RECOVERY OF PYROCHLORE FROM SLIMES DISCARDED AT NIOBEC BY COLUMN FLOTATION

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Recovery of Pyrochlore from Slimes Discarded by Column Flotation

To my parents

ABSTRACT

The Niobec concentrator recovers niobium minerals (principally pyrochlore) from carbonate and silicate gangue. In the process some 15% of the Nb is rejected in a slimes (-10 μm) product. Recovery of pyrochlore from the slimes was studied. There were two main objectives: to develop a reagent scheme for the selective flotation of pyrochlore and to determine if flotation columns were better suited to slimes processing than mechanical cells. The study was performed using plant water which contained about 200 ppm Ca, 100 ppm Mg and variable quantities of organics as the main contaminants. Selective pyrochlore collectors and selective silicate depressants were evaluated. Four possible routes to float pyrochlore selectively were found. All routes required prior carbonate flotation. Carbonate flotation was attempted using emulsified fatty acids and sodium silicate dispersant (as conducted at Niobec) in a flotation column. However, pyrochlore also floated because of inadequate slimes dispersion. Therefore, new dispersants were tested, which proved effective only at high concentration, because of reaction with Ca and Mg cations in the water. High dispersant concentration caused carbonate depression.

An additional problem in the flotation column was bubble coalescence and collapse of the frotn. This was caused by the fatty acids, and enhanced by the high dispersant concentration. This problem had to be solved in order to continue flotation testing. A novel method of quantifying coalescence using the change in gas holdup in the column was developed. A stable froth could be made at high fatty acid emulsifier concentration, but this promoted unselective flotation. Only by working at high percent solids (10% vs 1% by weight) could sufficient emulsifier be introduced into solution without exceeding the dosage (per unit mass of solids) above which flotation is

unselective. For such high percent solids work must be conducted at plant site. Preliminary plant column work was encouraging.

Since column flotation of carbonates from the slimes was not satisfactory, as a compromise, column plant tests were conducted at Niobec on the fine carbonate stream. Tests were conducted in parallel with the conventional circuit cells. Column flotation gave superior metallurgy. Extensive testwork on other streams of very fine feeds (80% passing 8 to 23 μm) confirmed that column flotation is ideal for selective flotation of fines primarily due to reduced gangue entrainment.

RÉSUMÉ

Le concentrateur de Niobec récupère les minéraux de Niobium (principalement le pyrochlore) d'une gangue formée de carbonates et de silicates. Toutefois, 15% du Nb est perdu avec les schlammes (-10 μm). Ce travail porte sur la récupération du pyrochlore de ces schlammes. L'étude avait deux buts: choisir les réactifs et leur séquence d'addition afin de promouvoir une flottation sélective du pyrochlore et déterminer si les colonnes de flottation étaient mieux appropiées (que les cellules mecanique) au traitement des schalmmes. L'eau utilisée lors de ces tests provenait de l'usine et contenait principalement 200 ppm de Ca, 100 ppm de Mg et des quantités variables de matières organiques. On a évalué le potentiel des collecteurs sélectifs au pyrochlore ainsi que des déprimants sélectifs aux silicates. Quatre procédés de flottation sélective au pyrochlore nous sont apparus possibles; tous requièrent une pré-flottation des carbonates. On a flotté les carbonates dans une colonne avec les réactifs employés à Niobec, des émulsions d'acides gras, et du silicate de sodium comme dispersant. Le pyrochlore flottait également par suite d'une dispersion inadéquate des schlammes. Nous avons donc testé d'autres dispersants et avons trouvé qu'une bonne dispersion n'était possible qu'à haut dosage de dispersant, ce dernier étant neutralisé à basse concentration, en réagissant avec les cations de Ca et de Mg contenus dans l'eau. Toutefois, un dosage trop élevé de dispersant déprime aussi les carbonates.

En presence d'acides gras, et à haut dosage de dispersant, on a pu

observer deux phénomènes qui nuisent considérablement a l'efficacité de flottation: la coalescence des bulles (dans la zone de récupération) et l'affaissement de l'écume (zone de nettoyage).

Pour étudier la coalescence des bulles causée par les acides gras et la concentration élevée de dispersant, on a mis au point une nouvelle méthode pour quantifier la coalescence en mesurant la fraction gazeuse dans la zone de récupération de la colonne. On a résolu les problèmes de coalescence des bulles et de stabilité de l'écume en émulsionnant l'acide gras avec du lannagol; toutefois, la flottation perd alors toute sélectivité. Seule une hausse du pourcentage de solides (10% vs 1%) nous a permis d'introduire assez d'émulsifiant en solution pour stabiliser l'écume sans toutefois excéder le dosage d'émulsifiant (par masse de solides) causant la perte de sélectivité. Pour une telle concentration en solides, le travail doit être fait au concentrateur. Les travaux preliminaires entrepris en ce sens semblent encourageants.

Puisque la flottation par colonne des carbonates des schlammes n'était pas satisfaisante, on a dû par compromis, effectuer des tests au moulin de Niobec sur leur courant de carbonates fins. Ces tests ont été conduits parallèlement à un circuit de cellules mécaniques. Les colonnes donnèrent des résultats supérieurs. D'autres tests sur d'autres circuits de matériel très fin (80% passant 8 a 23 µm) ont démontré que la colonne de flottation est idéale pour la flottation des fines à cause de la reduction de l'entrainement de la gangue.

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APPENDIX 1. Reports 3, 4 and 5 of Series Prepared for the Centre de Recherches Minerals, Quebec; and manuscript entitled; Effects of the water chemistry on pyroclore and silicates flotation (to be published in Minerals Engineering)

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THESIS STRUCTURE

This thesis is presented in part by four original manuscripts which are either published or in press. Common abstracts introduction and conclusions are given. The first chapter gives a full background and the other four present manuscripts. The chapters containing manuscripts give an introduction and additional material is also provided in detail. (Complementary information is provided in appendices). The references cited are all presented at the end of the thesis. A common reference format is used.

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STATEMENT ON CONTRIBUTION OF CO-AUTHORS AND OTHERS

Note: This statement is written in conformity with the requirements of The Faculty of Graduate Studies and Research.

The Author wishes to thank Professor Finch for selecting the Ph.D. topic: Recovery of Pyrochlore from Slimes Discarded at Niobec by Column Flotation.

Manuscript 1 (Chapter 2): Effects of the Type of Water on the Selective Flotation of Pyrochlore from Niobec. The author should be credited for all the work performed in this manuscript: e.g. sample preparation, sample characterization, the studies to select new pyrochlore flotation reagents, the electrokinetic study, the microflotation study and the evaluation of the effects of water quality in the selective flotation of pyrochlore, but he is indebted to his supervisors, Profs. J.A. Finch and A.R. Laplante for the discussion, the English corrections during writing and their advice on the presentation of results. The author also wants to recognize: a) Mr. R. Biss (Niobec) for helpful discussion about the problems in the pyrochlore and carbonate flotation circuits at Niobec; b) Dr. S.R. Rao (McGill) for helpful discussion on organic and surface chemistry matters .

Manuscript 2 (Chapter 3): Dispersion Study on Slimes from Niobec. The author should be credited for all the work performed in this manuscript: e.g. sample preparation, flotation tests and the new technique to evaluate dispersion, but he is indebted to his supervisors, Profs. J.A. Finch and A.R. Laplante for the discussion, the English corrections during writing and their advice on the presentation of results. The author also wants to recognize, Dr. S.R. Rao (McGill) for helpful discussion on organic and surface chemistry matters .

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CHAPTER 1

BACKGROUND

1.1 INTRODUCTION

The first part of this chapter presents a review of the main Nb-minerals, especially pyrochlore, their composition and the most common methods of recovery, to end with a description of the Niobec ore and concentration plant. In the second part, the flotation of fine particles is addressed, emphasising the main difficulties that presently limit their treatment and introducing column flotation as a potential technique for the treatment of fines. The objectives of this research are stated and the method of attack outlined. Finally, an overview of the chapter contents is given.

1.2 MAIN Nb-MINERALS AND THEIR PROCESSING

The element niobium was first discovered in 1802 and was originally named columbium. It was subsequently rediscovered in 1844 and was named niobium. Later, it was realized that columbium and niobium were the same element; however, both names are still used. In this work the name niobium will be used because of its wider acceptance in the scientific literature.

1.2.1 Mineralogy

Niobium is an essential constituent of approximately fifteen mineral groups, mostly oxides and hydroxides, some silicates and one borate (Perrault and Manker, 1981). From the economic point of view three groups are most important and supply practically all of the niobium used in the industrialized world (Biss, 1984a). These three groups are: 1) pyrochlore, 2) columbite-tantalite, and 3) perovskite-latrappite.

1) The pyrochlore group: The pyrochlore group is the most important economic group. Pyrochlore group minerals have a general formula; $A_2B_2O_6F$. Where 'A' is mainly Na(1+) and Ca(2+); 'B' is mainly Nb(5+), Ti(4+) or Ta(5+). Depending on the nature and abundance of the B cation, the group is divided into subgroups: pyrochlore (containing mainly Nb), betafite (containing mainly Ti), and microlite (containing mainly Ta). (Perrault and Manker, 1981; Hogarth, 1977). The nature and abundance of the A cations is the second argument for distinguishing species (Hogarth, 1977), where Na and Ca are partially replaced by K, Sn, Ba, Y+ (La-Lu), Pb, Bi and U. Interestingly this list does not include Fe, although as will be seen, pyrochlore from Niobec does show Na replacement with Fe.

The crystal structure of the pyrochlore group minerals is cubic. The color is quite variable, ranging from white, beige, yellow, green, gray to black. Some other physical properties are: hardness, 5 to 5.5, density, 4 to 4.4, luster, resinous; and refractive index, 2.0 to 2.2. Thus, it is an impossible mineral to identify positively from its physical properties alone. The most common deposits of the group are found in the carbonatites and pegmatites derived from alkalic rocks. Common associate minerals in addition to the forming minerals (calcite and dolomite in carbonatites; nepheline, orthoclase and albite in alkalic rocks) are zircon, and apatite (Razvozzhaev and Khodyreva, 1965; Potekhina et al, 1967; Levinskii and Nikonova, 1971; Biss, 1982; Caronneau and Caron, 1965; Harris and Jackson, 1966). Pyrochlore tends to be brittle. This brittleness is partly due to radiation damage as most of the species contain some uranium or thorium or both.

2) The columbite-tantalite group. The group is represented by columbite, $FeNb_2O_6$ and tantalite. The columbite forms a pseudo-solid solution series with tantalite in naturally occurring specimens. This group provided the raw material for the first, modern, industrially important production of niobium. After the discoveries of large pyrochlore deposits its importance decreased. The minerals of the group are found essentially in pegmatites.

3) The perovskite-latrappite group. This group is only marginally important. It is composed of several minerals characterized by different degrees of replacement of the original perovskite, $CaTiO_3$ by niobium or by the other elements. The minerals of the group can be found in carbonatites, kimberlites, basalts and skarns.

1.2.2 Concentration Methods

The choice of the concentrating method depends on the nature of the gangue, and on the minerals to be recovered. The pyrochlore group of minerals respond mainly to the flotation process, the columbite-tantalite group to gravity concentration and to a lesser degree, flotation. The response to flotation depends on the degree of weathering of the ore, the variety of Nb-species, the associated silicate types and content, and the choice of flotation reagents. For example, during the course of weathering (Harris and Jackson, 1966) the pyrochlore can be altered, e.g. to a microcrystalline form which readily breaks up into slime-sized particles, increasing the difficulties to recover pyrochlore by flotation. If the flotation response of the Nb-species present are quite different, several Nb-collectors may be required (Biss, 1982). If the silicate content is high, direct flotation may be impossible (Gorlouskii et al, 1966; Raicevic and

Noblitt, 1979; Biss, 1984a).

All these factors determine whether or not a Nb-ore can be recovered by flotation. However, in all the cases quite complex flowsheets are required, always producing a low grade Nb_2O_5 concentrate (e.g. less than 20%). Niobec mines has been the sole producer that has succeeded on a commercial scale with a direct flotation process to recover the pyrochlore from carbonatites. Niobec concentrates are typically 55% Nb_2O_5 (from about 0.6% Nb_2O_5 ore).

1.3 THE NIOBEC CONCENTRATION PLANT

The Niobec mill (Les Services TMG Inc) is located in St Honore P.Q., near Chicoutimi, about 525 km North-East of Montreal. The mill processes 2100 tonne per day of dolomitic and calcitic carbonatite ore, containing 0.5-0.7% Nb_2O_5, present primarily as pyrochlore.

1.3.1 Description of the Ore

The ore composition (see Table 1.1) is complex, with the Nb-minerals representing only about 1% of the total mass. The most abundant contaminants are carbonates (mainly calcite and dolomite), silicates (mainly microcline, biotite and chlorite) and to a lesser extent apatite. The mineralization occurs in two distinct zones, zone 1 and zone 2. Zone 1 contains about 0.70% Nb_2O_5; the main Nb mineralization is pyrochlore with a higher proportion of high Fe pyrochlore and columbite. Zone 2 contains a lower grade, about 0.63% Nb_2O_5. The local names Na-pyrochlore and Fe-pyrochlore are used to distinguish the low and high Fe types. Typically Na-pyrochlore contains about 8.6% Na and about 1% Fe, and Fe-pyrochlore, about 4% Na and about 4% Fe. Na-pyrochlore is the more common type (Biss, 1982), and it is easier to

TABLE 1.1 MINERALOGICAL COMPOSITION OF NIOBEC ORE.

MINERAL	Wt. (%)	IEP	REFERENCE
CARBONATES	65		
DOLOMITE		7	HANNA AND SOMASUNDARAN, 1976
CALCITE		8.2	
ANKERITE			
SULPHIDES	1		
PYRITE		1.5	NEY, 1973
PYRRHOTITE			NEY, 1973
OXIDES			
PYROCHLORE AND COLUMBITE (10:1)	1	2.6	DUVAL, 1980
MAGNETITE, HEMATITE AND ILMENITE.	2	6	FUERSTENAU AND PALMER, 1976
APATITE	7	5	HANNA AND SOMASUNDARAN, 1976
ZIRCON	0.2	5.8	FUERSTENAU AND PALMER, 1976
SILICATES	21		
BIOTITE		1.5	FUERSTENAU AND RAGHAVAN, 1976
CHLORITE		4.5	
Na,K,FELDSPARS		1.8	

treat. The ratio between pyrochlore (Na+Fe types) and columbite in zone 2 is 10:1. Typical compositions (Perrault, 1978; Perrault and Manker, 1981) of Na-pyrochlore, Fe-pyrochlore and columbite are given in Table 1.2.

With the present reagent conditions at Niobec it is known that as the proportion of Fe-pyrochlore and columbite increases, the overall Nb_2O_5 recovery decreases. This relationship is shown in Figure 1.1 (Perrault, 1978). When no Fe-pyrochlore or columbite is present, recoveries as high as 77% can be obtained; when present, recoveries can be as low as 55%.

1.3.2 Flowsheet Description

Recovery of pyrochlore is performed by flotation using a very complex flowsheet (Biss, 1982; Biss and Duval, 1982; Biss, 1984a). Direct pyrochlore flotation has proved difficult due to the similar flotation characteristics of the pyrochlore and the two main contaminants, carbonates and silicates. In particular, the silicates present a major problem due to their similar electrokinetic properties. Table 1.1, includes the isoelectric point of charge (IEP) obtained from the literature. The IEP of pyrochlore is about 2.6, biotite about 1.5, chlorite about 4.5, and Na,K Feldspars (e.g. microcline) about 1.8. These values are quite close limiting selective flotation, if based on physical adsorption of collectors.

Figure 1.2 shows the main sections of the flowsheet. The process consists of ore reduction to 95% -208 μm (resulting in about 95% pyrochlore liberation), desliming, carbonate flotation, desliming again, magnetic separation, pyrochlore flotation, sulphide flotation, leaching of the remaining carbonate and apatite, and finally, another sulphide flotation step. Figure 1.3 gives more details on the carbonate flotation and desliming stages. Carbonate flotation is performed in two separate circuits, a fines

Table 1.2. TYPICAL COMPOSITIONS OF THE Nb-MINERALS AT NIOBEC

	Nb_2O_5	TiO_2	Na_2O	CaO	FeO	U_3O_8	ThO_2	TOTAL
Na-PYROCHLORE	68.95	2.72	8.61	15.17	1.08	0.16	0.98	97.62
Fe-PYROCHLORE	68.22	3.19	3.8	13.87	4.18	0.22	2.15	95.63
COLUMBITE	72.74 MnO=1.71	3.21	0.29	1.39	16.8	0.09	1.52	97.75

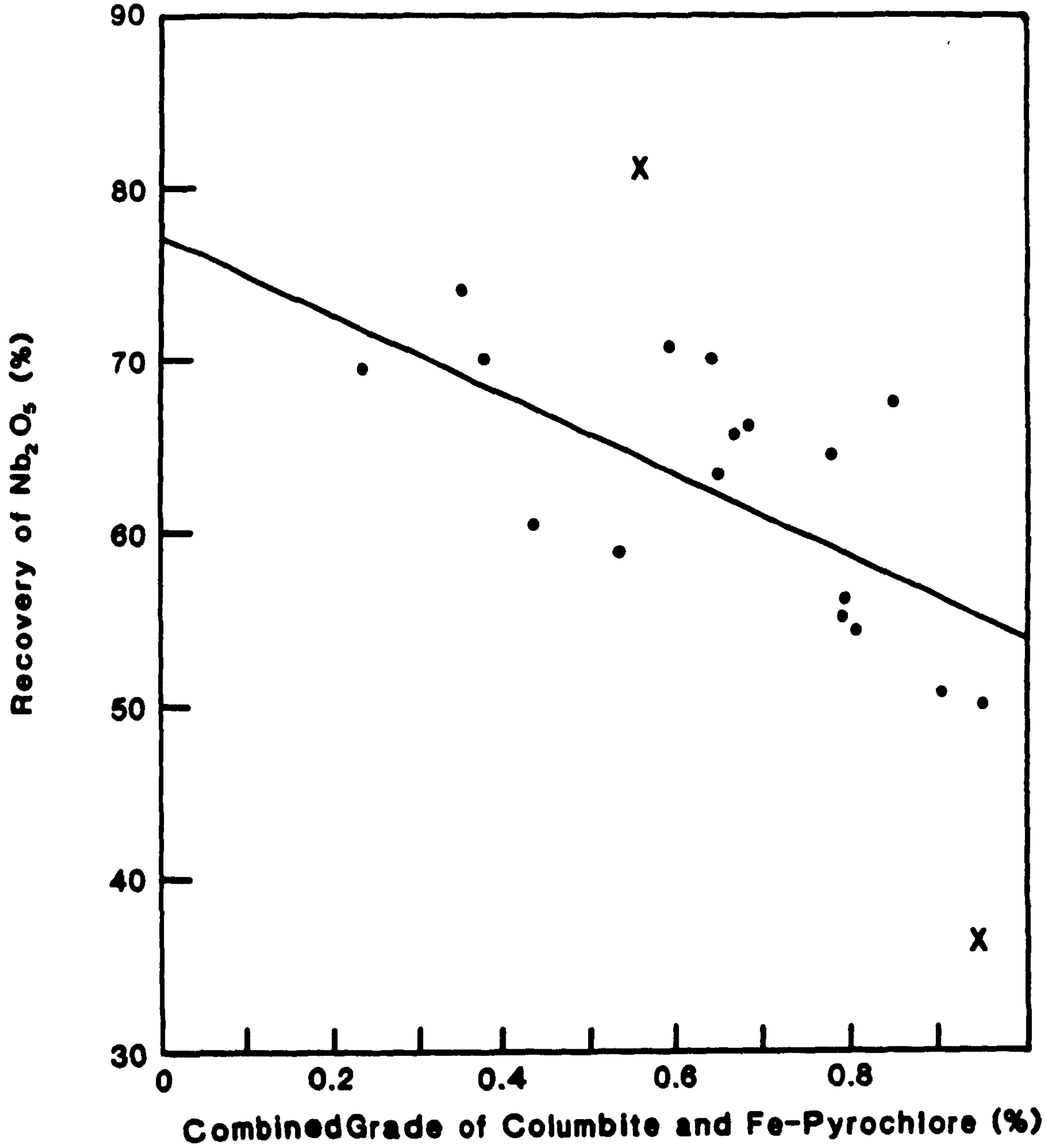
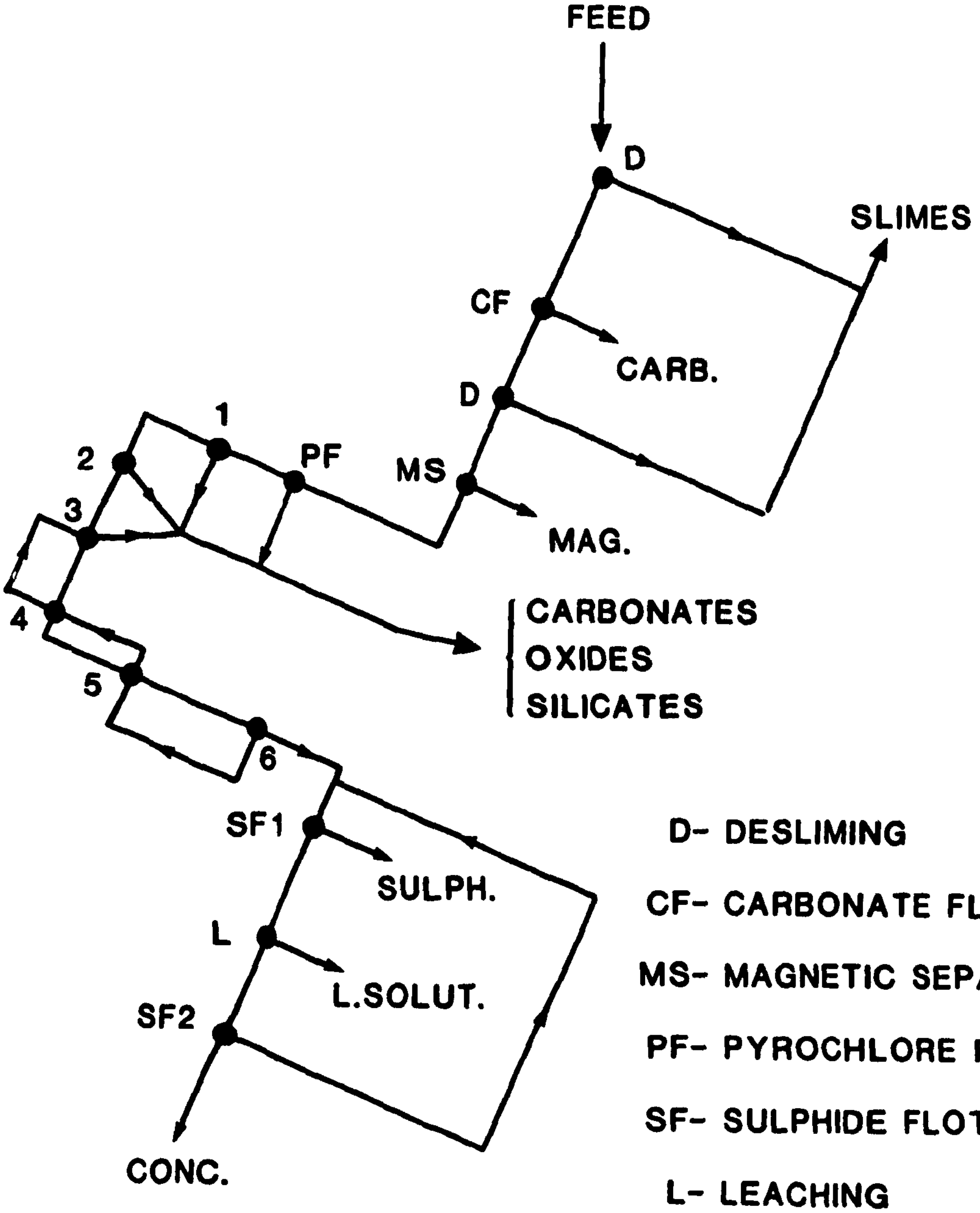


Figure 1.1 Overall Nb_2O_5 recovery vs the content of columbite and Fe-pyrochlore at Niobec.

Figure 1.2 **FLOWSHEET AT NIOBEC**



D- DESLIMING

CF- CARBONATE FLOTATION

MS- MAGNETIC SEPARATION

PF- PYROCHLORE FLOTATION

SF- SULPHIDE FLOTATION

L- LEACHING

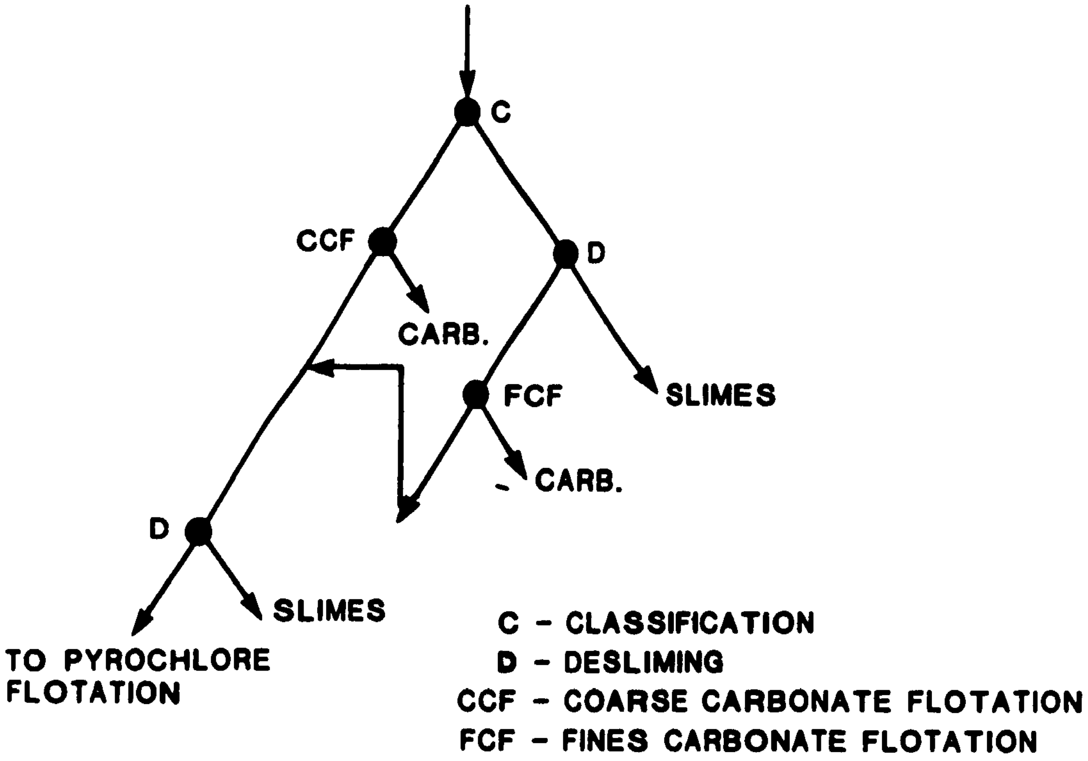


Figure 1.3 Details about the carbonate flotation circuit.

circuit and a coarse circuit after cyclone classification. This improves overall Nb_2O_5 recovery to the non-floats compared to treating the complete size range in a single circuit. Desliming of the carbonates is done before the carbonate and pyrochlore flotation, the slimes being discarded. Desliming is necessary for selective flotation and to reduce reagent consumption. About 15% of the Nb_2O_5 is lost in the slimes. Figure 1.4 shows a typical particle size distribution of these slimes. They are very fine: 80% -10 μm, 50% -3.5 μm and 30% -1 μm.

Table 1.3 presents the reagents used at Niobec and their consumption in terms of g/tonne of fresh feed. Carbonate flotation is performed with fatty acids, depressing the pyrochlore with sodium silicate and sometimes starch. Pyrochlore flotation is conducted with a cationic collector, 1-alkylamidoethyl-2-alkylimidazoline, manufactured by Hoechst and marketed as HOE-F2642 (F2642) (Biss and Nadeau, 1982). (F2642 is also quoted as an amine acetate by Hoechst, Hoechst, 1982). The selectivity of this cationic collector with respect to the gangue minerals is shown in Figure 1.5 (Biss, 1984a). Its selectivity against silicates is poor, being best at pH 2. However, selectivity against carbonates and oxides is high (Biss and Duval, 1982). Silicates are depressed by a combination of polyacrylamide, DK813 (from Allied Colloids), and sodium silicate. Sulphide flotation is performed at pH 10.5 with potassium amyl xanthate.

The pyrochlore flotation circuit consists of one roughing and six cleaning stages. In the cleaning stages the pH is gradually reduced from 6 to 3 to achieve selectivity while minimising acid consumption by residual carbonates. The pH modifiers used in the pyrochlore flotation are fluosilicic and oxalic acids. Oxalic acid is the preferred reagent. However, fluosilicic acid is used in the first cleaning stage because it does not

TABLE 1.3 SUMMARY OF REAGENT CONSUMPTION AT NIOBEC

CONSUMPTION PER TON. OF MILL FEED		
REAGENT	g/tonne	STEP
FATTY ACID	225	CARBONATE FLOTATION
FATTY ACID EMULSIFIER	80	
STARCH	10	
SODIUM SILICATE	450	
CATIONIC COLLECTOR: F2642	530	PYROCHLORE FLOTATION
COPOLYMER OF POLYACRYLAMIDE (DK-813)	65	
SODIUM SILICATE	60	
FLUOSILICIC ACID (H_2SiF_6)	1570	
OXALIC ACID ($C_2H_2O_4$)	2500	
POTASSIUM AMYL XANTHATE	5	SULPHIDE FLOTATION
TRIETHOXY BUTANE (TEB) (FROTHER)	10	
SODIUM HYDROXIDE	390	
COPPER SULPHATE	28	
HYDROCHLORIC ACID	2330	LEACHING

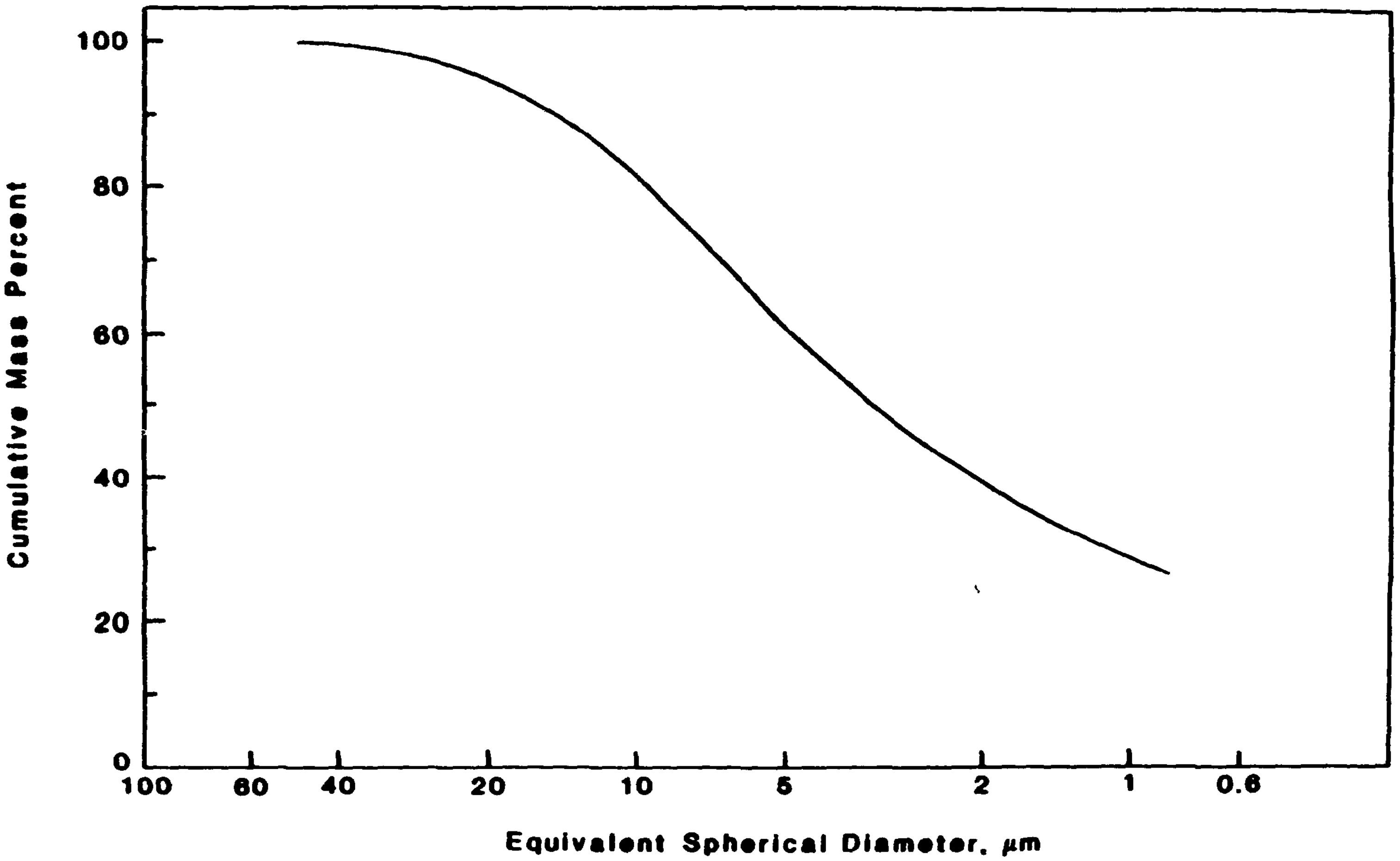


Figure 1.4. Typical particle size distribution of the slimes. This graph was obtained in the Sedigraph 5000.

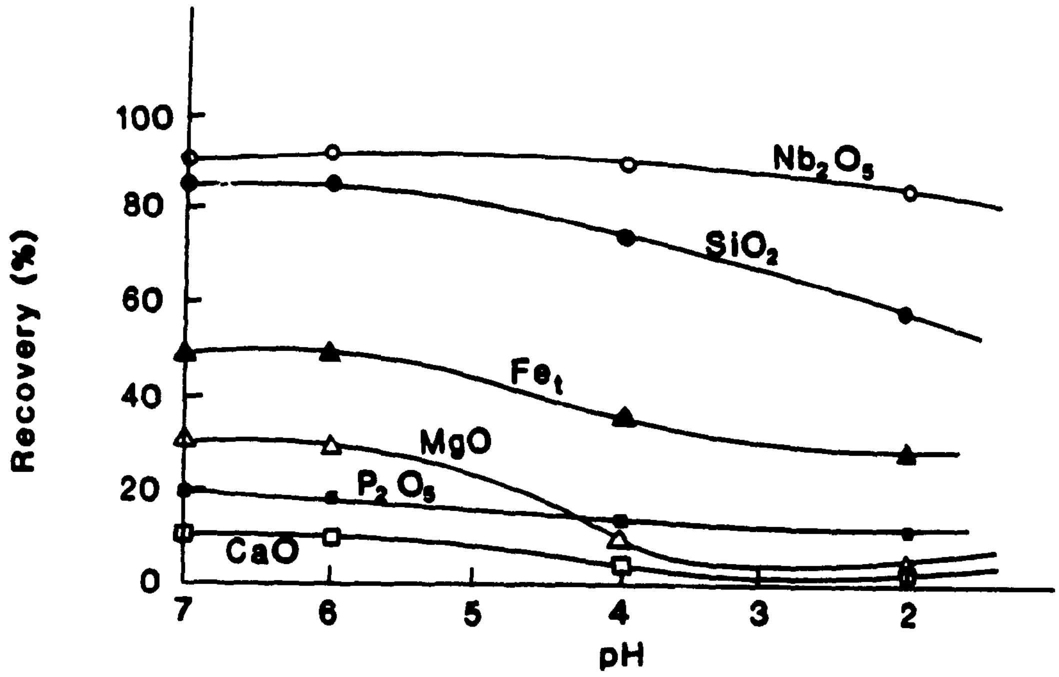


Figure 1.5 **Flotation of pyrochlore by F-2642 at different pH.** (Biss and Nadeau, 1982).

react with carbonates or their derived ions to any significant or troublesome extent. On the other hand, oxalic acid reacts with calcium ions to form calcium oxalate. This results in scale formation in the pipes and in the flotation cells, in increased acid consumption and in effervescence, which makes the pulp foam and difficult to control in flotation. For these reasons oxalic acid is used only in the latter cleaning stages.

Since up to 15% of the Nb_2O_5 is lost in the slimes, the recovery of even some of this would improve the overall plant recovery. Selective pyrochlore recovery from the slimes, however, is a complex problem and has not been addressed before at Niobec. The main problems confronting recovery of pyrochlore by flotation from the slimes are the extreme fineness and the similarity of flotation properties, especially with respect to the silicates.

1.4 FLOTATION OF FINES, GENERAL CONSIDERATIONS

Several books and reviews (Klassen and Mokrousov, 1963e; Somasundaran and Arbiter, 1978; Somasundaran, 1980; King, 1982; Somasundaran, 1986) have been presented on fine particles processing. Selective flotation of fines, particularly in non-sulphide processing, has proved difficult to achieve (Somasundaran, 1984). Consequently, in non-sulphide plants slimes are nearly always rejected in a desliming step. This is the case in the processing of pyrochlore (Raicevic and Noblitt, 1979). This results in loss of resources. For example, one-third of the phosphate mined in Florida, one-fifth of the World's tungsten, and one-half of the tin mined in Bolivia are discarded as slimes (Somasundaran, 1986). These losses have attracted much attention but, generally, recovery from slimes has been poor (Sresy et al, 1978; Banks, 1980; Iwasaki et al, 1980; Hu and Wang, 1982; Araujo et al, 1985).

Selective flotation becomes progressively more difficult as particle size decreases. This is due to a combination of physical factors such as an increased contact time with the bubble and entrainment of hydrophilic particles, and chemical factors, such as poor dispersion and less selective adsorption. These chemical factors, in turn, are influenced by water quality.

1.4.1 Physical Factors

Contact time. Particle collection efficiency is controlled by the product of collision and attachment/detachment efficiencies (Laskowski, 1974). For selective collection there must be a difference in attachment efficiency. However, for fine particles (less than 10 μm) a recent theory has suggested that attachment efficiencies become similar because contact time becomes very long (Dobby and Finch, 1986c). Thus there is an increased probability that the contact time of fine gangue particles will exceed the induction time for gangue particle attachment. This results in gangue attachment to bubbles (i.e. true flotation) and decreased selectivity.

Entrainment. Entrainment is the non selective recovery of particles which are transported into the froth with water carried in the wake of the bubbles. Since leaving the wake is largely a matter of gravitational settling fine particles (-10 μm) are preferentially entrained and recovered. Consequently, entrainment recovery increases with water recovery and decreasing particle size (Jowett, 1966; Trahar, 1981; Johnson et al, 1974). Trahar (1985) has found that the role of entrainment when treating ultrafine particles is often of such a magnitude that the true flotation of fines ceases to be the dominant recovery mechanism. The entrainment problem in mechanical cells has been studied in detail to try to reduce its magnitude (Johnson et al, 1974; Engelbrecht and Woodburn, 1975; Bisshop, 1976). Johnson et al (1974) suggested the use of more diluted feeds to reduce entrainment. Reducing entrainment has also been attempted by use of water spraying over the surface of the froth (Klassen and Mokrousov, 1963f; Kaya and Laplante 1987) and by the use of sonic vibration in the froth (Kaya and Laplante, 1987). Entrainment is eliminated in column flotation by maintaining a net downward flow of water through a deep froth (Dobby and Finch, 1985; Yianatos et al, 1987d). The column has great potential to improve separability of the fines and was the reason it was chosen in this study.

1.4.2 Chemical Factors

Dispersion. Dispersion is an essential step in any physical separation process (Yang, 1978; Somasundaran, 1986). Dispersion can be induced by mechanical agitation in systems with particles coarser than about 10 μm (Healy, 1979). However, as particle size decreases (and especially below 1 μm), van der Waals and electrostatic forces (among others) start to dominate and mechanical agitation is no longer sufficient to induce dispersion. This becomes more critical with non-sulfide and multimineral systems where it is more likely that species with opposite surface charge exist which promotes heterocoagulation.

Water quality. Contaminants in water have been shown to have a detrimental effect on selective flotation (Fuerstenau, 1975; Vreudge and Poling, 1975; Broman, 1980; Hoover, 1980; Rybinski et al, 1984; Ofori and Somasundaran, 1985). Water quality plays an important role in dispersion and non selective adsorption. For example, cations such as Ca and Mg, and residual non-polar organics from the process can adsorb on the particles, reducing electrostatic repulsion and inducing coagulation.

1.4.3 Alternative Techniques for Processing of Fines

There are some techniques that have the potential for effective treatment of fines: carrier flotation, selective flocculation, and flotation with controlled dispersion and chemisorbing collectors.

Carrier flotation. This technique consists in floating fines by using coarse particles as a carrier (Wang and Somasundaran, 1983). Carrier flotation is used, for example, to remove anatase impurities from clay, using coarse calcite as carrier particles, and oleate as collector along with Na_2CO_3 and Na_2SiO_3 as modifiers. The mechanism responsible is enhanced aggregation between anatase and calcite with almost no aggregation between clay and calcite (Wang and Somasundaran, 1983).

This aggregation is not due to electrostatic attractions because at the flotation pH, calcite and anatase have a strongly negative (-70 mV) zeta potential. Aggregation is rather caused by interactions between oleate layers adsorbed on anatase and calcite. Even though oleate is also adsorbed on clay, the strength of this attachment is very weak and the adsorbed layer can be easily detached by the strong agitation used in the process (Somasundaran, 1984).

Carrier flotation has also been used for the recovery of apatite from phosphatic slimes using sulfur as carrier mineral (Greene and Duke, 1962), Table 1.4 shows that the recovery from Florida phosphate slimes was improved by carrier flotation, while that of Tennessee slimes was not. For Tennessee slimes, with a higher fraction of plus 0.2 µm particles, coarser

TABLE 1.4 COMPARISON OF RECOVERY OF APATITE FROM TWO PHOSPHATIC SLIMES WITH AND WITHOUT SULPHUR AS CARRIER MINERAL

	% RECOVERY
FLORIDA SLIMES	
conventional method	3.6
carrier flotation	22.4
TENNESSEE SLIMES	
conventional method	54.5
carrier flotation	55.8

particles acted as carriers and sulfur particles did not.

Another application of carrier flotation was a laboratory study of separation of hematite fines (100% -38 μm) from quartz fines (100% -75 μm). The carrier was coarse hematite (-212 + 150 μm) in an oleic acid collector system (Cristovenau and Meech, 1985). In this system, hematite recovery was increased. However, carrier flotation was found to be very sensitive to changes in oleic acid addition, the relative proportion of fine and coarse material, conditioning time, and water quality. The authors conclude that application of carrier flotation is only suitable when flotation conditions can be stabilized.

Another drawback for this process may be the subsequent separation of valuables from the carrier particles, when the valuables minerals are being recovered in the froth.

Selective flocculation/flotation. This technique involves three steps: a) selective aggregation (flocculation) of well dispersed fines, through utilization of a bonding agent (polymer flocculant) that ties specific mineral particles together; b) separation of flocs and dispersed solids; and c) upgrading of the flocs to release entrapped particles.

For selective flocculation, several factors need to be considered: a) flocculant characteristics (type of functional group, physical and electrochemical properties); b) mineral characteristics (surface charge, solubility); and c) aqueous medium (water quality, pH, hydrodynamic forces).

Most flocculants are not very selective. However, selectivity can be increased through either incorporation (or substitution) of more selective donor atoms into the structure. Clauss et al (1976) incorporated hydroxamic acid groups into a polyacrylamide flocculant, producing a selective flocculant for cassiterite from mixtures with quartz. These investigators found that separation of cassiterite from quartz was possible at pH between 3.5 and 7.

Selective flocculation has been applied on a large scale in iron ore processing. The process (Colombo, 1978), involves grinding to 75-80% -500 mesh with NaOH and Na_2SiO_3 (0.57 lb per t). The product is treated at pHs 10.5-11.0 with causticized corn starch. This results in a strong selective flocculation of the hematite, the quartz and other silicates remaining well dispersed. Simple thickening removes about one-third of silica with only 7% of iron in the overflow. The underflow is treated by cationic flotation to remove the coarser remaining silica and produce a 65 to 66% iron concentrate at 70% recovery. This technique has also been shown to have potential in the recovery of sedimentary phosphate ores (Araujo et al, 1985).

Flotation with controlled dispersion and chemisorbing collectors. As mentioned, a vital condition for selective flotation is particle dispersion. In this technique after dispersion, selective chemisorbing collectors are added to be adsorbed only on desired species. Chemisorbing reagents, due to their lower solubility are added with oil. Oil has been found to increase recovery and decrease collector consumption. The oil-collector mixture is added as an emulsion and the minute oil droplets readily adsorb on the surfaces of minerals rendered hydrophobic by adsorbed chemisorbing reagents. Oil coated particles agglomerate, increasing flotation rate due to a particle size effect.

This technique has been laboratory tested (Yang, 1978) on Tilden taconite ore without desliming. After adding a dispersant (0.1 - 0.5 Kg/t ferric silicate), ore was conditioned for two minutes at pHs 8-9. Flotation was performed using 0.5 - 0.75 Kg/t of fuel oil. Results show 87% Fe recovery with a grade of 65.8% Fe compared to the commercial selective flocculation-cationic flotation process which yielded only 70% Fe recovery at comparable grade. This technique becomes difficult (Espinosa-Gomez et al, 1987b) when dispersion cannot be achieved at low dispersant concentrations which arise due to a high concentration of cations dissolved in the process water. Since large amounts of dispersants are required to get dispersion, there is difficulty in maintaining a balance between particle dispersion and particle flotation.

