percentage points below that of Finnfix 300 consistently at the various dosages tested (Figure 6.4).

Figure 6.9 shows the response contour map for the recovery of KCl as a function of collector and CMC ACZ dosage. The optimum dosage, for a KCl recovery of 98% is 207 g/t ACZ and 84 g/t collector. Figure 6.10 shows a recovery of insoluble slimes of about 25% coincidental with the domain of optimum KCl recovery. This is consistent with Figure 6.4 showing that CMC ACZ is a slightly more selective insoluble slimes depressant than Finnfix 300, although at a much higher dosage. This higher CMC ACZ depressant dosage is compensated by a much lower collector dosage required to achieve optimum KCl recoveries.

As the carboxymethyl cellulose of low molecular weight and low degree of substitution ACZ is also cheaper than Finnfix 300, there is potential for improvement in the Saskatchewan potash plants that are currently using Finnfix. This statement should be confirmed by a full scale plant tests at one of the potash plants.

Guar gum gave slightly lower sylvite recoveries than carboxymethyl celluloses, between 96 and 98 % (Figure 6.9). In general guar gum allowed recoveries of insoluble slimes between 5 and 10 %; whilst most of the CMC's allowed insoluble slimes

recoveries below 25 %. It is important to note that the drop in the recovery of insoluble slimes from 30 to 5 %, for the ore used in the testing program, means a decrease from 1.5 % to 0.8 % in the insoluble slimes assay of the concentrate.

The contour map for the recovery of KCl when guar gum was used as insoluble slimes depressant (Figure 6.14) reveals that the optimum reagent dosages are about 157 g/t of guar gum and 102 g/t of collector, for a KCl recovery of about 98%. Figure 6.15 shows the response contour map of insoluble slimes recovery with an optimum reagent dosage of 293 g/t of guar gum and 57 g/t of collector producing an insoluble slimes recovery of 5%. This implies a superb depressing performance for guar gum, when compared with the other reagents tested. The superimposed contour maps for KCl and insoluble slimes recoveries (Figure 6.16) shows that for the domain of optimum KCl recovery the expected insoluble slimes recovery is about 6-8%.

Polyacrylamides in general produced fairly high sylvite recoveries, sometimes exceeding 95 % (Figure 6.13) but recovery of insoluble slimes was also high in the range from 30 and 60 % (Figure 6.14), at the same modifier dosages.

In general, the lower the dosage of the polyacrylamide, the higher the recovery of insoluble slimes, and the lower the recovery of KCl. The series of tests using Percol 351 as a modifier show that at low dosages of that reagent the slimes float instead of the sylvite. The little amount of sylvite that is floated is mostly composed of very fine particles. An increase in the modifier dosage produces a steady reduction in the recovery of insoluble slimes, associated with a dramatic increase in sylvite recovery (Figures 6.15 and 6.16).

Figure 6.21 shows the response contour map for KC recovery when using the high molecular weight polyacrylamide Percol 351 as insoluble slimes depressant. Figure 6.21 shows a fairly extended domain of optimum KCl recovery (98%) with optimum reagent dosages of about 146 g/t of Percol 351 and 179 g/t of collector, which are both fairly high dosages.

The contour map for the insoluble slimes recovery (Figure 6.22) shows a domain of maximum insoluble slimes recovery with optimum dosages of 103 g/t of depressant and 93 g/t of collector. The superimposed response surface contour maps (Figure 6.23) shows that in the domain of the optimum KCl recovery the expected insoluble slimes recovery is in excess of 50%. This seems to indicate that although the polyacrylamide shows fairly good slimes blinder properties, its depressing action is far from acceptable.

The following Figures 8.1 and 8.2 consist of some of the results of the tests used to develop the statistical models, re-arranged in such a way to allow a direct comparison between the three main depressant types under study.

Figure 8.1 shows KCl recovery when using a carboxymethyl cellulose (Finnfix 300), a guar gum (Rantec 4000) and a non-ionic polyacrylamide (Percol 351) as modifier, at a constant collector dosage of 100 g/t.