Flotation columns can be applied in conjuction with any of the three previous techniques. Flotation columns would avoid entrainment and would increase the potential of such techniques. In this study column flotation was planned in conjuction with an approach similar to the third technique.

1.4.4 Flotation Column

A flotation column is shown schematically in Figure 1.6. There are two basic zones in a column, the collection and the cleaning zones. In the collection zone, particles from the feed slurry are contacted counter current with a bubble swarm produced by a gas sparger at the bottom of the column. Hydrophobic particles attached to the bubbles are transported to the cleaning zone. In the cleaning zone, water is added near the top of the froth, providing a net downward liquid flowrate called a positive bias. This positive bias prevents the entrainment of fine hydrophilic particles into the concentrate (Dobby and Finch, 1985a; Yianatos et al, 1987d). The column has been used primarily in cleaning applications and can achieve in a single stage upgradings comparable to several stages of mechanical cells often with improved recovery (Coffin and Miszczak, 1982; Amelunxen and Redfern, 1985;

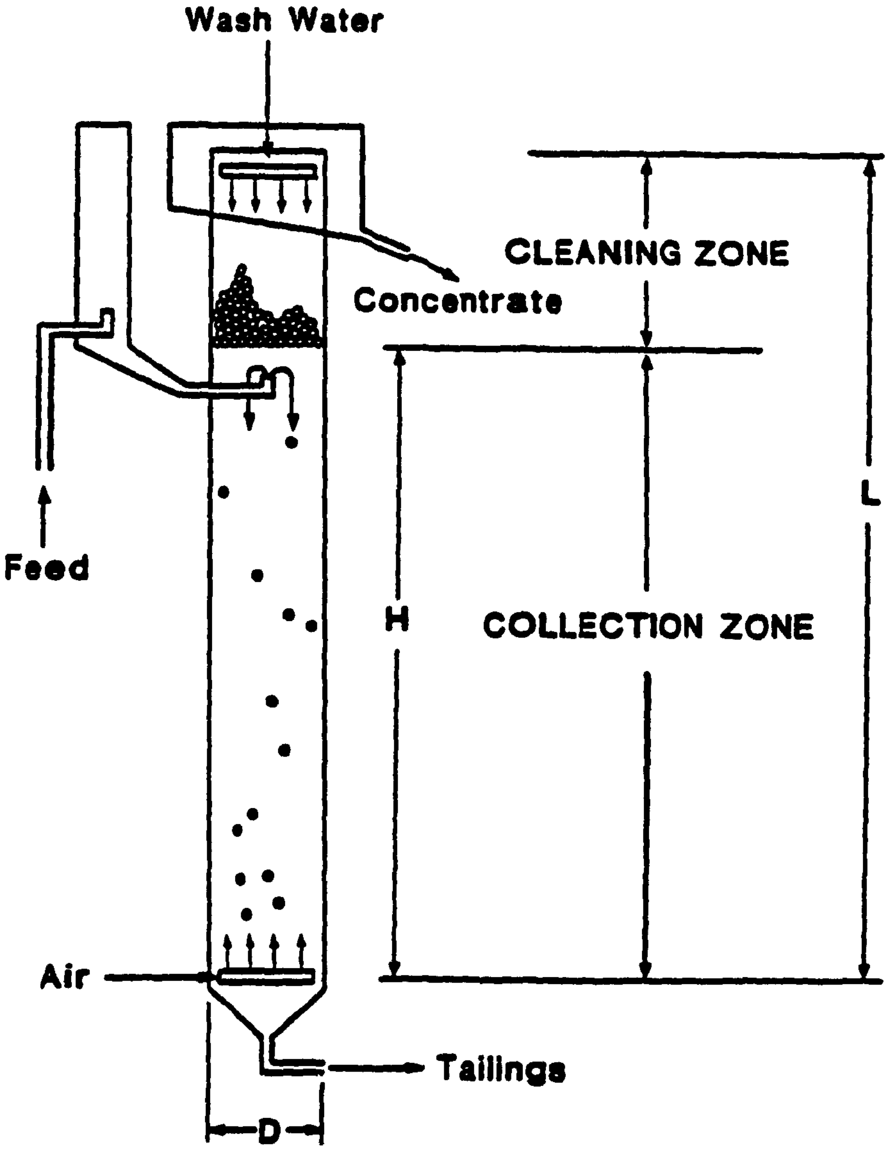


Figure 1.6. Schematic representation of a flotation column

Wheeler, 1985).

1.5 SIMILARITY OF FLOTATION PROPERTIES OF PYROCHLORE AND CONTAMINANTS MINERALS

Selective pyrochlore flotation is rendered difficult because of the similar flotation characteristics of pyrochlore and gangue minerals (particularly the silicates). The pyrochlore and the silicates have similar electrokinetic properties as evidenced by the similarity in their IEP values (Table 1.1). These close values limit selective flotation based on collector adsorption by electrostatic mechanisms. Reagents that exploit selective chemical interactions are required. Improved selectivity may permit direct pyrochlore flotation with the possible advantages of higher niobium recoveries, lower reagent consumption and a simpler circuit. Several reagents exploiting selective chemisorption have been studied but without great success (Bogdanov, et al, 1973; Pickett, 1959; Kennecot Copper Corp., 1958).

A potential route to increase the recovery and selectivity of the Nb ores at Niobec is the use of more selective (e.g. chemisorbing) reagents which allow recovery of the Nb lost in the slimes, while maintaining selectivity against the silicates. To control entrainment, the flotation device of choice is the flotation column. More selective reagents and the flotation column together should result in a simpler circuit than that currently used at Niobec.

1.6 STATEMENT OF THESIS OBJECTIVES

The two main objectives of this work were: 1) to develop a reagent scheme for the selective flotation of pyrochlore from the slimes discarded at Niobec, and 2) to determine if flotation columns were better suited to slimes processing than mechanical cells.

1.7 METHOD OF ATTACK

To achieve these two objectives the approach was broken down into the following steps:

i) Characterization of the slimes (mineralogical composition and particle size distribution).

ii) Evaluation of the effects of the water contaminants on selective flotation. This needed to be addressed since water is recycled at Niobec, and the only way to evaluate and select conditions is using plant recycle water. The effects of two types of water (recycle and potable) from Niobec were evaluated by electrokinetic and microflotation studies.

iii) Identification of selective niobium flotation collectors and selective silicate depressants based on chemisorption rather than electrostatic interactions.

iv) Microflotation testing of the reagents, with single minerals.

v) Recycle water treatment, to determine if there is any improvement in selectivity with a better water quality.

vi) Dispersion study using recycle water to select best dispersion conditions.

vii) Column flotation testing of slimes with flotation and dispersion conditions identified in iv) and vi).

viii) Plant testing to compare the column performance to that of the mechanical cells with streams treating very fine particle sizes (e.g. less than 20 μm).

1.8 OVERVIEW OF CHAPTER CONTENTS

Chapter 2. Chapter 2 describes the basic work to develop the reagent scheme for the selective flotation of pyrochlore. This work involved a literature search for selective chelating agents for Nb-minerals flotation. The reagents selected were evaluated by microflotation using pure samples of pyrochlore and the main silicate contaminants. An electrokinetic study was conducted to help interpretation of results. During the testwork plant water was used to try to reproduce plant conditions. Further results and discussion are given in a manuscript entitled 'Effects of the type of water on the selective flotation of pyrochlore from Niobec.' From this work, some options were suggested to float selectively the pyrochlore from the slimes. (More details about the flotation work are given in an Appendix which includes three reports (prepared for the Centre the Recherches Minerals, Quebec) and a subsequent manuscript entitled 'Effects of the water chemistry on pyrochlore and silicate flotation').

Chapter 3. Chapter 3 describes the column flotation work, including sample preparation. In the first part it is shown that the column results on the slimes do not match plant results on the deslimed material due to poor dispersion. A dispersion study was conducted. A manuscript gives the results of the dispersion study and also the effect of dispersants on flotation, and on coalescence and froth stability. The manuscript is entitled 'Dispersion study on slimes from Niobec.' These coalescence and froth stability problems appeared when the dispersants were used in flotation. Finally, in the last section, additional details on the column flotation results are presented using the dispersion conditions selected in the dispersion study. It is also shown that coalescence and froth stability problems persisted, preventing further testing. (More details about sample preparation, and flotation results and conditions are given in three appendices).

Chapter 4. Chapter 4 describes a detailed study of the coalescence and froth collapse problems in the frother-fatty acid-dispersant-solid system. This study was performed to ascertain the origin of these problems. A novel method of quantifying coalescence through measured changes in the gas holdup in the collection zone of the flotation column was developed. The chapter also reviews coalescence and describes the basic principles of its quantification by the method of gas holdup change. The chapter concludes with a manuscript entitled 'Coalescence and froth collapse in the presence of fatty acid', which gives an overview and includes the results and discussion.

Chapter 5. Chapter 5 describes the column flotation of fines. Column flotation of carbonates from the slimes in the laboratory was not satisfactory due to inadequate dispersion and froth collapse/coalescence problems. It was therefore not possible to determine whether the column was the device best suited to treat the slimes which was one of the original objectives. As a compromise plant tests were conducted at Niobec with the fines carbonates stream as this offered the closest available size distribution to the slimes. In a latter part of this chapter a manuscript entitled 'Column flotation of very fine particles' is included which shows additional work conducted by the author in other fine particle streams, including the results at Niobec, which supports the superior performance of column flotation on fines.

CHAPTER 2

FLOTATION OF Nb AND SILICATE MINERALS FROM NIOBEC: SINGLE MINERAL MICROFLOTATION TESTS AND EFFECT OF WATER QUALITY

2.1 INTRODUCTION

This chapter describes the basic work conducted to select the chemical reagents and conditions for recovery of the Nb-minerals from the slimes discard at Niobec. The work involved: a) an electrokinetic study, b) a literature search for selective Nb-mineral flotation collectors, and c) a microflotation study to select best reagents and conditions from b). Experimental details are given in the first part of this chapter, the second part being a manuscript entitled 'Effects of the type of water in the selective flotation of pyrochlore at Niobec (Colloids and Surfaces, Vol. 26, pp. 333-350 (1987)). This manuscript emphasises the effects of the type of water on the selective flotation of pyrochlore. Further details are given in Appendix 1, which includes three reports (prepared for the Centre de Recherches Minérals, Québec, and included with permission) and a subsequent manuscript entitled 'Effects of the water chemistry on pyrochlore and silicates flotation' (to be published in Minerals Engineering).

2.2 SAMPLE CHARACTERIZATION AND PREPARATION

2.2.1 Sample Source

Pyrochlore. It is difficult to obtain sufficiently pure pyrochlore for the planned single mineral testwork from run-of-the-mine ore. The compromise was to use a pyrochlore concentrate from Niobec. This will contain residual flotation reagents. The solution was to isolate a coarse size fraction (106-150 μm), clean it to remove contaminant minerals and residual reagents, and grind (- 16 μm) to generate a large number of fresh surfaces.

A pyrochlore concentrate was obtained while the plant was processing zone 1 ore. This concentrate was subjected to a mineralogical examination (including X-ray diffraction and optical and electron microscopy), to identify the Nb-carrying species and contaminant minerals. Initial work employed loose grain and polished sections of size fractions 106-150 μm, 53-75 μm and 43-53 μm. The main contaminants were microcline, biotite and chlorite.

To aid in the identification of the Nb-minerals a sample of the 53-75 μm size fraction was processed on a Frantz Isodynamic Magnetic Separator, (Frantz) (Gaudin and Spedden, 1943; Hess, 1959) working at a side slope of 20. Magnetic fractions from 250 mA to 1500 mA were isolated. X-ray diffraction (using a Philipps PW 1710 diffractometer) was performed on each magnetic fraction, and selected fractions were subjected to electron microprobe analysis (using a Camebax Electron Microprobe, Model MB1, wavelength analysis, probe beam less than 1 μm size) primarily to determine Nb and Fe content of the mineral particles.

The results of the microprobe examination are summarized in Table 2.1. The dominant Nb-species is pyrochlore ranging from relatively strongly magnetic with Fe = 0.41% to relatively weakly magnetic with Fe=0.03%. These pyrochlore types are referred to as Fe-pyrochlore and Na-pyrochlore, respectively. A minor Nb-species is columbite (about 6% of the sample). The iron content in the types of pyrochlore present here is lower than those reported by Perrault (1978) and Perrault and Manker (1981) (Chapter 1, Table

TABLE 2.1 SUMMARY OF THE MICROPROBE STUDY ON THE PYROCHLORE CONCENTRATE

Frantz range (mA)	% mass in range	Nb mineral	Mineral Grade			% Mineral in fract.	% Mineral in sample
			% Nb	% Fe	% Na		
-250	6.0	columbite					
		Fe pyrochlore	66.1	0.41	8.31	95	5.7
		Na pyrochlore	69.0	0.00	8.4	5	0.3
+250	94	columbite	72.8	9.3	0.46	6	5.6
		Fe pyrochlore	64.0	0.63	5.34	26	24.5
		Na pyrochlore	67.4	0.03	8.36	68	63.9
overall sample		columbite				6	
		Fe pyrochlore				30	
		Na pyrochlore				64	

1.2), i.e. an iron content of 4.2% and 1.04% for the Fe-pyrochlore and Na-pyrochlore, respectively. The Na content of the Fe-pyrochlore in the -250 and +250 mA fractions was 8.31 and 5.34%, respectively. The Na content in the -250 mA fraction is similar to that reported by Perrault and Manker (1981) in Na-pyrochlore (8.6%); the Na content in the +250 mA fraction is closer to that in the Fe-pyrochlore (4.18%).

Other minerals. Microcline, biotite and chlorite were the minerals of interest. They were obtained as hand-picked specimens from the Niobec region and from Ward's Natural Scientific Co.

2.2.2 Samples for the Electrokinetic Study

Pyrochlore, microcline and biotite were obtained from Niobec. Pyrochlore was isolated from the -150 +106 μm fraction of the concentrate, using heavy liquid (s-tetrabromoethane) separation at s.g.=2.9. The sink (pyrochlore) fraction was washed repeatedly with acetone to dissolve any residual heavy liquid. Microcline and biotite were hand-picked specimens. They were ground and upgraded using the Frantz. Microcline was isolated as the non-magnetic product at the maximum setting of 1500 mA. Biotite was isolated between currents 350 mA and 650 mA. Repeated processing on the Frantz was required. Sample purity in all cases was checked by X-ray diffraction and optical microscopy; no contaminants were found.

The particle size required for electrophoretic mobility measurements in the device used is -3 μm (Yoon, 1971; Lozyk, 1978; and Kelebek, 1980). Upgraded samples of the 106-150 μm size fraction, weighing about 2 g, were used to obtain this particle size range. Each sample was first dry ground in an agate mortar and then transferred to a 500 ml glass cylinder and dispersed for 20 minutes using an ultrasound bath. The sample was then allowed to settle and the top 5 cm of suspension was recovered. Different settling times were allowed for each mineral to obtain a top size of 3 μm (see Appendix 2). The suspension thus recovered was transferred to a 200 ml flask and stored for future use. Deionized water was used throughout.

2.2.3 Preparation of Samples for Microflotation Tests

The pyrochlore samples were obtained from the same 106-150 μm size fraction isolated from the concentrate, as described. The silicates (microcline, biotite and chlorite) were obtained from Ward's Natural Scientific Co.. All samples were ground in a vibratory porcelain mill and a primary classification stage was conducted in a 5.08 cm (2 inch) hydrocyclone (cyclone) to eliminate the -2 μm fraction to the overflow. The underflow was further classified using a Warman Cyclosizer (Kelsall and MacAdam, 1962). The content of cones 1-5 were collected and most of the finest (-cone 5) fraction. To obtain the feed for the microflotation tests the contents from cones 3, 4, 5 and -5 were mixed. The particle size distribution (PSD) of these materials was determined using a particle size analyzer, the Sedigraph 5000D (Sedigraph) (See Appendix 3). The PSD was about 1 to 14 µm for all the samples. This is close to the size range used in the column flotation tests on the slimes (Chapter 3). The electrokinetic properties of these silicates were similar to those of the Niobec samples (see values in Appendix 4).

2.3 ELECTROKINETIC STUDY

Much of this study is addressed in the manuscript (section 2.5) with additional details in Appendix 1 (Reports 3 and 4).

This section is intended to provide more detail on the apparatus used and the calculation of electrophoretic mobility. This section also summarizes the values of the isoelectric point of charge (IEP) of the minerals.

2.3.1 Apparatus.

Electrokinetic mobility of the particles was measured on a Mark II micro-electrophoresis apparatus (Rank Brothers) (see Figure 2.1). It consists of a transparent flat-type quartz cell in which observations of moving particles are made under voltage applied through platinum electrodes. The flat cell has a section of 0.1 cm x 1 cm. The inter-electrode distance, found by calibration with 0.100 M KCl at 25 °C, was 7.08 cm. Movement of the particles was observed through the eye-piece micrometers, which were located on each side of a timer accurate to 1/10 of a second. One of the eye-pieces is fitted with a scale of 104 μm/division, the other 36 μm/division. The cell is immersed in a water bath whose temperature was determined during the observations with a total immersion thermometer with graduations of 0.1°C. The temperature used was 25 +/-0.5 °C.

2.3.2 Electrophoretic Mobility Determination

Procedure. The experimental procedure for the electrophoretic mobility measurement is similar to that of Yoon (1971), Lozyk (1978), and Kelebek (1980). The micrometer positions of the inner faces of the front and back walls were located by focussing on the solid particles which adhered to them. These positions were recorded and the difference between them was calculated to obtain the cell thickness. Cell thickness was then multiplied by a factor of 0.197 to determine the distance in millimeters from the front

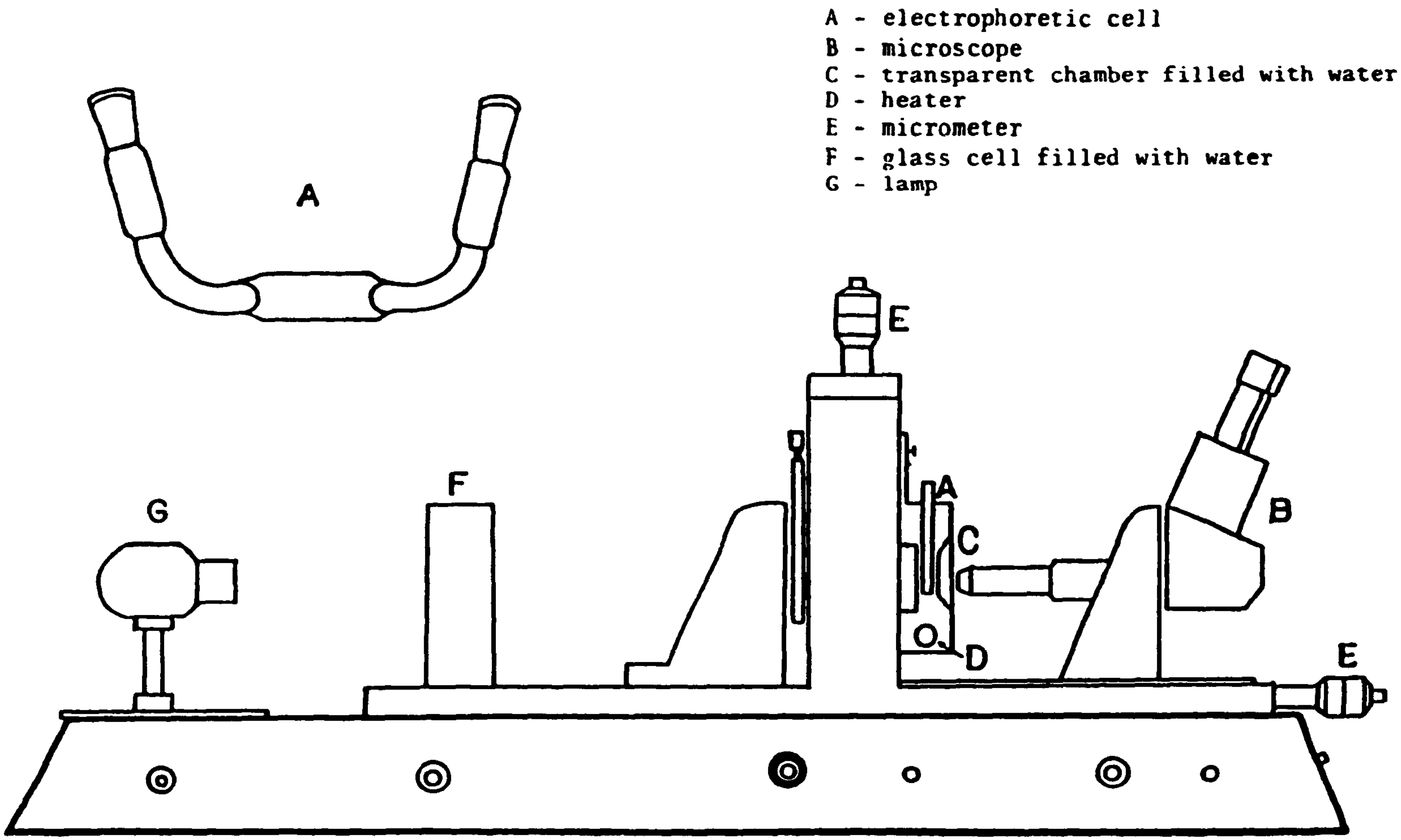


Figure 2.1. Schematic representation of the micro-electrophoresis Apparatus

and back cell walls of the location of the zero fluid velocity where the characteristic electrokinetic mobility of the migrating particles can be measured accurately. The eyepiece was focussed at these locations.

For each pH value, about 40 different particles were observed in order to calculate an average mobility value. In the case of extremely low pH values, due to electrolysis of the water during the prolonged observation time (necessitated by slow movement of particles) the number of particles which could be observed without gas liberation was about 8 to 20.

The apparent charge on the particle surface was determined by observing the direction of motion of the particles under the influence of the applied field and taking into consideration the reversal of the image by the lens.

Before each determination the cell was washed in concentrated hydrochloric acid and then several times in distilled water. Next it was washed in a 0.1 N NaOH solution and washed several times in distilled water again. To measure electrophoretic mobility, about 2 ml of suspension was transferred from a flask containing the sample suspension to a beaker with about 50 ml of the type of water to be tested. The pH was adjusted and allowed to equilibrate for about 4 hrs. After this time, the pH was measured again and part of the solution was used to rinse thoroughly the flat cell. About 5 ml of the suspension was transferred to the cell to measure the electrophoretic mobility. The remaining suspension was used to rinse the electrodes.

Calculation of electrophoretic mobility. The electrophoretic mobility, u, was calculated using the following relation:

$$u = \frac{\text{particle velocity}}{\text{potential gradient}} = \frac{d/t}{E/l} \quad \frac{(\text{cm/s})}{(\text{volt/cm})}$$

where:

d: distance travelled by the particle (cm)

t: average time required to travel distance d, (s)

E: applied field (volts)

l: inter-electrode distance (cm)

2.3.3 Summary of the IEP Values

Table 2.2 summarizes the isoelectric point of charge (IEP) values obtained. The minerals studied were Fe-pyrochlore, pyrochlore (combined Fe and Na types) and the two main silicate contaminants: microcline and biotite. Subsequent work did not distinguish between types of pyrochlore since the electrokinetic behaviour was not significantly different. Initial testwork was conducted with four types of water and three pH modifiers. Subsequent work involved only the pH modifiers used at Niobec (oxalic and fluosilicic acid), and the two types of water used at Niobec (potable and recycle).

2.4 SELECTIVE Nb FLOTATION REAGENTS

This section outlines the procedure followed to select collectors and depressants for pyrochlore and depressants for silicates. Selective reagents would simplify the complex flowsheet which is at present required to recover the pyrochlore at Niobec. Chelating agents were considered because they can be highly specific for cation sites on a mineral surface.

TABLE 2.2 SUMMARY OF THE ISOELECTRIC POINT OF CHARGE OBTAINED WITH THREE pH MODIFIERS AND FOUR TYPES OF WATER

MINERAL	pH MODIFIER	(*) IEP TYPE OF WATER			
		ORGANIC FREE	DISTILLED	POTABLE	RECYCLE
PYROCHLORE (**)	HCl	2.5	3.5	2.7	
PYROCHLORE (**)	$C_2H_2O_4$			2.5	
PYROCHLORE (**)	H_2SiF_6			2.4	1.8
FELDSPARS	$C_2H_2O_4$			2.0	
FELDSPARS	H_2SiF_6			2.0	1.8
BIOTITE	$C_2H_2O_4$			1.5	
BIOTITE	H_2SiF_6			1.9	2.0
PYROCHLORE (***)	HCl		4.6	2.3	
PYROCHLORE (***)	$C_2H_2O_4$			2.2	
PYROCHLORE (***)	H_2SiF_6			2.3	

(*) IEP, isoelectric point of charge (pH at which the mobility of the particle is zero).

(**) Combined Na, Fe-pyrochlore types

(***) Fe-pyrochlore

(A chelating agent contains at least two polar groups which are close enough to interact and occupy simultaneously more than one position in the coordination sphere of the cation). The reagents selected in this study were tested by microflotation. The best reagents and conditions were later to be tested on the slimes by column flotation.

A literature survey in analytical chemistry of Nb was conducted to look for possible selective chelating agents (Yale, 1943; Moshier and Schwamberg, 1957; Majumdar and Chowdhury, 1958; Langmyhr and Hongslo, 1960; Laitinen, 1960; Charlot and Bezier, 1960; Brandt, 1960; Moshier, 1964; Buser et al, 1970; Gibalo, 1970; Chatterjee, 1978; Agrawal, 1979; Cheng et al, 1982). Chelating agents can be extremely selective forming stable compounds with specific metal ions. In principle, therefore, chelating agents would react with Nb whether in pyrochlore or columbite. Formation of an adsorbed chelating agent with appropiate hydrocarbon (hydrophobic) groups could cause flotation (Aplan and Fuerstenau, 1962; Nagaraj and Somasundaran, 1977; Muthuswami et al, 1983; Barbery and Cecile, 1983; Somasundaran and Nagaraj, 1984; Fuerstenau and Pradip, 1984). Analogous reasoning was applied in the search for chelating agents for depression (of either Nb-minerals or the silicates) in which case the adsorbed chelating agent should carry hydrophylic groups such as hydroxyl. Also considered in the search were flotation reagents already tested for Nb and Ta, (Faucher, 1964; Gorlovskii et al, 1966; Abeidu, 1974; Biss and Nadeau, 1982; Burt, 1982; Luo and Chen, 1985; Fuerstenau and Pradip, 1984; Collins et al, 1984), and flotation reagents suggested by some manufacturers, e.g. Hoechst Canada Inc. and Allied Colloids.

Tables 2.3 and 2.4, summarize most of the organic reagents found during this search. Table 2.3 shows the reagents suitable as Nb collectors.

TABLE 2.3 ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS.

ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS

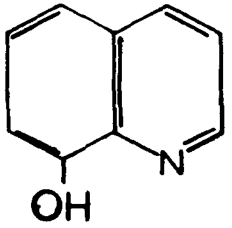
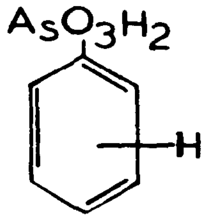
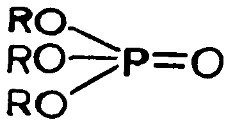
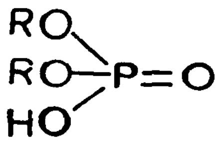
NAME	STRUCTURE	SELECTIVITY	REMARKS
8-Hydroxyquinoline.		Poor	In acetate solutions react with Al,Fe,Nb,Ta,Th,U,V,Cu,Ag.
Benzenearsonic acid (phenylarsonic acid).		Good	In acid medium, Nb ions are precipitated above pH,4.8 Fe is also precipitated. Very stable Nb compounds.
Trialkyl phosphate (TBP).		Poor	Fe and Nb are extracted. It is convenient to work at very low pH.
Dialkyl phosphate (HDEHP).		Poor	Fe and Nb are extracted. It is convenient to work at very low pH.

TABLE 2.3 ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS. (CONTINUATION).

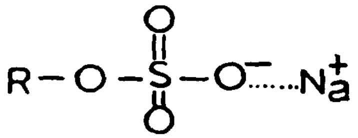
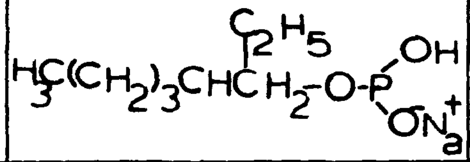
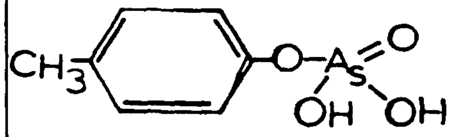
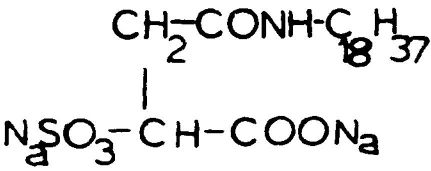
ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS			
NAME	STRUCTURE	SELECTIVITY	REMARKS
Sodium alkyl sulphate.	 $R-O-\overset{O}{\underset{O}{S}}-O^{-}\cdots Na^{+}$	Good	Good selectivity in the pH range of 2.5-3.5.
Alkyl phosphonic acids; e.g. 2-ethylhexyl phosphonic acid.		Poor	Strongly depress by Fe(III).
Alkyl (aryl) arsonic (or antimonic) acids. RAsO4H2 (RSbO4H2), such as p-tolylarsonic acid.		Good	Precipitate very easy with Fe(III), Nb(V) and Ca(II).
Sulfosuccinamate: N-octadecyl sulfosuccinamate (Aerosol 18).	 $\begin{array}{l}CH_2-CONH-C_{18}H_{37}\\ \quad\quad\; \vert \\ NaSO_3-CH-COONa\end{array}$	Good if iron can be complex.	Optimum pH for Ta at 2.4. Fe(III) can be complex with citric acid.

TABLE 2.3 ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS. (CONTINUATION).

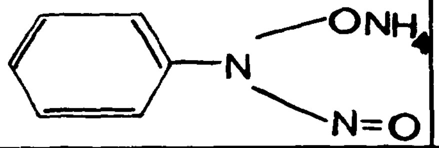
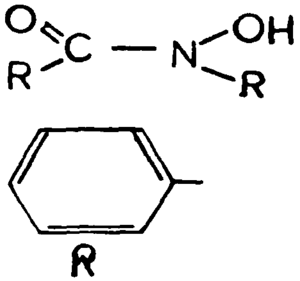
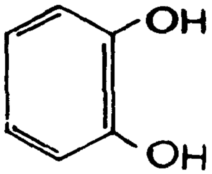
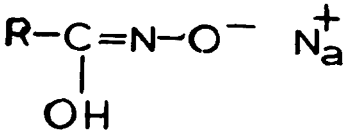
ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS			
NAME	**STRUCTURE**	**SELECTIVITY**	**REMARKS**
Cupferron (N-Nitrosophenyl hydroxylamine, ammonium salt).		Poor	Under strong acid conditions Fe(III),Nb,Sn,Ta,Ti,V and Zr precipitate.
N-Benzoyl-N-phenyl-hydroxylamine (N-phenylbenzohydroxamic acid, BPA, NBPHA, BPHA).		Good,in a very narrow pH range.	Nb (V) can be selectively separated from Al,Ca,Fe(III),Mg,Ta,Th,and U(VI) at pH 3.5 - 3.6, with tartarate, EDTA.
Catechol (Pyrocatechol 1,2-dihydroxybenzene).		Poor	It forms colored chelates with Fe(III), Mo(VI), Ti,V,Nb and Ta,but most of the reactions are of little practical importance.
Alkyl hydroxamates.	 $\mathrm{R{-}\underset{\displaystyle OH}{\underset{\vert}{C}}{=}N{-}O^{-}\ \overset{+}{Na}}$	Poor	Optimum flotation conditions at pH-7.

TABLE 2.4 ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE.

ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE

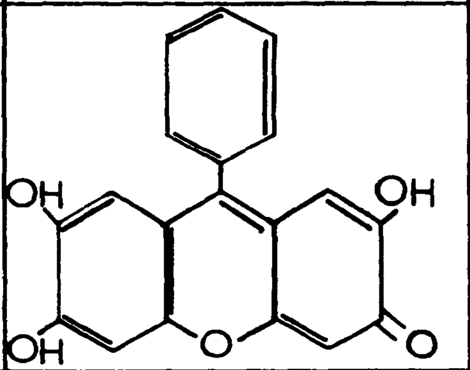
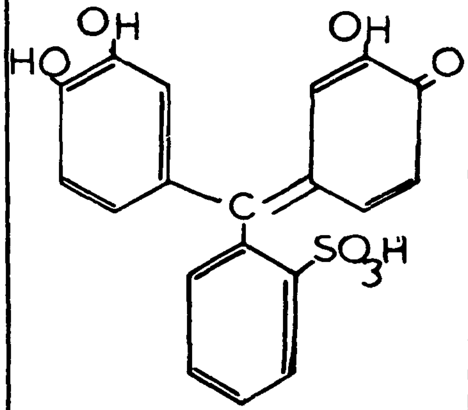
NAME	STRUCTURE	SELECTIVITY	REMARKS
Phenylfluorone (2,3,7-Trihydroxy-9-phenyl-fluorone,2,6,7-thrihydroxy-9-phenylisoxanthene-3-one.		Poor	Selective for Nb(V) and Fe(III). Works better with 0.8% H_3PO_4.
Pyrocatechol violet (3,3',4-Trihydroxyfuchsone-2"-sulfonic acid).		Good	Selective for Nb(III) at pH 4-5 Selective for Al(III) at pH -6 Selective for Fe(III) al pH 5.7-6.4.

TABLE 2.4 ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE. (CONTINUATION).

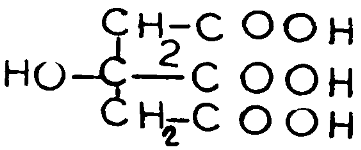
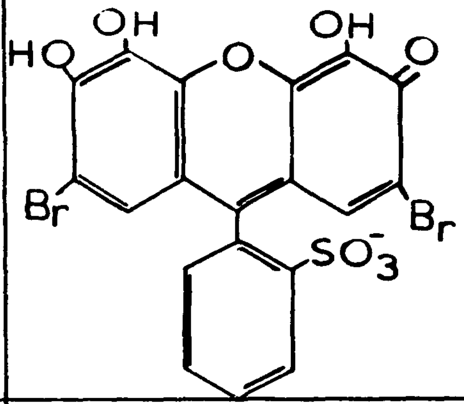
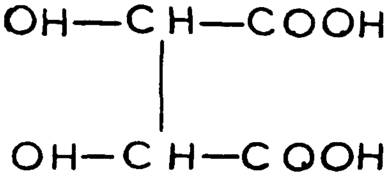
ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE			
NAME	STRUCTURE	SELECTIVITY	REMARKS
Citric acid (2-hydroxypropane-1,2,3-tricarboxylic acid).	 $\begin{array}{l} CH_2-COOH \\ \vert \\ HO-C-COOH \\ \vert \\ CH_2-COOH \end{array}$	Good selectivity with iron ions.	Citric acid can complex Fe(II) and Fe(III) cations. It is used as a depressant, for quartz, mica, carbonates and iron oxide.
Bromopyrogallol Red (5,5'-Dibromopyrogallol sulfonepthalein, BPR).		Good	This reagent in combination with Di-n-octylmethylamine can complex Nb at pH 5.
Tartaric acid (1,2-Dihydroxyethane-1,2-dicarboxylic acid).	 $\begin{array}{l} OH-CH-COOH \\ \quad\ \ \vert \\ OH-CH-COOH \end{array}$	Poor	Tartaric acids forms the most stable complex with Nb.

TABLE 2.4 ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE. (CONTINUATION).

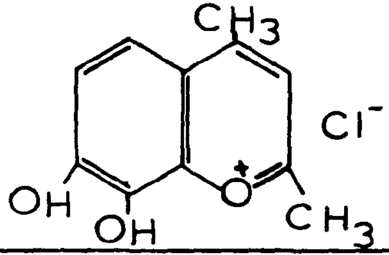
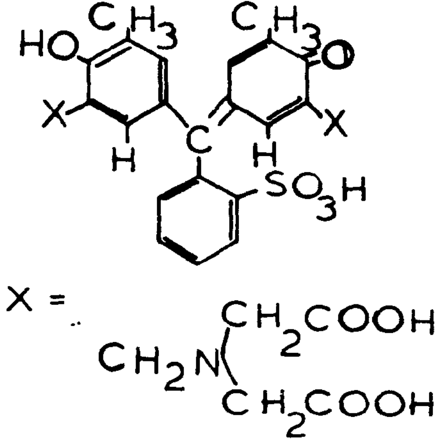
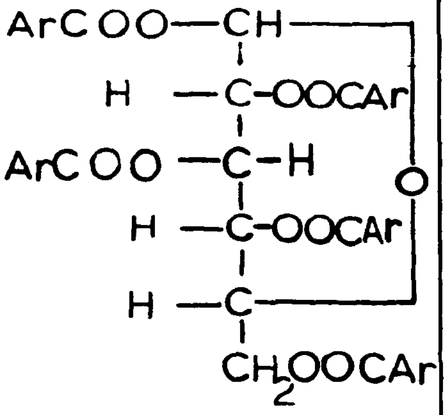
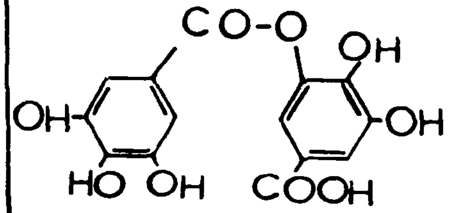
ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE			
NAME	STRUCTURE	SELECTIVITY	REMARKS
o-Dihydroxy chromenols (7,8-dihydroxy-2,4 -dimethybenzopyrylium chloride).		Good	Selective depressant for Ge,Zr,Ti,Th,Mo,W,V,Ta,Nb,Sn.
Xylenol Orange		Poor	Reacts with xylenol orange at pH 2-3.

TABLE 2.4 ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE. (CONTINUATION).

ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE

NAME	STRUCTURE	SELECTIVITY	REMARKS
Tannin (Rose Tannin).	 Ar, indicates the residue of the metadigallic acid, which has the following formula: 	Good	Ta,Ti(IV) and Nb are quantitatively precipitated by tannin from weakly acid oxalate solutions at pH 4.5. Al and Fe are not precipitated. Separation is even better in presence of complexone III .

Some interesting points are 1) none of the reagents is selective for Nb only (although selective complexing gangue depressants to be used in conjuction are recommended in some cases); 2) for most of the reagents, Fe(III) is a major interferant; and 3) some reagents have apparently good selectivity but only within a very narrow pH range, for example, the N-benzoyl-N-phenyl-hydroxylamine (BPA) can react selectively with Nb but only at pH 3.5-3.6 and using tartarate and ethylene-diamine-tetraacetic acid (EDTA) to mask Al, Ca, Mg and Fe(III). Table 2.4 presents some organic depressants apparently selective for Nb. This could permit inverse pyrochlore flotation. Among them are pyrocatechol violet, tannin (at pH about 4.5) and bromopyrogallol red.

From the observations in Tables 2.3 and 2.4 the following flotation routes to achieve selective pyrochlore flotation were considered:

A) Direct flotation of pyrochlore (with prior carbonate flotation); B) Inverse flotation of pyrochlore (with prior carbonate flotation), i.e., floating the silicates; C) Direct flotation of pyrochlore (without prior carbonate flotation).

A) Direct flotation of pyrochlore (with prior carbonate flotation): One of the most important points noted from Table 2.3 is the need for low pH to achieve any selectivity. Since up to 70% of the Niobec ore is carbonate, which is soluble in acid, it is necessary to remove the carbonates prior to lowering the pH (as is done at Niobec for this reason, Chapter 1). After the removal of as much of the carbonates as possible, the main problem would be the selectivity between pyrochlore and the silicates. Pyrochlore/silicate separation was contemplated using the following routes:

1) Pyrochlore flotation with N-benzoyl-N-phenyl- hydroxylamine and tartarate and ethylenediamine tetracetic acid (EDTA) as silicate

depressants;

2) pyrochlore flotation with the different Nb-collectors (Table 2.3) using citric acid as silicate depressant, and also to mask Fe(III). The use of citric acid as an Fe complexing agent to produce a hydrophilic compound is suggested in Table 2.4. In addition, citric acid acts as a depressant for quartz, mica, carbonates and iron oxides (Cyanamid, 1976). It is also quite important to find the best pH for separation with each individual reagent. For example, Table 2.3 suggests optimum pH conditions for alkyl hydroxamates, phenyl arsonic acid and 8-hydroxyquinoline, as 7, 4.8, and 4.0, respectively. These potential Nb-collectors were also tested using other silicate depressants, e.g. sodium silicate, a copolymer of acrylamide (DK813, from Allied Collids), tannic acid, tartaric acid, and DA811 (an anionic dispersant from Allied Colloids).

B) Inverse flotation of pyrochlore (with prior carbonate flotation): The inverse flotation of pyrochlore from silicates also looks attractive. At all pHs above 4.5 the silicates present in the ore bear a negative charge. They can then be floated using cationic collectors while depressing the pyrochlore. This approach was contemplated using the following routes:

1) depression of pyrochlore using the more selective Nb depressants, as listed in Table 2.4, such as tannin, pyrocatechol violet and bromopyrogallol red; followed by cationic flotation of silicates. The pH has to be above 4.5;

2) depression of pyrochlore using less selective Nb depressants, such as tartaric acid, followed by cationic flotation of silicates at pH above 4.5.

C) Direct flotation of pyrochlore (without prior carbonate flotation). This approach was contemplated using two types of depressants, sodium hexametaphosphate to depress carbonates and apatite, and citric acid to depress silicates. Pyrochlore flotation was to be attempted using hydroxamic acid-based Nb collectors at pH 5.

Subsequent to completing the work other Nb chelating agents that could be used as collectors were identified-- e.g. cinnamyl hydroxamic acid (Thiele and Pickard, 1899; Majumdar and Mukherjee, 1960). The idea behind this hydroxamic acid was to to provide a longer hydroxamic chain in an attempt to reduce the high reagent consumption required for some hydroxamic acids tested (e.g. benzohydroxamic acid, see manuscript). Others were methyl-benzyl arsonic acid (Luo and Chen, 1985); potassium octyl hydroxamate (Fuerstenau and Pradip, 1984); iso-monyl imino-bis-methylene phosphonic acid and dodecyl imino-bis-methylene phosphonic acid (Collins et al, 1984).

There are clearly many more additional combinations than can be tested during this work. As will become clear problems encountered in conducting column flotation in the laboratory further reduced the number of combinations which could be evaluated.

2.5 MICROFLOTATION TESTS

The manuscript gives most of the details on the microflotation tests, which were conducted using many of the reagent combinations suggested in the previous section. (Additional details are given in Appendix 1, Reports 4 and 5). All the tests were conducted using plant water to try to simulate plant conditions. (Appendix 1, in the Manuscript also shows the results of further tests conducted in a continuation of the study of the effects of the water chemistry on pyrochlore and silicate flotation/depression).

2.6 MANUSCRIPT 1:

EFFECTS OF THE TYPE OF WATER ON THE SELECTIVE FLOTATION OF PYROCHLORE FROM NIOBEC

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ABSTRACT

The effects on selective flotation of fine (1-16 μm) pyrochlore of two types of water used at the Niobec concentrator, potable and plant recycle, were assessed. Several pyrochlore collectors and silicate depressants were tested.

Water effects were different for each mineral-reagent(s) combination. In general plant recycle water decreased selectivity and increased reagent consumption. Interpretation of results is given in terms of electrokinetic effects, hydrolysis of cations, and mineral-collector interactions. Two techniques of treatment for plant recycle water, cation precipitation and zeolite adsorption, were assessed. The former was found to be more effective under the tested conditions but recycle water continued to give poorer selectivity than the potable.

INTRODUCTION

Water quality plays an important role in selective flotation. Specific effects have been reported by many workers (Rybinski et al, 1984; Somasundaran, 1984; Fuerstenau, 1975; Vreugde and Poling, 1975; Broman, 1980; Hoover, 1980) and in general the better the water quality the more selective is flotation. Water composition depends on origin and the minerals being processed. For example pulp containing salt-type minerals (calcite, apatite, dolomite) will contain more Ca and Mg cations than that from a sulphide deposit. Another source of water contamination is the flotation reagents added to the pulp, particularly if water recycling is practiced.

At the Niobec concentrator the water supply comes from the municipality (potable water) and plant recycle.

The ore composition at Niobec is very complex (see Table 1), as is the flowsheet used to recover the Nb-containing minerals, pyrochlore and columbite (Biss, 1984a). This flowsheet comprises desliming, carbonate flotation, magnetic separation, pyrochlore flotation at six different pHs, two sulphide flotation stages and leaching. Together pyrochlore and columbite represent only about 1% of the total mass of the ore. The most abundant minerals are carbonates (mainly dolomite and calcite), silicates (biotite, chlorite and Na,K,feldspars) and to a lesser extent apatite. Other constituent minerals are magnetite, hematite, pyrite and zircon. Selectivity is hindered by the similar flotation characteristics of the pyrochlore and the two main contaminants, carbonates and silicates. The silicates for example have electrokinetic properties similar to pyrochlore (see Table 1).

TABLE 1. MINERALOGICAL COMPOSITION OF NIOBEC ORE.

MINERAL	Wt. (%)	IEP
CARBONATES	65	
DOLOMITE		7
CALCITE		8.2
ANKERITE		
SULPHIDES	1	
PYRITE		1.5
PYRRHOTITE		
OXIDES		
PYROCHLORE AND COLUMBITE (10:1)	1	2.6
MAGNETITE, HEMATITE AND ILMENITE.	2	6
APATITE	7	5
ZIRCON	.2	5.8
SILICATES	21	
BIOTITE		1.5
CHLORITE		4.5
Na,K,FELDSPARS		1.8

The isoelectric point (IEP) of pyrochlore is about 2.6, biotite about 1.5, chlorite about 4.5, and Na,K feldspars about 1.8. These close values limit selective flotation based on collector adsorption by electrostatic mechanisms. In addition to the close IEP between pyrochlore and silicates, their compositions are not fixed causing variations in their IEP. Typical Nb recoveries are of the order of 70% with about 15% Nb lost in the slimes rejected before flotation (Biss, 1984a).

Potential routes to improve Nb recovery in the overall circuit are by: a) increasing Nb recovery in pyrochlore flotation and b) recovering some of the Nb lost in the slimes. Both approaches require more selective flotation systems. Improved selectivity may permit direct pyrochlore flotation with the possible advantages of a) higher Nb-recoveries, b) possibly a lower reagent consumption, c) simpler circuits.

Niobium recovery from the slimes is being studied. The investigation involves five phases a) characterization of the slimes (composition and particle size distribution); b) determination of electrokinetic properties of pyrochlore and the principal silicate contaminants; c) evaluation of selective Nb-flotation reagents based on chemisorption rather than electrostatic interactions; d) micro-flotation testing of the reagents with single minerals; and finally e) column flotation testing of slimes with flotation conditions identified in d). All testwork is being performed using potable and recycle water obtained from the plant. Results from phases b), c) and d) are presented here.

EXPERIMENTAL METHODS AND MATERIALS

Water treatment.

Two techniques of recycle water treatment, Ca precipitation and zeolite adsorption, were assessed.

Precipitation was achieved by adjusting pH with oxalic acid to values of 3.6 or 1.9 and removing the precipitates produced. Zeolite adsorption consisted of adding 3.25 g of zeolite (theoretical zeolite exchange capacity for Ca and Mg plus 25%) per liter of water and stirring for 5 or 10 minutes at a pH of 10.5 or 7.2. A commercially available zeolite (Aldrich Chemical Co) identified as 'zeolite mixture' was selected.

Sample Preparation

Pyrochlore, feldspar (microcline) and biotite were obtained from Niobec for the electrokinetic study. Pyrochlore was isolated from a -150 +106 μm concentrate using heavy liquid separation at s.g.= 2.9. The sink (pyrochlore) fraction was washed repeatedly with acetone to dissolve any residual heavy liquid. Feldspar and biotite were upgraded using a Frantz Isodynamic Magnetic Separator. Purity was checked by X-ray diffraction (XRD, Philips PW 1710) and microscopic examination; no contaminants were found. Silicate samples for microflotation were obtained from Ward's Scientific Co.. Their electrokinetic properties were similar to those of the Niobec samples.

Electrophoretic mobility.

The electrokinetic study was conducted using a Mark II micro-electrophoresis apparatus (Rank Brothers) (Collins and Jameson, 1976). Electrophoretic mobility of the particles was measured at pH values 2 to 8.2, adjusted with oxalic acid ($C_2H_2O_4$) or fluosilicic acid (H_2SiF_6). Particle size was 1 to 3 μm. Precipitates formed upon treating recycle water were also tested.

Microflotation.

A microflotation cell specially designed for fine particles was used, Figure 1. The design is similar to that proposed by Siwek et al (1981), but with a modified feed location. Single mineral samples were used during the tests. The particle size range was 1-14 μm (close to the size range of the slimes). The optimum cell operating conditions were sample size, 0.5 g, air flow rate, 74 mL/min and froth depth, 4 cm. The test procedure involved preparation of the pulp (0.5 g of mineral and 40 mL of water), pH adjustment with oxalic acid or potassium hydroxide, and addition of the reagent(s) with further pH adjustment as necessary. After conditioning (for 4 or 10 mins, depending on reagents) the pulp was transferred to the microflotation cell with the air already on. Level was adjusted with the test water containing frother. During flotation the froth level was continuously adjusted. Normally 3 floated products were collected in each test, at 1, 2 and 4 minutes of flotation, as well as the non-floats. The results are reported as mass remaining in the cell (%) vs. volume of liquid recovered to floats, instead of flotation time as flotation time, gave a less consistent correlation.

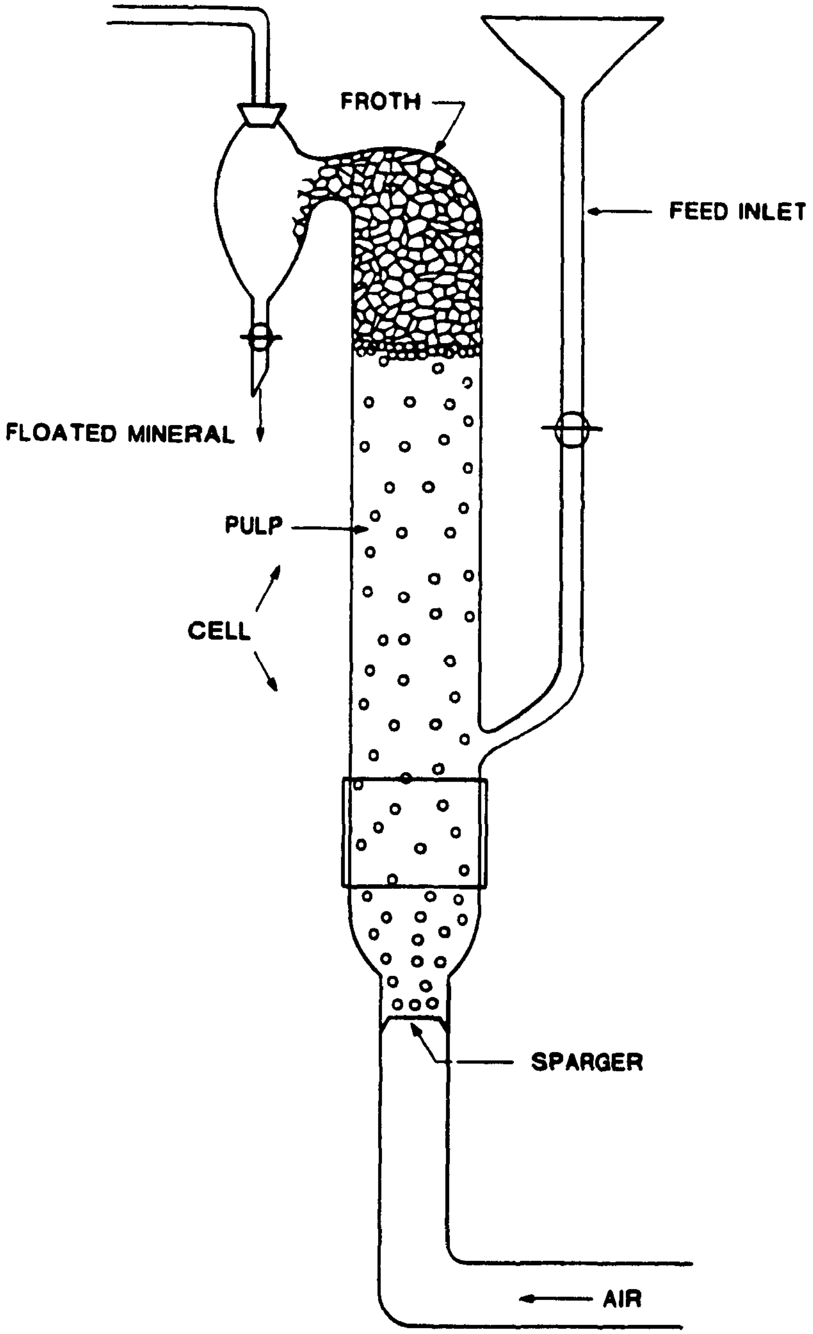

Figure 1. Microflotation cell

Selective Nb flotation reagents.

The search for selective reagents for pyrochlore has been attempted before without great success (Bogdanov et al, 1973; Pickett, 1959; Kennecott Copper Corp., 1958). However, many potential reagents remain to be tested (Moshier, 1964; Buser et al, 1970; Gibalo, 1970; Charlot and Bezier, 1960; Laitinen, 1960; Cheng et al, 1982; Biss and Nadeau, 1982; Faucher, 1964; Abeidu, 1974; Gorlovskii et al, 1966; Burt et al, 1982). In this work, two basic groups of reagents were tested, listed in Table 2, one suited to the flotation of pyrochlore, the second suited to the selective depression of silicates.

F-2642, CA-540 and BHA are readily soluble in water; BPA and oxine are not. BPA was dissolved in ethyl alcohol and diluted with water. The oxine was dissolved with acetic acid and diluted with water. Apart from the three depressants listed, tannic acid, hexametaphosphate, citric acid, EDTA, tartaric acid, starch, DA811 (Allied Colloids), DA813 (Allied Colloids, 1982), AQ55D (from Eastman Chemical Products) and sodium silicate were also tested.

The criteria for optimum pH and reagent(s) addition were as follows: a) select best pH for pyrochlore flotation using a given collector addition, b) at the best pH select minimum collector addition required to float pyrochlore, and c) select maximum depressant addition that does not affect pyrochlore flotation. The concept behind b) and c) is to work in a very sensitive flotation regime to facilitate identification of any effect on pyrochlore selectivity. Conditioning time was 3 minutes for F-2642 and 10 minutes for the others; conditioning times were established by tests on pyrochlore.

TABLE 2. FLOTATION REAGENTS REPORTED IN THIS WORK

NAME	PYROCHLORE COLLECTOR			
	TYPE	SELECTIVITY / REAGENT CONSUMPTION **		pH
		POTABLE	RECYCLE	
* AMINE ACETATE (F-2642) WITH DK-813	CATIONIC	V.G. / 1	V.G. / 1	2-4
BENZOHYDROXAMIC ACID (BHA)	ANIONIC	V.G. / 100	V.G. / 300	6
N-BENZOYL-N-PHENYLHYDROXYLAMINE (BPA)	ANIONIC	G / 21	G / 21	3.5
SULFOSUCCINATE (CA-540) WITH TARTARIC ACID	ANIONIC	V.G. / 1	G / 4	9
	SILICATE DEPRESSANT			
* POLYACRYLAMIDE (DK-813) WITH F-2642	CATIONIC	V.G.	V.G.	2-4
TANNIC ACID WITH F-2642	ANIONIC	V.G. / 2.5	F / 1.5	6
TARTARIC ACID WITH CA540	ANIONIC	V.G. / 10	G / 10	9

* REAGENT USED AT NIOBEC

** V.G. = VERY GOOD, G = GOOD, F = FAIR, P = POOR
CONSUMPTION IN Kg / TONNE

RESULTS

Water treatment

Table 3 shows the analysis of the as-received potable and recycle water samples. The major difference is the much higher Ca (199 vs 26 ppm), Mg (98 vs 1 ppm) and sulphate (110 vs 4 ppm) content of the recycle water.

Table 4 shows the Ca, Mg content after treatment, a) by precipitation with oxalic acid, b) by zeolite adsorption. Precipitation at pH 3.6 has a minor effect; at pH 1.9 the concentration of Ca^{++} decreases to less than 10 ppm although that of Mg^{++} is little affected. The precipitate formed was identified by XRD as Ca oxalate. Zeolite adsorption showed little effect; 10 mins stirring at pH 10.5 did reduce the concentration of Mg^{++} less than 50 ppm.

Electrophoretic mobility.

Mobility vs. pH curves were obtained for pyrochlore, microcline and biotite using potable water with oxalic acid and fluosilicic acid. Figure 2 shows the curves obtained with oxalic acid. The IEP for pyrochlore, feldspar and biotite were 2.5, 2.0 and 1.5 using oxalic acid, and 2.4,2.0 and 1.9 using fluosilicic acid. The mobility for both microcline and biotite was about the same; pyrochlore mobility was consistently lower.

Oxalic acid could not be used with recycle water due to the large amount of precipitate formed. Figure 3 shows the mobility vs. pH for pyrochlore, microcline and biotite using recycle water and fluosilicic acid. Figure 3 also includes for comparison the pyrochlore mobility using potable water. Note that: a) the absolute electrophoretic mobility values for potable water are in general higher than when using recycle wate., b) recycle water

TABLE 3. WATER ANALYSIS

ANALYSIS (ppm)	POTABLE WATER	RECYCLE WATER
Total alkalinity in CaCO.	57	140
Bicarbonate in CaCO.	1	1
TOC (*)	0.1	2.1
Phosphate	0.03	0.03
Sulfate	4	110
Aluminium	0.1	0.1
Calcium	25.6	199
Iron	0.02	0.02
Magnesium	1.3	98
pH	6.9	7.3

(*) Total organic carbon

TABLE 4a. ANALYSIS OF RECYCLE WATER
TREATED BY PRECIPITATION

TYPE OF TREATMENT	ELEMENT (ppm)	
	Ca	Mg
UNTREATED	199	98
TREATED pH 3.6	116	97
TREATED pH 1.9	9	92

TABLE 4b. ANALYSIS OF RECYCLE WATER
TREATED WITH ZEOLITES

FIXED CONDITIONS : 3.5 g OF ZEOLITE / 1

STIRRING TIME (min)	pH	ELEMENT (ppm)	
		Ca	Mg
UNTREATED	7.1	199	98
5	10.5	196	83
10	7.2	196	73
5	7.2	200	59
10	10.5	200	48

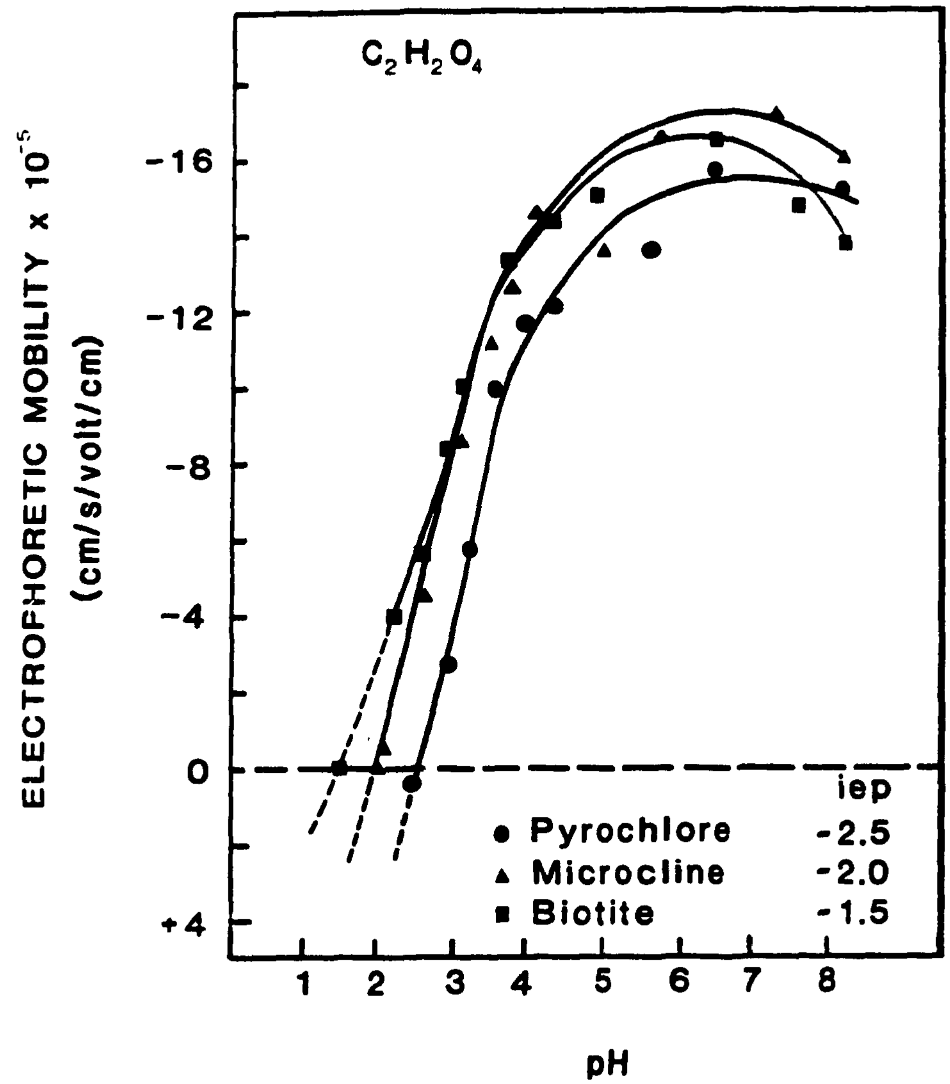

Figure 2. electrophoretic mobility vs pH curves using potable water.

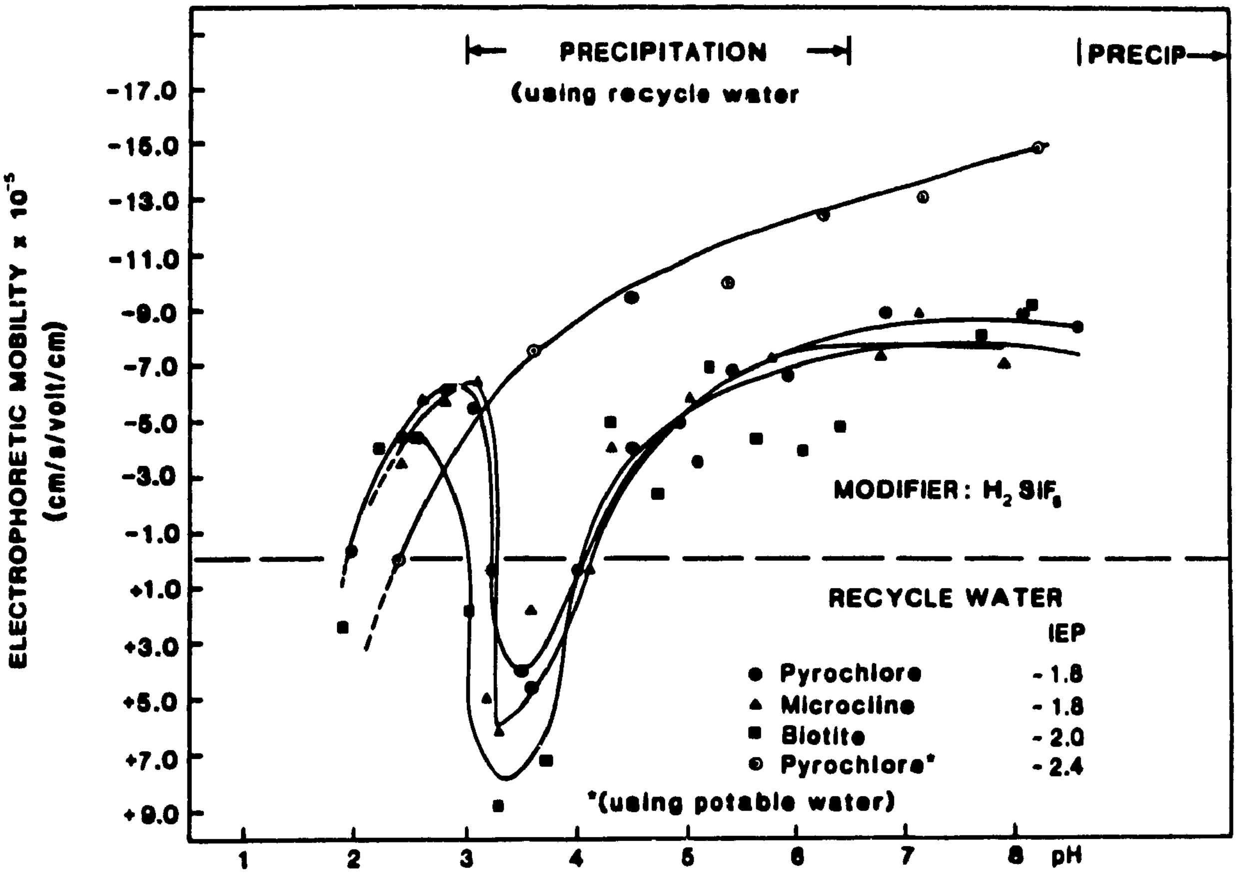

Figure 3. Electrophoretic mobility using recycle water.

reverses the zeta potential of the three minerals, from negative to positive in the pH range 3 to 4.2, c) using recycle water the three minerals become electrokinetically indistinguishable, d) recycle water produced precipitates with H_2SiF_6 in the pH range 3 to 6.5, and above pH 9, and e) the lowering and reversal in the zeta potential is closely related to the formation of precipitates.

Figure 4 shows the mobility of the precipitate formed at pH 3-6.5 along with pyrochlore (Figure 3) as a function of pH. The precipitates show a similar charge reversal.

Flotation.

The flotation results represent the most selective flotation conditions found for pyrochlore vs the silicates. For pH modification oxalic acid was selected because fluosilicic acid reduced pyrochlore flotation. Of the silicate depressants tested, only the three in Table 2 proved effective.

Amine acetate (F-2642).

Figure 5 shows flotation as a function of pH and type of water with F-2642 and a polyacrylamide copolymer (DK-813). Both reagents are currently in use at Niobec. In this case pyrochlore selectivity was tested vs microcline only, because this silicate is the one which causes problems with this reagent combination (Biss, 1984b). Flotation behavior is quite different between potable water and recycle water. In general selectivity is increased as the pH is decreased below about 5.

Figure 6 shows the flotation of pyrochlore, microcline, chlorite and biotite with F-2642 and tannic acid, using potable water. Selectivity was achieved only with potable water. During the tests with recycle water a

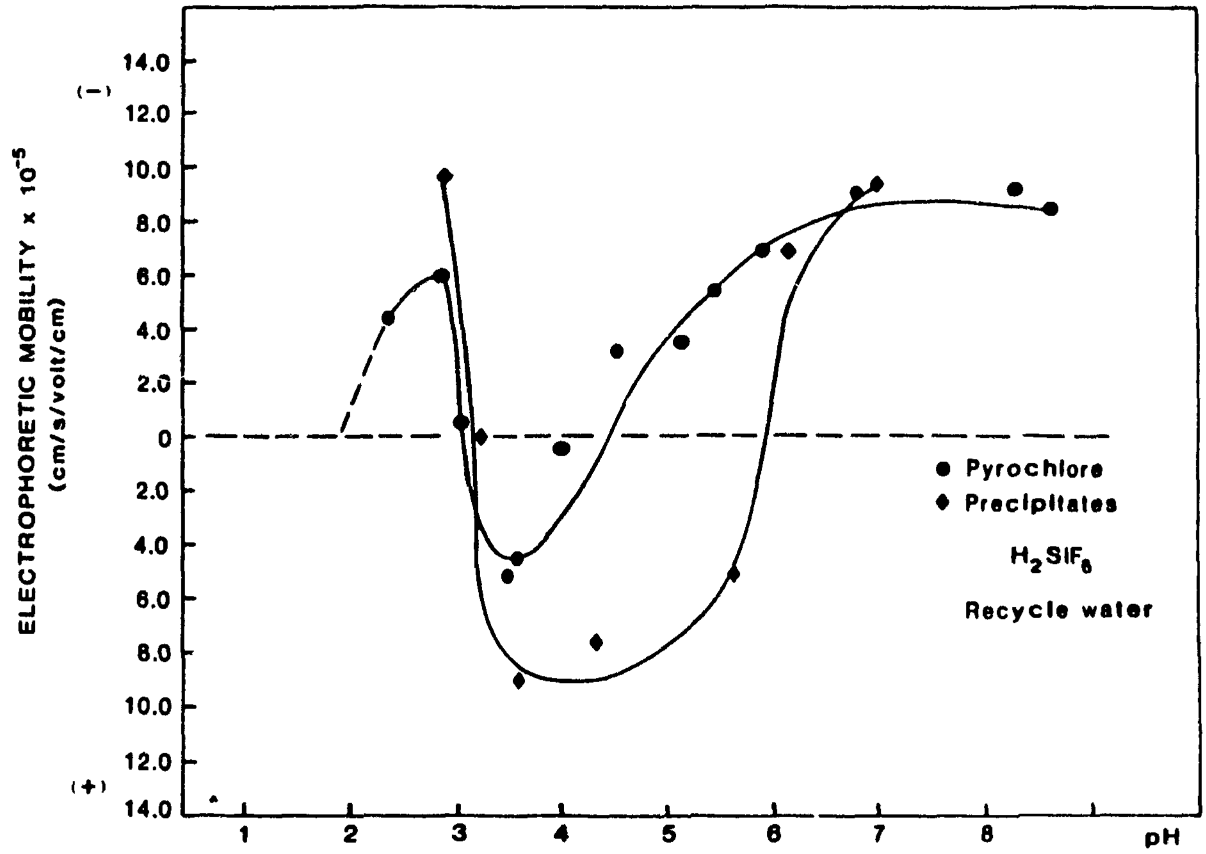

Figure 4. Electrophoretic mobility of pyrochlore and precipitates using recycle water.

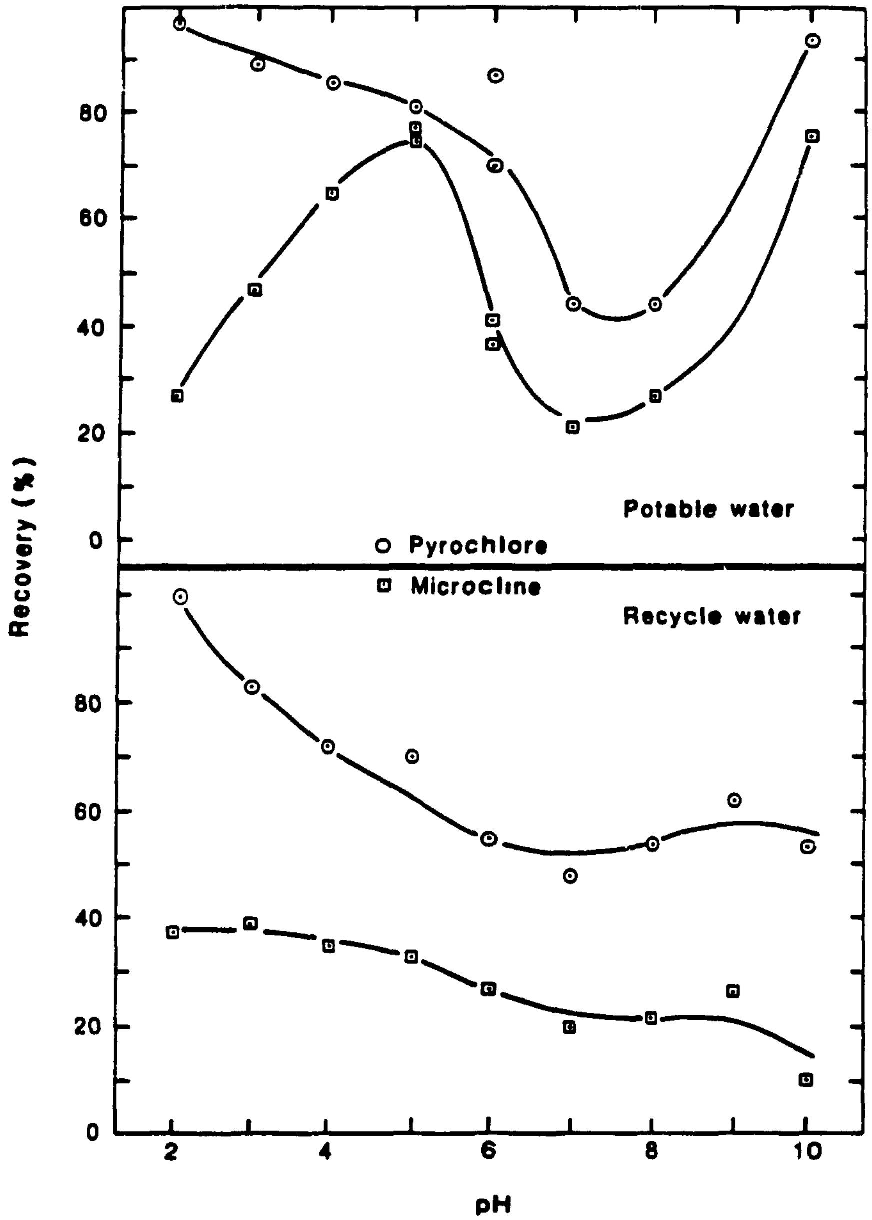

Figure 5. Recovery vs pH for pyrochlore and microcline with the collector F-2642 (1 kg/tonne) and the depressant DK813 (5 kg/tonne), and using two types of water.

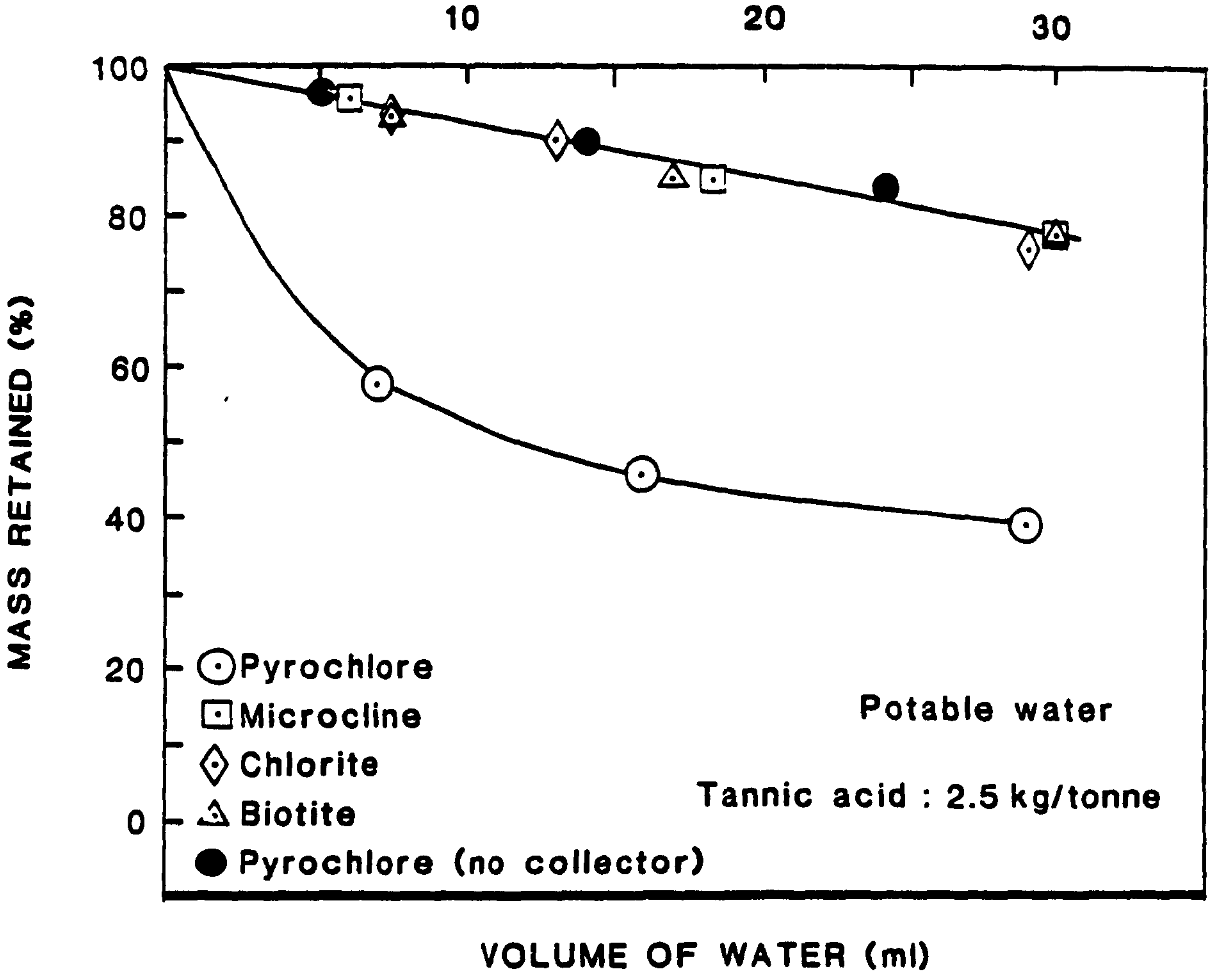

Figure 6. Flotation performance with the collector F2642 (1 kg/tonne) and the depressant tannic acid using potable water at pH 6. The filled circles represent pyrochlore recovery by entrainment.

lower addition of tannic acid (1.5 kg/tonne instead of 2.5 kg/tonne) was required to avoid depression of all minerals. The filled circles represent pyrochlore recovery by entrainment, suggesting this mechanism is also the cause of microcline, chlorite and biotite recovery in potable water.

Figure 7 shows the results of flotation using recycle water treated by precipitation, a) at pH 1.9, b) at pH 3.6. The full lines indicate a) flotation with potable water and b) flotation with untreated recycle water, for comparison. Results for treated recycle water at pH 1.9 approach the results for potable water. Results for treated recycle water at pH 3.6 appear similar to the untreated recycle water.

Benzohydroxamic acid (BHA). Figure 8 shows the flotation performance using BHA with potable and recycle water. Selectivity was achieved even without depressants. The effect of recycle water was to increase the BHA consumption from 100 kg/tonne to 300 kg/tonne. However, in either cases consumption is excessive.

N-benzoyl-N-phenyl-hydroxylamine (BPA). Figure 9 shows the flotation performance using BPA with potable and recycle water. In both cases selectivity was achieved against microcline and biotite, but not chlorite. Several depressants for chlorite were tested but none was effective.

Sulfosuccinate (CA540). Figure 10 shows the flotation performance with CA540 and tartaric acid as a depressant with potable and recycle water. Recycle water in this case increased CA540 consumption (from 1 kg/tonne to 4 kg/tonne) and enhanced microcline and chlorite flotation.

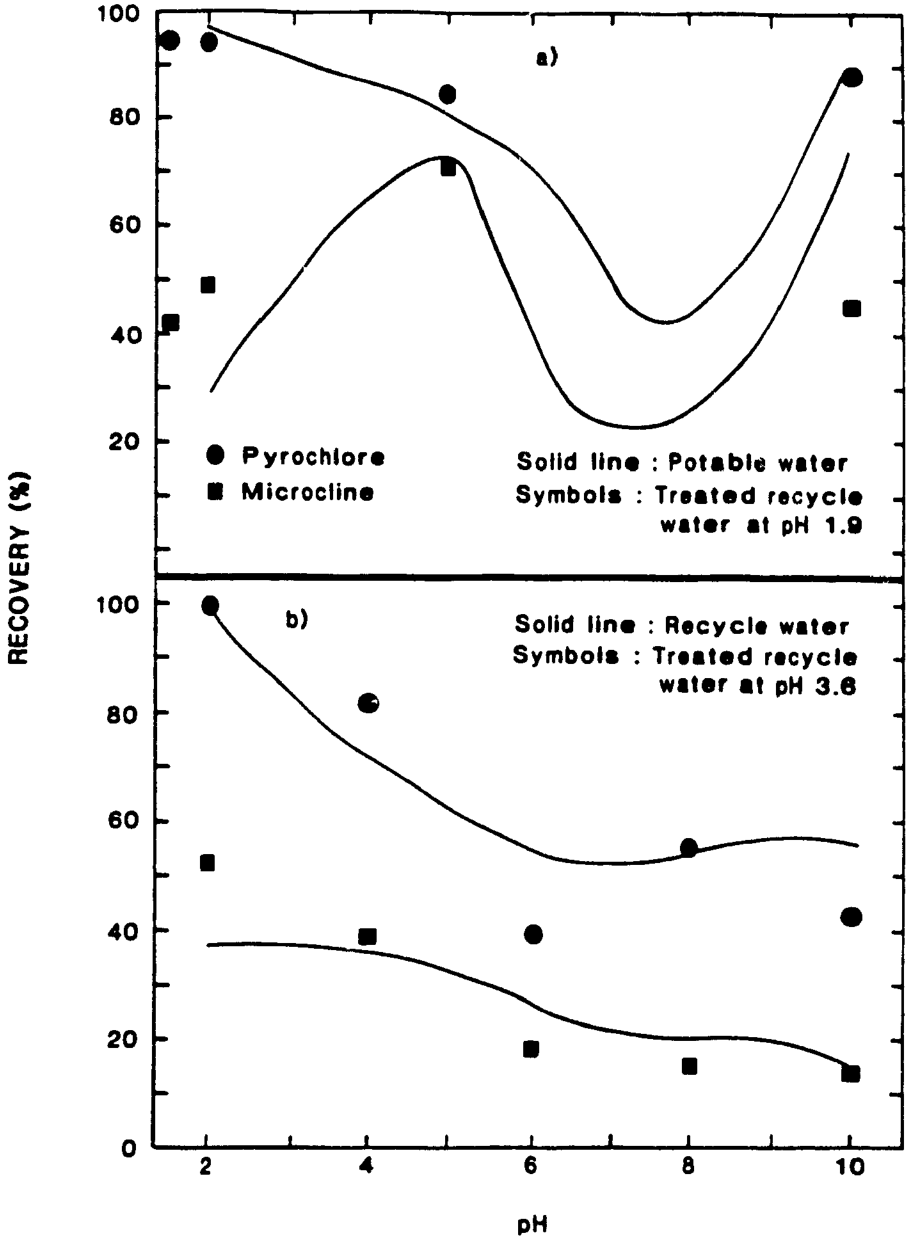

Figure 7. Recovery vs pH for pyrochlore and microcline with F-2642 (1 kg/tonne) and DK813 (5 kg/tonne), and using four types of water.

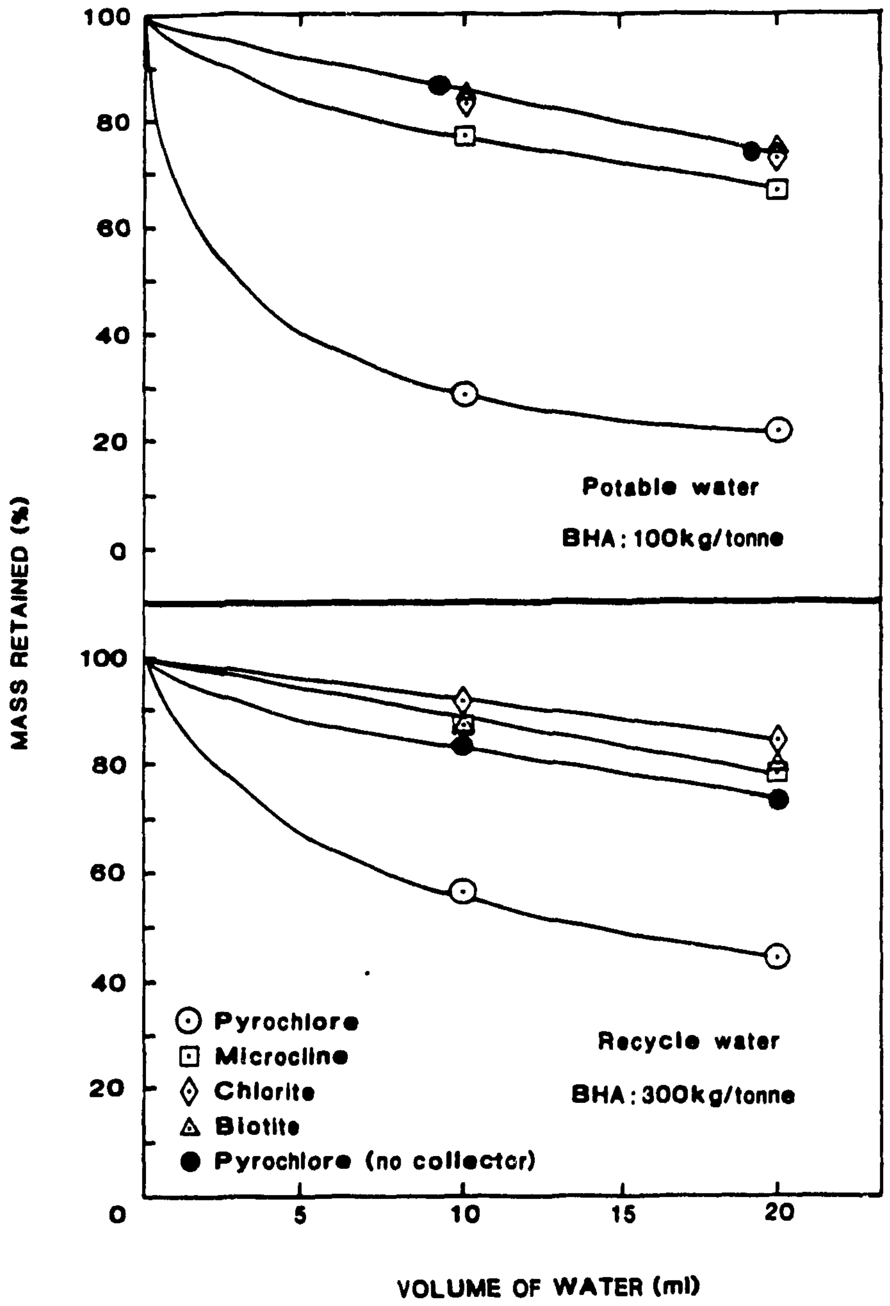

Figure 8. Flotation performance with the collector Benzohydroxamic acid (BHA), no depressant, and 0.1 % oil (volume basis on the pulp). Two types of water were used, at pH 6.0. The filled circles represent pyrochlore recovery by entrainment.

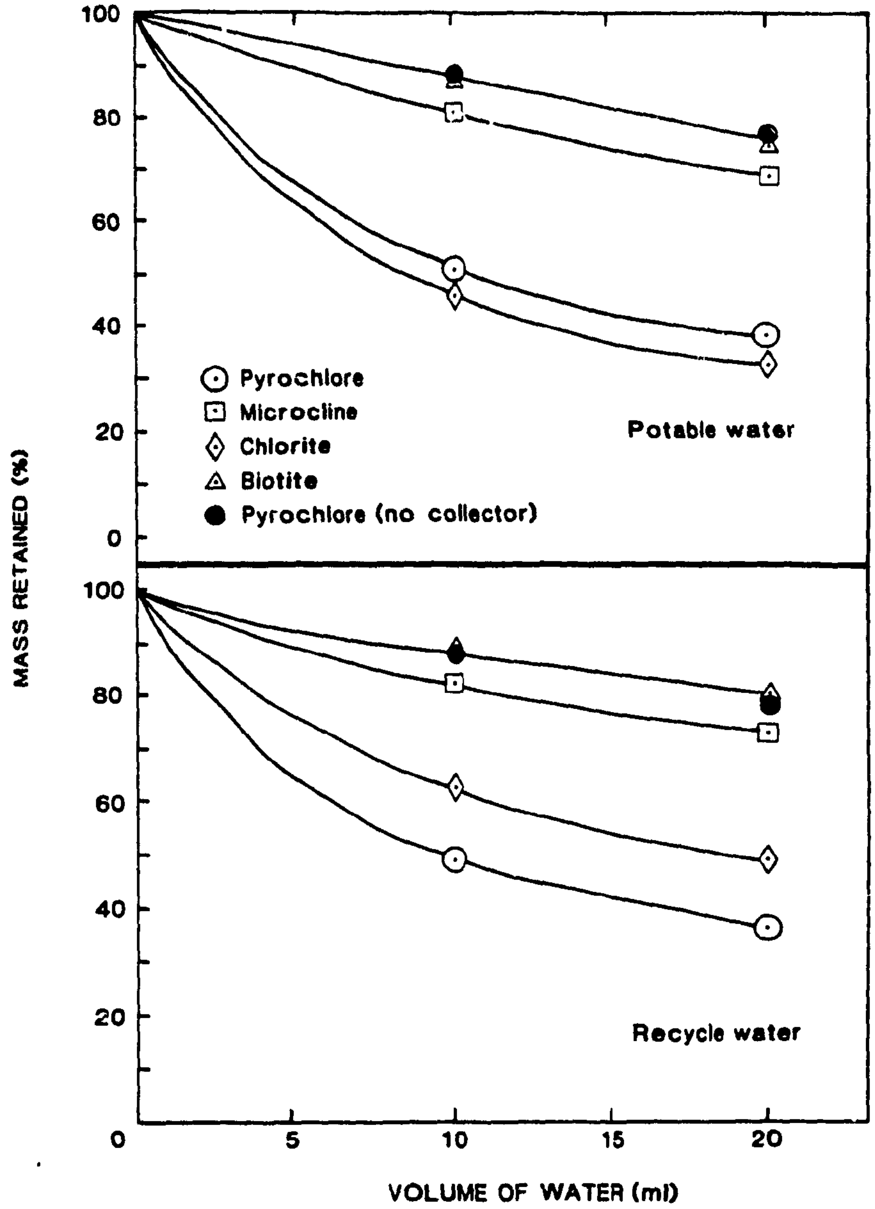

Figure 9. Flotation performance with the collector N-benzoyl- N-phenyl hydroxylamine (BPA) (21 kg/tonne), no depressant, and 0.1 % oil (volume basis on the pulp). Two types of water were used at pH 3.6. The filled circles represent pyrochlore recovery by entrainment.