It can be seen from Figure 8.1 that KCl recovery remains very high, well over 95% for all three modifiers, at dosages that range from 50 g/t to 250 g/t. This would indicate that all three polymers are efficient blinders for insoluble slimes.
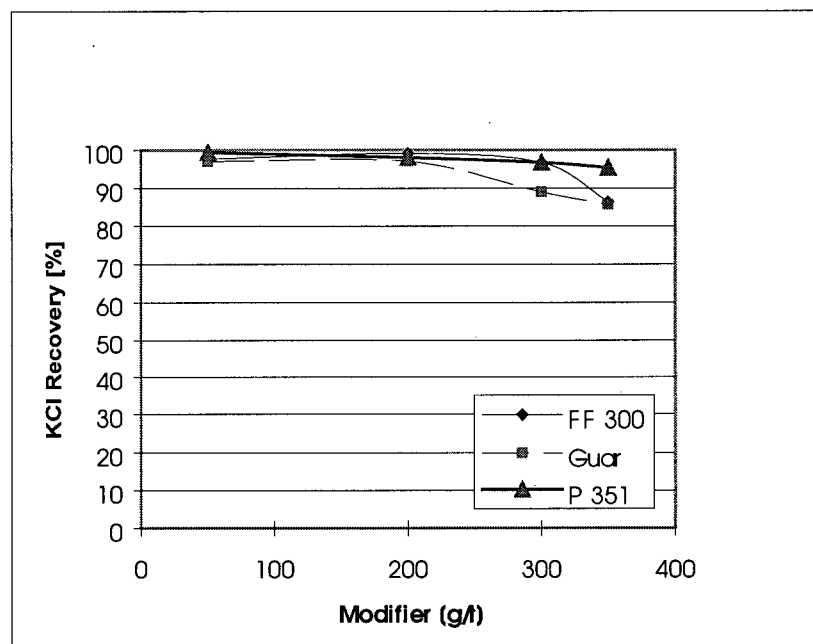
Figure 8.1: KCl recovery using various modifiers at a collector dosage of 100 g/t.

KCl recoveries when using guar gum as modifier are slightly lower than when using carboxymethyl cellulose or the non-ionic polyacrylamide, although the difference between them is no more than two percentage points.

KCl recoveries start to decrease at modifier dosages in excess of 250 g/t. This effect is more pronounced in the case of guar gum (Figure 8.1). This may be an indication of the inconvenience of overdosing the modifier.

Figure 8.2 shows insoluble slimes recovery when using a carboxymethyl cellulose (Finnfix 300), a guar gum (Rantec 4000) and a non-ionic polyacrylamide (Percol 351) as modifier, at a constant collector dosage of 100 g/t.
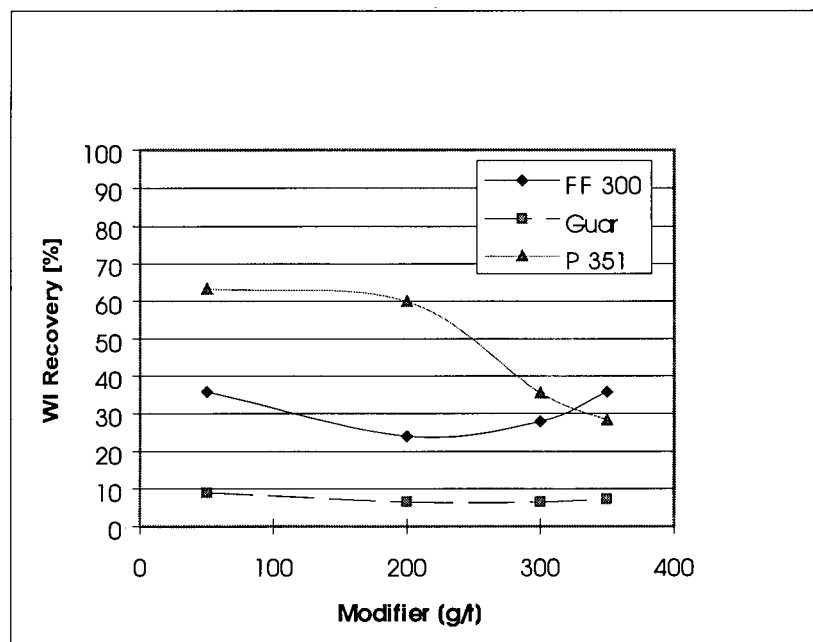
Figure 8.2: WI recovery using various modifiers at a collector dosage of 100 g/t.

Figure 8.2 allows for a clear differentiation of the performance of the three polymers as slimes depressants. The non-ionic polyacrylamide can hardly be considered, based on Figure 8.2, as an insoluble slimes "depressant" since considerably more than half of the insoluble slimes present in the feed are reporting to the concentrate at polyacrylamide dosages ranging from 50 to 200 g/t. Only when the polyacrylamide dosage exceeds 250 g/t, does the modifier delivers a reasonably low insoluble slimes recovery. As polyacrylamides are usually very expensive reagents, this high dosage requirement makes them unable to compete with the cheaper and more effective carboxymethyl cellulose and guar gum.