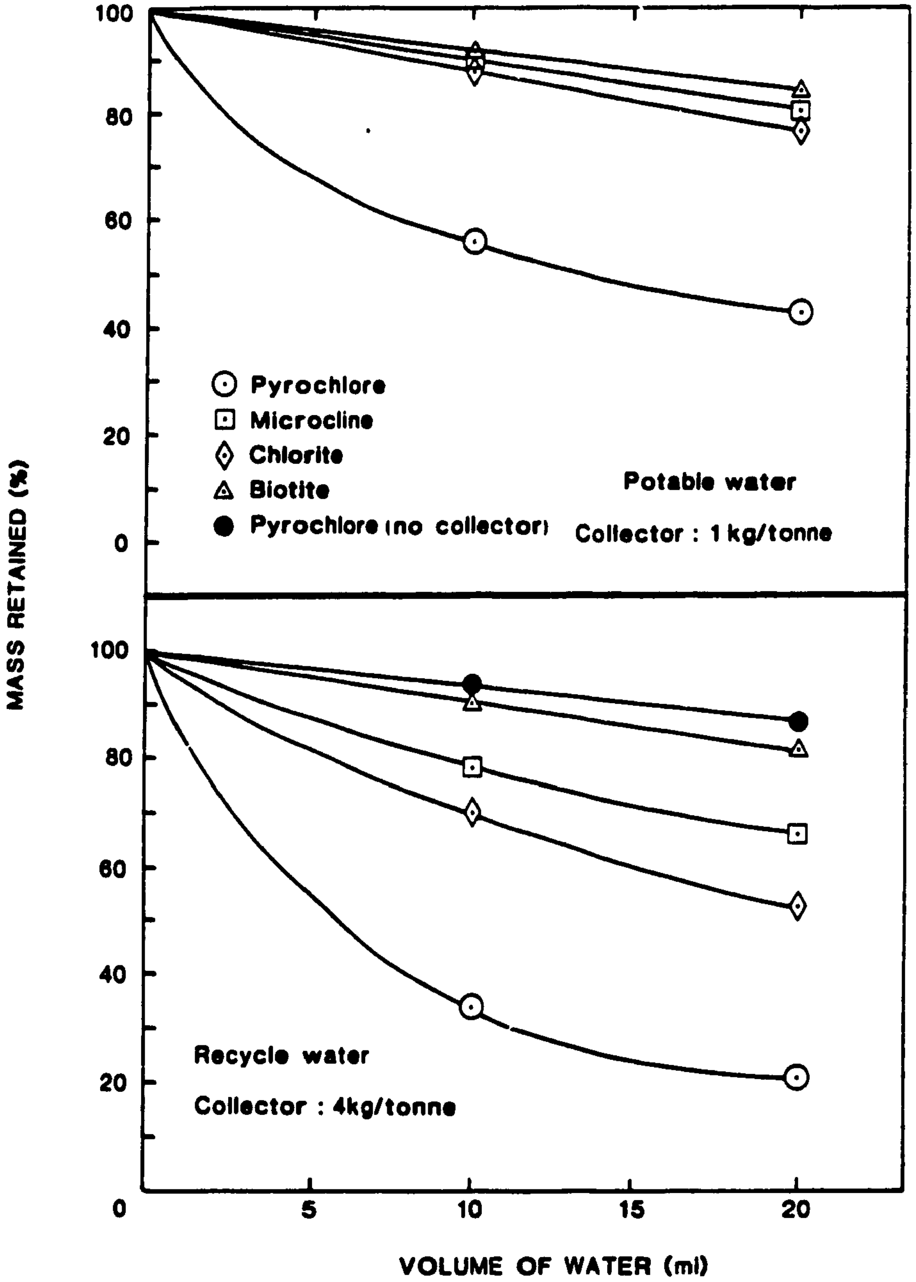

Figure 10. Flotation performance with the collector sulfosuccinate (CA540), and tartaric acid (10 kg/tonne) as a depressant.
Two types of water were used at pH 9.
The filled circles represent pyrochlore recovery by entrainment.

DISCUSSION

Electrophoretic mobility

The higher concentration of cations in the recycle water, 199 ppm vs 26 ppm for Ca and 98 ppm vs 1 ppm for Mg, plays a major role in the lowering of the electrophoretic mobility of the particles. The reversal in the zeta potential is probably related to the high concentration of these cations.

The fact that recycle water causes the three minerals to become electrokinetically similar is detrimental to selective flotation using physisorbed collectors. However, this effect may be reduced using chemisorbed reagents either as selective depressants for the silicates, or as collectors for pyrochlore.

The reversal in the zeta potential (from negative to positive) could be a source of pyrochlore losses at Niobec, since a cationic collector is used.

The presence of precipitates probably alters the zeta potential. These precipitates may adsorb on the mineral surface converting the zeta potential to that of the precipitate substance. This is known to occur and has been suggested as a means of metal ion removal from contaminated water (Broman, 1975). This effect is suggested by Figure 4 where pyrochlore mobility follows the same trend as that of the precipitates. Adsorption of precipitates may be more significant in this static electrokinetic study than in the dynamic flotation system. Consequently extrapolation to flotation must be done cautiously.

Flotation

F-2642 and DK-813 (Niobec Reagents). Figure 5 shows the importance of the type of water in controlling flotation performance. With potable water, flotation selectivity is achieved at a pH below 3. At pH 6 to 8 both pyrochlore and microcline are depressed; at pH 10 both pyrochlore and microcline float well but without selectivity. The depression at pH 6 to 8 may be related to a maximum in the degree of ionization of the depressant, but this has yet to be closely examined.

Recycle water induced a similar flotation response for pyrochlore and microcline, although there is some selectivity as pyrochlore is consistently more floatable. Again, the acid pH range shows the best selectivity. At pH 10 both pyrochlore and microcline flotation is suppressed. This is probably due to the high concentration of Ca and Mg cations in the recycle water. These cations are specially active above pH 10, reducing the negative charge and consequently the collector electrostatic interaction with mineral surfaces (Fuerstenau and Palmer, 1976).

The purpose of treating the recycle water was to improve selectivity, especially at acid pH values. Recycle water treated at pH 3.6 gives little improvement. Treating the water at pH 1.9 shifted the response to one similar to that of potable water. This effect is achieved presumably because of the large removal of Ca cation (from 199 to 9 ppm).

Finally, the adsorption mechanism pyrochlore-F2642 may be partly of a chemical type, otherwise flotation should decrease significantly at pH values from 3 to 4 and below pH 2.

F-2642 and Tannic Acid. Tannic acid depressed selectively the silicates when potable water was used. However with recycle water pyrochlore selectivity was markedly affected. Pyrochlore selectivity was pursued using recycle water treated at pH 1.9, but no substantial increase was attained. This suggests that not only Ca but also Mg plays a significant role in pyrochlore selectivity.

Benzohydroxamic acid (BHA). Recycle water caused a large increase in BHA consumption, although selectivity remained good. The higher consumption could be due to complexation with Ca and Mg cations (Fuerstenau and Pradip, 1984); consumption was not reduced when using water treated at pH 1.9, presumably because Mg was not removed.

The adsorption mechanism in this case may also be partly chemical, to explain this anionic reagent interaction with the negatively charged pyrochlore surface. An important point is that this reagent is very selective for pyrochlore vs microcline, biotite and chlorite, suggesting a collector based on hydroxamic acid may be worth considering. It may be possible to reduce consumption if a larger chain hydroxamate was used, for example octyl hydroxamate (with 8 C atoms)

N-benzoyl-N-phenyl-hydroxylamine (BPA). In this case both types of water produce a similar Nb flotation response (Figure 9). This illustrates the reactivity of this reagent with Nb (Majumdar and Bijoli, 1961). The flotation of chlorite may be due to electrostatic interaction since at pH 3.6 chlorite is positively charged (see Table 1).

Sulfosuccinate (CA540). Recycle water increased reagent consumption (Figure 10). Increased selectivity was attempted using treated recycle water at pH 1.9; no improvement was found.

SUMMARY AND CONCLUSIONS

The effects of two types of water (potable and recycle) on the selective flotation of fine (1-16 μm) pyrochlore mineral from Niobec vs the main silicate contaminants (microcline, biotite and chlorite) was studied using several combinations of pyrochlore collector and silicate depressant. Overall results are summarized in Table 2. In general recycle water decreased selectivity and increased reagent consumption. Recycle water reduces the electrophoretic mobility of the particles and the surface potential of the minerals becomes nearly indistinguishable. Consequently pyrochlore collectors which exploit chemical rather than electrostatic-type interactions are required. The four flotation collectors reported here were selective. Selectivity was further enhanced using the organic silicate depressants DK-813, tannic acid and tartaric acid. Only BHA and BPA did not require silicate depressants to attain selectivity. No depressant was found to be effective to depress microcline when using oxine.

Two types of treatment for recycle water were tested, zeolite adsorption and Ca precipitation using oxalic acid. The zeolite used did not improve selectivity probably because it did not adsorb Ca cations. Precipitation at high oxalic acid concentrations (pH 1.9) removed Ca efficiently but not Mg. The water thus treated this way did regain some of the selectivity of the potable water. With the data available it seems that selective reagents with untreated water, rather than water treatment, offers the best scope for success.

As a result of this research three options are contemplated to selectively float fine pyrochlore from the slimes: using BPA at pH 3.9; using sulfosuccinate with tartaric acid at pH 9; and using the reagent combination used at Niobec. BHA (at pH 6), while technically attractive, is discounted due to high reagent consumption. In all cases a previous carbonate flotation, as conducted at Niobec, is required.

2.7 SUMMARY AND CONCLUSIONS

A search for selective chelating agents for Nb flotation was conducted and several reagents and conditions were suggested. These were evaluated by microflotation tests using pure samples of fine (1-16 μm) pyrochlore mineral from Niobec, and the main silicate contaminants (microcline, biotite and chlorite). Flotation tests were conducted using two types of plant water, potable and recycle. An electrokinetic study on pyrochlore, microcline and biotite was conducted to help interpretation of results.

Main findings were:

1) In general, recycle water decreased selectivity and increased reagent consumption. Recycle water reduced the electrophoretic mobility of the particles and the surface potential of the minerals became nearly indistinguishable. Consequently pyrochlore collectors which exploit chemical rather than electrostatic-type interactions are required.

2) Four of the Nb flotation collectors tested were selective. Selectivity was further enhanced using the organic silicate depressants DK-813, tannic acid and tartaric acid. Only benzohydroxamic acid and N-benzoyl-N-phenyl-hydroxylamine did not require silicate depressants to attain selectivity.

3) Two types of treatment for recycle water were tested, zeolite adsorption and Ca precipitation using oxalic acid. The zeolite used did not improve selectivity probably because it only adsorbed Mg cation to any extent. Precipitation at high oxalic acid concentrations (pH 1.9) removed Ca efficiently but not Mg. The water thus treated did regain some of the selectivity of the potable water.

4) With the data available it seems that selective reagents with untreated water offer the best scope for success.

5) As a result of this research three options are contemplated to float fine pyrochlore selectively from the slimes: using BPA at pH 3.9; using sulfosuccinate with tartaric acid at pH 9; and using the reagent combination used at Niobec. Benzohydroxamic acid (at pH 6), while technically attractive, is discounted due to high reagent consumption. In all cases a previous carbonate flotation step, as conducted at Niobec, is required.

6) There are still many potentially selective reagents that could be tested. As will become clear, problems encountered in conducting column flotation in the laboratory reduced the number of combinations evaluated.

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CHAPTER 3.

PREFLOTATION OF CARBONATES FROM SLIMES USING THE NIOBEC PROCESS: DISPERSION/ FLOTATION STUDY.

3.1 INTRODUCTION

In the previous chapter, three options were suggested to float selectively the pyrochlore from the slimes. All three options required the preflotation of carbonates as is the practice at Niobec (Chapter 1). Column flotation of carbonates using the Niobec procedure was to be used. This chapter describes the column flotation work, including the sample preparation. It will be shown that the column flotation results on the slimes do not match the plant results on the deslimed material. This suggested poor dispersion, the pyrochlore (with about 1% of the total mass) being heterocoagulated with the large (about 60 per cent) mass of carbonates.

Coagulation becomes progressively more difficult to eliminate as particle size decreases, a difficulty enhanced in the present case by the presence of Ca and Mg cations in the plant water, which reduce the surface potential (e.g. see Chapter 2, Manuscript, Figure 3). A dispersion study was therefore initiated. The state of dispersion was monitored using a particle size analyser, which gives a measure of apparent particle size. Details of the method are given. Results from the dispersion study and the effect of dispersants on flotation, and a discussion of the coalescence and froth stability problems which appeared when the dispersants were used in flotation are given in a manuscript. This manuscript is entitled: "Dispersion study on slimes from Niobec" (to be published in the CIM Bulletin). Finally, additional details on the column flotation results are presented using the dispersion conditions selected in the dispersion study.

3.2 SAMPLE PREPARATION

The slimes for the column flotation study were generated from two locations in the plant while processing zone 1 ore, the rod mill feed (- 3.8 cm), and the slimes discard. The rod mill feed was used because it represented fresh unreagentized material while the slimes from the plant are partially reagentized with potentially unknown effects on flotation.

Throughout the preparation, dispersion problems were evident. To help alleviate the problems it was decided to remove the -1 μm fraction (typically 20-30% of the slimes, e.g. see Figure 1.4 in Chapter 1). Thus the objective was production of about 1-10 μm column feed. Subsequent work intended to progressively return the -1 μm fraction to determine the limiting content of this fraction for acceptable flotation performance.

Rod mill feed. 200 kg of ore (- 3.8 cm) was cone crushed to about 0.32 mm and dry ground to 210 μm (65 mesh). Grinding was accomplished in 1 kg lots in a 19 cm x 19 cm Abbe mill with a 10.7 kg ball charge. Grinding was typically for 3 minutes with the product screened at 65 mesh, the +65 mesh returned to the mill. A size by size study of the -210 μm fraction showed that the Nb_2O_5 assay was virtually uniform throughout most of the size range, and slightly lower in the finest size fractions (0.5% vs 0.7% Nb_2O_5, Table 3.1). The 1-10 μm (column feed) was isolated from the -210μm material by cycloning. Figure 3.1a shows the final cyclone configuration used and Figure 3.2 shows the particle size distribution of the column feed, curve 1. This final column feed product represented about 6 % (12 kg) of the initial feed (200 kg).

Slimes. Slimes from Niobec were received at McGill U. in sealed

TABLE 3.1 METAL AND SIZE DISTRIBUTION FOR THE MILL FEED

Product (microns)	Wt (%)	Assay (%) Nb_2O_5	Distribution (%)
Feed	100.0	0.63	
210	0.3	0.08	0.04
210/150	12.2	0.47	9.64
150/106	11.9	0.56	11.20
106/75	14.1	0.64	15.17
75/53	8.8	0.67	9.91
53/45	8.0	0.74	9.65
45/38	5.4	0.71	6.44
-38	39.3	0.57	37.65
Calc. Feed		0.59	100.0
Feed (-38)	39.3	0.57	
38/25	6.3	0.75	7.57
25/15	5.2	0.65	5.41
15/10	10.7	0.64	10.97
-10	17.1	0.50	13.70
Calc. Feed		0.60	

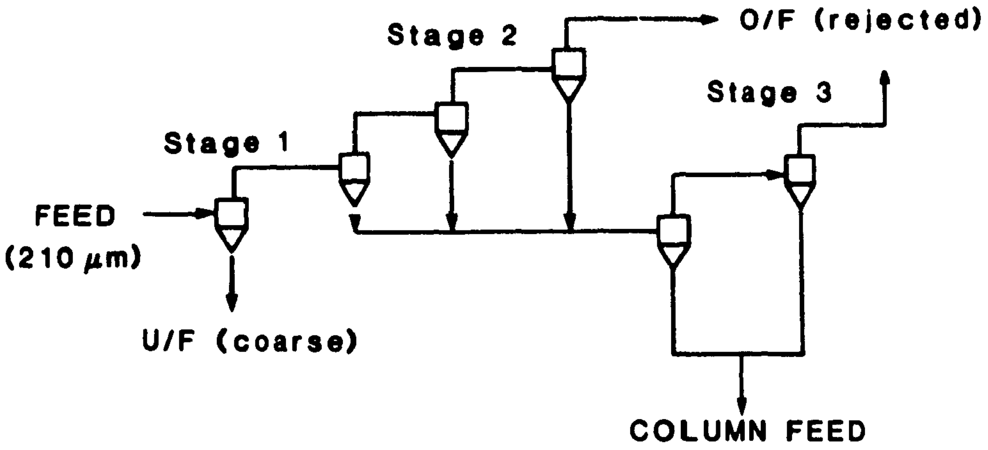

Figure 3.1a Column feed preparation using rod mill feed.

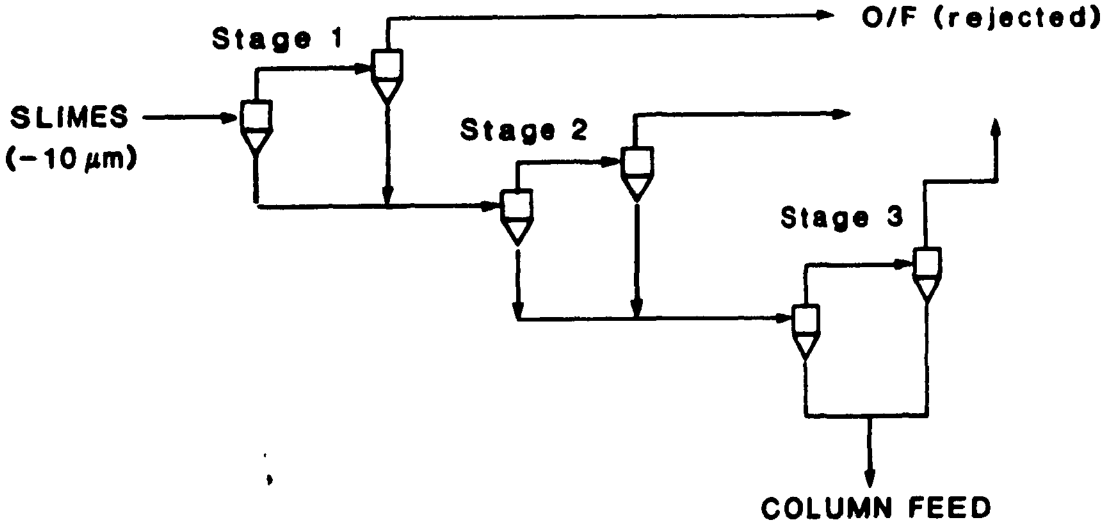

Figure 3.1b Column feed preparation using slimes discard.

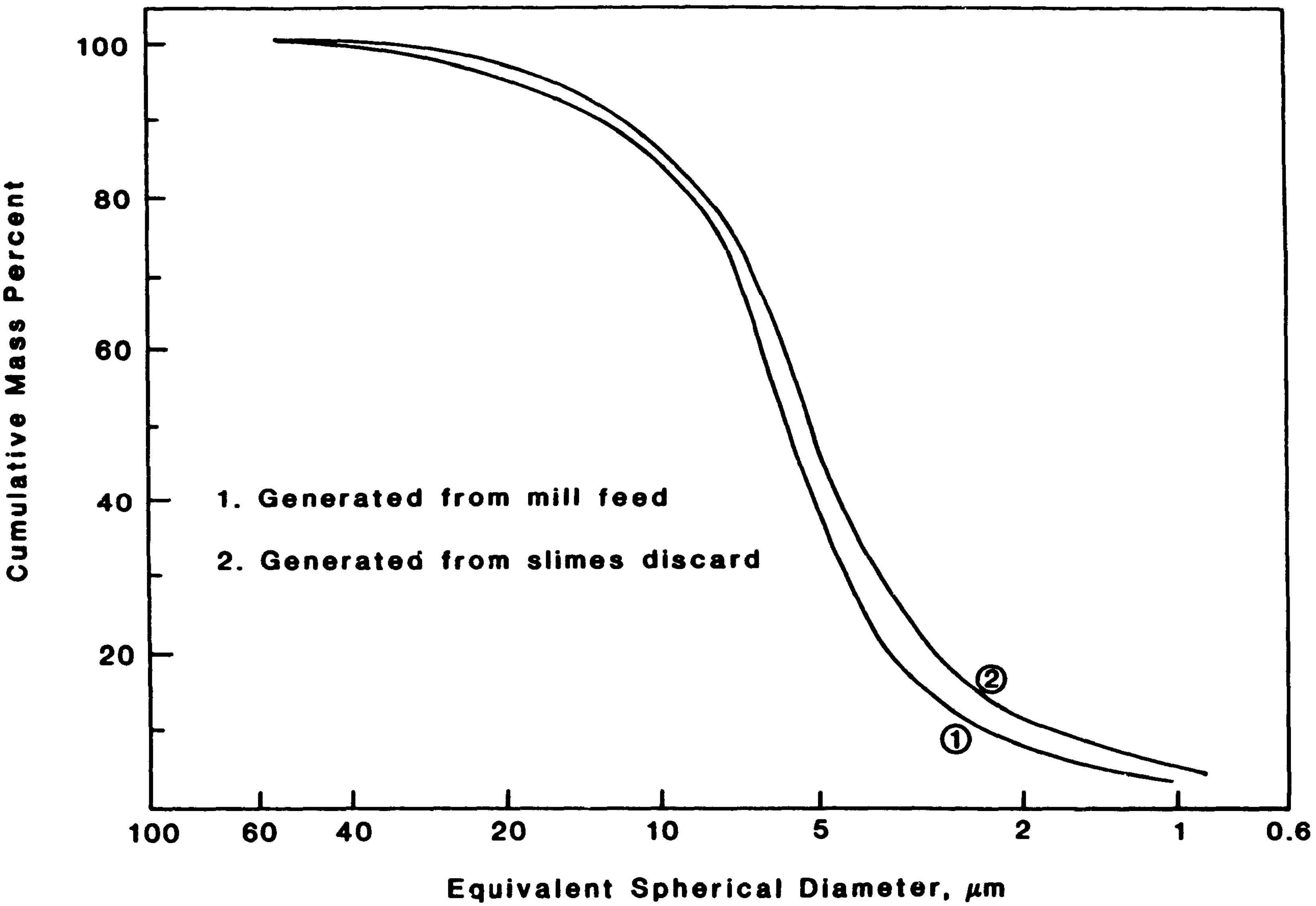

Figure 3.2. Particle size distribution of the column feed (Zone 1)

plastic 200 L drums. The slimes were classified to remove the -1 μm fraction. The cyclone configuration used to remove the -1 μm fraction is shown in Figure 3.1b and the particle size distribution in Figure 3.2, curve 2.

A 5.08 cm (2 inch) hydrocyclone (cyclone) was used to perform the preparation of the column feed. Plitt's equation (1976) was used to guide selection of the desliming conditions. A particle size analyzer, the Sedigraph 5000D (Sedigraph) was used to monitor the particle size distribution (PSD) (particularly the - 1 μm fraction in the final cyclone product). Table 3.2 summarizes the cyclone conditions, the d50 estimated by Plitt's equation and the -1 μm fraction in the cyclone underflow. More details about the selection of these cyclone conditions (e.g. operating conditions and number of stages) and the PSD of the products are given in Appendix 5.

Column feed preparation, particularly from the rod mill feed, was time-consuming. This was partly due to the large volumes of diluted (e.g. 1% w/w solids) pulps required to enhance the -1 μm fraction removal which limited slimes preparation to batches of only 3.5 kg . Another factor was the multiple cyclone stages required to eliminate the - 1 μm fraction. These stages could be reduced to 1 stage (e.g. see Appendix 5, Table A.5.1) but only by using large concentrations (e.g. 1 % w/w in water) of dispersant during classification. This route was avoided to eliminate the risk of altering the surface properties of the column feed. The difficulties in preparing column feed prompted the use of dilute feeds (e.g. 1% w/w solids) in column flotation to preserve sufficient material to perform all the testwork.

TABLE 3.2 SUMMARY OF CYCLONE CONDITIONS DURING COLUMN FEED PREPARATION

STAGE #	CYCLONE DIMEN. Di	 Dn cm	 Du	P kPa	% SOLIDS IN FEED	d50 (*) (μm)	(-1μm) in U/F (%)
			ROD MILL FEED				
1	1.06	1.43	0.48	68.90	2.10	12.34	N.A.
2	1.06	1.43	0.32	413.68	1.23	22.59	6
	1.06	1.43	0.32	413.68	1.00	22.26	6
	1.06	1.43	0.48	413.68	1.23	16.58	17
3	1.06	1.43	0.32	413.68	0.60	21.71	5
	1.06	1.43	0.32	413.68	0.60	21.71	5
			SLIMES				
1	1.06	1.43	0.32	413.68	1.27	22.64	N.A.
	1.06	1.43	0.32	413.68	0.33	21.34	N.A.
2	1.06	1.43	0.48	413.68	0.33	15.67	N.A.
	1.06	1.43	0.32	413.68	0.33	21.34	N.A.
3	1.06	1.43	0.32	413.68	0.33	21.34	4
	1.06	1.43	0.32	413.68	0.33	21.34	8

(*) d50 estimated by Plitt's equation
(see more details in Appendix 5)

3.3 PRELIMINARY FLOTATION TESTS

3.3.1. Experimental Set Up and Conditions.

Column tests were performed in a 3.8 cm diameter 200 cm high column using a porous ceramic sparger (Fisher Scientific Limited, Cat. N. 11-139A). Typically, the column was operated at a froth depth of 30 cm, a superficial gas rate of 0.6 cm/s, a superficial wash water rate of 0.6 cm/s, a feed density of 1% solids, and a superficial feed rate, 0.7 cm/s. Figure 3.3 shows the experimental set up. The water and pulp flowrates were controlled using peristaltic (Masterflex) pumps. Air flowrates weremeasured with a gas flowmeter previously calibrated to the air pressure used (normally 68.9 kPa (10 psi)); calibration curve is shown in Appendix 6. In order to reproduce plant results, tests were always done using plant recycle water. Two samples of plant recycle water were used: recycle water 1, containing 200 ppm Ca, 98 ppm Mg and total organic carbon (TOC) of 2 ppm, and recycle water 2, containing 107 ppm Ca, 80 ppm Mg, and TOC 43 ppm. Unless indicated the results reported are with recycle water 1.

Samples were taken after steady state conditions had been reached (minimum 10 minutes). Chemical assays were performed using X-ray fluorescence. Samples were assayed for Nb_2O_5, SiO_2, CaO, Al_2O_3, Fe_2O_3, and MgO. These assays were subsequently adjusted using a mass balance program which also calculated the metal recoveries. (Appendix 7, gives the experimental and adjusted assays, and recoveries from the flotation tests conducted). Typical conditions at Niobec are: pH 8, sodium silicate, addition of 0.05-0.4 kg/tonne, fatty acid (Acintol FA2) (FA), dosage of about 0.6 kg/tonne. The FA is always added as an emulsion with lannagol (emulsifier) in a proportion of 3:1.

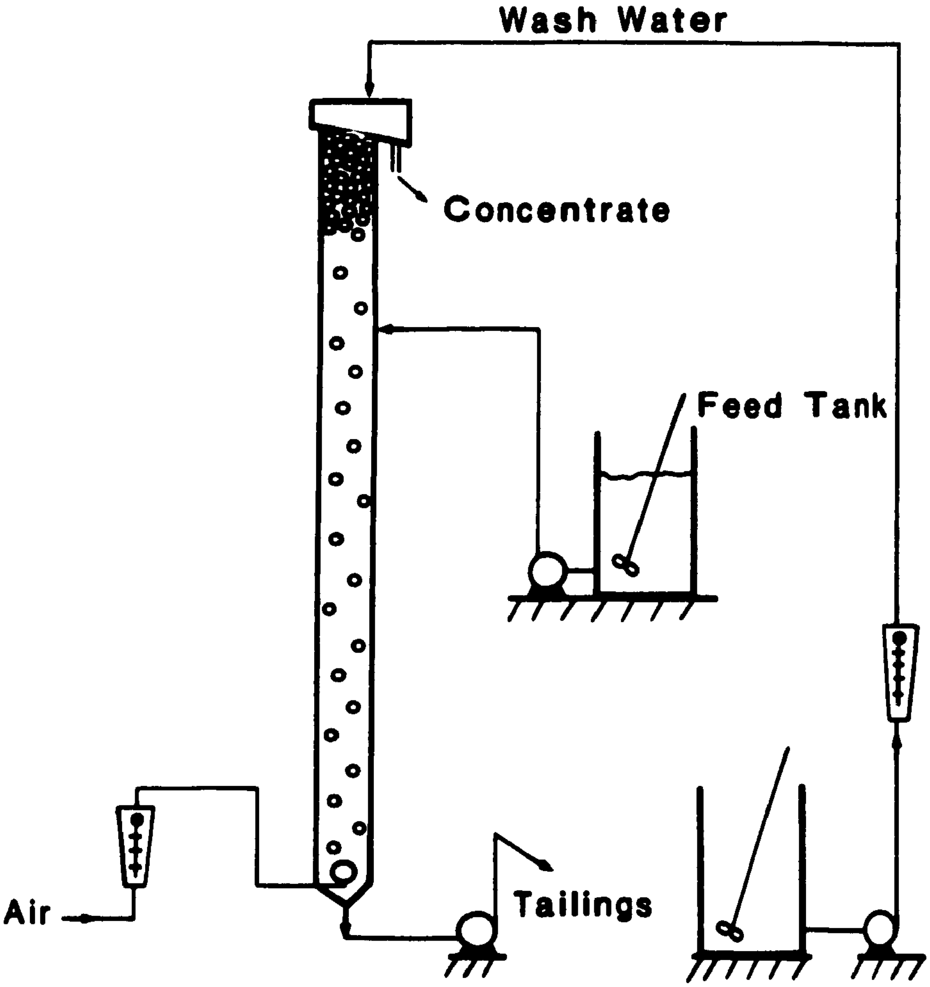

Figure 3.3 Column flotation experimental set up.

3.3.2. Results

The column flotation tests were aimed at reproducing the carbonate flotation results obtained by Niobec on the deslimed material using their conditions. Typical plant results are illustrated in Figure 3.4. Results are expressed as recovery of Nb_2O_5, SiO_2, and CaO vs mass recovery to the float product. This representation permits simultaneous illustration of which species are being floated and which are being depressed.

Column flotation tests were performed to optimize sodium silicate and fatty acid additions. Results of the test series, using sodium silicate and FA, are given in Figures 3.5 and 3.6, respectively. In all cases rejection of pyrochlore (and silicates) is superior at Niobec. For example, at 60% CaO recovery at Niobec (Figure 3.4), only about 8% of the Nb_2O_5 and SiO_2 is floated with the carbonates while in the column tests (Figures 3.5 and 3.6), about 40% Nb_2O_5 and 25% SiO_2 are floated. In addition, Figure 3.5 indicates that the main effect of increasing sodium silicate addition is to depress the overall mass of material without any gain in carbonate selectivity. Figure 3.6 shows the selectivity with the fatty acid increases slightly at about 4.5 kg/tonne. Conditions selected for further testwork were 3 kg/tonne Na_2SiO_3 and 4.5 kg/tonne FA.

This poor rejection of Nb_2O_5 and SiO_2 in the column is attributed to a lack of dispersion between the carbonates, pyrochlore and silicates. This suspicion of poor dispersion was based partly on visual inspection and partly on knowledge of the system. For example, at pH 8 the carbonates are close to their point of zero charge (Hanna and Somasundaran, 1976) thus the surface charge is small and electrostatic repulsion is lost. Preliminary flotation tests using dispersants (e.g. lignosulphonates and polyphosphates (calgon)) were conducted. These tests were not successful, consequently, a

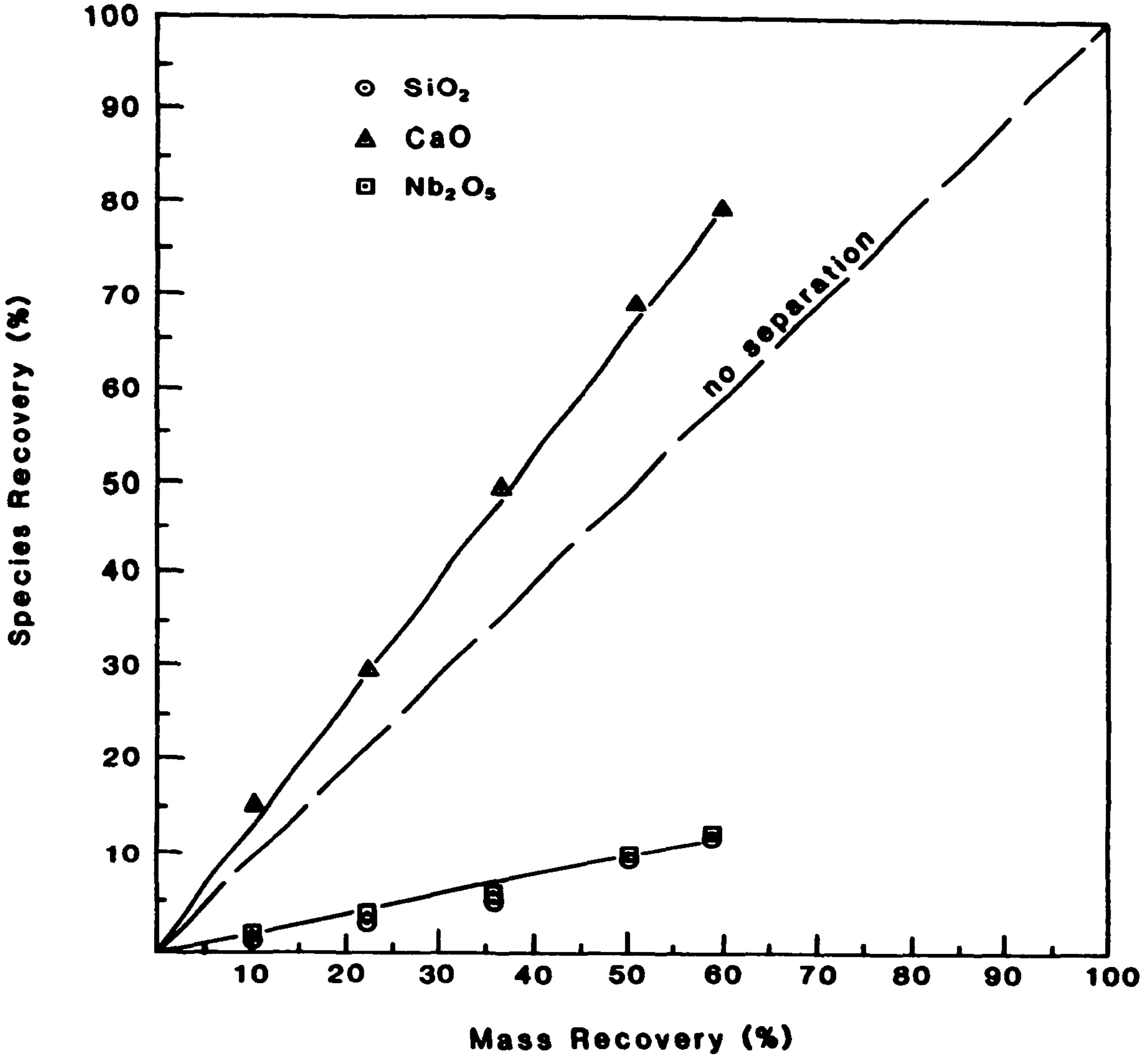

Figure 3.4 Species recovery (%) vs mass recovery (%) at Niobec.

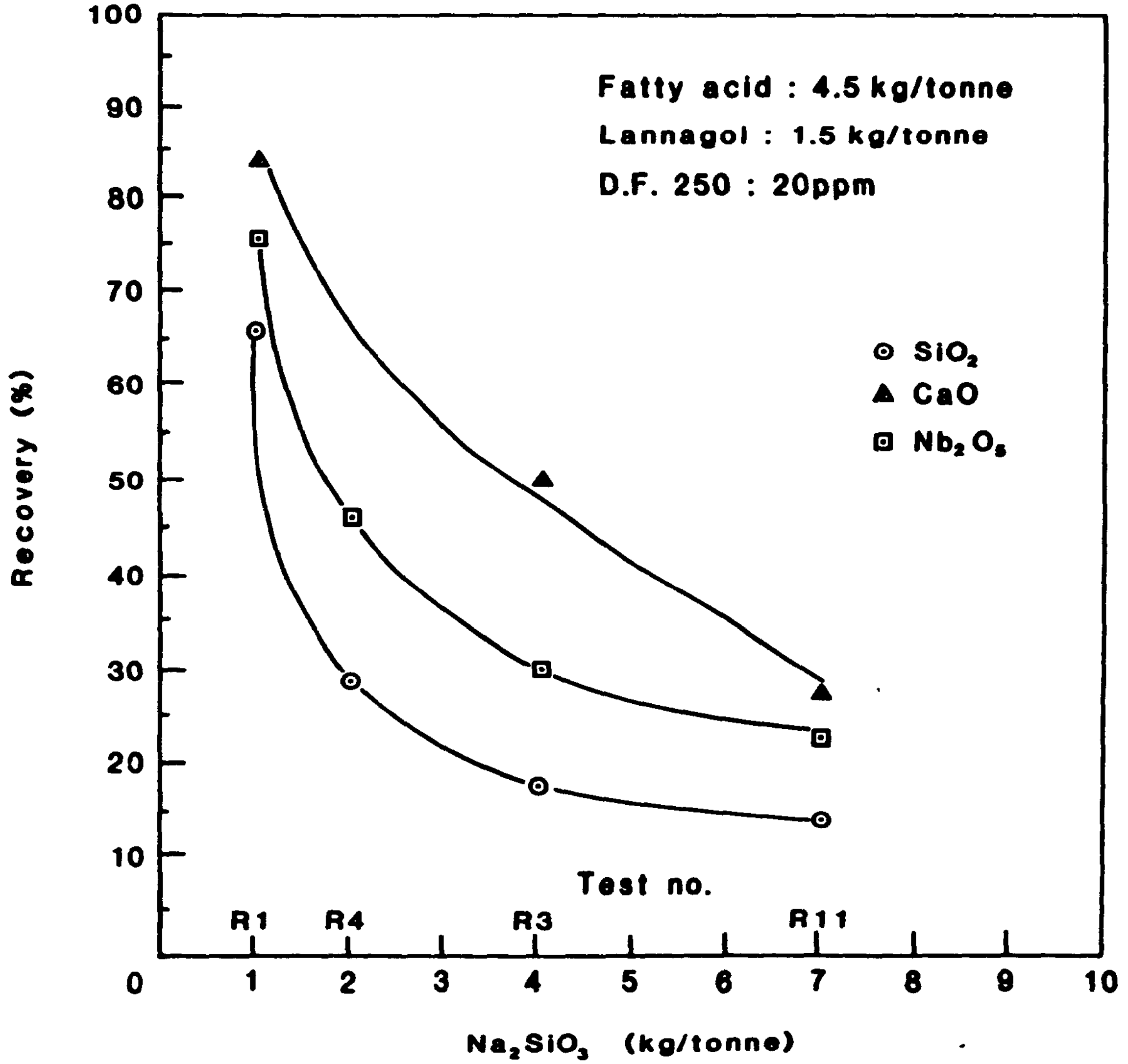

Figure 3.5 Effect of sodium silicate

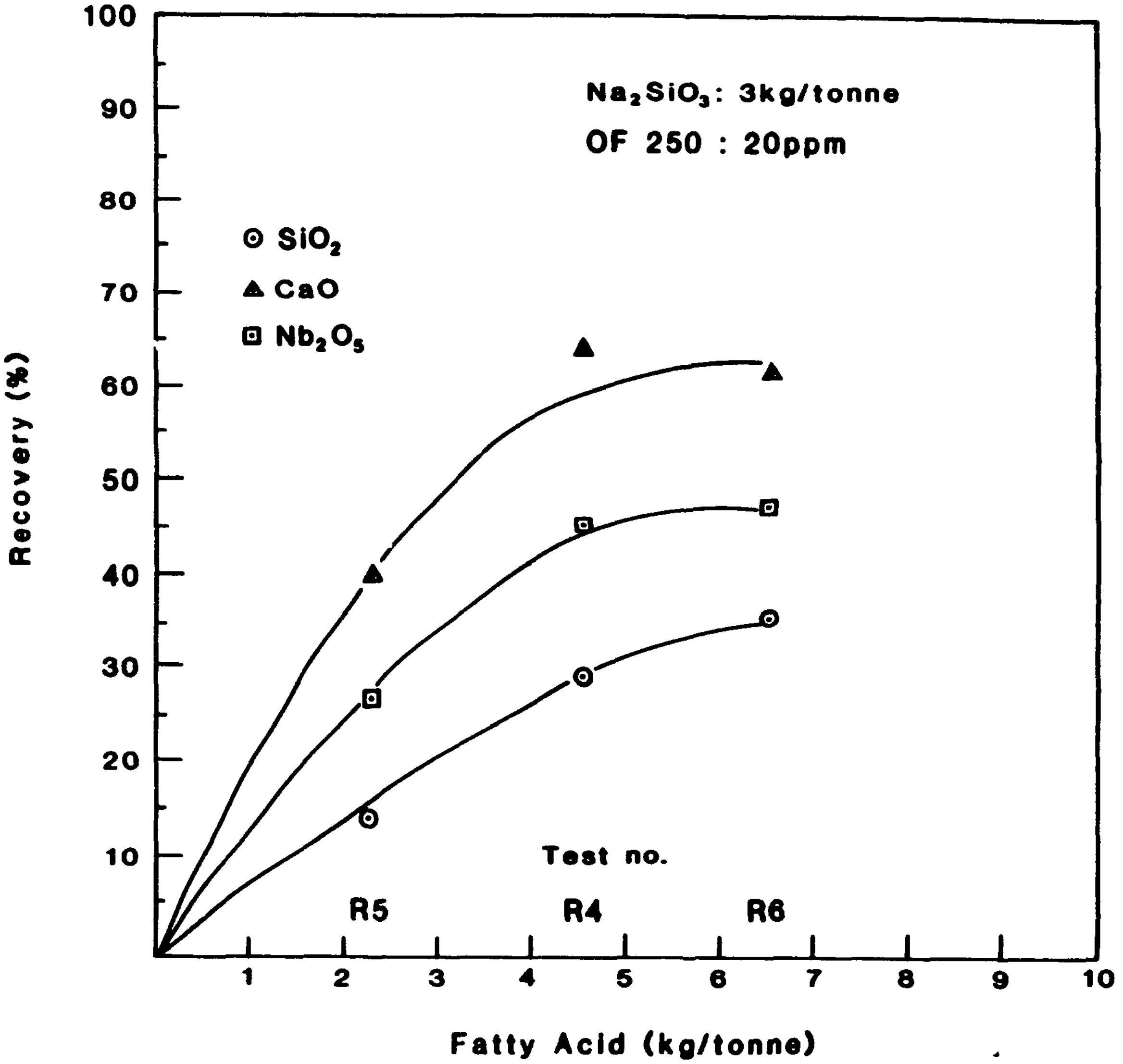

Figure 3.6 Effect of fatty acid.

systematic dispersion study was undertaken to find the best dispersants and the optimum dispersion conditions.

The results and conditions of these flotation tests are given in Appendix 7 and 8, respectively. More tests were conducted than are reported, since, due to the strong color differences between the carbonates and the silicates and the visually clear indication of coagulation and froth collapse, it was possible to judge the success of a particular flotation test. At this stage the froth collapse problems were partially reduced by increasing the superficial gas rate from 0.6 to 1.0 cm/s.

3.4. MEASUREMENT OF DISPERSION

Dispersion can be measured by sedimentation techniques and evaluated in terms of a ratio between an initial and final concentration (Rinelli and Marabini, 1980). It has also been evaluated by measuring the transmittance of the colloidal dispersions (Correa de Araujo et al, 1986). In this work dispersion was evaluated using a X-ray sedimentometer, the Sedigraph 5000D (Sedigraph) which gives a measure of apparent particle size. This technique has been used by the author in other applications (Espinosa-Gomez, 1981).

The Sedigraph employs a finely collimated X-ray beam to measure the change with time of particle concentration during settling in a sample cell. The cell also descends relative to the beam, to reduce the time required for analysis. For good reproducibility constant temperature and sample size are required. To illustrate the use of the Sedigraph for determining dispersion an example from the manuscript (section 3.5) will be used.

Figure 3.7 gives a typical plot produced by the Sedigraph. Results are directly expressed as cumulative mass percent vs equivalent spherical diameter (μm). Curve 1 represents the finest size distribution obtained,

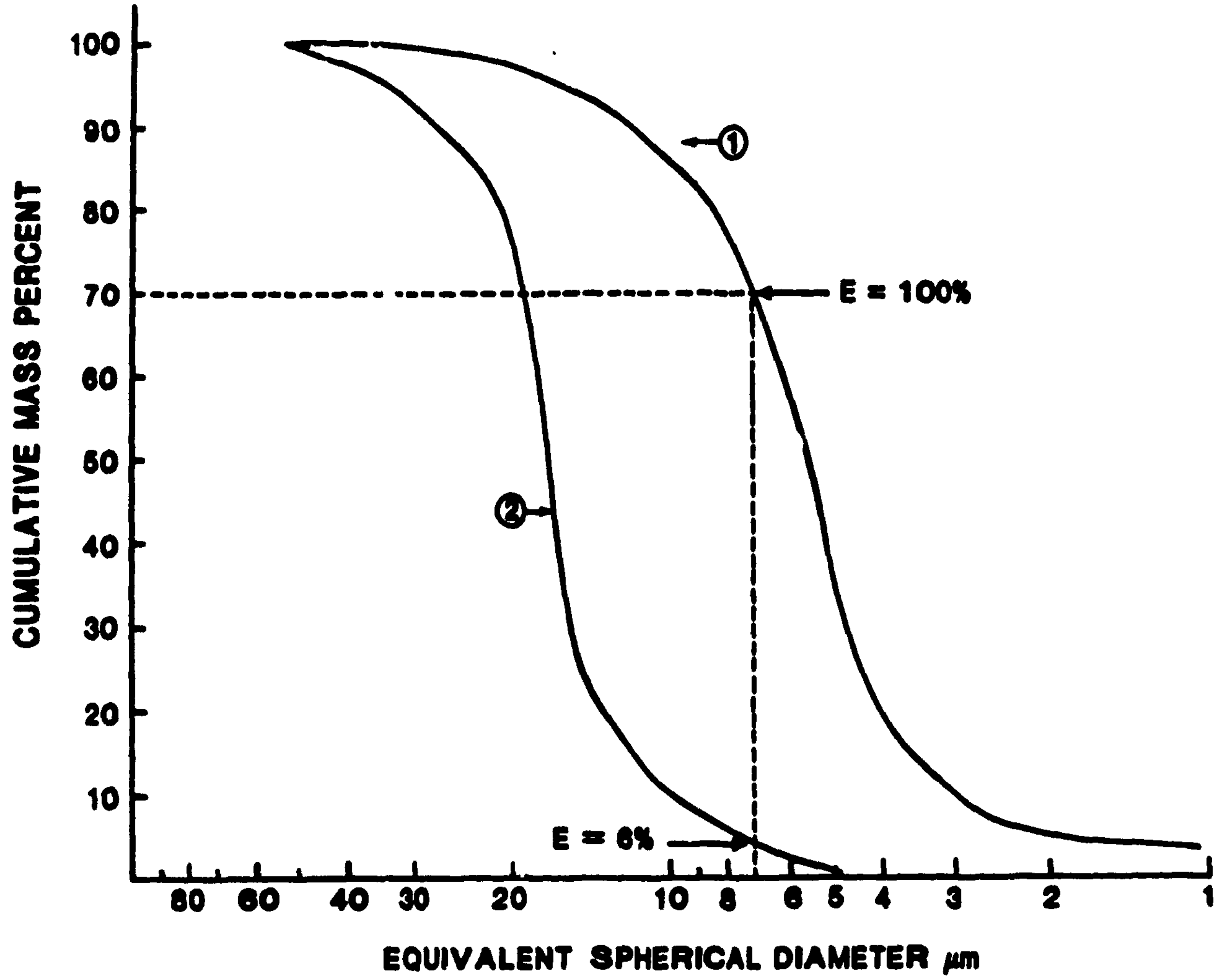

Figure 3.7. Particle size distribution of the column feed at two dispersion conditions

using distilled water with 0.1% $(NaPO_3)_{13}.Na_2O$ (referred to as calgon), 10 minutes of mechanical agitation and five minutes in an ultrasonic bath. Curve 1, therefore, represents full dispersion. Curve 2 was obtained without any dispersant and using plant recycle water. The PSD represented by curve 2 is coarser because of coagulation. By comparing the two curves a measure of dispersion relative to complete dispersion (or dispersion efficiency) can be developed. One way to achieve this is to take the ratio of cumulative percent passing a selected particle size. A convenient size in this case is 7 μm being about the mean of the fully dispersed PSD. The efficiency of dispersion, E, therefore is:

$$E = \frac{\%\ -7\ \mu\text{m (at tested conditions)}}{\%\ -7\ \mu\text{m (fully dispersed)}} * 100$$

The range in E is 0 to 100 %. It cannot exceed 100%, by definition. If the material is so poorly dispersed that none appears finer than the selected size this registers as E=0 regardless of the observed PSD of the material.

3.5 MANUSCRIPT 2:

DISPERSION STUDY ON SLIMES FROM NIOBEC

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ABSTRACT

The Niobec concentrator rejects up to 15% of the Nb as slimes (-10 μm). The recovery of Nb from these slimes by column flotation is being studied. An essential condition for selective flotation is slimes dispersion. A particle size analyzer, the Sedigraph 5000D, was used to monitor dispersion in plant waters with combinations of organic and inorganic dispersants and flotation reagents. Flotation reagents were particularly detrimental to dispersion with sodium silicate. The best dispersion conditions were selected for column flotation tests. These tests revealed that reagent combination had a pronounced effect on froth stability. Careful frother selection achieved good froth stability under conditions of good dispersion. Plant water increased the required dispersant concentration causing mineral depression.

INTRODUCTION

Slimes (-10 µm) rejected at Niobec contain up to 15% of the overall Nb content (Biss, 1984a). Recovery of Nb from these slimes would significantly increase overall recovery. A suggested recovery route involves two phases: 1) carbonate flotation (to remove most of the carbonates) depressing silicates and pyrochlore (as currently done at Niobec); and 2) selective (direct) pyrochlore flotation from the silicates. Phase 2 has been addressed and several routes to achieve selective flotation are available. Phase 1 was attempted with the selective reagent scheme used at Niobec but a considerable amount of pyrochlore floated with the carbonates due to poor slimes dispersion.

Slimes dispersion is essential to avoid pyrochlore flotation with the carbonates. Two factors inhibit dispersion of the slimes: fine particle size (80% passing 8 m and 10% passing 1 µm), and Ca and Mg cations in the plant water. Ca and Mg are adsorbed in the surface of the silicates and pyrochlore (Espinosa-Gomez et al, 1987a) compressing the electrical double layer and enhancing coagulation due to the low surface potential. In addition, at the pH of flotation (about 8), the carbonates are close to their isoelectric point of charge or are positive, enhancing coagulation with the negatively charged silicates.

To attempt slimes dispersion several dispersants were tested, with a stress on those less affected by Ca and Mg cations to avoid high consumption and carbonate depression. This dispersion study is presented here; and also shown are some preliminary flotation results which were conducted in a flotation column.

Dispersion has been traditionally measured by sedimentation techniques and evaluated in terms of a ratio between an initial and final concentration (Rinelli and Marabini, 1980; Shimoiizaka et al, 1980). It has also been evaluated by measuring the transmittance of the colloidal dispersions (Correa de Araujo et al, 1986). In this work, dispersion was evaluated using a particle size analyser, which gives a measure of apparent particle size.

EXPERIMENTAL METHODS AND MATERIALS

Reagents / Water

Table 1 shows the reagents used in this work, classified as dispersants, pyrochlore depressants, carbonate collectors, and frothers. For many organic dispersants only the trade name was known.

The material used in this study is identified at Niobec as slimes from Zone 1. Zone 1 is characterized by its high columbite content. Columbite losses are high at Niobec, because the reagent combinations used in plant were designed for pyrochlore recovery.

A sample of slimes (100% - 15 μm) was deslimed at about 1 μm using a 5 cm hydrocyclone. Two batches were prepared; one was used in the dispersion study (4% -1 μm) and the other was used as column feed (9% -1 μm). Subsequent work will progressively return the - 1 μm fraction to determine the limiting content of this fraction for acceptable flotation performance.

Two samples of plant recycle water were used: recycle water 1, containing 200 ppm Ca, 98 ppm Mg and total organic carbon(TOC) of 2 ppm, and recycle water 2 containing 107 ppm Ca, 80 ppm Mg, and TOC 43 ppm. Unless indicated, the results reported will be with recycle water 1.

TABLE 1. REAGENTS USED IN THIS WORK

NAME	SUPPLIER	REMARKS
	DISPERSANTS	
Procol DA811	Allied Colloids	anionic dispersant
Alcomer 75L	Allied Colloids	strong Ca,Mg, resistance
Alcomer 74L	Allied Colloids	good Ca,Mg resistance
Dispex N40	Allied Colloids	dispersant
AQ55D	Eastman Chem. Pros.	silicate dispersant
SFX	Reeds Co.	Na lignosulphonate
Kelig 32	Reeds Co.	Na lignosulphonate
Lignosol WT	Reeds Co.	Na lignosulphonate
SFX65	Reeds Co.	Na lignosulphonate
Sodium silicate	Niobec	silicate depressant
$(NaPO_3)_{13} \cdot Na_2O$ (Calgon)	Fisher Sci. Co.	dispersant
Ethylenediaminetetraacetic acid (EDTA)	Aldrich Chem. Co.	Ca and Mg complexing agent
Alkasperse A-5	Alkaril Chem. Ltd	dispersant
	PYROCHLORE DEPRESSANTS	
Tapioca starch	Niobec	
Heated Tapioca starch	Niobec	Heated at 165 C for 15 min.
Hydrosols: $FeCl_3 \cdot 6H_2O$ $ZnSO_4 \cdot 7H_2O$ $ZrOSO_4 \cdot H_2SO_4 \cdot 3H_2O$ $Na_2SO_3 + FeSO_4$ $(NH_4)_2SO_4$	Niobec	Mixture of a salt and Na2SiO3
	CARBONATE COLLECTORS	
Acintol FA-3	Niobec	Fatty Acids
Lannagol DP6	Niobec	Emulsifier
	FOTHERS	
Dowfroth 250	Dow Chem.	Polypropylene glycol methyl ethers
Dowfroth 400	Dow Chem.	
Dowfroth 1400	Dow Chem.	
Pine oil		
MIBC		
TEB (1-1-3-triethoxy butane)	Niobec	

Dispersion.

A Sedigraph 5000D was used to monitor dispersion. The efficiency (E) of dispersion was evaluated as:

$$E\ (\%) = \frac{\%-7\ \mu m\ \text{(at tested conditions)}}{\%-7\ \mu m\ \text{(fully dispersed)}} * 100$$

The choice of 7 μm is arbitrary; it is approximately the arithmetic mean of the slime size range.

Fully dispersed conditions were achieved using distilled water and $(NaPO_3)_{13} \cdot Na_2O$ (calgon) with 5 minutes of ultrasonic dispersion (Figure 1, curve 1). For good reproducibility constant temperature and sample size were used in all the dispersions tests. The 95% confidence interval for the mean of E was +/-2%.

Dispersants were first tested at natural pH (8.1) using a high concentration, and only those which gave good dispersion were tested at several concentrations and pHs to determine the optimum. Mainly basic pHs, used for carbonate flotation, were tested.

The suspension stability with the best dispersants was then tested in the presence of the carbonate flotation reagents.

Flotation Column / Froth Coalescence

Column tests were performed in a 3.8 cm diameter 200 cm high column using a porous ceramic sparger (Fisher Scientific Limited, Cat. No. 11-139A). Typical operating conditions were: froth depth, 30 cm; gas rate, 1 cm/s; wash water rate, 0.6 cm/s; % solids, 1; and feed rate, 0.5 L/min.

Froth coalescence was evaluated only qualitatively. It was classified as: a) some, when froth bubble size increased from about 2 to 3 mm to about 4 to 6 mm; b) strong, when froth bubble size increased to more than 6 mm; and, c) very strong, when a froth bed could not be maintained.

RESULTS

Dispersion.

Figure 1 shows typical particle size distribution (PSD) curves and efficiencies (E). Curve 1 shows fully dispersed conditions. In untreated recycle water, efficiency was only 6% (curve 2). The use of sodium silicate promoted coagulation in recycle water, E = 0% (curve 3), but excellent dispersion in distilled water, E = 93% (curve 4).

Table 2 shows the best dispersants and their optimum concentration and pHs, defined by efficiencies higher than 85%. AQ55D produced good dispersion over a wide pH range. Figure 2 shows the influence of pH on dispersion for Alcomer 74L and Procol DA811. For Alcomer 74L dispersion is reduced from 86% (curve 1), at pH 11.2, to 40% (curve 2), at pH 8.1. On the other hand, for Procol DA811 dispersion is reduced from 86% (curve 3), at pH 8.1, to 3% (curve 4), at pH 11.2. The best dispersants (DA811, AQ55D, calgon) were tested using recycle water 2. Dispersion was the same for DA811 and calgon, but for AQ55D, E was reduced to 57%.

Table 2 also shows the stability of dispersion after carbonate flotation reagents were added. Only the dispersion produced by DA811, AQ55D and calgon remained unaffected by reagent addition. Dispersion with the lignosulphonates (SFX, Kellig-32, and Lignosol WT) was the most unstable in the presence of flotation reagents, especially in the presence of sodium silicate. Figure 3 shows how the 86% dispersion produced by Kellig-32 (at pH

TABLE 2. OPTIMUM DISPERSANTS AND CONDITIONS - WATER SAMPLE 1

NAME	CONCENTRATION (%)	OPTIMUM pH	EFFICIENCY E (%)(*)	STABILITY (1)
DA-811	0.025-0.05	8.1	86	excellent
Alcomer 75L	0.03 -0.05	8.8-11.2	91	fair (2)
Alcomer 74L	0.05	11.2	86	fair
AQ55D	0.05	4.6-11.2	100	excellent (3)
SFX	0.1	11.5	85	poor (4)
Kelig 32	0.1	8.1	86	poor
Lignosol WT	0.1	11.1	91	poor
Calgon	0.21	11.1	100	excellent

(*) The 95 % confidence interval for the mean of E was +/- 2%.
(1) Stability of dispersion after carbonate flotation reagents (CFR) were added.
CFR : Na_2SiO_3, Starch and Fatty Acid.
(2) E was reduced to about 65 %.
(3) E was reduced to 57 % using recycle water 2.
(4) E was reduced to 15 %.

FIGURE 1. PARTICLE SIZE DISTRIBUTION CURVES FOR THE COLUMN FLOTATION FEED OBTAINED AT SEVERAL DISPERSION CONDITIONS. CURVE 1, FULLY DISPERSED; CURVE 2, IN RECYCLE WATER (WITHOUT DISPERSANTS); CURVE 3, IN RECYCLE WATER WITH Na SILICATE (4 kg/tonne) AS DISPERSANT; CURVE 4, IN DISTILLED WATER WITH Na SILICATE (4 kg/tonne).

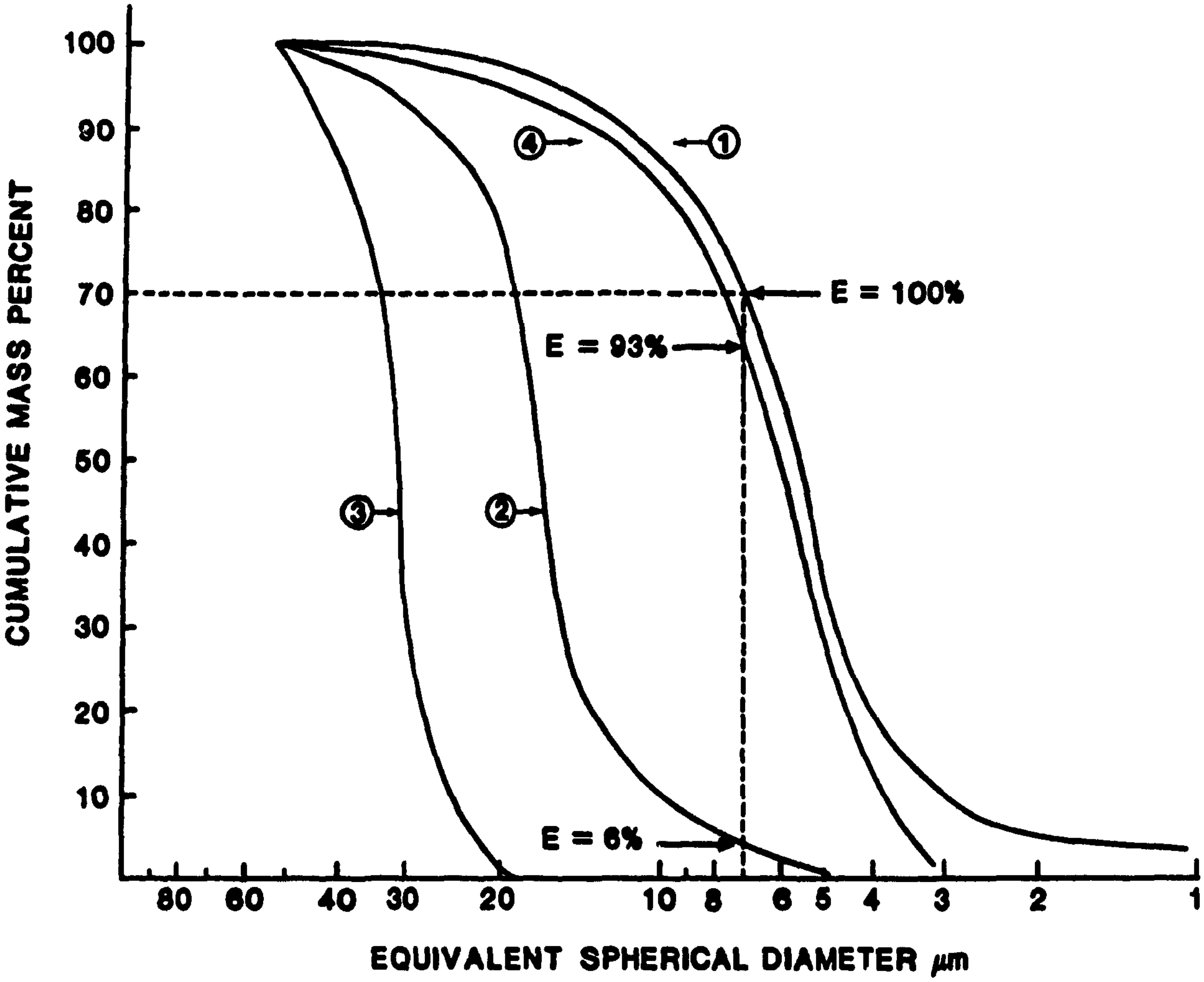


FIGURE 2. EFFECT OF pH ON DISPERSION. CURVE 1, IN RECYCLE WATER + ALCOMER 75L (0.05 %) AT pH 11.2; CURVE 2, SAME CONDITIONS AS CURVE 1 BUT pH 8.1; CURVE 3, IN RECYCLE WATER + PROCOL DA811 (0.05 %) AT pH 8.1; CURVE 4, SAME CONDITIONS AS CURVE 3, BUT pH 11.2.

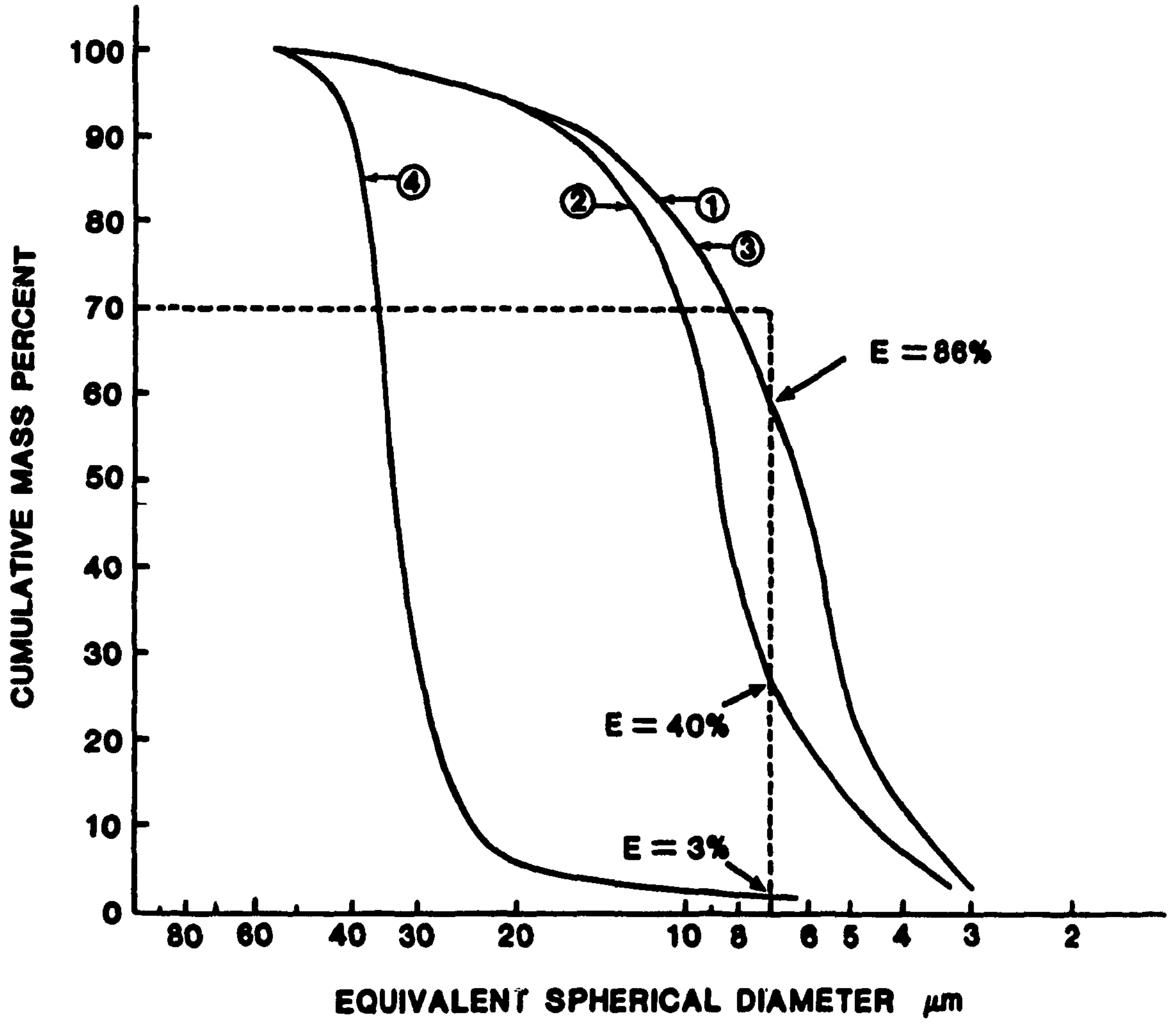
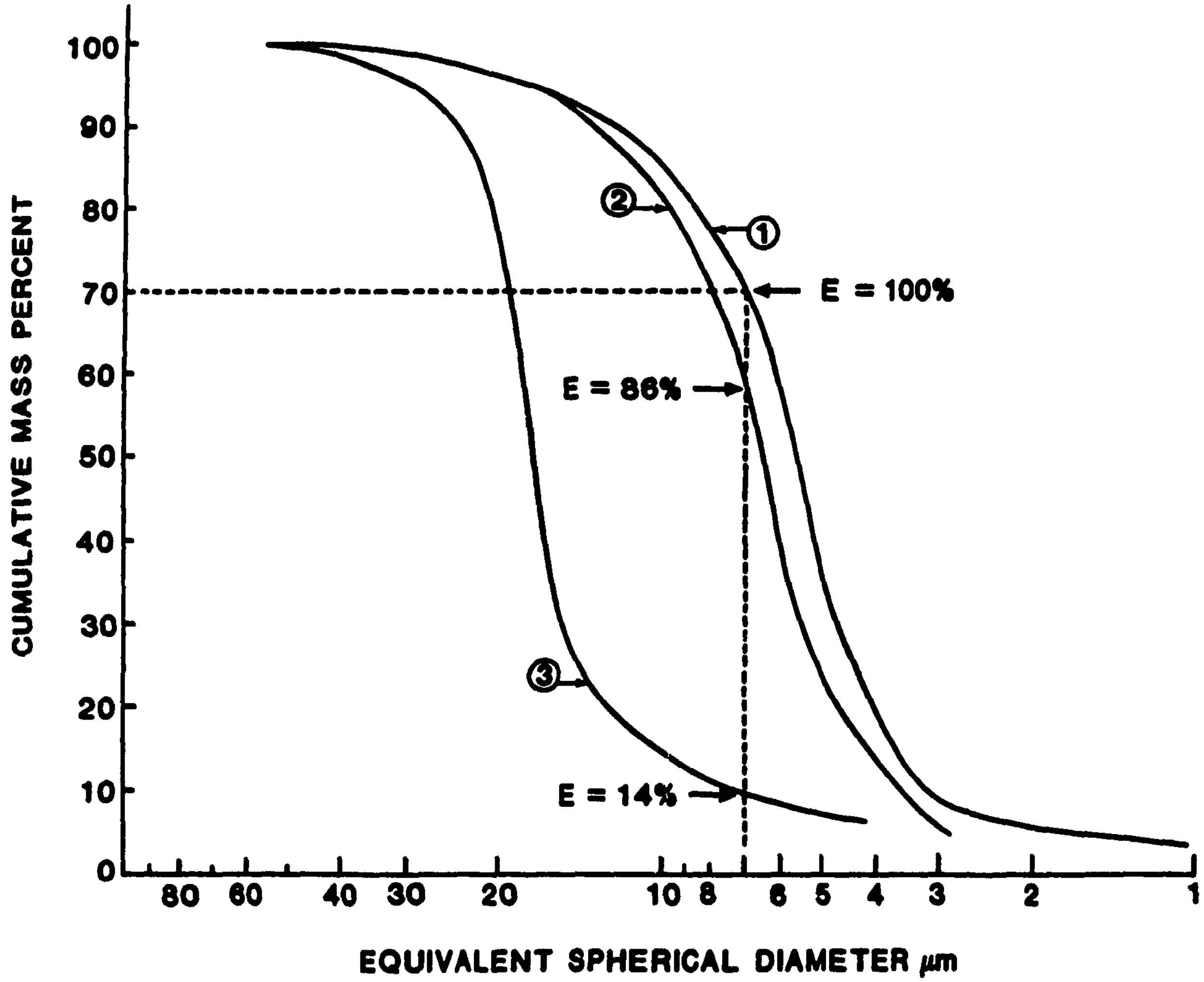


FIGURE 3. EFFECT OF FLOTATION REAGENTS ON DISPERSION WITH KELIG 32 (0.1 %) IN RECYCLE WATER. CURVE 1, FULLY DISPERSED; CURVE 2, KELIG 32 ALONE; CURVE 3, KELIG 32 + FLOTATION REAGENTS.



8.1), curve 2, is reduced to 14%, curve 3, when flotation reagents are added.

Flotation

The best dispersion conditions (Table 2) were used in flotation. However, two problems were encountered: a) bubble coalescence, and b) depression of carbonates.

a) Bubble coalescence. Coalescence was observed from the onset of the column work with DF250. The froth depth (30 cm) could only be maintained at increased gas rate (1 cm/s). It was established by tests without solids that this coalescence was due to reagent combinations. Coalescence increased when organic dispersants were added. With some reagent combinations a froth could not be maintained. Froth collapse in fatty acid systems has been reported elsewhere (Klassen and Mokrousov, 1963c).

Table 3 summarizes the reagent combinations that promote coalescence. In general fatty acids tended to cause coalescence, and coalescence increased when fatty acid was combined with DA811. Coalescence is greatest when fatty acids combined with sodium silicate and organic dispersants. Several frothers (Table 1) were evaluated to overcome the coalescence. The best frother was TEB at a concentration of 20 ppm. TEB eliminated coalescence caused by fatty acids, although it only partially reduced coalescence caused by combination of the fatty acid, sodium silicate, and organic dispersant.

b) Depression of carbonates. Carbonate flotation was affected by high dispersant concentrations. Reducing the amount of dispersant resulted (as expected) in coagulation. A combination of less dispersant and ultrasonic treatment (Kusnetsov et al, 1979) did not reduce the amount of dispersant to

TABLE 3. REAGENT COMBINATIONS THAT PROMOTE BUBBLE COALESCENCE

REAGENT COMBINATION	COALESCENCE(1)	
	Dowfroth 250(2)	TEB(3)
Fatty Acid (F.A.)(4)	some	none
Na silicate + F.A.	some	none
Alcomer 75L + F.A.	some	none
DA-811 + F.A.	strong	none
Alcomer 75L + Na silicate + F.A.	very strong	strong
DA-811 + Na silicate + F.A.	very strong	strong

(1) - F.A. was always used with the emulsifier Lannagol DP6
(2) - frother: polypropylene glycol ether
(3) - frother: 1,1,3-triethoxybutane
(4) Coalescence was stronger in the presence of solids and using water sample 2 (higher residual organic)

a level which did not affect flotation. Another approach was to precipitate Ca and Mg from the recycle water with EDTA or polyphosphates (calgon) and then disperse using lower dispersant concentrations. This approach also did not reduce the amount of dispersant to a level which did not affect flotation. In theory an increase in pH from 8 to 10.5 should increase silicate dispersion due to the higher electrostatic repulsion (Fuerstenau and Palmer, 1976). However, as soon as pH was increased, strong coagulation occured.

Preliminary carbonate flotation tests were performed using the same conditions employed at Niobec, namely: sodium silicate (as silicate depressant), starch (as pyrochlore depressant), fatty acids (mainly oleic and linoleic acids), and emulsifier (Lannagol DP6). Table 4 shows some typical results. Niobium depression is incomplete; 21% of it is recovered in the carbonate float, which assays 0.34% Nb_2O_5. Two possible reasons for this high Nb content are : 1) incomplete slimes dispersion, and 2) incomplete columbite depression (Biss, 1984b).

Seven pyrochlore depressants (polysilicates and heated starch (Iwasaki and Lai, 1965) were also tested (Table 1), but none gave better depression than starch alone. This second series of tests with polysilicates and heated starch were conducted using recycle water 2, (43 ppm instead of 2 ppm of TOC). With the same reagent conditions as those in Table 4, the Nb_2O_5 grade in the carbonate concentrate increased to 0.46% from 0.34%. This suggests the residual organics significantly reduce flotation selectivity.

TABLE 4. FLOTATION RESULTS USING NIOBEC CONDITIONS(*)

PRODUCT	Wt(%)	ASSAY (%)				RECOVERY (%)			
		Nb_2O_5	SiO_2	MgO	CaO	Nb_2O_5	SiO_2	MgO	CaO
CARBONATES	29.6	0.34	6.5	12.8	28.1	21.2	14.4	29.0	37.6
TAILINGS	70.4	0.53	16.3	13.2	19.6	78.8	85.6	71.0	62.4
FEED GRADE		0.47	13.4	13.1	22.1				

(*) CONDITIONS: fatty acid, 4.5 kg/tonne; emulsifier, 1.5 kg/tonne; starch, 0.5 kg/tonne; Na silicate, 3 kg/tonne.

DISCUSSION

Dispersion.

Two inorganic and eleven organic dispersants were tested. Dispersant efficiency was affected by water composition, pH, and flotation reagents (Table 2).

The two inorganic dispersants tested were sodium silicate and calgon. Dispersion produced by sodium silicate (the reagent used at Niobec) was markedly different between recycle and distilled water (Figure 1, curves 3 and 4). Sodium silicate induced coagulation with recycle but produced an excellent dispersion with distilled water. This implies that good dispersion should be achieved with sodium silicate if recycle water is first treated. Ca and Mg in recycle water may promote coagulation due to particle bridging through polymeric silicates developed on the particle surface (Stumm et al, 1967; Greenberg and Sinclair, 1955). Calgon produced excellent dispersion; however, consumption was high partly due to sequestering of Ca and Mg ions and further interactions with calcite and dolomite surfaces (Li and Lu, 1983).

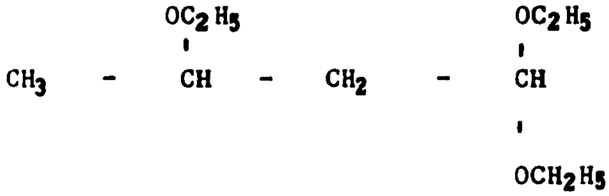
A detailed discussion of organic dispersant interactions is not possible because not all chemical structures are available. However, it can be concluded that, in general, their behavior depends on structure and their affinity for Ca and Mg cations. For example, the consumption of Alcomer 74L is higher than that of Alcomer 75L due to its higher affinity for Ca and Mg (Allied Colloids, 1982); lignosulphonates stability was similarly reduced by sodium silicate possibly due to the same type of interactions with sodium silicate. Also EDTA, due to its shorter organic chain and high affinity for

Ca and Mg, works better as a complexing agent than as a dispersant.

Froth Coalescence.

Froth coalescence was a major problem. It was eliminated in many cases using TEB instead of Dowfroth 250. One possible explanation of the difference lies in frother structure:

TEB structure:



$$CH_3 - \overset{OC_2H_5}{\overset{|}{CH}} - CH_2 - \underset{\underset{OCH_2H_5}{|}}{\overset{OC_2H_5}{\overset{|}{CH}}}$$

Dowfroth structure:

$$CH_3 - (O - C_2H_4)n - OH$$

The OH group of Dowfroth 250 and the carboxyl group (COOH) of the fatty acid may interact forming an ester (esterification). No interaction is expected between the ether structure of TEB and the carboxyl group. Fatty acids failed to produce sufficient froth on their own. No comments can be made regarding the coalescence caused with organic dispersants, as their structure is not known.

Froth coalescence was an unexpected problem and deserves more attention. There is relatively little literature on bubble coalescence in flotation studies although this phenomenon may be process limiting in some cases. Quantification of coalescence is required for systematic study.

Depression of carbonates.

Depression of carbonates occured at the dispersant concentrations required for dispersion. Possible reasons include $(NaPO_3)_{13}$ $.Na_2O$-carbonate and EDTA-carbonate interactions (Li and Lu, 1983). Even when stoichiometric quantities (with regard to Ca and Mg in solution) were added, depression was not eliminated.

Thus far, good dispersion without flotation problems has not been achieved. To resolve the problems of selectivity in flotation, more emphasis is required in the study of real systems, even though interpretation is more difficult.

SUMMARY AND CONCLUSIONS

This paper describes dispersion of slimes in a carbonate flotation stage of Nb-mineral processing. Two inorganic and eleven organic dispersants were tested.

The main findings are:

1) The Sedigraph is a practical, precise, and fast means of evaluating / quantifying dispersion.

2) Dispersant behavior is affected by water composition, pH, and flotation reagents. For example, sodium silicate is an excellent dispersant with distilled water, but promotes coagulation with recycle water due to sodium silicate / Ca, Mg interactions.

3) Dispersants caused two flotation problems, bubble coalescence and depression of carbonates. Froth coalescence was related to reagent combinations, e.g., DF250 with fatty acid caused froth collapse, possibly because the OH group in the frother interacted with the COOH group of the collector (esterification).

3.6 FURTHER FLOTATION RESULTS

Flotation tests were conducted using the best dispersion conditions (Table 1 in the manuscript). It became apparent that the system was not very stable, and periodic froth collapse would occur. The problem became worse upon two apparently minor changes in the system, the use of recycle water 2 and a new batch of fatty acid, in both cases because the originals lots had been exhausted. (Appendix 7 summarizes the flotation results and Appendix 8 the flotation conditions, for most of the tests conducted).

The main difference between recycle water 1 and recycle water 2 is their total organic carbon (TOC), 2 vs 43 ppm, respectively. It is common at Niobec to experience this magnitude of change in the TOC. The most obvious effect of recycle water 2 was virtually complete mass flotation at the same fatty acid addition (4.5 kg/tonne) used with some success with recycle water 1. Tests were conducted to try to optimize the fatty acid addition (Figure 3.8). Fatty acid addition had to be reduced to about 1.5 kg/tonne with recycle water 2. Also, these new tests required a pyrochlore depressant, starch. Starch is sometimes used at Niobec, and was selected here after testing several pyrochlore depressants (e.g. heated starch, and several hydrosols; flotation results are shown in Appendix 8). However, the depression produced by the starch using recycle water 2 is similar to that obtained in recycle water 1 (e.g. Figure 3.6) with no starch.

The second change was use of the new batch of fatty acid. With this new batch the collapse problems became worst making carbonate flotation impossible. Table 3.3 shows a comparison of the coalescence and froth collapse obtained with the first and the second fatty acid batches. With the second batch, even flotation with only sodium silicate as a dispersant could

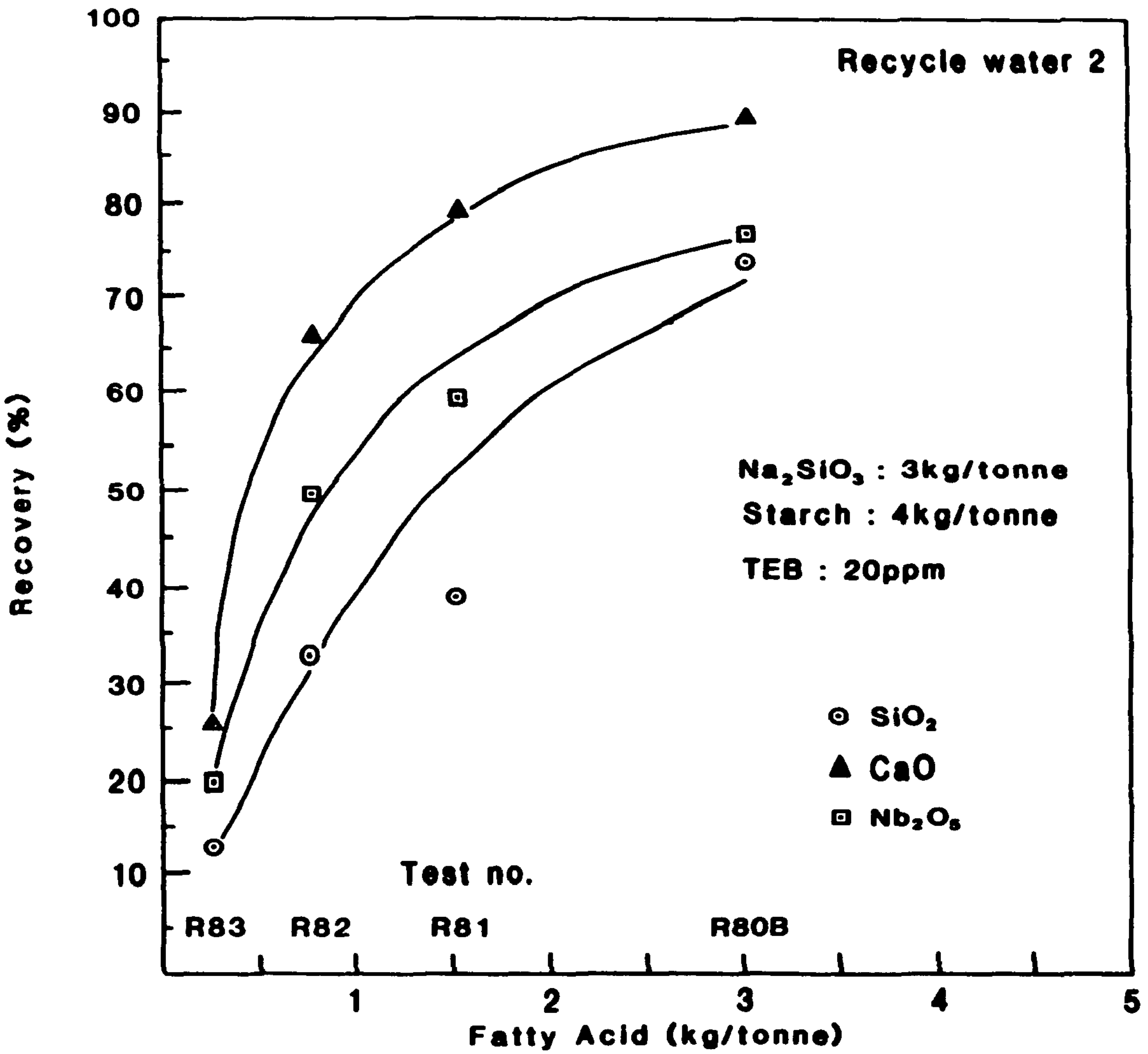


Figure 3.8 Effect of fatty acid using recycle water 2.

TABLE 3.3 .REAGENT COMBINATIONS THAT PROMOTE COALESCENCE

REAGENT COMBINATION (*)	BATCH 1 OF FATTY ACID (1)			BATCH 2 OF FATTY ACID		
	CONCENT. (PPM)	COALESCENCE	FROTH PERSISTS	CONCENT. (PPM)	COALESCENCE	FROTH PERSISTS
FATTY ACID (F.A.)(**)	10-40	NONE	YES	10-20	CONSIDERABLE	YES
ALCOMER75L + F.A.	300 10-40	NONE	YES	N.T.		
Na silicate + F.A.	10-40 10-40	NONE	YES	20 10-30	VERY STRONG	NO
DA811 + F.A.	250 40	NONE	YES	N.T.		
ALCOMER75L + Na silicate + F.A	300 30 40	STRONG	NO	N.T.		
DA811 + Na silicate + F.A.	250 40 40	STRONG	NO	N.T.		

(*) FROTHER: 1,1,3, TRIETHOXY BUTANE

(**) FATTY ACID (ACINTOL FA3) WAS ALWAYS MIXED WITH AN EMULSIFIER (LANNAGOL DP6) IN A 3:1 RATIO

NOTE: ALCOMER 75L AND DA811 WERE SUPPLIED BY ALLIED COLLOIDS; OTHER REAGENTS BY NIOBEC MINES not be performed. The exact nature of the difference between fatty acid batches is not known.

At this point it was decided to investigate coalescence/froth collapse in the fatty acid system prior to attempting further flotation.

3.7 SUMMARY AND CONCLUSIONS

This chapter described the preliminary column carbonate flotation results. There was inadequate rejection of Nb_2O_5. Poor dispersion was suspected and confirmed. A systematic dispersion study was conducted. Dispersion was quantifyed using a particle size analyser (Sedigraph). Two inorganic and eleven organic dispersants were tested, the main findings of the dispersion study were:

1) The Sedigraph is a practical, precise, and fast means of evaluating / quantifying dispersion.

2) Dispersant behavior is affected by water composition, pH, and type and dosage of flotation reagents. For example, sodium silicate is an excellent dispersant with distilled water, but promotes coagulation with recycle water due to sodium silicate / Ca, Mg interactions.

3) Dispersants caused two flotation problems, bubble coalescence and depression of carbonates. Bubble coalescence was apparently related to reagent combinations.

Coalescence and froth collapse problems intensified when using a new type of recycle water and a new batch of fatty acid. Flotation tests then became impossible to conduct.

CHAPTER 4

COALESCENCE AND FROTH COLLAPSE

4.1 INTRODUCTION

Coalescence and froth collapse problems were described in the previous chapter. Attempts were made in particular to avoid froth collapse (see Sections 3.3, 3.5 and 3.6). Of the frothers tried, TEB (1,1,3 triethoxy butane) first appeared to stop the froth collapse. However, it became evident during the test work (see Section 3.6) that the system was still unstable, and that coalescence and froth collapse problems would repeatedly re-occur.

As a consequence a more detailed study of coalescence and froth collapse in the TEB-fatty acid-dispersant-solid system was undertaken to ascertain the origin of these problems. A novel method of quantifying coalescence through measured changes in the gas holdup in the collection zone of the flotation column was developed. This chapter briefly reviews coalescence and describes the basic principles of its quantification by the method of gas holdup change. Following this (Section 4.3) is a manuscript entitled "Coalescence and froth collapse in the presence of fatty acid" (to be published in the journal "Colloids and Surfaces"), which gives an overview and includes the results and discussion.

4.2 COALESCENCE

Flotation requires a stable dispersion of fine (e.g. 1-2 mm) bubbles in the pulp phase. Fine bubbles give higher particle collection rates (Laskowsky, 1974) than coarse bubbles and also provide a higher specific surface area and thus higher carrying capacity for particles. A stable froth is required, partly to effect some upgrading (Yianatos et al, 1987c; Moys, 1978; Cutting et al 1981) and partly to transport the collected solids over the concentrate weir. Coalescence of bubbles in the pulp and in the froth (leading to froth collapse) are, therefore, detrimental to flotation.

Coalescence of bubbles can occur whenever two or more bubbles remain in contact long enough for the liquid film to become sufficiently thin that it ruptures. In flotation, stable bubble dispersions and stable froths are maintained principally by frother addition. Typical frothers include polypropylene glycol methyl ethers (e.g. DF250), 1-1-3- triethoxy butane (TEB) and methyl isobutyl carbinol (MIBC). Conditions which give fine bubble dispersion in the pulp are not necessarily the same as those which give stable froths. A froth may persists even when strong bubble coalescence in the pulp occurs (Klassen and Mokrousov, 1963b). The presence of solid particles also influences froth stability. A high concentration of solids in the froth often promotes stability (Szatkowski, 1987), sometimes giving rise to poor control over frothing (Lovell, 1986). In the case of low concentrations of fine well dispersed hydrophobic solids, however, the froth may tend to collapse (Dudenkov, 1967; Dippenaar, 1978).

Classical methods to study coalescence include:

(i) refractive index matching (in liquid extraction and effluent treatment processes) (Allak and Jeffreys, 1974), in which, for example, the refractive index of the dispersed (drops) and continuous (liquid) phases are chosen to be similar, then one or more colored drops are fed into the transparent band and their behaviour followed;

(ii) using a single drop (in liquid extraction processes) (Lang and Wilke, 1971a), where a drop (phase-1) of a more dense liquid falls through a less dense fluid (phase-2) to the interface between phase-1 and phase-2 liquids. The drop often remains at the interface a period of time before it coalesces with the bulk phase-1 material. This time period is measured as a quantification of coalescence;

(iii) by photographic techniques (Koide et al, 1968; Crabtree and Bridgewater, 1971; Yianatos et al, 1986; Argyriou et at, 1971; Dekee et al, 1986; Szatkowski, 1987); and,

(iv) contacting two bubbles (Lessard and Zieminsqki, 1971; Klassen and Mokrousov, 1963a): a number of pairs of bubbles are contacted and coalescence percentage is determined.

For purposes of this discussion coalescence refers to an increase in the mean bubble size in the collection (pulp) zone of a column. Froth collapse is treated separately and only described qualitatively. The method used to quantify coalescence is through a measured change in the gas holdup in the collection zone of the flotation column. Coalescence causes an increase in bubble diameter and thus a decrease in gas holdup. This change in gas holdup can be converted mathematically to an estimated change in bubble diameter (Yianatos et al, 1987a).