106

It is clear from Figure 8.2, which is consistent with the statistical models (see Figure 6.14) that guar gum is a more effective insoluble slimes depressant than the carboxymethyl cellulose. The depressing action of guar gum is strong even at low dosages (50 g/t), however at higher dosages no significant increase in depression is observed. Carboxymethyl cellulose shows the maximum depression at a dosage around 200 g/t, which is considered fairly high. At its optimum point, the amount of insoluble slimes reporting to the concentrate when carboxymethyl cellulose is used as a modifier is still more than twice the insoluble slimes recovery using guar gum (and the carboxymethyl cellulose dosage is four times higher).

The results shown in Figure 8.2 are consistent with those from Shortridge et al., who investigated the effect of chemical composition of polysaccharide depressants on the flotation of talc. Shortridge et al., concluded that the low depressing action of the carboxymethyl cellulose when compared to that of guar gum could be indicative of a substantially higher adsorption of guar onto the talc particles. Shortridge et al. also observed that guars were much more effective depressants than CMC at higher ionic strengths of the system.

As carboxymethyl celluloses are usually more expensive than guar gum, Figures 8.1 and 8.2 show that a serious evaluation has to be performed when deciding which modifier to use at an industrial scale, in order to determine whether the extra KCl recovery is enough to pay off for the use of a more expensive depressant at a higher dosage.

8.2 Flocculation-Flotation of Slimes

Figure 7.2 shows that the anionic flocculants (Flocculants B and C) seem to produce a higher insoluble slimes recovery than the non-ionic flocculant (Flocculant A), although the difference is very small. It is also important to note that the recovery of KCl to the concentrate of insoluble slimes increases with the use of anionic flocculants as well (Figure 7.1). It can be observed from the Figures 7.1 and 7.2 that the difference in performance between Flocculant B (charge density = 3%) and Flocculant C (charge density = 10%) is negligible.

Figures 7.3 and 7.5, showing KCl recovery to the slimes concentrate, do not seem to show a plateau for an optimum reagent dosage probably because a combination of entrainment and true flotation is causing the KCl particles to float. The figures indicate though, based on the contour maps, that a higher dosage of the collector results in a higher amount of KCl lost to the insoluble slimes concentrate. This would explain why in the flocculation-flotation of slimes it is so important to maintain good control of the collector dosage and a good control of the brine recovery (linked to the level of the pulp inside the flotation cells) in order to minimize KCl losses.

Based on the model output depicted in Figures 7.4 and 7.6 for slimes recovery in the slimes concentrate, the anionic flocculant increases insoluble slimes recovery by approximate 5% over the non-ionic flocculant (both used at almost the same dosage).

The problem observed when strongly anionic flocculants are used is that especially at high dosages they produce a very tight froth. Such a froth tends to stick to the side

walls of the flotation cell and to the impeller shaft making it increasingly difficult to remove out of the cell. This phenomenon was observed during a full-scale plant test at Agrium using Percol E 10 (a high molecular weight, anionic polyacrylamide).

Finally, it can be seen from Figure 7.7 that the flocculation-flotation of insoluble minerals is a flocculant dependent process. The addition of an "insolubles collector" or a frother, or no reagent at all merely affects the kinetics of the process. It is likely that the reagents like ether amine (Procol CK 910) used in this test program act as a froth regulator instead of a true collector for insoluble slimes.

8.3   Conclusions

1. Modifiers like carboxymethyl celluloses and guar gum are good slime depressants, producing slime recoveries below 30 %.

2. ACZ carboxymethyl cellulose characterized by a low molecular weight and a low degree of substitution, is slightly more selective than Finnfix 300.

3. Guar gum provides slightly lower sylvite recoveries than carboxymethyl celluloses, but it is very selective (insoluble slime recoveries between 5 and 10 %).

4. Polyacrylamides produce fairly high sylvite recoveries, but recovery of insoluble slimes is also high (from 30 to 60 %, when they are used).

5. Anionic polyacrylamide flocculants produce a higher insoluble slimes recovery than non-ionic flocculants, although the difference is very small.

6. Anionic flocculants when used at high dosages, produce a very tight froth which tends to stick to the side walls of the flotation cell and to the impeller shaft making it increasingly difficult to the froth remove out of the cell.

8.4   Recommendations

From the results of this work it is my understanding that little progress would be made without extensive research work on adsorption of blinders on the slimes minerals. It is my impression that guar gum might adsorb differently than CMC on the slime minerals under the high ionic strength conditions of the system and that probably explains the differences in the slime recovery between Figures 6.7 and 6.15.

Also, I think it would be very important to develop viscosity determinations of the modifier solutions under high ionic strength conditions. I think a detailed analytical interpretation of these two combined phenomena, viscosity and adsorption hold the key to understand the mechanism responsible for depressing slimes in saturated brine. For example, differences in viscosity between CMC and Guar in brine might probably be an indication of coiling of the molecules that would eventually affect the way the polymer is adsorbed on the slime particles. These are the areas that would require further work in order to obtain a better understanding of the way the polymers work as depressants.