4.2.1 Quantification of Coalescence by Change in Gas Holdup

Gas holdup. The gas holdup is defined as the volume occupied by gas as a fraction of the total volume of gas and liquid (or slurry). Gas holdup (ϵg) can be measured by level rise (Chapman et al, 1983), by pressure difference (Argyriou, 1971; Yoshida and Miura, 1983; Oedjoe and Buchanan, 1966), and by conductivity (Dhanuka and Stepanek, 1978; Achwal and Stepanek, 1975; Turner, 1976; Begovich and Watson, 1978). Pressure difference was the method selected due to the simplicity and accuracy of measurement (Yianatos et al, 1986).

The static pressure at levels A and B in a column (e.g. see Figure 4.1.a) can be expressed mathematically as:

$$PA = P\ atm + \rho_{sl}\ g\ (1 - \epsilon_g A)\ LA + \rho_g\ \epsilon_g\ LA\ g \qquad (1)$$

$$PB = P\ atm + \rho_{sl}\ g\ (1 - \epsilon_g B)\ LB + \rho_g\ \epsilon_g\ LB\ g \qquad (2)$$

where P atm is the atmospheric pressure, ρ_{sl} and ρ_g are the slurry and gas density, respectively, (ρ_g is sufficiently small that it can be neglected). ϵ_g A and ϵ_g B are the average gas holdup above the points A and B, respectively. Thus the differential pressure, ΔP, between points A and B is:

$$PA - PB = \Delta P = \rho_{sl}\ g\ (1 - \epsilon_g)\ \Delta L \qquad (3)$$

where ϵ_g is the average gas holdup between A and B. Upon re-arranging

$$\epsilon_g = 1 - \frac{\Delta P}{\rho_{sl}\ g\ \Delta L} \qquad (4)$$

ΔP can be measured using water manometers (see Figure 4.1.b); thus,

$$PA = \rho_w\ g\ (\Delta L + h1) + P\ atm \qquad (5)$$

$$PB = \rho_w\ g\ h2 + P\ atm \qquad (6)$$

and,

$$\Delta P = \rho_w\ g\ (\Delta L + h1 - h2) \qquad (7)$$

$$\Delta P = \rho_w\ g\ (\Delta L - \Delta h) \qquad (8)$$

and the ϵ_g is given by:

$$\epsilon_g = 1 - \frac{\rho_w}{\rho_{sl}}\ (1 - \frac{\Delta h}{\Delta L}) \qquad (9)$$

If $\rho_{sl} = \rho_w$ (i.e. two-phase system, no solids), the solution simplifies to

$$\epsilon_g = 1 - (1 - \frac{\Delta h}{\Delta L}) \qquad (10)$$

or,

$$\epsilon_g = \frac{\Delta h}{\Delta L} \qquad (11)$$

Most of the work here involves the air-water system, consequently

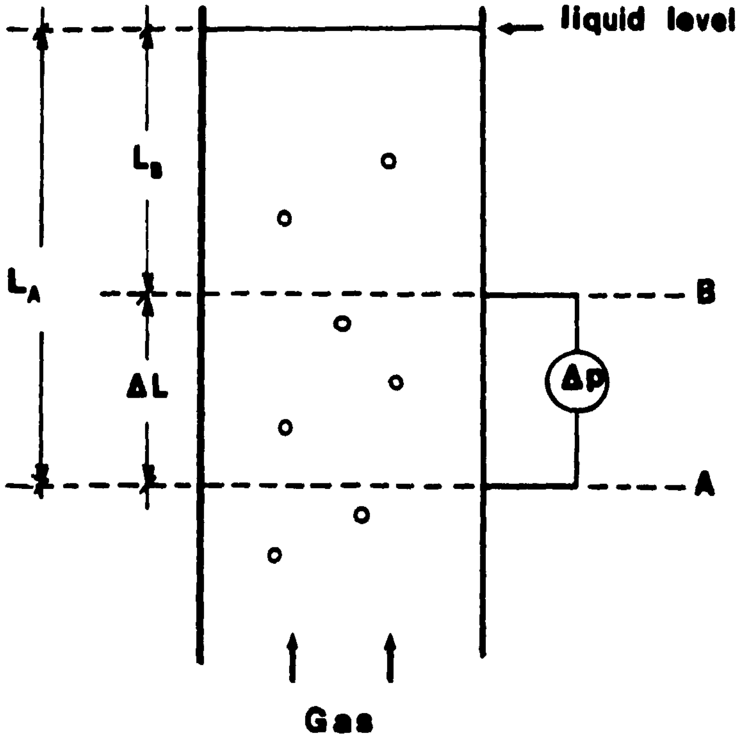


Figure 4.1.a. Static pressure at levels A and B in a column

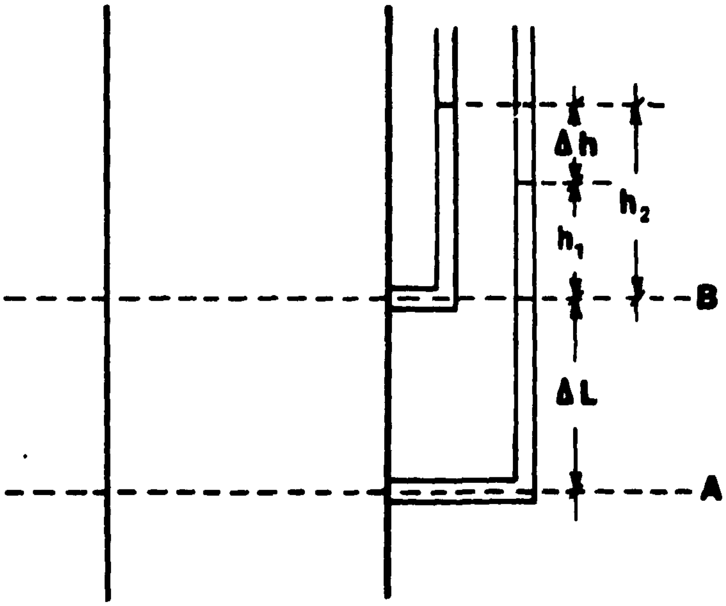


Figure 4.1.b. Differential pressure between points A and B in a column

Eq. (11) is used. There are some tests with 1%, w/w solid feeds. For a solid specific gravity of 3.2, 1% w/w is equivalent to $\rho_{sl} = 1.007$. By comparing Eq. (9) with Eq. (11) the assumption $\rho_{sl} = \rho_{water}$ introduces an error of less than 1% in the value of ϵ_g from Eq. (11). At higher pulp densities there is an appreciable difference between ϵ_g given by Eq. (9), the actual holdup, and Eq. (11), the observed holdup. This is illustrated in Appendix 9.

In the method described here (e.g. see Figure 1 in the manuscript) ϵ_g is measured at several sections along the column. In this particular work coalescence occurred close to the sparger, thus the measured ϵ_g was similar in all the sections and the average ϵ_g was used. The method does, however, provide a technique for following coalescence with distance from the bubble generator. For columns higher than about 2 m a correction factor must be applied to account for bubble diameter changes from section to section due to the hydrostatic head. Details on the experimental setup (e.g. see Figure 1 in the manuscript) are described in the manuscript (Section 4.3).

Bubble diameter. Gas holdup varies with gas velocity (Jg), liquid velocity (Jl), surfactant concentration, and can also vary with the presence of solids (Yianatos et al, 1987a; Xu et al, 1987). Jg and Jl are expressed in terms of superficial gas and liquid velocities (cm/s) i.e. volumetric flowrate per unit column cross-sectional area. Figure 4.2 shows qualitatively the relationship between ϵ_g and Jg with changes in Jl and frother dosage (i.e. bubble diameter, db). The linear ϵ_g vs Jg relationship is typical of bubbly flow conditions (Yianatos et al, 1987a; Xu et al, 1987), the desirable flow regime for column operation (Amelunxen et al, 1985). Clearly, if gas rate and liquid rate are held constant, any change in

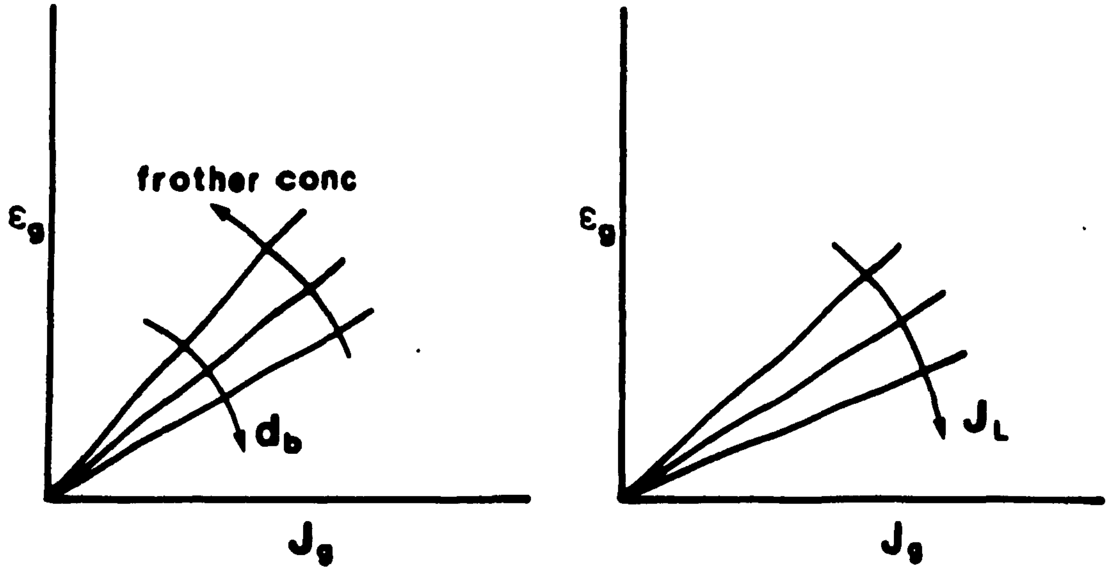


Figure 4.2 Relationship between gas holdup vs superficial gas rate: variations with frother concentration and superficial liquid velocity. (Arrow direction is for increasing values)

ϵg is attributable to a change in db.

Yianatos et al (1987a) developed a method of determining db from measurements of ϵg, Jg and Jl. This can be adapted to estimate the change in db from changes in ϵg. The method is described below.

4.2.2 Method to Estimate Bubble diameter

The proposed method consists of determining the slip velocity (using Eq. (12) from experimental conditions and then calculating the bubble diameter that gives the same slip velocity using Eq (13).

$$Us = \frac{Jg}{\epsilon g} + \frac{Jl}{1 - \epsilon g} \qquad (12)$$

$$Us = \frac{- \; g \, db^2 (1 - \epsilon g)^{m-1}}{18 \mu (1 + 0.15 Reb^{0.687})} \qquad (13)$$

Equation 12 is the definition of slip velocity (relative gas to liquid velocity) for a countercurrent gas-liquid system. Equation 13 is derived by analogy with the expression of Masliyah (1979) for hindered settling in a multi-species particulate system, where db is the bubble diameter (cm), g is the acceleration due to gravity (cm/s^2), and μ is the liquid viscocity (g/cm.s). Reb is the bubble Reynolds number defined as:

$$Reb = \frac{db\ Us\ \rho l\ El\ (1 - \epsilon g)}{\mu} \qquad (14)$$

The parameter m is related to bubble Reynolds number. According to Richardson and Zaki (1954):

for 1<Re<200

$$m = (4.45 + 18\ (db/dc))\ Re^{-0.1} \qquad (15)$$

where dc is column diameter, (cm)

and, for 200<Re<500

$$m = 4.45\ Re^{-0.1} \qquad (16)$$

where the Reynolds number is defined (Richardson and Zaki, 1954) as:

$$Re = \frac{db\ Ut\ \rho l}{\mu l} \qquad (17)$$

where

$$Ut = \frac{Us}{(1- \epsilon g)^{m-1}} \qquad (18)$$

Because db occurs directly and indirectly (in Reb and m) in Eq. (13) an iterative solution is required.

To preserve the analogy with particle settling, Reb must be smaller than 500 (Yianatos et al, 1987a) corresponding to db < 2 mm. Bubbles larger than this rise in a tortuous path and the drag coefficient is different.

The method of solution is illustrated in Figure 4.3 (Xu et al, 1987), for Jg = 1.2 cm/s and Jl = 0.5 cm/s. The Us from Eq. 12 is plotted against ϵg. The actual Us, ϵg combination depends on db. The Us from Eq. 13 is plotted against ϵg for various db. Where the two curves intersect

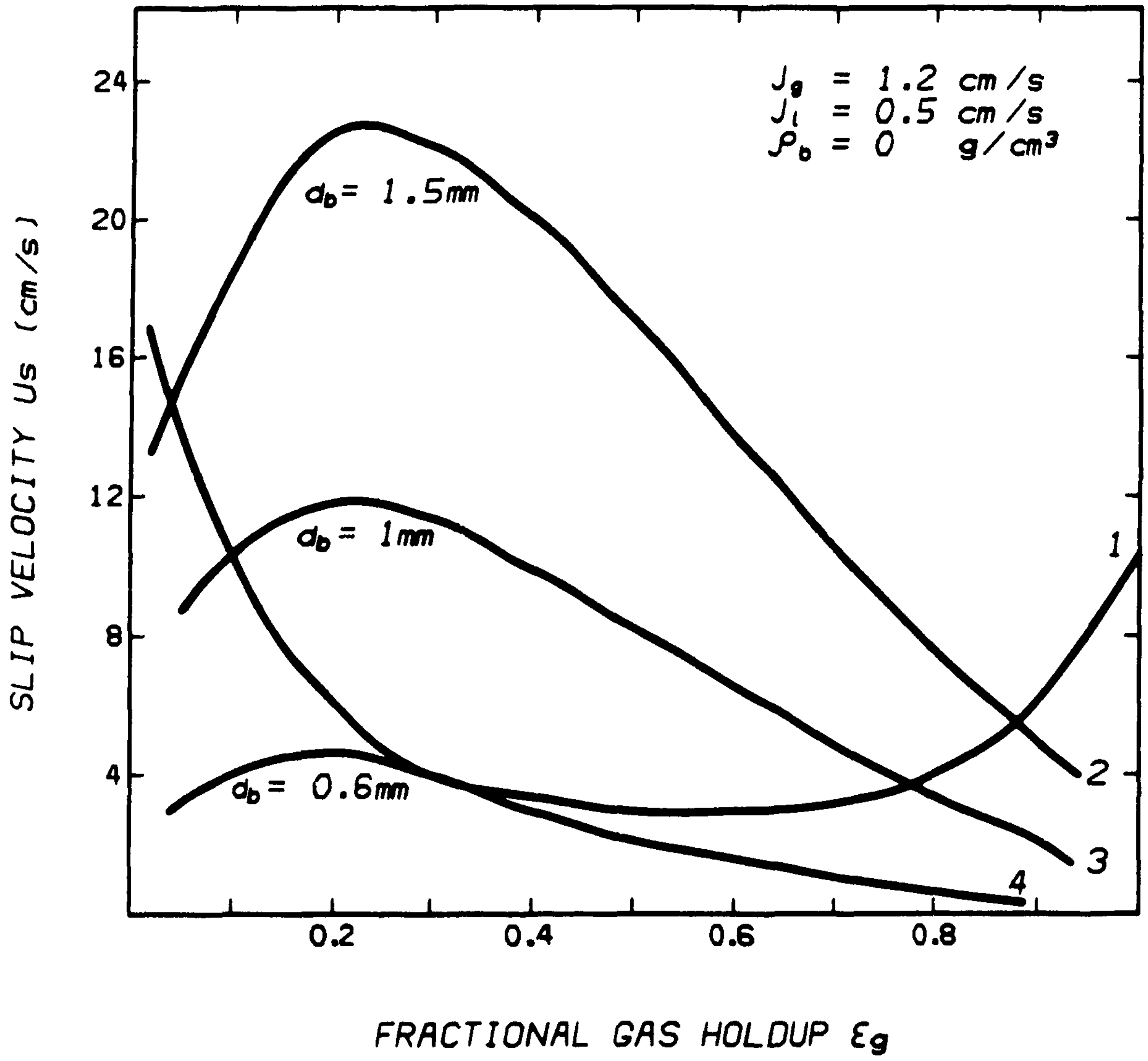


Figure 4.3 Determination of db. Illustration of method of solution

gives the ϵ_g for that db. For example, if for the given conditions of Jg, Jl, ϵ_g is measured to be 10% then db must be about 1.0 mm; if ϵ_g is measured to be 30% then db must be about 0.6 mm. There is no solution for db less than 0.6 mm. The physical interpretation of this is that at Jg = 1.2 cm/s, Jl = 0.5 cm/s the minimum db is about 0.6 mm. Bubbles smaller than 0.6 mm will cause instability and loss of the bubbly flow regime (i.e. transition to churn-turbulent flow (Xu et al, 1987)--where Eq. 13 no longer applies.

The method has been tested for a variety of column geometries and chemical conditions, e.g. see Table4 1 (column 1 is the same column used in this work) and Figure 4.4 from Yianatos at al (1987a). In all situations the agreement is within +/- 15% of the measured mean bubble diameter which is of the order of the error in measuring db (Yianatos et al, 1987a).

The model permits bubble diameter to be estimated from the gas holdup. It is used in this coalescence study to convert the change in gas holdup upon change in solution chemistry into the corresponding change in db.

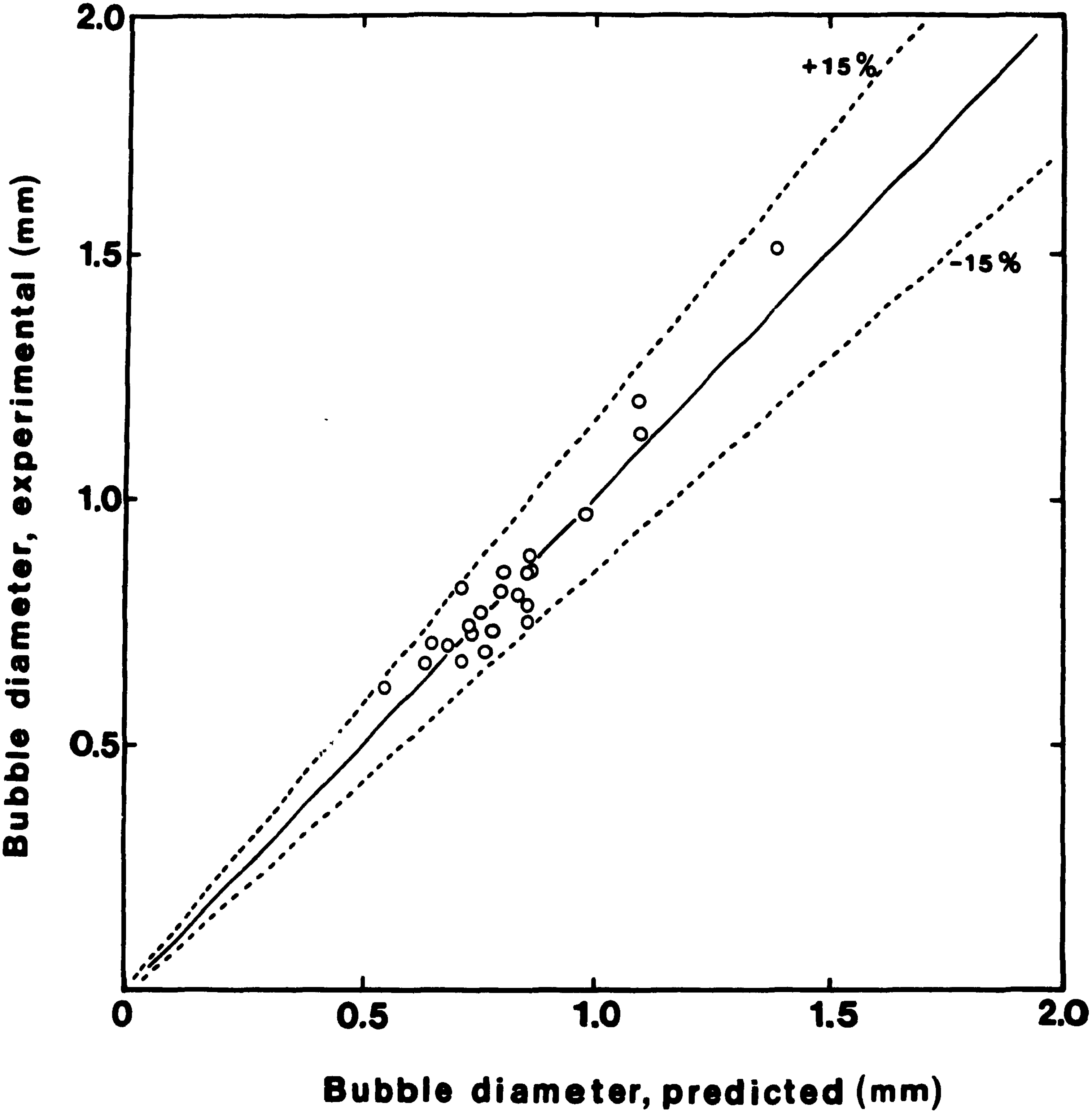


Figure 4.4 Bubble diameter: experimental vs predicted

TABLE 4.1 CHARACTERISTICS OF THE COLUMNS WHERE THE METHOD OF YIANATOS ET AL HAS BEEN TESTED

COLUMN	SHAPE	DIAMETER (side)cm	HEIGHT cm	SPARGER
1	circular	3.81	200	ceramic
2	circular	5.71	450	ceramic
3	rectangular	2.5x10	180	steel

4.3 MANUSCRIPT 3:

COALESCENCE AND FROTH COLLAPSE IN THE PRESENCE OF FATTY ACID

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ABSTRACT

Bubble coalescence and froth collapse occurred during carbonate/silicate separation tests in a flotation column using a commercial fatty acid. To study this a method of quantifying coalescence is developed using the change in gas holdup in the collection (pulp) zone of the flotation column and the calculated change in bubble diameter.

Fatty acid (mainly oleic acid), in 3:1 ratio with lannagol emulsifier, caused significant coalescence. For example, 10 ppm of fatty acid added to 20 ppm of TEB (1,1,3 triethoxy butane), a frother, caused a 55 % decrease in gas holdup (corresponding to a 50 % increase in bubble diameter) and collapsed the froth completely. Higher concentrations of lannagol stabilized the froth, but failed to prevent bubble coalescence. Solids in the presence of sodium silicate, a dispersant, further enhanced coalescence and froth collapse.

It is observed that there was no simple relationship between coalescence and froth collapse as a persistent froth could be maintained even with high coalescence.

A mechanism of coalescence is proposed based on hydrophobic coating of the bubble causing water drainage.

INTRODUCTION

Flotation requires a stable dispersion of fine bubbles in the pulp phase to collect mineral particles and a stable froth to carry the collected solids over the concentrate weir. Coalescence of bubbles in the pulp and froth collapse are, therefore, detrimental to flotation.

Stable bubble dispersions and stable froths are maintained principally by frother addition. However, the relationship between the two is not necessarily simple; a froth may persists even when strong bubble coalescence in the pulp occurs (Klassen and Mokrousov, 1963b). An additional factor is the presence of solids. For example, fine dispersed hydrophobic solids sometimes promote froth collapse (Dudenkov, 1967; Dippenaar, 1978)

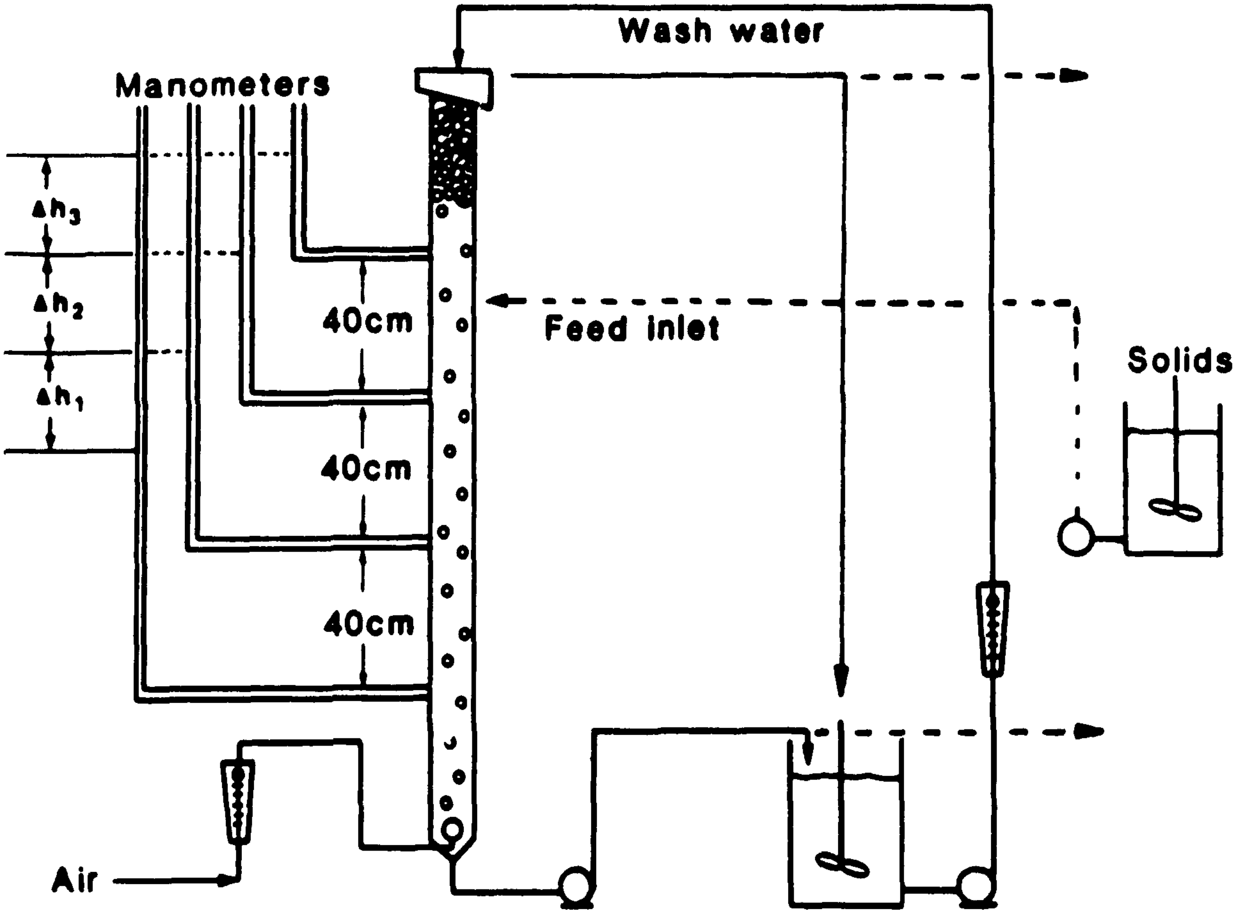
In a previous column flotation study carbonates were floated from slimes discarded at the Niobec concentrator (Les Services TMG Inc) (Espinosa-Gomez et al, 1987b) using plant recycle water and a fatty acid, Acintol FA3 (hereafter referred to as FA). Bubble coalescence and froth collapse problems were described. Typical conditions were a FA dosage of 2 kg/tonne solids and pulp densities of 1 % w/w (low pulp densities were necessitated by availability of sample). Of the frothers tried, TEB (1,1,3, triethoxy butane), yielded the most stable froth. However, coalescence and froth collapse problems repeatedly re-occured, and prevented further testing (Espinosa-Gomez, 1987).

As a consequence a more detailed study of coalescence and froth collapse in the presence of FA was undertaken. Classical methods to study coalescence include: refractive index matching (in liquid extraction and effluent treatment processes) (Allak and Jeffreys, 1974); using a single drop (in liquid extraction processes) (Lang and Wilke, 1971a); by photographic techniques (Koide et al, 1968; Crabtree and Bridgwater, 1971; Yianatos et al, 1986; Argyriou et al, 1971; Dekee et al, 1986); and contacting two bubbles (Lessard and Zieminsqki, 1971; Klassen and Mokrousov, 1963a). A novel method of quantifying coalescence through measured change in the gas holdup in the collection zone of a flotation column is described here. Coalescence causes an increase in bubble diameter and thus a decrease in gas holdup. The change in gas holdup can be converted mathematically to an estimated change in bubble diameter. The technique is used to examine the TEB-fatty acid-dispersant-solid system. At the same time froth stability is described qualitatively.

QUANTIFYING COALESCENCE

Gas holdup: Fractional gas holdup, ϵg, was calculated from the pressure difference over a section of length L (Figure 1). Pressure is measured by water-filled manometers, thus ϵg is given by $\epsilon g = \Delta h/L$, where Δh is the difference in manometer readings. The flotation column was operated in closed and open circuit. When working in closed circuit the average gas holdup over the three sections (Figure 1) was taken. When working in open circuit, the liquid superficial velocity above the feed point is different from that below, affecting gas holdup. Thus, only two sections below the feed point are used.

The estimated absolute error in the readings of the manometers was ± 0.5 %. The standard deviation of ϵg was estimated at 0.4 % from full test repeats. At a constant gas flowrate and chemical environment a decrease in gas holdup indicates an increase in bubble diameter, i.e. coalescence. (For columns 2 m or less, changes in bubble size and gas holdup due to hydrostatic head can be ignored).



$$\text{LOCAL } \varepsilon_g = \frac{\Delta h_i}{40} \qquad \text{OVERALL } \varepsilon_g = \frac{\Sigma \Delta h_i}{120}$$

Figure 1.: Full line, closed circuit arrangement, dashed line open circuit arrangement.

Results from the coalescence study are reported as per cent change in gas holdup relative to the gas holdup with 20 ppm TEB only, $\Delta\epsilon_g$, i.e.

$$\Delta\epsilon g = \frac{(\epsilon g(x) - \epsilon g(20))}{\epsilon g(20)} \times 100 \qquad (1)$$

where $\epsilon g(x)$ is the gas holdup at condition 'x' and $\epsilon g(20)$ the gas holdup at 20 ppm TEB. From full test repeats the standard deviation on $\Delta\epsilon g$ was 8 %.

Bubble diameter: Bubble diameter was estimated from the average gas holdup measurement and the column operating conditions using the technique described by Yianatos et al (1987a). The term bubble diameter in this paper is the 'mean bubble diameter' of the bubble swarm. A typical relative standard deviation of the mean bubble diameter in the presence of frother is about 20 % (Yianatos et al, 1987a). The conditions used in this work are similar to those used by Yianatos et al., bubble Reynolds numbers less than about 500 and bubble diameters between 0.5 to 1.5 mm.

The approach to estimating db consists of determining the slip velocity (Eq. 2) from experimental conditions and then calculating the bubble diameter that gives the same slip velocity velocity (Eq. 3):

$$Us = \frac{Jg}{\epsilon g} + \frac{Jl}{1 - \epsilon g} \qquad (2)$$

$$Us = \frac{g\, db^2 (1 - \epsilon g)^{m-1}}{18\, \mu (1 + 0.15 Reb^{0.687})} \qquad (3)$$

Equation 2 is the definition of slip velocity (relative gas to liquid velocity) for a countercurrent gas-liquid system, where Jg and Jl are the superficial gas and liquid velocities (cm/s), respectively, and ϵg is the fractional gas holdup. Equation 3 is an adaptation of the expression of Masliyah (1979) for hindered settling in a multi-species system, where db is the bubble diameter (cm), g is the acceleration due to gravity (cm/s^2), μ is the liquid viscocity (g/cm.s), Reb is the bubble Reynolds number, and m is a parameter related to bubble Reynolds number (Yianatos et al, 1987a; Richardson and Zaki, 1954). Reb is defined as:

$$Reb = \frac{db\ Us\ \rho_l\ \epsilon_l}{\mu} \qquad (4)$$

Because db occurs directly and indirectly (in Reb and m) in (Eq. 3) an iterative solution is required.

The method has been tested for a variety of column geometries and chemical conditions (Yianatos et al, 1987a). Table 1 summarizes results obtained on the column used here with three frother types. Estimated bubble diameter is within ± 15 % of the measured.

TABLE 1. BUBBLE DIAMETER: MEASURED VS ESTIMATED

FROTHER CONCENT. (PPM)	FROTHER TYPE (*)								
	DF250			TEB			MIBC		
	GAS HOLDUP (%)	BUBBLE DIAM. MEAS. (mm)	ESTIM.	GAS HOLDUP (%)	BUBBLE DIAM. MEAS. (mm)	ESTIM.	GAS HOLDUP (%)	BUBBLE DIAM. MEAS. (mm)	ESTIM.
5	9.5	1.2	1.15	11.23	0.97	1.01	13.2	0.78	0.89
10	12.9	0.86	0.9	13.15	0.85	0.89	13.3	0.75	0.89
15	15.8	0.77	0.79	14.42	0.85	0.84	13.6	0.8	0.88
20	15.5	0.69	0.8	17.7	0.82	0.74	15.3	0.73	0.81
25	16.2	0.73	0.77	21.53	0.71	0.67	18.0	0.67	0.75

(*) DF250 IS POLYPROPYLENE GLYCOL METHYL ETHER
TEB IS 1-1-3 TRIETHOXY BUTANE
MIBC IS METHYL ISOBUTYL CARBINOL

EXPERIMENTAL PART

Figure 1 shows the experimental set up. A Plexiglas column, 3.8 cm in diameter and 200 cm in height, was used. Bubbles were generated with a ceramic ball sparger of surface area 19.6 cm^2, nominal pore diameter of 60 μm and estimated porosity 160 holes/cm^2. (Xu, 1987). During all the experiments temperature was kept at 25 ± 2 °C. Wall effects should be negligible as the ratio of column diameter to bubble diameter is about 40, much greater than the limiting value of 10 suggested by Steiner et. al. (1977), and Collins (1967).

The column was operated with and without solids. It was operated in closed circuit when working without solids, in which case the only feed to the system was wash water. With solids the column was operated in open circuit, the solids being added through the feed inlet (Figure 1), and the concentrate and tailings discarded. Unless otherwise stated the feed was at 1 % w/w solids of particles about 1-15 μm (Espinosa-Gomez et al, 1987b) and wash water contained the same reagent concentration as the feed water. For the typical conditions of 2 kg FA/tonne solids, at 1 % w/w solids this translates to about 20 ppm FA. Consequently, FA dosages were in the 0 to 40 ppm range to simulate the previous conditions (Espinosa-Gomez et al, 1987b). Column operating conditions were: superficial gas rate (Jg), 0.7 cm/s; superficial wash water rate (Jw), 1.0 cm/s; and froth depth, about 30 cm. The usual identification of collection zone and froth zone for the zones below and above the interface, respectively, will be used (Dobby and Finch, 1986a). The superficial liquid rate (Jl) in the column was 0.65 cm/s and 1.36 cm/s, for closed and open circuit, respectively.

The flotation reagents used were: purified oleic acid (identified as low in linoleic acid, from Fisher Scientific); fatty acid (Acintol FA3, from

Arizona Chemicals), which contains mainly oleic and linoleic acids; fatty acid emulsifier (lannagol DP6, from Diamond Shamrock), sodium silicate (silicate mineral depressant/dispersant, from National Silicate), and the frother 1,1,3-triethoxybutane (TEB, from National Chemical Products). These are the reagent suppliers used by Niobec Mines.

Table 2 summarizes the test conditions. Sixteen tests were performed, seven of them with solids. Tests 12,13 and 14 re-create the previous tests conditions (Espinosa-Gomez et al, 1987b) with only frother in the wash water. Test 16 involved 10 % w/w solids feed.

RESULTS

Figure 2 shows the estimated change in bubble diameter vs the change in gas holdup at $J1 = 0.65$ cm/s (closed circuit) and $J1 = 1.36$ cm/s (open circuit). The gas holdup at 20 ppm TEB ($\epsilon g(20)$) was 13.3 % in closed circuit, and 17.8 % in open circuit.

Figure 3 shows the change in gas holdup as a function of FA concentration, using Montreal tap water and Niobec plant recycle water. The error bars indicate the 95 % confidence interval on the mean. Gas holdup decreased by 45 % at FA concentrations greater than 20 ppm, corresponding to a bubble diameter increase of about 56 % (Figure 2). As indicated, the froth collapsed below 10 ppm, but persisted above this value.

Apart from the difference in gas holdup at 5 ppm FA, both tap and recycle water gave similar holdup and froth stability results. For present purposes Montreal tap water provides an adequate simulation. The large volumes of water required, 14-20 L per test, precluded the use of recycle water (and distilled water) which was in restricted supply.

Table 3 shows the effect of lannagol on gas holdup and froth

TABLE 2. SUMMARY OF TEST CONDITIONS

TEST (*) #	REAGENT COMBINATION (**)	REAGENT CONCENTRATION RANGE (PPM)	SOLIDS (***)	FEED SYSTEM (****)
1	F.A.	(5-40)	NO	CLOSED
2	F.A.	(5-40)	NO	CLOSED
3	F.A.	(5-40)	NO	CLOSED
4	F.A.	(5-20)	NO	OPEN
5	F.A.	(5-20)	YES	OPEN
6	F.A.	(5-40)	NO	CLOSED
7	F.A./Na silicate	(5-40)/(40)	NO	CLOSED
8	F.A./Na silicate	(5-20)/(20)	NO	OPEN
9	F.A./Na silicate	(5-20)/(20)	YES	OPEN
10	LANNAGOL	(10)	YES	OPEN
11	OLEIC ACID	(5-40)	NO	CLOSED
12	F.A./Na silicate	(80)/(20)	YES	OPEN (*****)
13	F.A./Na silicate	(40)/(20)	YES	OPEN (*****)
14	F.A./Na silicate	(20)/(20)	YES	OPEN (*****)
15	F.A./LANNAGOL	(5)/(1.7-5)	NO	CLOSED
16	F.A./Na silicate	(300)/(300)	YES	OPEN (*****)

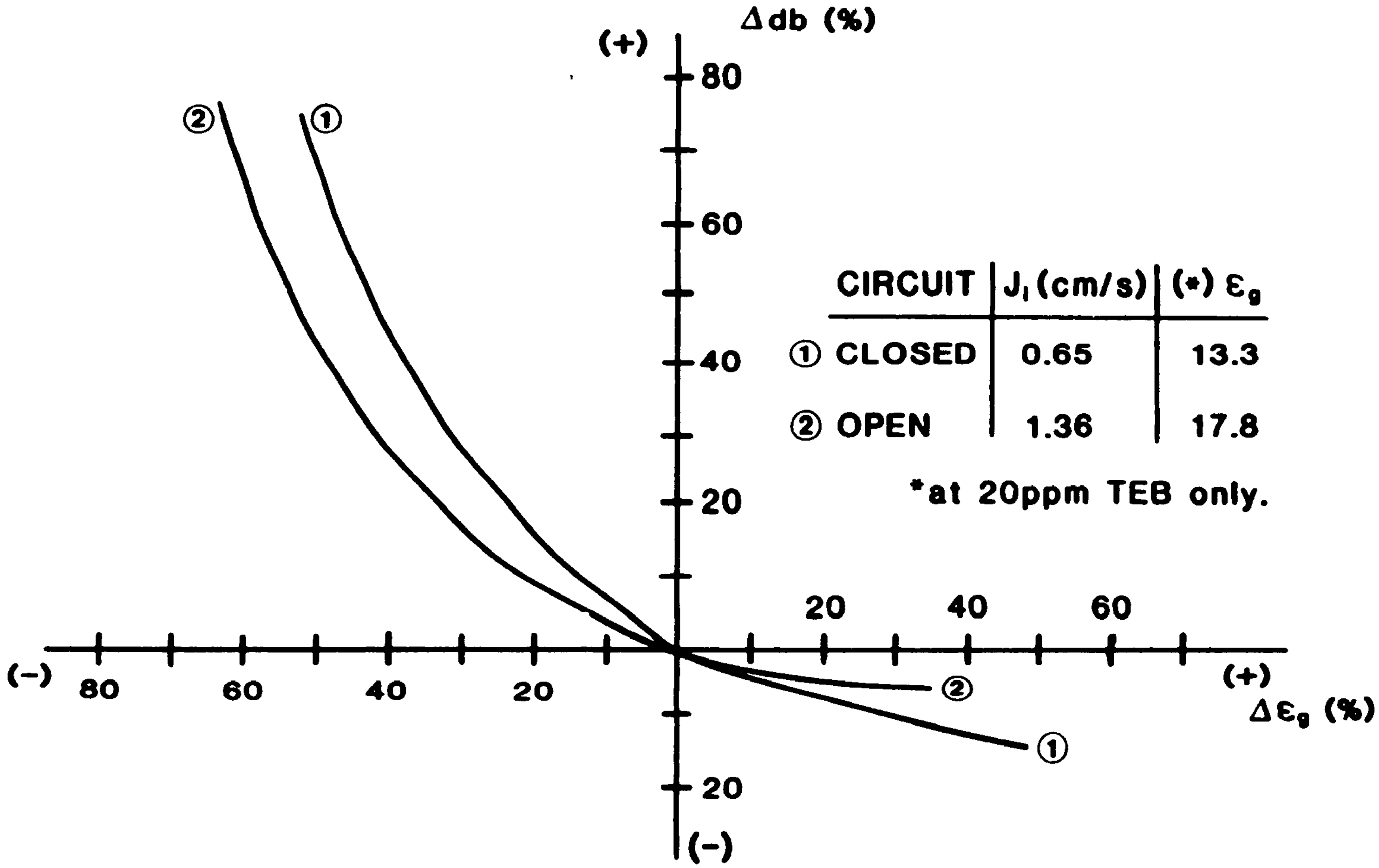
(*) ALL THE TESTS WERE PERFORMED WITH MONTREAL TAP WATER EXCEPT TESTS 2 AND 3, DONE WITH PLANT RECYCLE WATER

(**) F.A., FATTY ACID (ACINTOL FA3) WAS ALWAYS ADDED AS AN EMULSION, WITH THE EMULSIFIER LANNAGOL DP6 IN A 3:1 (ACID : LANNAGOL) RATIO, EXCEPT IN TEST 15

(***) SOLIDS WERE FED AT 1 % W/W SOLIDS, EXCEPT IN TEST 16 (10% SOLIDS)

(****) OPEN CIRCUIT WAS ESSENTIAL WHEN WORKING WITH SOLIDS

(*****) ONLY FROTHER ADDED IN THE WASH WATER



CIRCUIT	J_l (cm/s)	(*) ε_g
① CLOSED	0.65	13.3
② OPEN	1.36	17.8

*at 20ppm TEB only.

Figure 2 .: Relationship between change in bubble size (Δdb) vs change in gas holdup (Δε_g), J_g = 0.70cm/s

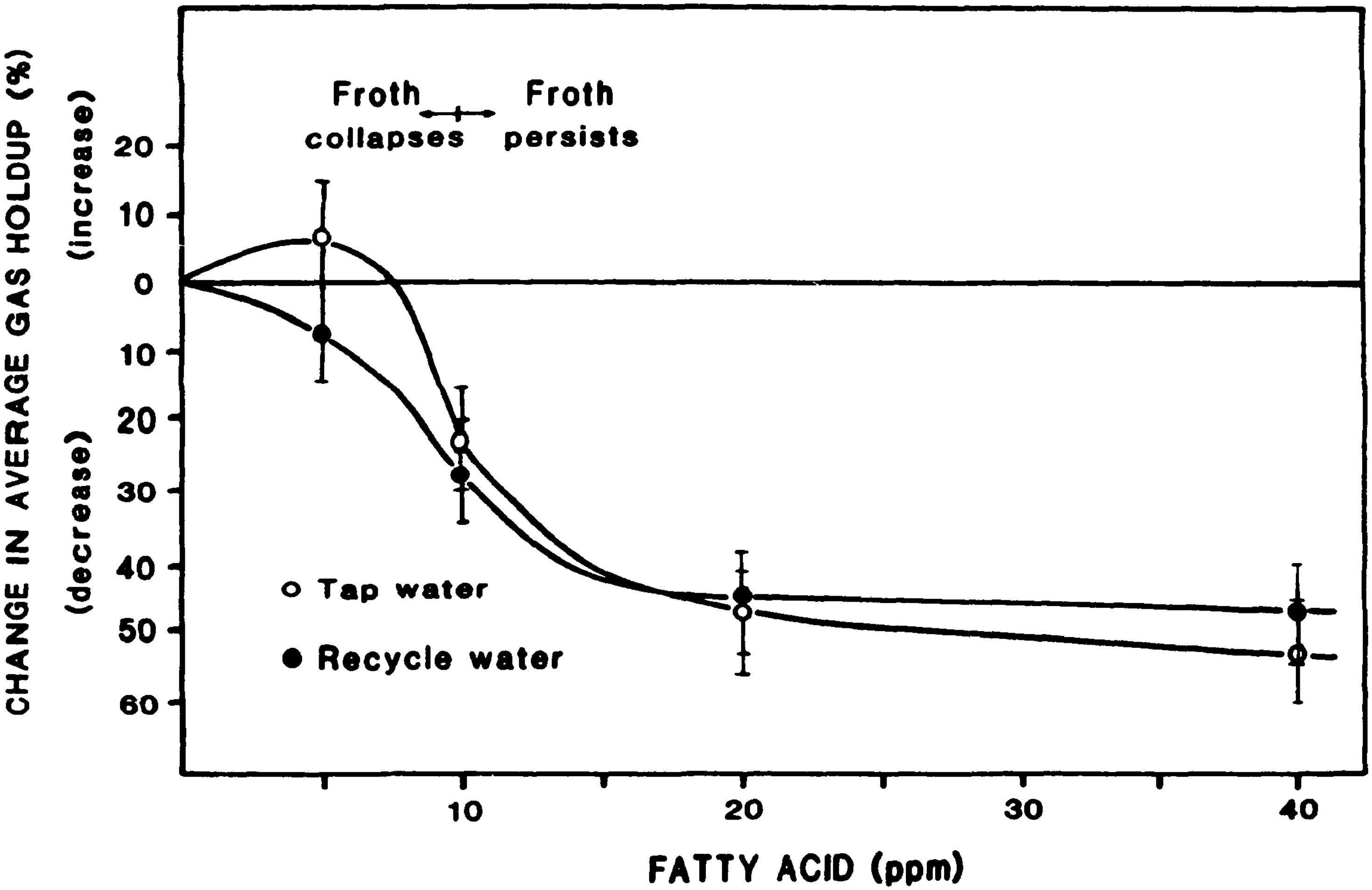


Figure 3.: Effect of fatty acid concentration in the average gas holdup using two types of water. Working in closed circuit

Table 3. Effect of Lannagol on Froth Stability and Gas Holdup at 20 ppm TEB and 5 ppm Fatty Acid (F.A.)

LANNAGOL (PPM)	RATIO OF F.A./LANNAGOL	INCREASE IN AVERAGE GAS HOLDUP (%)	FROTH CHARACTERISTICS
1.66	3 : 1	2.5	COLLAPSES
2.50	3 : 2	8.5	UNSTABLE (*)
5.00	3 : 3	11.0	PERSISTS

(*) Large bubbles, periodic collapse

stability at 20 ppm TEB and 5 ppm FA (a combination which causes froth collapse at FA:lannagol ratios of 3:1 (Figure 3)). Froth stability increased as the lannagol concentration increased, with a persistent froth above 5 ppm lannagol. The gas holdup increased slightly with lannagol.

Tests using purified oleic acid without lannagol showed froth collapse even up to 40 ppm; the froth was restored upon adding 10 ppm lannagol. Lannagol on its own gave stable froths above 2-3 ppm.

Figure 4 shows the effect of solids (Figure 4a) (conducted in open circuit) and sodium silicate (Figure 4b) (conducted in closed circuit). The general conclusion is that neither solids nor sodium silicate on their own significantly affected coalescence or froth stability. In both cases the froth collapsed at concentrations below 5 ppm FA, and it persisted at concentrations greater than 10 ppm. In detail it is noted that for the closed circuit tests at 5 ppm FA, 40 ppm sodium silicate increased the gas holdup by a further 10 % (cf Figure 3).

Figure 5 shows the combined effect of 20 ppm sodium silicate and solids. Coalescence increased over that obtained for either on their own. For example, gas holdup decreased an additional 8 % at 20 ppm FA, corresponding to an additional increase in bubble diameter (from Figure 2) of 28 %. With solids, the froth collapsed at 5 ppm and only an unstable froth could be formed at 10 ppm. Above 20 ppm FA the froth persisted.

Three tests (Tests 12-14) were done using only frother in the wash water, since this was the method used in the previous work (Espinosa-Gomez et al, 1987b). In these tests the minimum FA concentration required to form a froth was 40 ppm. This concentration compares with the maximum concentration (30 ppm) used previously when trying to make a persistent froth.

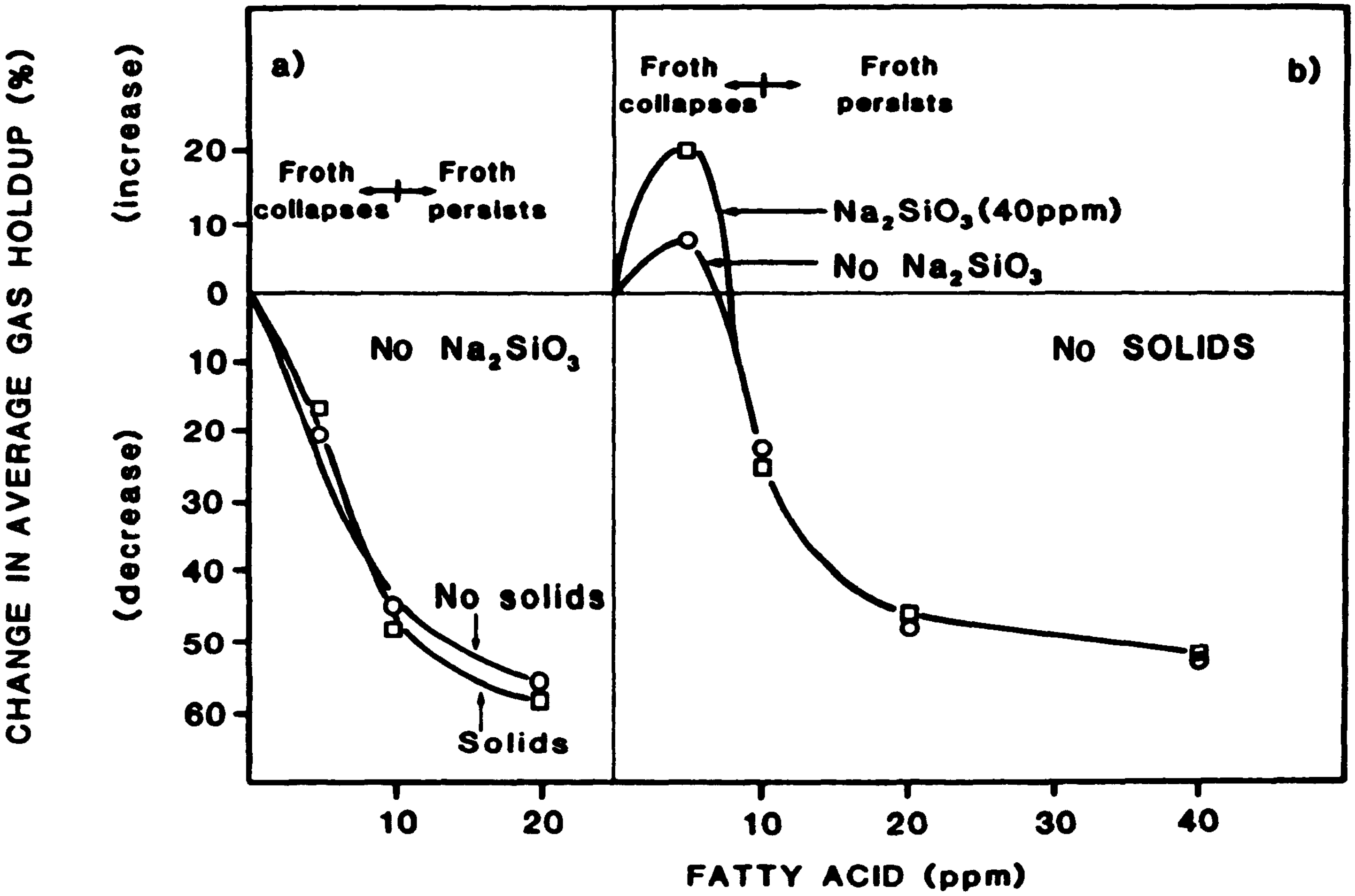


Figure 4.: Effect of solids and sodium silicate on gas holdup at several fatty acid concentrations. (TEB conc. : 20ppm)

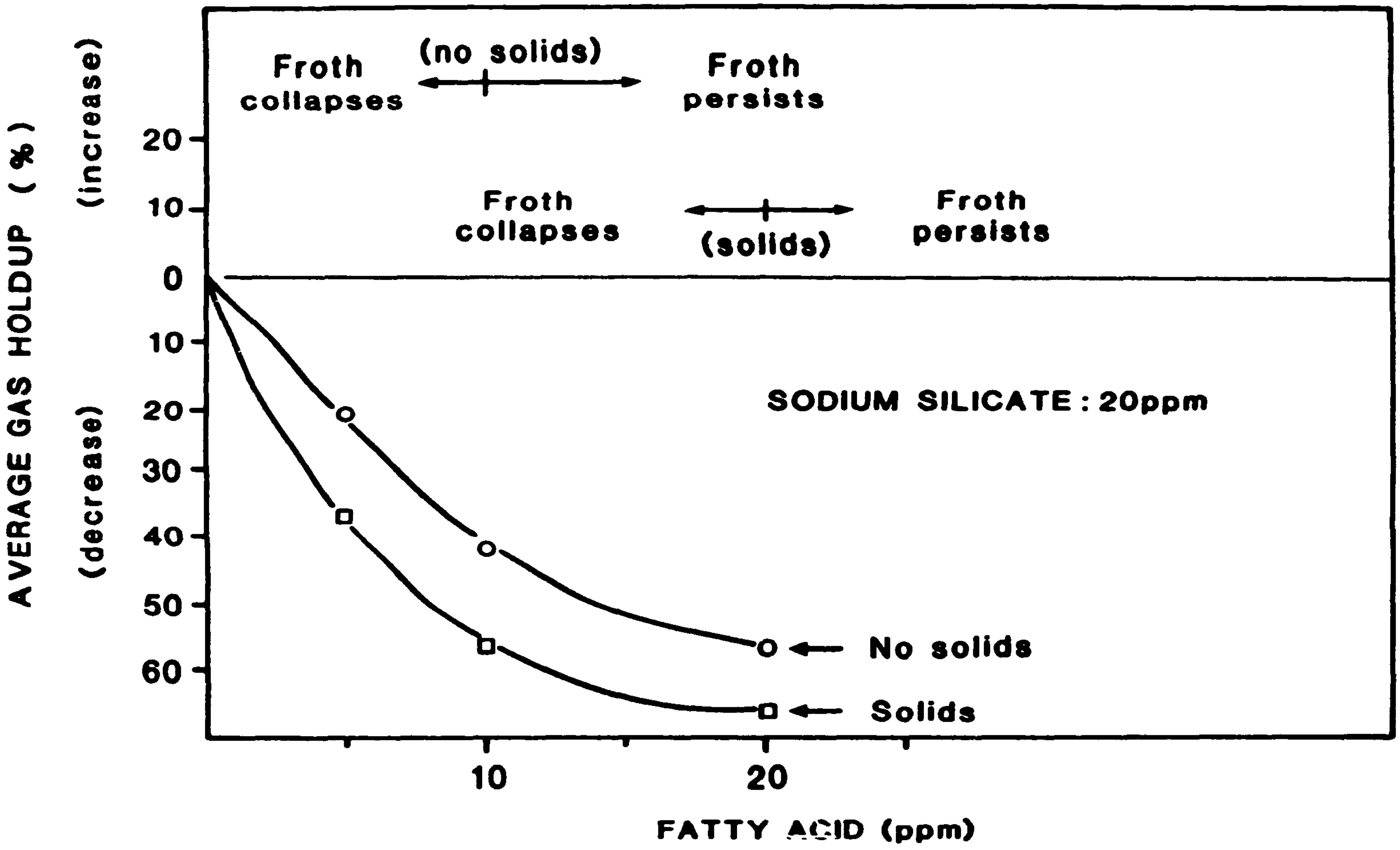


Figure 5.: Effect of solids and sodium silicate in the average gas holdup at several fatty acid concentrations working in open circuit. (TEB conc. : 20ppm)

One test (Test 16), was performed at 10 % solids instead of 1 %. The FA addition was 3 kg/tonne solids. At 1 % solids this is equivalent to 30 ppm FA and 10 ppm lannagol. At 10 % solids, the equivalent concentration in solution increases to about 300 ppm F A and 100 ppm lannagol, respectively. A persistent froth was readily formed at 10 % solids. Subsequent pilot-scale column flotation tests at the plant site with 15 % solids and 0.6 kg FA/tonne solids and no frother gave persistent froth (Espinosa-Gomez, 1987).

DISCUSSION

Method of determining coalescence: Gas holdup is a sensitive indicator of changes in bubble diameter in a bubble swarm. As the absolute estimated error in the manometer readings is 0.5 % any gas holdup change greater than 1.4 % is significant at a 95 % confidence interval. It should prove a useful new tool in the study of coalescence. In addition, provided the restrictions in (Eq. 3) are respected, the corresponding bubble diameter change can be calculated. The method could also be adapted to detect progressive coalescence along the column. In the present case coalescence was not progressive, it occurred close to the sparger.

Mechanisms of coalescence/froth collapse: Frothers retard bubble coalescence and froth collapse by a combination of mechanisms including electrostatic repulsion, the Marangoni effect and stabilization of water on the bubble surface (Bikerman, 1958).

It has long been known that FA produces coarse bubbles and poor froths (Klassen and Mokrousov, 1963b). The effect of FA here may be to compete with the frother at the bubble surface. A possibility is that the FA forms small droplets at the bubble surface because of its low solubility (0.6×10^{-6} mol/L) (Leja, 1982a). The droplets will have a low surface tension and thus will spread. The bubble, therefore, acquires a hydrophobic coating which promotes film drainage. This effect was plainly evident in the absence of lannagol where 'oil patches' were visible in the froth. With sufficient lannagol, about 7-10 ppm (in the presence of dispersed solids), a stable froth can be maintained in the presence of FA, presumably because of lannagol's own frothing power.

Hydrophobic coating as a mechanism of bubble coalescence has been suggested by Harris (1982). Examples of other substances which promote coalescence through formation of hydrophobic coatings are fuel oil and heavy metal xanthate precipitates (Klassen and Mokrousov, 1963).

The change from froth collapse to persistent froth was not accompanied by any significant change in coalescence (e.g. see Figure 5). This observation supports that of others (Klassen and Mokrousov, 1963) showing there is no simple relationship between bubble diameter and froth stability. It is possible that similar competing mechanisms are at play in both the collection and froth zones but in the dynamic environment of the collection zone coalescence forces predominate, while in the more static froth, film stabilizing forces prevail.

The increase in coalescence and froth collapse in the presence of solids and sodium silicate (Figure 5) may also be the result of hydrophobic coating. Sodium silicate is known to disperse these solids (Espinosa-Gomez et al, 1987b). The effect of dispersed hydrophobic solids on froth stability has been studied by Dudenkov (1967) and by Dippenaar (1978). Both researchers found that well dispersed hydrophobic particles accelerated the coalescence of bubbles and de-stabilised froths. It was postulated that as bubbles with some hydrophobic particles on their surface came into contact each interface would try to establish its equilibrium position on the hydrophobic particle. This would reduce the thickness of the film between the bubbles to almost zero on either side of the particle and lead to immediate rupture of the film. It was also shown that flocculated fine particles tended to reduce the effect of froth destruction, probably due to water retention in the aggregate. This may be why undispersed solids (Figure 4a) did not affect the froth.

Metallurgical implications: The objective of the previous work (Espinosa-Gomez, 1987a) was to study carbonate flotation from the Niobec slimes reject using a flotation column. Because of the need to run the column continuously (minimum 10 mins to reach steady state) preparation of even 1 % w/w solids pulps of 1-15 μm particles required processing over 250 L of slime pulp. This pulp had to be cycloned in several stages to eliminate the colloidal (-1 μm) fraction (Espinosa-Gomez, 1987). Thus, to prepare samples in the laboratory for continuous column runs with 10 % or 15 % w/w solid pulps was simply impractical; the one run at 10 % w/w solids consumed all the remaining sample.

The use of dilute feeds does not normally introduce problems. For example, even lower values are used in microflotation tests (Espinosa-Gomez et al, 1987a) and similar values to those here are reported in pilot and plant flotation columns (Dobby, 1984; Dobby and Finch, 1985a; Laplante et al, 1988). Indeed, diluting the feed to flotation cleaning stages is recommended for improving concentrate grades (Johnson et al, 1974). In this case, however, the dilute feed presented a problem.

The problem is related not to the presence or absence of solids as such but to the corresponding lannagol concentration in solution. Reagent addition rate is usually related to the solids; in this case FA addition rate is typically 2 kg/tonne solids. At 1 % w/w solids this translates to about 7 ppm lannagol in solution and consequent coalescence and froth instability problems; at 15 % w/w (as at the plant) this translates to about 100 ppm lannagol and no coalescence/froth collapse problems. The simple expedient of increasing addition rates in the laboratory to compensate was ineffective since this induced unselective flotation (Espinosa-Gomez, 1987).

The only viable solution is to conduct the experiments at plant-site where there is no restriction on solids availability. This conclusion is supported by Test 16 and the preliminary test results at Niobec refered to here, and reported in detail elsewhere (Espinosa-Gomez, 1987).

CONCLUSIONS

1) A novel, sensitive technique of quantifying coalescence by the change in gas holdup in a flotation column is described. Absolute changes in gas holdup greater than 0.5 % should be detectable. The change in gas holdup can be converted to a change in bubble size.

2) The FA in 3:1 ratio with lannagol, increases coalescence up to about 20 ppm FA.

3) Coalescence was not related to froth stability. Stable froths could be maintained at lannagol concentrations greater than about 3 ppm (in the absence of solids), with no change in coalescence.

4) Well dispersed hydrophobic fine particles promoted further coalescence and froth instability. Lannagol concentrations greater than about 7 ppm were required for froth stability, in the presence of solids.

5) The effect of fatty acid and solids on coalescence and froth collapse is tentatively related to hydrophobic coating of the bubble, promoting drainage of the water film.

6) The coalescence and froth collapse problems are related to using low 1 % w/w solid pulps as feed to a flotation column. At typical addition rates of 2 kg FA/tonne solid, pulps densities greater than 1 % w/w are required to ensure adequate lannagol in solution to promote a stable froth.

4.4 SUMMARY OF CONCLUSIONS

This chapter reviews coalescence and describes the basic principles of its quantification by a method of gas holdup change. Also, it presents in a manuscript, a detailed study of the coalescence and froth collapse problems in the frother-fatty acid-dispersant-solid system.

The main findings were:

1) A novel, sensitive technique of quantifying coalescence by the change in gas holdup in a flotation column was developed. Absolute changes in gas holdup greater than 0.5% should be detectable. The change in gas holdup can be converted to a change in bubble size.

2) The fatty acid (FA) in a 3:1 ratio with lannagol increases coalescence up to about 20 ppm FA.

3) Coalescence was not related to froth stability. Stable froths could be maintained at lannagol concentrations greater than about 3 ppm (in the absence of solids), with no change in coalescence.

4) Well-dispersed hydrophobic fine particles promoted further coalescence and froth instability. Lannagol concentrations greater than about 7 ppm were required for froth stability, in the presence of solids.

5) The effect of fatty acid and solids on coalescence and froth collapse is tentatively related to hydrophobic coating of the bubble, promoting drainage of the water film.

6) The coalescence and froth collapse problems are related to using low solid pulps, 1% w/w, as feed to a flotation column. At typical addition rates of 2 kg FA/tonne solid, pulps densities greater than 1% w/w are required to ensure adequate concentration of lannagol in solution to promote a stable froth.

CHAPTER 5.

COLUMN FLOTATION OF FINES

5.1 INTRODUCTION

Column flotation of carbonates from the slimes in the laboratory was not satisfactory due to inadequate dispersion and the froth collapse/coalescence problems encountered. It was, therefore, not possible to determine if the column was the device best suited to treat these slimes, which was part of the original objective. As a compromise, plant tests were organized to compare the column with the mechanical cells treating the fines stream (d80 about 25 μm) at Niobec (see Figure 3, Chapter 1). This stream was selected rather than the slimes themselves since the laboratory experience had indicated considerable pre-treatment (e.g. sizing and chemistry) of the slimes would be required. The fines stream offers the compromise of the closest available size distribution to the slimes with a chemistry already well established. In addition, comparison with mechanical (conventional) cells could be made which would test whether the column was capable of superior results on fine feeds.

This chapter describes the column flotation of the fines stream. Comparison with the coventional circuit indicated that the column provided superior metallurgy. This example of improved fine particle separation using a column was supported by work with other fine particle streams conducted by the author at Mt.Isa Mine Ltd, Australia. Together, the results from these two plants convincingly demonstrate the superior cleaning action of the column on fine feeds. The work is presented in a manuscript, 'Column Flotation of Very Fine Particles' (to be published in Volume I, No. 1, of

Minerals Engineering).

5.2 PILOT PLANT TESTING AT NIOBEC

Column flotation performance was evaluated on the fines carbonate circuit at Niobec. Column flotation results were compared in detail with the rougher flotation section of the circuit and with the overall circuit final product. Column flotation was run in parallel with the plant circuit. The prime objective was to compare the performance of the column with that of the conventional circuit. Auxiliary objectives were a) to measure entrainment and mixing characteristics of the column (using a tracer), and b) to determine rate constants to test a method of scale-up.

5.2.1 Experimental Testwork

Experimental set up. The experimental set up is shown in Figure 5.1. The column dimensions were 5.7 cm diameter by 1030 cm high. Capacity was about 150 g/min at about 15% solids (about 1.1 L/min of pulp). Feed tank capacity was 200 L which enabled constant operation for at least 2.5 hours. Feed was taken directly from the feed tank of the plant fines carbonate circuit.

Feed and tailing flows to the column were kept constant using Moyno progressive cavity pumps (Moyno: 2L2-CDQ). Wash water was added at the top of the column through a circular piece of 0.32 cm copper pipe perforated with 0.08 cm holes using a Masterflex pump. A second Masterflex pump was required to deliver launder water to remove the floats.

Air was added at a constant pressure of 137.9 kPa (20 psi) using a stainless steel sparger of approximately 60 cm surface area. (Calibration of the air flowmeter is given in Appendix 10).

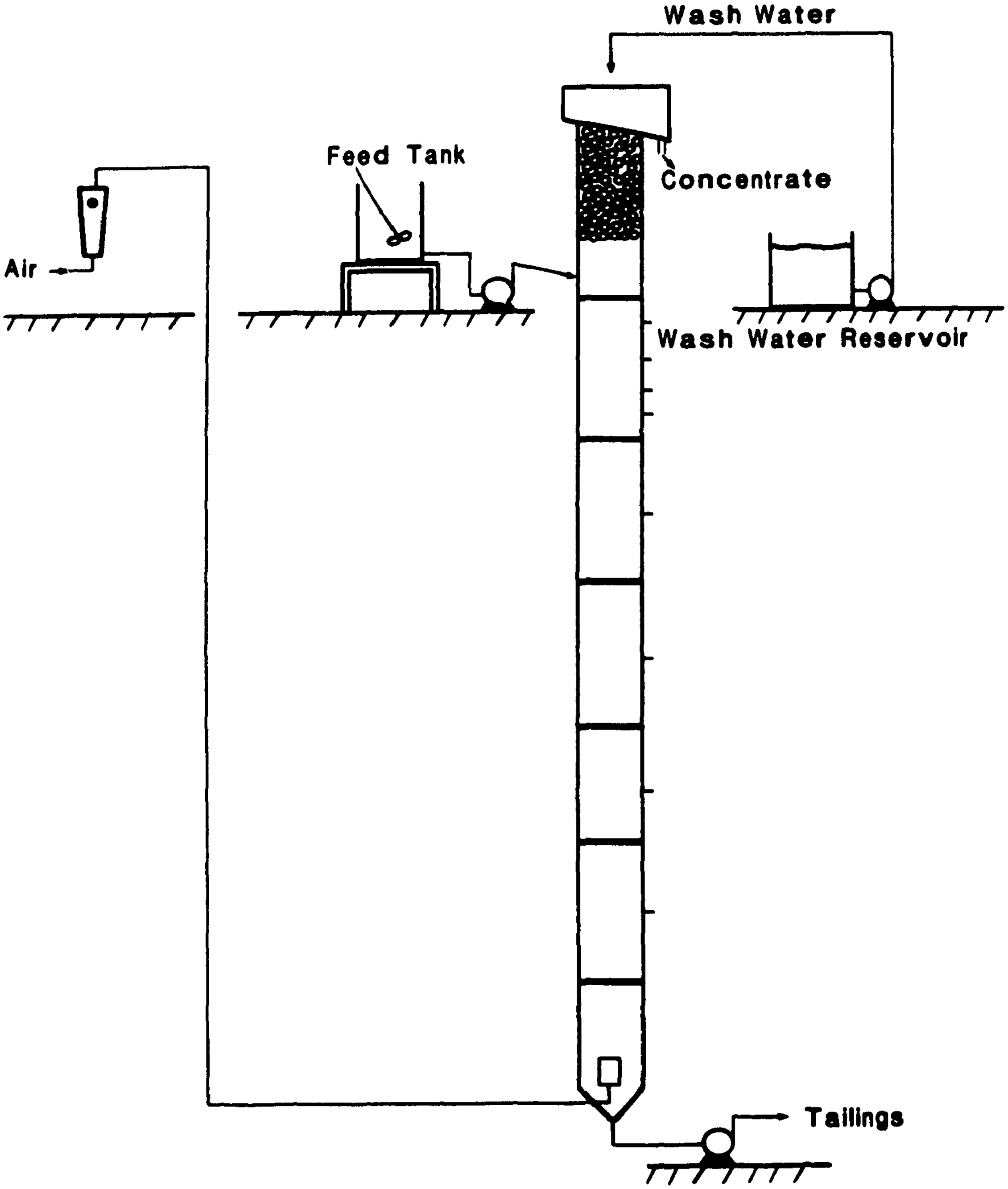


Figure 5.1 Experimental set up.

Flotation. Six tests were performed comprising repeats at three superficial gas velocities (Jg), 1.0, 1.5, and 2.0 cm/s. Table 5.1 summarizes the conditions. To compare with column performance, samples of the plant rougher feed, rougher floats, rougher non-floats, and second cleaner (final) floats were taken during the column tests. (The term floats is used rather than concentrate since the carbonates which float represent the reject). Column samples were taken after steady state conditions had been reached (about two times the nominal residence time of the test). Samples were assayed for Nb_2O_5, SiO_2, CaO, Fe_2O_3, and MgO. These assays were subsequently adjusted using a mass balancing routine and distributions calculated. (Results are reported in Appendix 11). The particle size distribution (PSD) of the products was measured at McGill University using a particle size analyzer (Sedigraph).

Tracer tests. The tracer was LiCl; 1 g was added, dissolved in 10 ml of water, via a 20 ml syringe directly into the feed line. No solids were used. Table 5.1 gives the prevailing conditions.

Rate constant determinations. Samples intended for rate constant determinations were taken from 8 taps, 0.32 cm in diameter, located at column heights of 205, 355, 505, 655, 740, 790, 840, and 870 cm.

Fatty acid frothability without solids. A test was done to determine if the fatty acid (Acintol, FA) can hold a froth without solids. A FA addition (0.6 kg/tonne) similar to that used with the solids was used. FA was added as an emulsion in a 3:1 ratio with lannagol, and no frother was added, as is the practice at Niobec in the carbonate flotation circuit).

TABLE 5.1 SUMMARY OF CONDITIONS

TEST	PRODUCT	% SOLIDS	DISTRIB. Wt (%) (*)	J_b	J_l (cm/s)	J_{ww}	τ (min) (**)	FROTH DEPTH (cm)
T-1	feed	14.8	100.0	0. 3	1.5	0.39	16.8	90
	floats	19.9	50.6					
	non-floats	5.8	49.4					
T-2	feed	13.9	100.0	0.16	1.5	0.39	16.3	90
	floats	16.3	33.5					
	non-floats	7.1	66.5					
T-3	feed	14.3	100.0	0.25	1.0	0.4	17.5	85
	floats	23.5	53.3					
	non-floats	5.3	46.7					
T-4	feed	14.2	100.0	0.1	2.0	0.41	18.6	110
	floats	19.7	53.6					
	non-floats	6.0	46.4					
T-5	feed	13.9	100.0	0.14	1.5	0.42	18.6	110
	floats	22.5	77.5					
	non-floats	2.9	22.5					
T-6	feed	14.7	100.0	0.24	1.0	0.41	17.4	110
	floats	28.3	56.2					
	non-floats	5.2	43.8					
Tracer Test		0.0		0.20	1.5	0.48	15.4	95

(*) Using adjusted values

(**) Nominal residence time

5.2.2 Results

Flotation. In general, the column tests ran smoothly. Figure 5.2 shows recovery and grade of floats as a function of Jg for CaO, Nb_2O_5, and SiO_2. Also included are the recovery and grade values obtained from the rougher floats and forthe overall circuit. Pyrochlore rejection was best at a Jg = 2.0 cm/s. For example, at 65% CaO recovery to the floats, the Nb_2O_5 recovery is only about 10% and 5% for the SiO_2. This is quite different from the results on the slimes obtained so far (e.g. see Figures 3.5 and 3.6 in Chapter 3), where at 60% CaO recovery, 40% Nb_2O_5 and 25% SiO_2 are floated.

Compared with the rougher circuit, the column tended to recover more carbonates (65% instead of 60%), with less Nb_2O_5 (only 12% instead of 26%). Comparison of grade/recovery with the overall fines carbonate flotation circuit was not possible because the circuit non-float product joins that of the coarse circuit prior to the sampling point. However, the Nb_2O_5 grade obtained in the final floats product, after two cleaning stages, is comparable to the floats from the (single-stage) column, 0.14% vs 0.12% for the column and the circuit, respectively.

Figure 5.2 also includes the 95% confidence intervals, for CaO and Nb_2O_5, considering the experimental error in the repeat tests and in the chemical assays. The error in the chemical assays was small compared with the error on the repeats. The only values which showed a marked variation were at Jg = 1.5 cm/s.

Figures 5.3 to 5.5 show the particle size distribution (PSD) for feed, floats, and non-floats, respectively, for three column tests (Jg = 1.0, 1.5, and 2.0 cm/s) and the Niobec circuit. Figure 5.3 shows that PSD for all column tests was reasonably constant. It also shows that the feed is very fine: 80% -25 μm, 50% -15 μm, and 30% -10 μm. Figure 5.4, curve R.F.

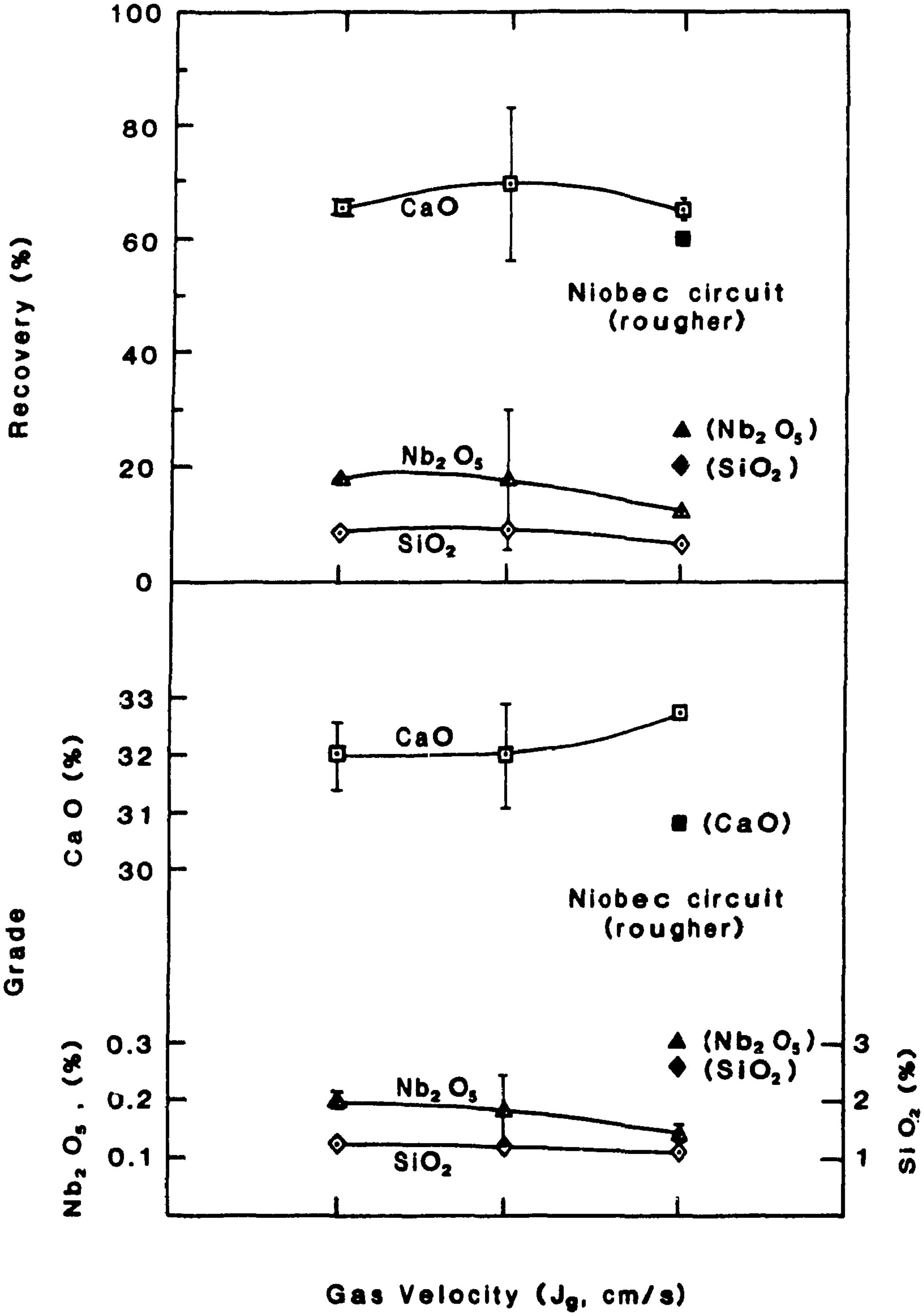


Figure 5.2 Recovery vs J_g and grade vs J_g curves.

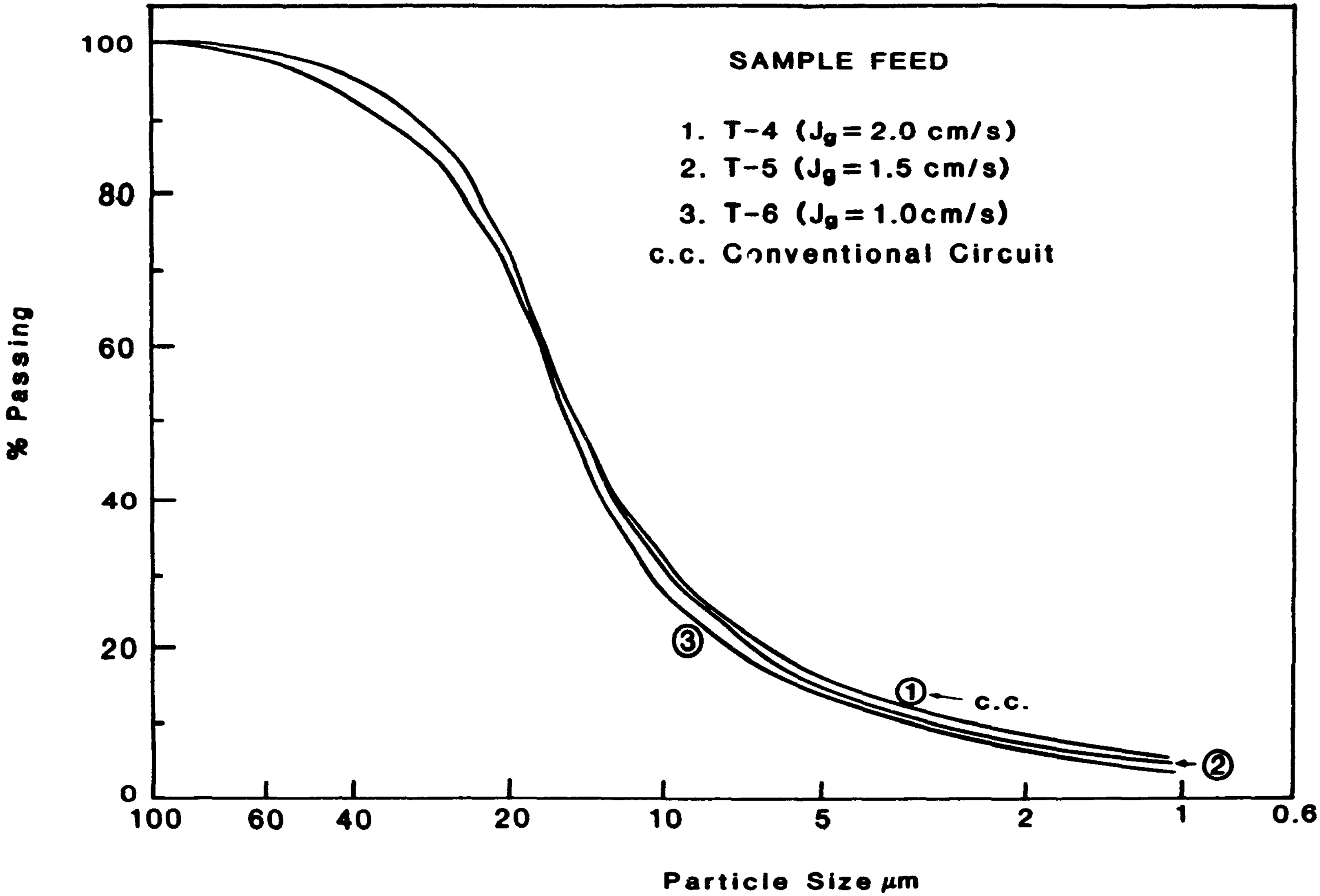


Figure 5.3. Feed particle size distribution

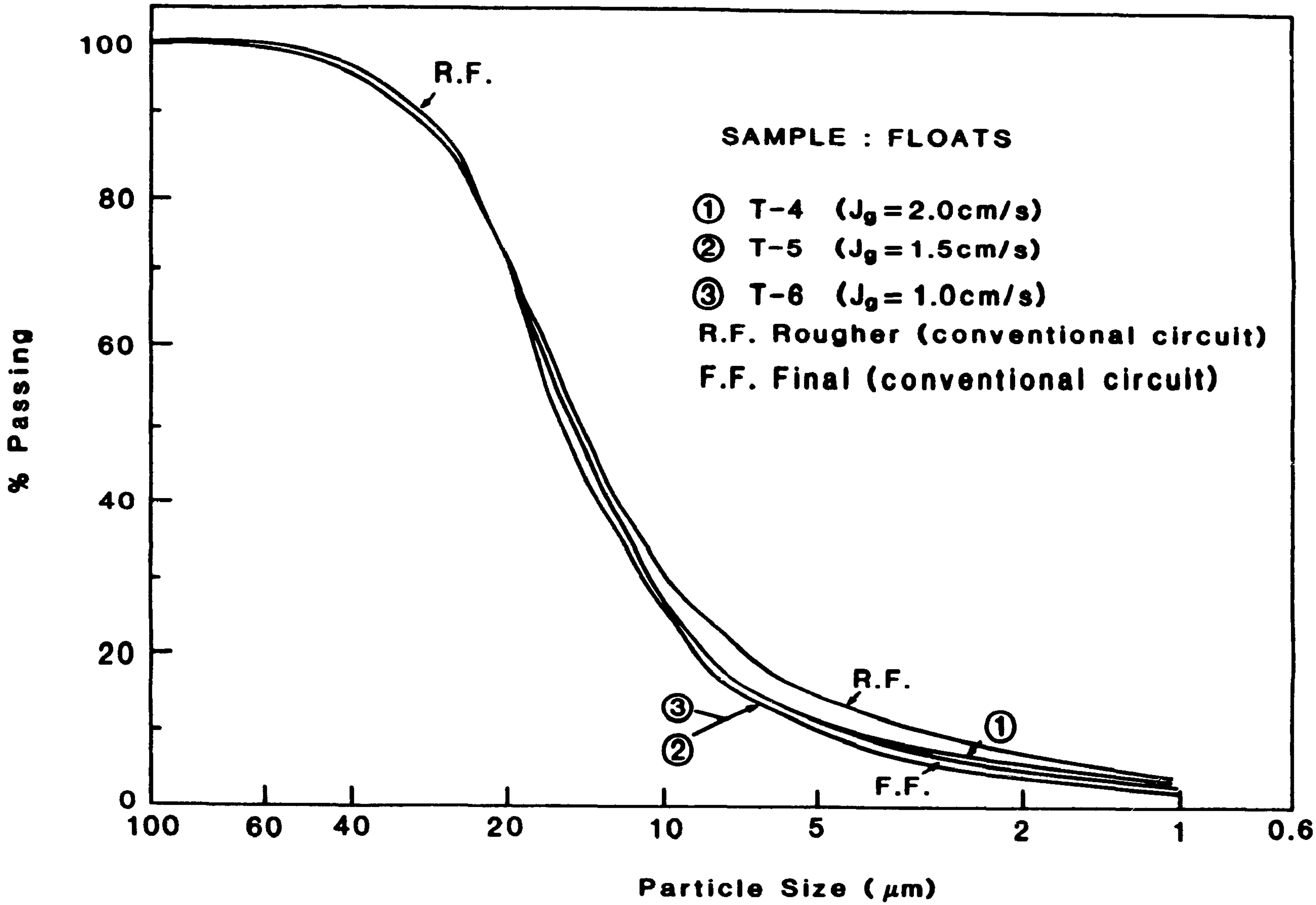


Figure 5.4 Floats particle size distribution

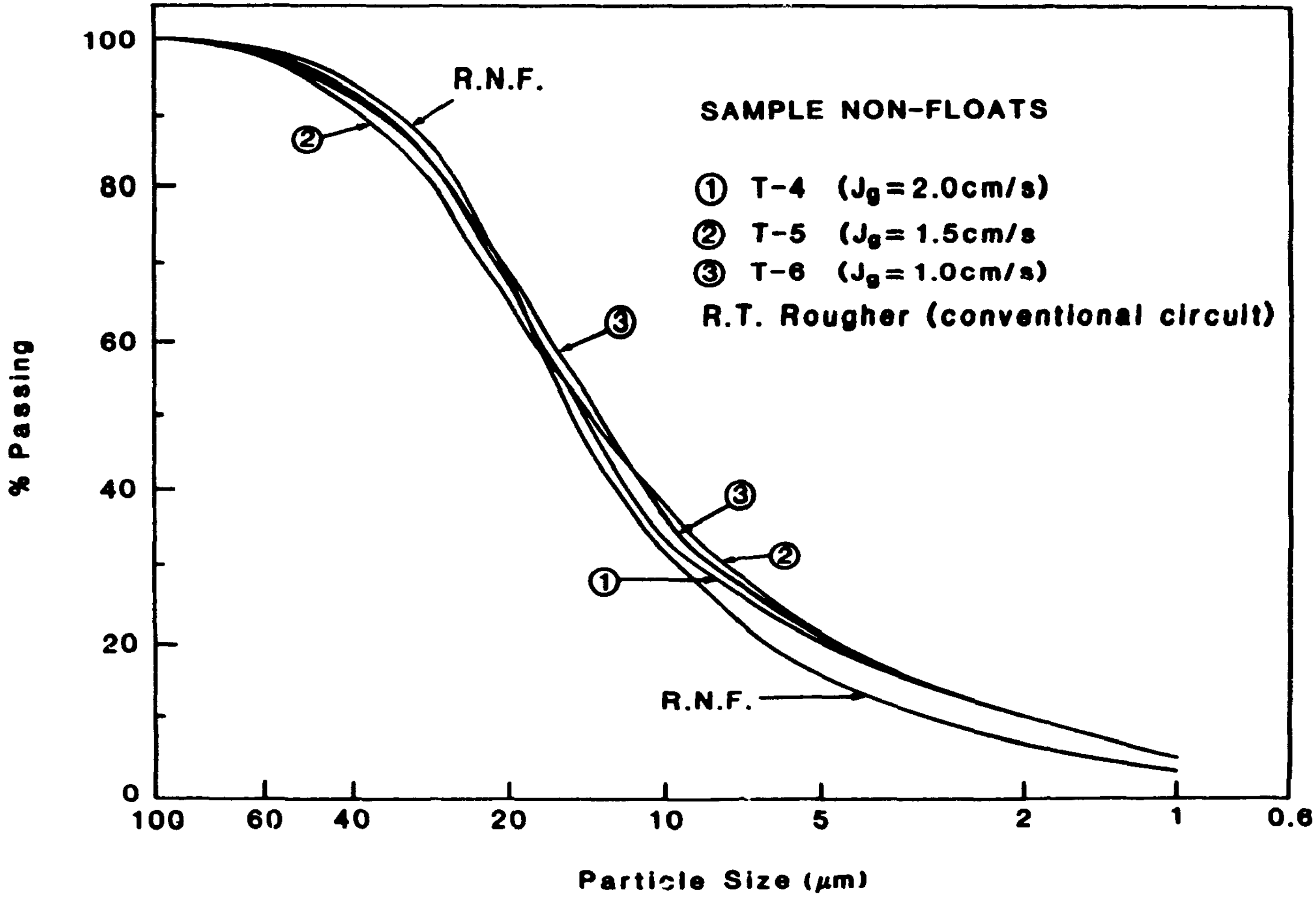


Figure5.5Non-floats particle size distribution.

(rougher floats), shows that the conventional circuit tends to float finer material which may be due to fines entrainment. This point is further suggested in curve F.F. (final floats), where after two cleaning stages the amount of fines in the floats is reduced and the PSD tends to that of the column floats. Figure 5.5, curve R.N.F. (rougher non-floats), shows that the non-floats from the circuit rougher tends to be coarser than from the column. This PSD agrees with the results from Figure 5.4, since more fines are removed in the rougher floats, leaving a coarser material in the non-floats.

Tracer tests. Figure 5.6 shows a plot of Li concentration vs time in the concentrate (curve 1), and the tailings discharge (curve 2). Curve 1 indicates that essentially no water from the feed goes to the concentrate, i.e. there is zero recovery by entrainment. The dispersion coefficient (D) for tailing discharge was estimated from the relative variance of the residence time distribution curve (RTD) curve (as for a closed end vessel); the value was 0.0050 m^2/s, showing a high level of plug flow transport, as expected from such a large column height to diameter ratio (Levenspiel, 1972).

Rate constant determinations. Within experimental error all the samples showed the same assays (Table 5.2). Thus, this approach to determining rate constants was not successful.

Fatty acid frothability without solids. In these tests coalescence and froth colapse occured, confirming the observation made at McGill under similar conditions. This is in sharp contrast to the testing with solids were no such problems were encountered.