Regarding to flocculation-flotation of slimes, further work should be carried out to improve our understanding of the mechanism of KCl flotation with the concentrate of insoluble slimes concentrate. The results of Chapter 7 seem to indicate that there is a

combined effect of entrainment and true flotation causing fine KCl particles to report to the insoluble slimes concentrate. A more thorough understanding of this would help to improve the efficiency of flocculation-flotation as a desliming technique by minimizing KCl loses to the slime concentrate.

9.0 REFERENCES

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117

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APPENDIX A: Brine Correction Calculation Spreadsheet.

Test #: __193__
Feed: - 20-mesh Tyler Ore
Desliming: Mechanical
Modifier: 200 gpt Superfloc 362
Collector: 50 gpt C-18
Frother: 50 gpt Dowfroth 319
Extender Oil: - gpt
Air: 7.5 lpm
Impeller Speed: 1500 rpm

	Sample Weight	Calculated				Distribution		Recoveries	
		KCl	NaCl	W.I.	Total	KCl	W.I.	KCl	W.I.
Feed									
Wet	1000.0	40.2	55.2	4.6	100.0				
Dry	1000.0	402.0	552.0	46.0	1000.0				
Brine	0.0	0.0	0.0		0.0				
Debrined Solids		402.0	552.0	46.0	1000.0	100.0%	100.0%		
Debrined Solids, %		40.2%	55.2%	4.6%	100.0%				
Concentrate									
Wet	475.5	84.6	12.2	2.4	99.2				
Dry	423.0	357.9	51.6	10.3	423.0				
Brine	77.2	7.8	16.2		24.1			**Float Recoveries**	
Debrined Solids	398.9	350.0	35.4	10.3	398.9	87.1%	22.4%	87.1%	22.4%
Debrined Solids, %		87.7%	8.9%	2.6%	100.0%				
Concentrate Grade in %K2	**55.3%**								
Tails									
Wet	583.5	8.0	90.1	1.9					
Dry	533.3	42.6	480.3	10.3	533.3				
Brine	73.8	7.5	15.5		23.0				
Debrined Solids	510.3	35.1	464.8	10.3	510.3	8.7%	22.5%		
Debrined Solids, %		6.9%	91.1%	2.0%	100.0%				
Slimes									
Wet	81.3	30.6	36.3	33.1					
Dry	73.7	22.5	26.7	24.4	73.7				
Brine	11.3	1.1	2.4		3.5				
Debrined Solids	70.1	21.4	24.4	24.4	70.1	5.3%	53.0%		
Debrined Solids, %		30.5%	34.8%	34.7%	100.0%				
Balance		-1.1%	5.0%	2.1%	2.1%			**Net Recoveries**	
Assay Grade		406.5	524.6	45.0	979.4			86.1%	22.9%
Assay Grade, %		41.5%	53.6%	4.6%	100.0%				
Comments:									

APPENDIX B: List of Reagents Tested

Part I: Depressants

Guar

Name	Supplier	Active Content	MW	DS
KP 4000	Rantec Corp.			

CMC

Name	Supplier	Active Content	MW	DS
ACZ	S&G Resources	65%	90,000	0.55
ACZ HVSG	S&G Resources	65%	100-200,000	0.65
ACZ HVG	S&G Resources	65%	200-300,000	0.65
PE 23EX	S&G Resources	96%	700-800,000	0.75
Finnfix 300	Rantec Corp.	98%	200-300,000	0.82
Depramin 96	Hollimex	65%	200-300,000	0.85
P 30 Super	Amtex S.A.	65%	250,000	0.50

CMStarch

Name	Supplier	Active Content	MW	DS
Starchem 96	Hollimex	96%	200-300,000	0.55

Carboxymethyl Isopropyl Guar

Name	Supplier	Active Content	MW	DS
Galactasol 650	Hollimex	96%	200-300,000	0.65

Polyacrylamides

Name	Supplier	Ionicity	MW	% charge
Percol E 10	CIBA S.C.	anionic	15-20 M	5
Percol 351	CIBA S.C.	non-ionic	10-15 M	0
Percol 156	CIBA S.C.	anionic	10-15 M	40
Superfloc 362	CYTEC	cationic	10 M	40
AccoPhos 950	CYTEC	anionic	1 M	5

Part II: Flotation Desliming

Flocculants

Name	MW	Charge Density
CM 309	17-21 M	0
Vanfloc 1809	22 M	0
Vanfloc 1530	17-21 M	3
Vanfloc 1830	22 M	3
Vanfloc 1710	17-21 M	10