TABLE 5.2. GRADE PROFILE TESTS TO MEASURE RATE CONSTANTS

TEST	LOCATION OF TAP (cm) (**)	SOLIDS (%)	GRADE (%)		
			Nb_2O_5	CaO	SiO_2
T-1(*)	FEED	14.8	0.70	27.07	8.24
	870	13.5	0.501	29.81	5.54
	840	13.1	0.543	26.79	6.11
	790	13.1	0.551	28.25	6.12
	740	12.3	0.540	27.12	6.5
	655	12.2	0.556	28.36	6.31
	505	12.3	0.589	27.30	6.46
	355	12.4	0.589	27.69	6.95
	205	11.1	0.607	27.52	7.38
	TAILS	5.8	1.20	21.17	15.13
T-3(*)	FEED	14.3	0.67	27.04	8.07
	870	N.A.	0.575	29.20	6.07
	840	11.9	0.550	27.93	6.23
	790	N.A.	0.567	17.51	6.24
	740	12.0	0.563	28.60	6.20
	655	N.A.	0.568	28.07	6.31
	505	11.7	0.592	28.33	6.44
	355	12.4	0.563	29.15	6.38
	205	10.8	0.606	26.45	7.36
	TAILS	5.3	1.17	21.3	15.8

(*) Test conducted after flotation samples had been taken. For details about test conditions see Table 5.1.

(**) Distance in cm from the bottom of the column.

TABLE 5.2. GRADE PROFILE TESTS TO MEASURE RATE CONSTANTS (CONTINUATION)

TEST	LOCATION OF TAP (cm) (**)	SOLIDS (%)	GRADE (%)		
			Nb_2O_5	CaO	SiO_2
T-4(*)	FEED	14.2	0.63	27.14	8.13
	870	12.2	0.491	28.53	6.07
	840	12.3	0.488	29.15	6.06
	790	13.1	0.507	17.55	6.22
	740	N.A.	0.492	28.69	5.88
	655	N.A.	0.503	28.41	6.05
	505	13.3	0.507	28.22	6.09
	355	14.3	0.496	28.55	5.80
	205	10.7	0.630	25.74	8.19
	TAILS	6.0	1.22	20.38	16.47

(*) Test conducted after flotation samples had been taken. For details about test conditions see Table 5.1.

(**) Distance in cm from the bottom of the column.

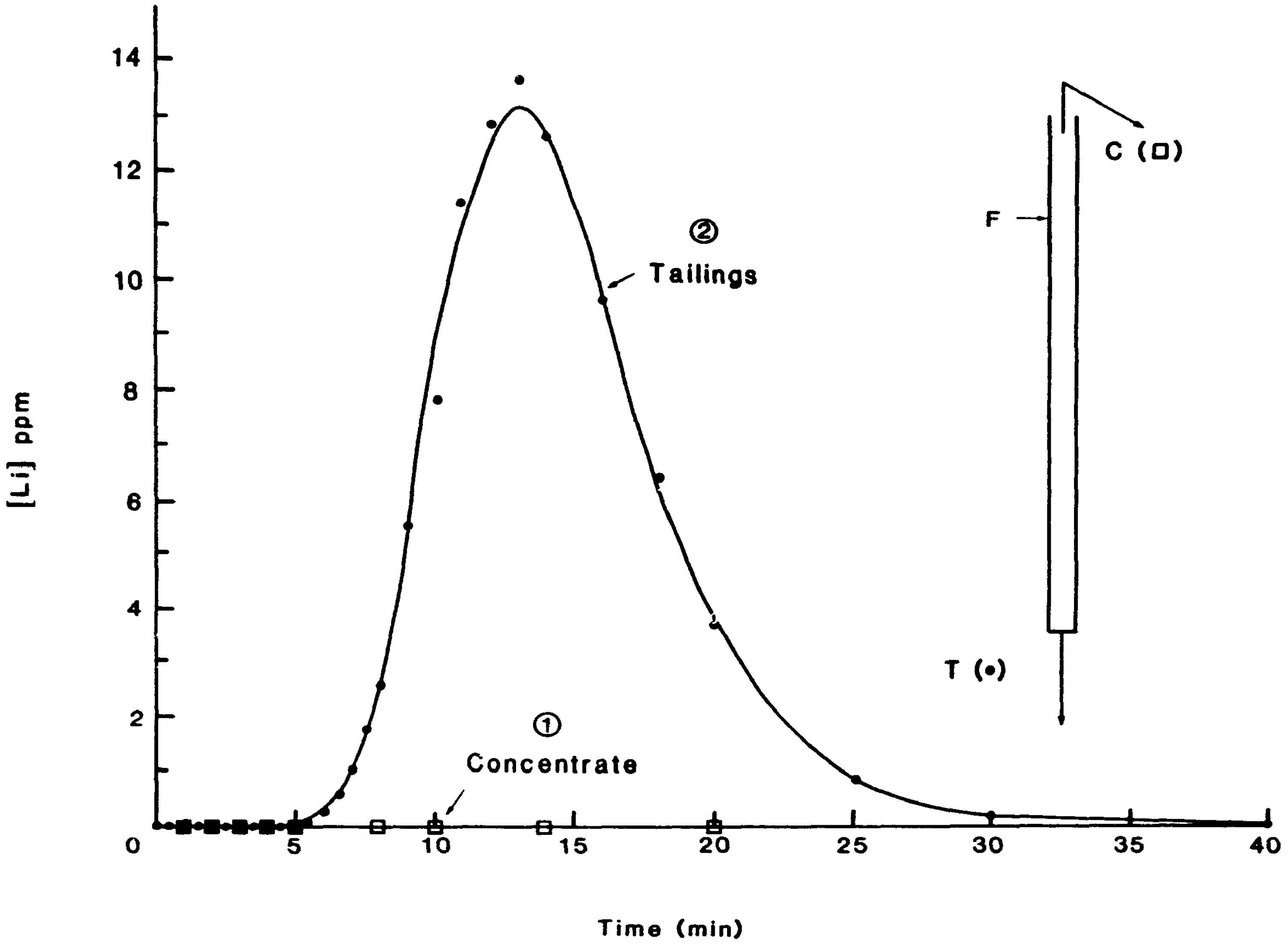


Figure 5.6. Column tracer test

5.2.3 Discussion

Flotation. In general, the column performance was superior to the rougher circuit, and at least equalled the circuit in grade. Column performance could probably have been further improved with experience. The use of the columns to treat these fines feed deserves worth further evaluation: by extension the column should also prove superior on the slimes.

It is noted that more Nb_2O_5 is recovered to the froth product than SiO_2. This either means greater Nb-mineral/carbonate locking, some true flotation of the Nb-phase, or agglomeration with carbonates. The suspicion is that true flotation occurs.

Tracer tests. Feed water recovery approaches zero. This, it has been established, is due to the wash water, or more specifically the bias water, that fraction which goes down through the froth (Yianatos et al 1987d). Provided Jg is less than about 2 cm/s, feed water is rejected within about 10 cm of the interface (Yianatos et al, 1987d). The current results, therefore, further confirm this efficient feed water rejection. By extension, if feed water recovery is zero then particle recovery by entrainment must be zero.

Rate constant determinations. Having established largerly plug flow transport then the samples withdrawn with increasingly distance from the feed inlet represent increasing flotation time. Consequently, recovery (R) vs time (t) can be determined and the rate constant determined from a plot of ln (1-R) vs t (Lynch et al, 1981). This was the idea. The method, however, was not successful since the assays were virtually identical down the column. This is attributed, in part, to the sample extraction technique which simultaneously extracts pulp which is wanted and loaded bubbles which are not. This method of estimating rate constants has been abandoned in favor of varying mean retention time by varying tailings rate, in a variation of the traditional method of batch determination of flotation rate constants.

5.3 MANUSCRIPT 4:

COLUMN FLOTATION OF VERY FINE PARTICLES

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ABSTRACT

This work presents plant-site evaluations of six feeds comprising very fine particles (80% passing size, 8-23 μm). Five of the streams contained mainly sulphides, (Mount Isa Mines (MIM) Limited, Australia), and one contained oxide minerals, (Niobec, Canada). All the testwork was performed using pilot-scale columns. For each stream, column metallurgical performance was compared with mechanical cells in the plant and in the laboratory. Column results were better than plant and laboratory results in all cases, giving concentrate grades up to 6% higher (in absolute terms) at the same recoveries. Entrainment into the column concentrate was measured by the recovery of the -10 μm non-sulphide gangue (NSG) fraction and by the recovery of feed water. Entrainment was much lower in the column than for the mechanical cells and accounted for most of the improved performance for the column. Some evidence of improved selectivity in the column was found which accounted for the additional improvement observed in some cases.

INTRODUCTION

Column flotation applications are becoming increasingly common worldwide (Amelunxen and Redfern, 1985; Coffin and Miszczak, 1982; Wheeler, 1985; Huggins et al, 1987; Narasinham et al, 1972; Brunskill, 1986; Mauro and Grundy, 1984; Clingan and McGregor, 1987). One of the main advantages of columns is the virtual elimination of entrainment by means of wash water addition (Yianatos et al, 1987d; Dobby and Finch, 1985). Entrainment poses a serious limitation to upgrading of very fine particles in mechanical cells

(Johnson et al, 1974; Klassen and Tikhonov, 1964).

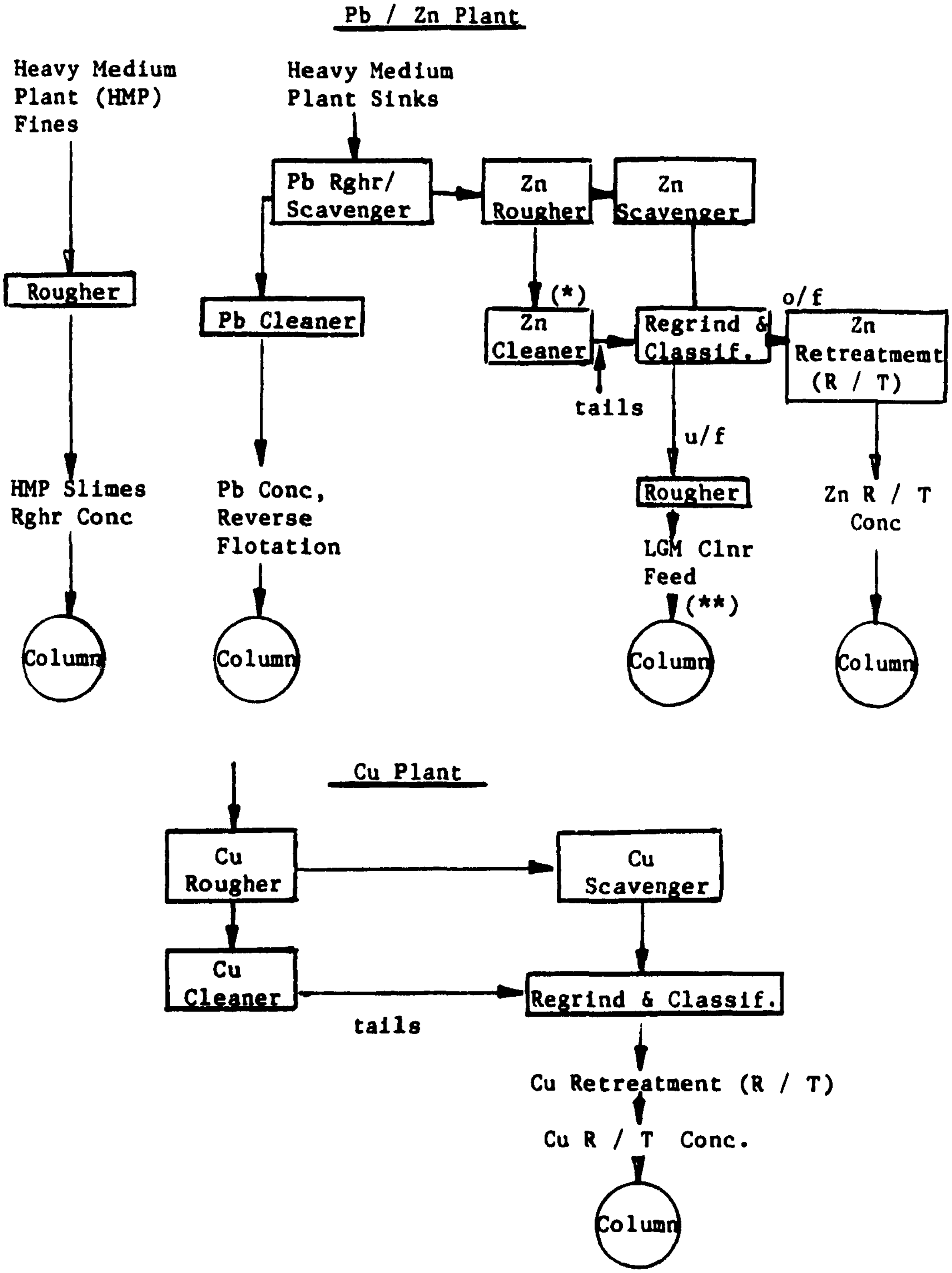
This paper presents plant-site evaluation of the metallurgical performance on fine particle feeds in a pilot plant column and a comparison with conventional cells. Six streams were evaluated, five at Mount Isa Mines (MIM) Limited, Australia, and one at Niobec, Canada. For brevity only three of the streams are discussed in detail.

EXPERIMENTAL PART

CHARACTERISTICS OF THE STREAMS TESTED

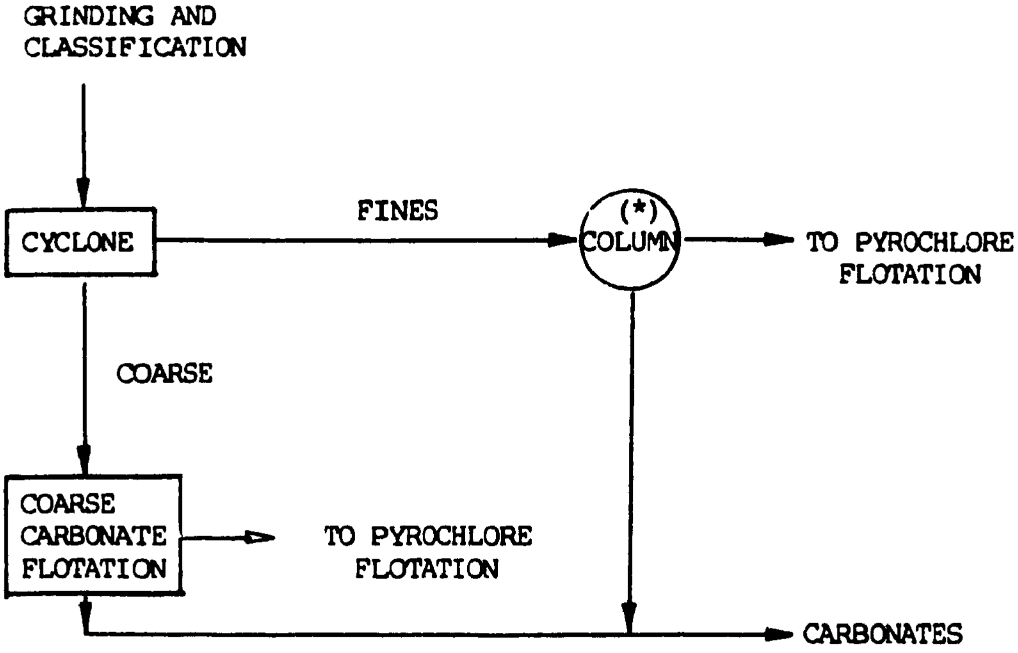
Figures 1A and 1B are simplified flowsheets identifying the streams tested at MIM and at Niobec, respectively. Tables 1A and 1B show the particle size distribution of the streams and Tables 2A and 2B show their composition. At MIM sizing was performed using a Haultain infrasizer. Table 3 presents the cut sizes produced by the infrasizer according to mineral density. At Niobec, size distributions were determined using a Sedigraph 5000D. The 80% passing size (d(80)) varied from 9 to 23 μm, hence the reference to 'very fine particles'.

Figure 1A. Flowsheet Indicating Streams Tested by Flotation Column at Mount Isa Mines (MIM) Ltd.



(*) Zn cleaner block feed. Circuit involves 3 cleaning stages.
(**) LGM plant cleaning circuit involves 2 cleaning stages.

FIGURE 1B. FLOWSHEET INDICATING THE STREAM TESTED BY FLOTATION COLUMN AT NIOBEC



(*) FINES CARBONATE CIRCUIT AT THE PLANT, INVOLVES A ROUGHER AND TWO CLEANING STAGES

TABLE 1-A. FEED PARTICLE SIZE DISTRIBUTION OF THE STREAMS TESTED AT MOUNT ISA MINES (MIM)

PARTICLE SIZE (*)	STREAM (% PASSING)				
	Cu R/T CONC.	Zn R/T CONC.	LGM	HMP SLIMES RGHR CONC.	Pb FINAL CONC REVERSE FLOT.
+53 μm	90.1				
+38 μm	88.9		96.7	92.9	
F3	83.4	97.2	88.3	83.3	81.5
F5	74.6	82.7	59.4	65.6	64.9
F6	61.8	74.7	49.4	54.3	56.2
d(80) (μm)	16	9	20	15	17

(*) SIZING WAS PERFORMED USING AN INFRASIZER. SEE TABLE 3 FOR CUT SIZES DEPENDING ON MINERAL SPECIES.

TABLE 1-B. PARTICLE SIZE DISTRIBUTION OF THE STREAM TESTED AT NIOBEC

PARTICLE SIZE(*) (μm)	% PASSING
	FINES CARBONATE FEED
40	95
20	70
10	30
5	17
2	10
d(80)	23

(*) THE SEDIGRAPH 5000 PARTICLE SIZE ANALYZER

TABLE 2-A. METAL AND MINERAL COMPOSITION OF THE STREAMS TESTED AT MIM

ASSAY (%)	STREAM				
	Cu R/T	Zn R/T	LGM	HMP SLIMES RGHR CONC.	Pb FINAL CONC. REVERSE FLOT.
Cu	8.5-11.6	0	0	0	0
Pb	0	2.2-2.7	8-11	30-40	47
Zn	0	31.5-35.2	24-31	10-15	6
FeS_2	30-40	16-22	18-25	11-26	23
NSG	33-40	20-24	16-25	11-19	11

TABLE 2-B. METAL COMPOSITION OF THE STREAM TESTED AT NIOBEC

ASSAY (%)	FINES CARBONATE FEED
Nb_2O_5	0.63-0.71
MgO	14.0-14.4
CaO	27.0-27.2
SiO_2	7.9-8.3
P_2O_5	2.5-2.9
Fe(T)	7.4-7.6

TABLE 3. CUT SIZES (MICRONS) GIVEN BY THE INFRASIZER

MINERAL	S.G.	CUT SIZE (MICRONS)				
		F3	F4	F5	F6	F7
QUARTZ	2.65	28	20	14	10	-10
SPHALERITE	4.00	23	16	11	8	-8
CHALCOPYRITE	4.20	23	16	11	8	-8
PYRITE	5.00	20	15	10	7	-7
GALENA	7.50	17	12	8	6	-6

COLUMN SET UP AND OPERATING CONDITIONS

Figure 2 shows the basic set up, and Table 4 gives the column dimensions and the accessories used. The interface level was observed visually in all cases, and controlled manually by wash water addition using a Masterflex pump; the feed and tailing flowrates were controlled using the pumps described in Table 4.

Table 5 is a summary of operating conditions. Superficial fluid velocities are quoted, i.e. the volumetric flow per unit column cross-sectional area (cm/s). Superficial gas velocity (Jg) was varied from 0.8 to 3.2 cm/s, the usual range being 1.0-2cm/s at Niobec, and 2.5-3 cm/s at MIM. Superficial wash water velocity (Jww), 0.4-1.0 cm/s; superficial tailing velocity (Jt), 0.5-2.2 cm/s. The superficial bias rate (Jb), which is defined as the difference between the superficial water rate in the tailing (Jt) and in the feed (Jf), varied from -0.1 to 0.9 cm/s. Jb was varied by adjusting Jf, to maintain a constant residence time. The nominal residence time (i.e. residence time without allowing for the gas), was 6-66 minutes. Changing nominal residence time was used to generate grade/recovery relationships. Froth depth was 40-150 cm.

The column was operated as one or two stages. In the two stage operation, the tailings from the first stage were stored and re-processed. The purpose of the two-stage operation was to increase the total residence time, since the maximum residence time in one column stage was about 35 minutes.

Table 6 shows the values for (undiluted) feed densities for each stream. At MIM they ranged from 25% to 50%; 15% solid was typical for the

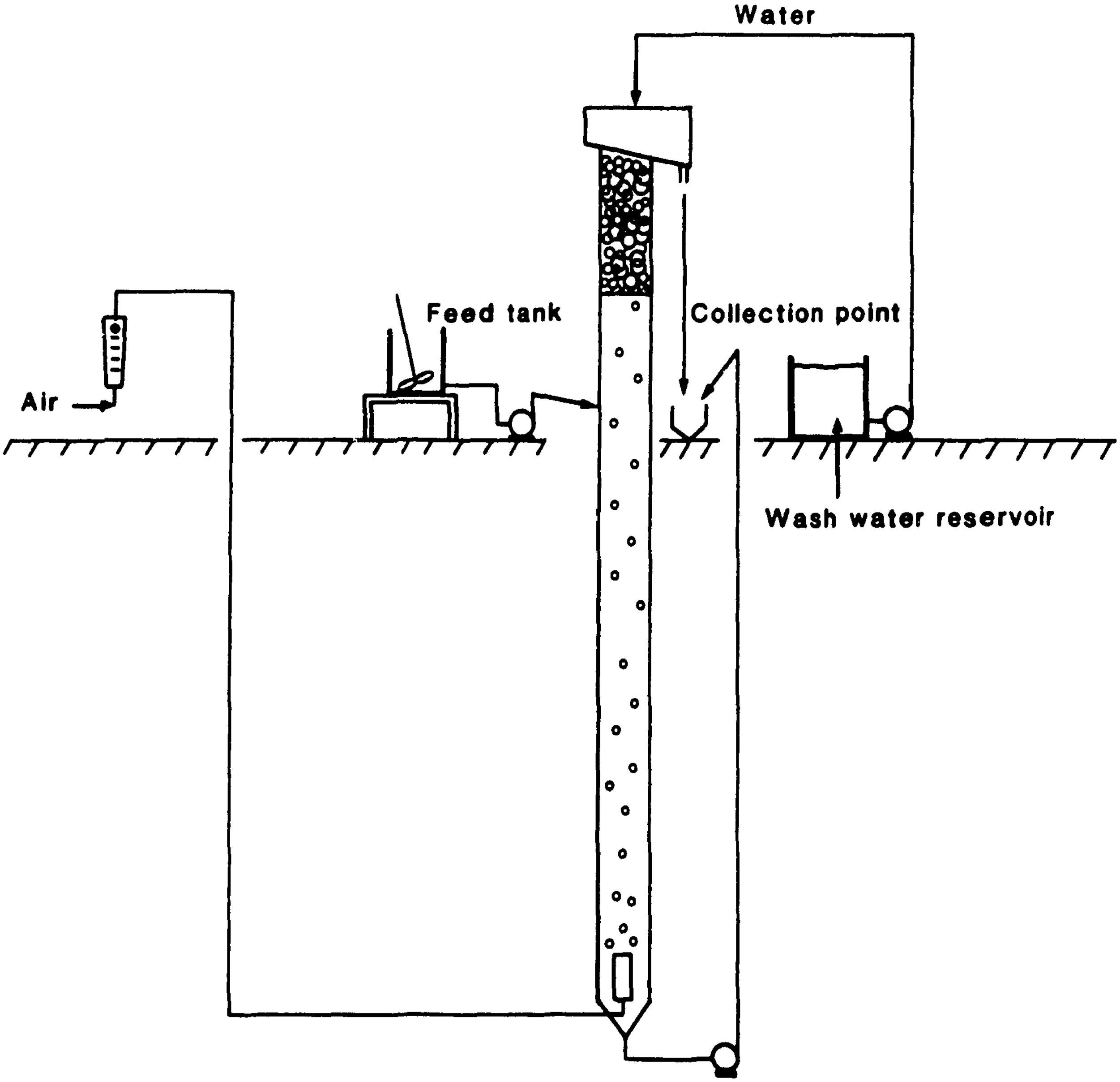


Figure 2.: Experimental set up

TABLE 4. COLUMN CHARACTERISTICS AND ACCESSORIES

LOCATION	COLUMN DIMENSIONS DIAMETER X HEIGHT (cm x cm)	SPARGER	PUMP
MIM	5.08 x 1035	STEEL (*)	MONO
NIOBEC	5.72 x 1030	STEEL (*)	MOYNO (2L2-CDQ)

(*) POROUS STAINLESS STEEL, SURFACE AREA = 56 cm^2

TABLE 5. SUMMARY OF OPERATING CONDITIONS

COLUMN DIMENSIONS D x L	SUPERFICIAL VELOCITIES (cm/s)				τ(5) (min)	FD(6) (cm)
	Jg(1)	Jb(2)	Jww(3)	Jt(4)		
5.08 x 1035 5.72 x 1030	0.8-3.2	-0.1-0.9	0.4-1.0	0.5-2.2	6-66	80-150

(1) Jg, SUPERFICIAL GAS RATE
(2) Jb, SUPERFICIAL BIAS RATE
(3) Jww, SUPERFICIAL WASH WATER RATE
(4) Jt, SUPERFICIAL TAILS RATE
(5) τ, NOMINAL RESIDENCE TIME
(6) FD, FROTH DEPTH

TABLE 6. SUMMARY OF FEED DENSITIES

STREAM	RANGE (% SOLIDS)
Cu R/T CONC	20-28
Zn R/T CONC	25-38
LGM	35-48
HMP SLIMES RGHR CONC	46-50
Pb FINAL CONC REVERSE FLOTAT	36-43
FINES CARBONATE FEED	15

feed at Niobec. In some tests the column feed was diluted to reduce loading (Espinosa-Gomez et al, 1988). This increased mineral recovery in comparison to tests with undiluted feed at the same residence time.

When possible the column was run in parallel with the existing plant circuit. On occasion this involved temporary changes in the plant circuit. In cases where comparison was not possible standard laboratory tests in mechanical cells (3L Agitair) were performed.

EVALUATION OF METALLURGICAL PERFORMANCE

To evaluate performance, grade-recovery relationships were established. These were compared, column vs circuit and/or vs laboratory.

Size-by-size analyses were performed, principally to estimate non sulphide gangue recovery by entrainment.

Finally, the impact of operating variables, such as bias rate and gas rate, was evaluated.

ESTIMATION OF ENTRAINMENT

Entrainment was estimated by measuring recovery of F7 (about -10 μm) non-sulphide gangue (NSG) and feed water recovery (using salt tracers). Entrainment was compared with the estimated entrainment in the conventional circuit cells at MIM where the feed to the conventional cells was assumed to be the same as for the column. The NSG (silicates plus carbonates) content was determined by difference, as the total sulphide content is known. Occasionally insoluble (Insol) determinations were made which reflects silicate gangue.

Entrainment in conventional cells can be calculated if water recovery to the concentrate is known (Johnson et al, 1974). By obtaining

%solid values for the feed and products of a conventional cell, the resulting water balance allows calculation of the water recovery. At MIM it has been established (Johnson et al, 1974), for F7 NSG of specific gravity 2.7, that for each unit of water recovery in the concentrate, 0.8 unit of NSG is recovered. Thus, multiplication of the water recovery by 0.8 gives an estimation of the expected entrainment in conventional cells.

Feed water recovery to the column concentrate was determined by tracer tests. A LiCl tracer was added in the column feed and samples from concentrate and tailings were collected at controlled time intervals. Tracer recovery in the column concentrate is a measure of the recovery of feed water in the concentrate which is the boundary case for entrainment.

RESULTS

METALLURGICAL PERFORMANCE

Figure 3 shows the lead and zinc grade-recovery relationship obtained on the low grade middlings (LGM) cleaner feed, by the column and the plant (two stages of closed circuit cleaning) (see Figure 1A). In this stream a Zn-Pb concentrate is produced. For the same lead recovery, e.g. 85%, similar lead grades (13%) were obtained in the column as in the plant, but with higher column zinc recoveries (92% vs 86%) and grades (35% vs 33%).

Figure 4 shows a grade-recovery relationship on the zinc retreatment concentrate (Zn R/T Conc), for the column and laboratory, the laboratory results being for one and three stages of cleaning. At a zinc recovery of 85%, the column concentrate grade was about 6% higher (50% vs 44%) than the best laboratory result.

Normally, the zinc cleaner block feed contains zinc rougher concentrate and zinc retreatment concentrate. By temporary divertion of the

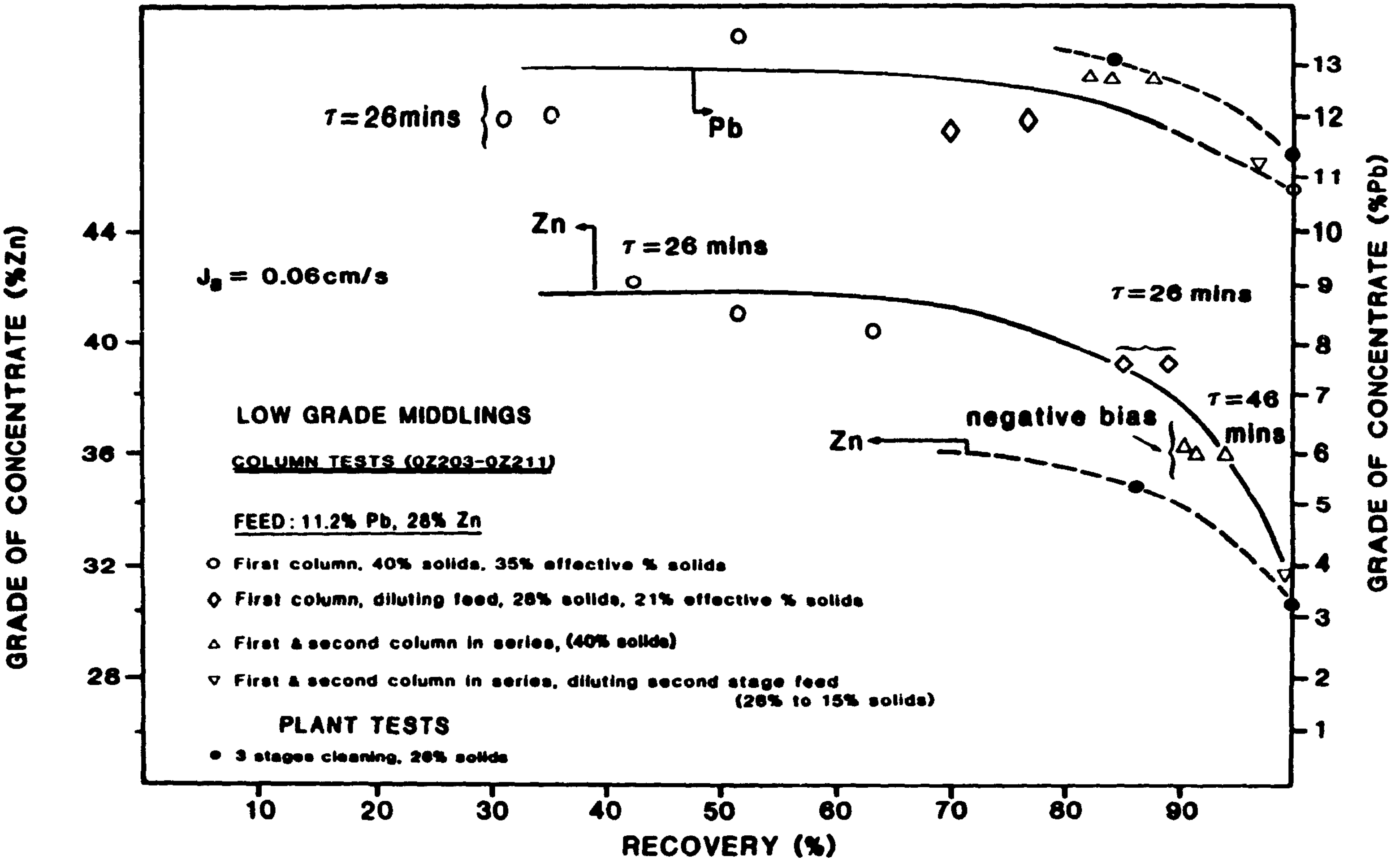


Figure 3.: Lead and zinc grade-recovery relationship on the LGM (column vs plant)
(The % solids figure refers to feed)

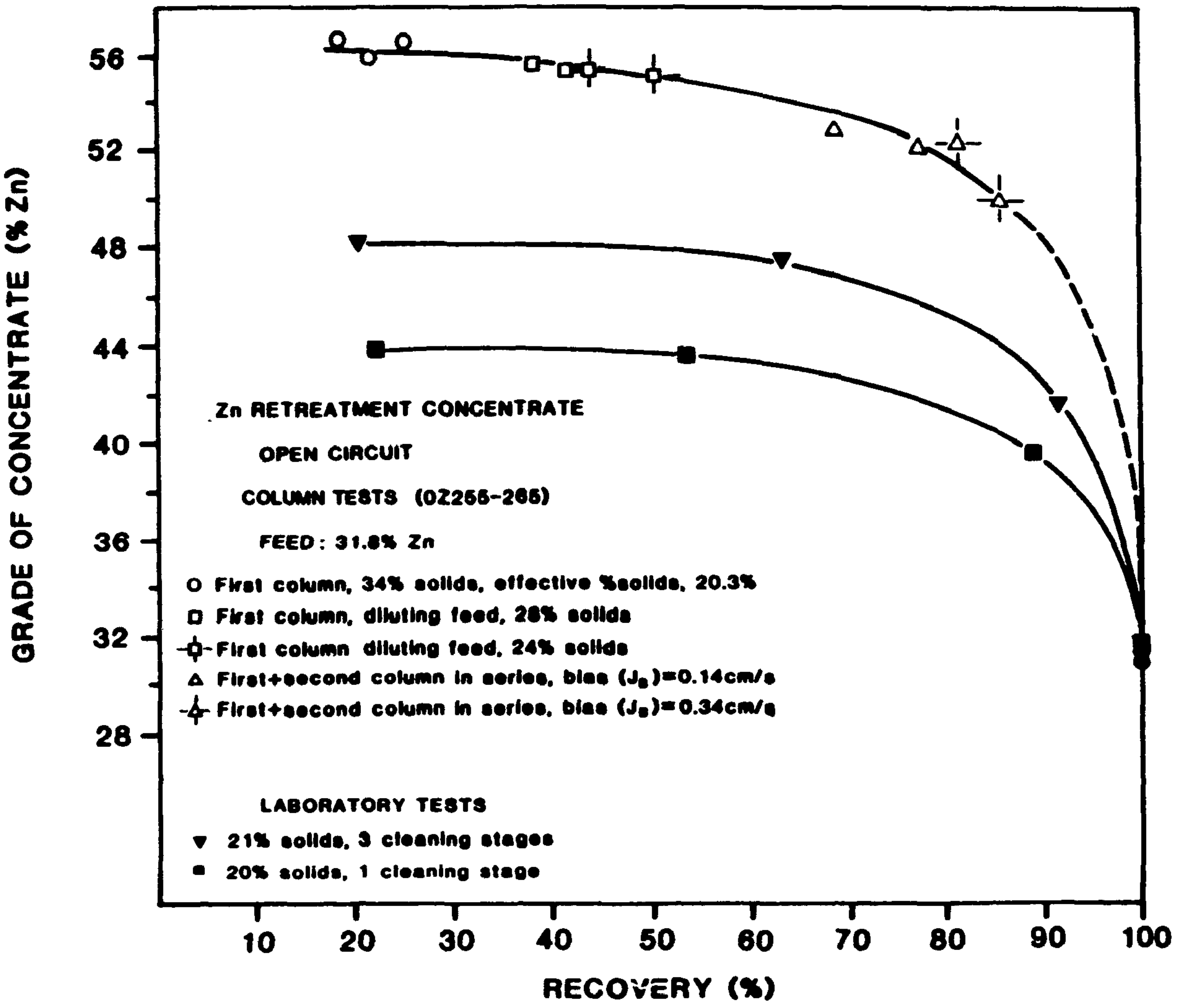


Figure 4.: Grade-recovery relationship on the Zn retreatment concentrate (column vs laboratory)

Zn R/T Conc to final concentrate (i.e. opening the circuit) the upgrading of this stream in the column could be compared to the plant upgrading of the Zn rougher concentrate (this is the envisaged circuit arrangement using columns). The comparison is shown in Figure 5. The column on more difficult feed matched the performance of the plant.

Figure 6 shows a grade-recovery relationship, on the copper retreatment concentrate (Cu R/T Conc) (see Figure 1A), for the column vs laboratory cells. The column gave improved separation. For example, at 97% copper recovery, the column produced a grade of 22% Cu vs 15.3% Cu in the laboratory. Dilution of the feed in the laboratory tests was used to take advantage of its known effect in reducing entrainment. (The value of 15% is the effective %solids starting from 21% solids if all the wash water in the column were added to the feed). It is clear that the column gives enhanced cleaning above that due to simple dilution.

Figure 7 shows a grade-recovery relationship on the fines carbonate flotation feed (Niobec), for the column and the plant roughing stage only. The grade-recovery curves do not cover the same range, but the improved grade/recovery of the column is evident. By extrapolation to 65% CaO recovery, the CaO grade is higher for the column, by about 2%. Column concentrate produced in one stage was compared with circuit concentrate after two cleaning stages. In both cases the grade was 32.5% CaO. Recovery for the fines circuit could not be determined, as the fines circuit product is mixed with other products prior to sampling.

SIZE BY SIZE BEHAVIOUR AND ENTRAINMENT

Figures 8 and 9 show the recovery-size relationship on the LGM, for the column (one stage, the point at 89% Zn recovery on Figure 3) and plant

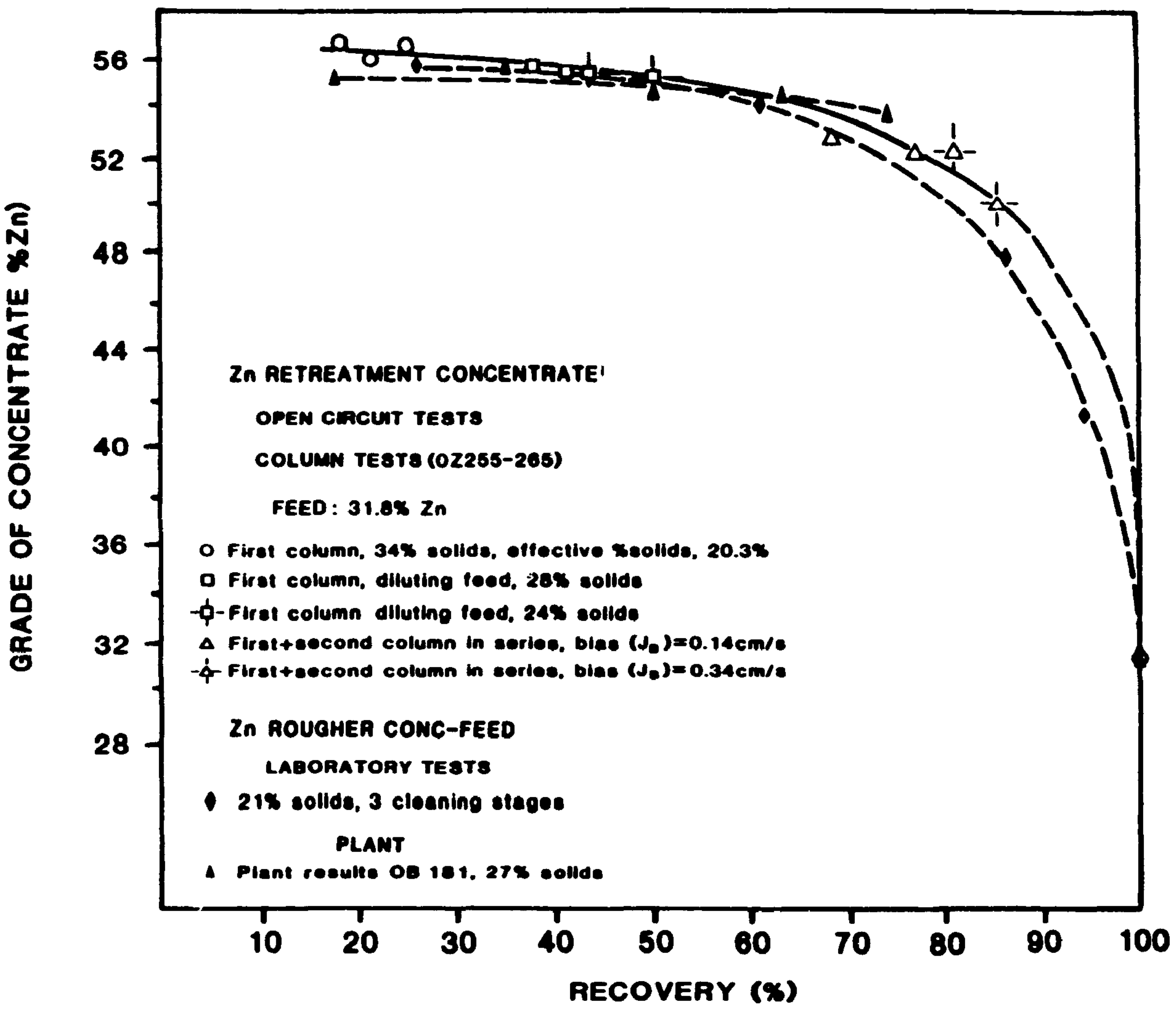
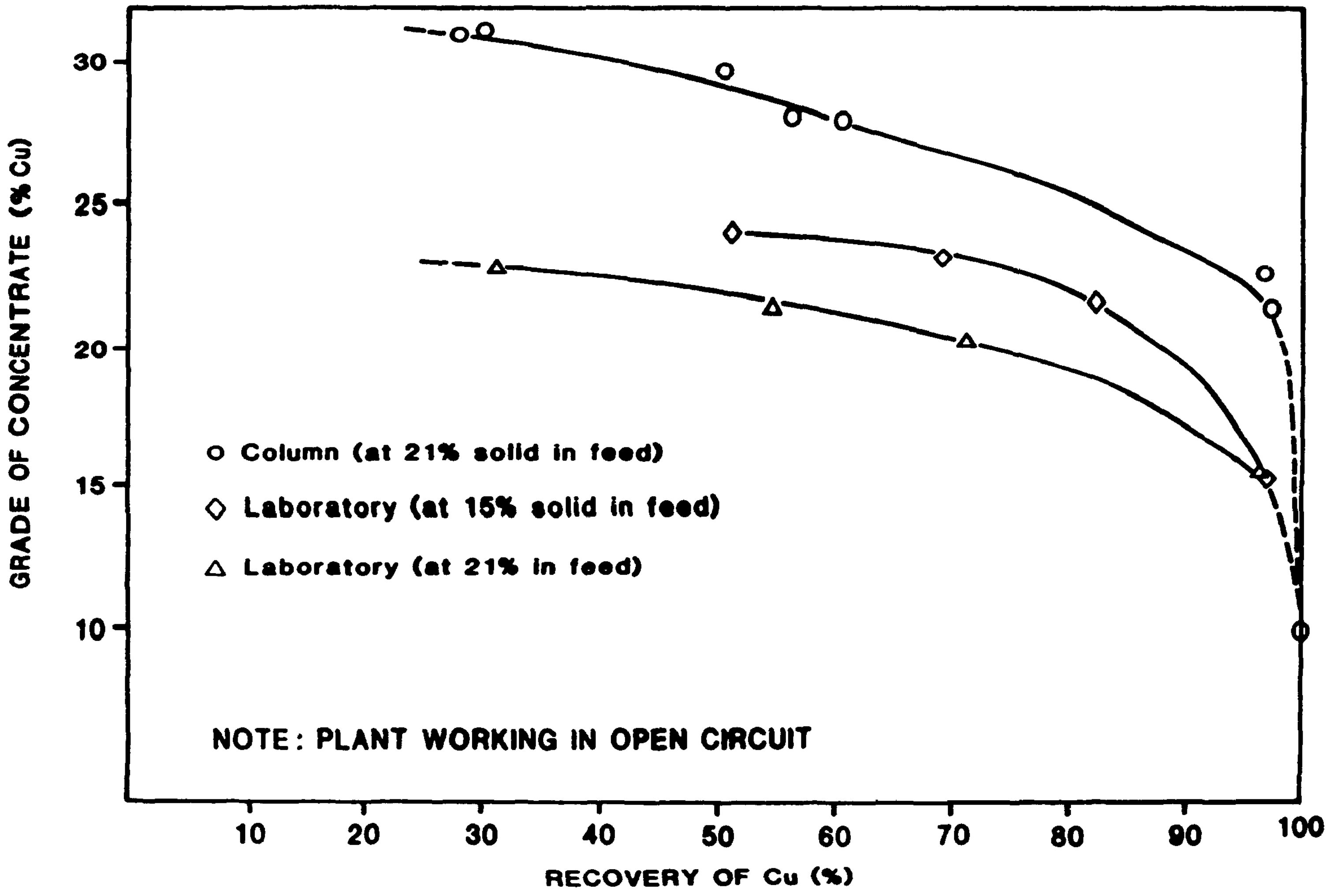


Figure 5.: Grade-recovery relationship for the column on the Zn retreatment concentrate and for the laboratory and plant on the Zn cleaner block feed



NOTE: PLANT WORKING IN OPEN CIRCUIT

Figure 6.: Grade-recovery relationship on the copper retreatment concentrate. (column vs laboratory)

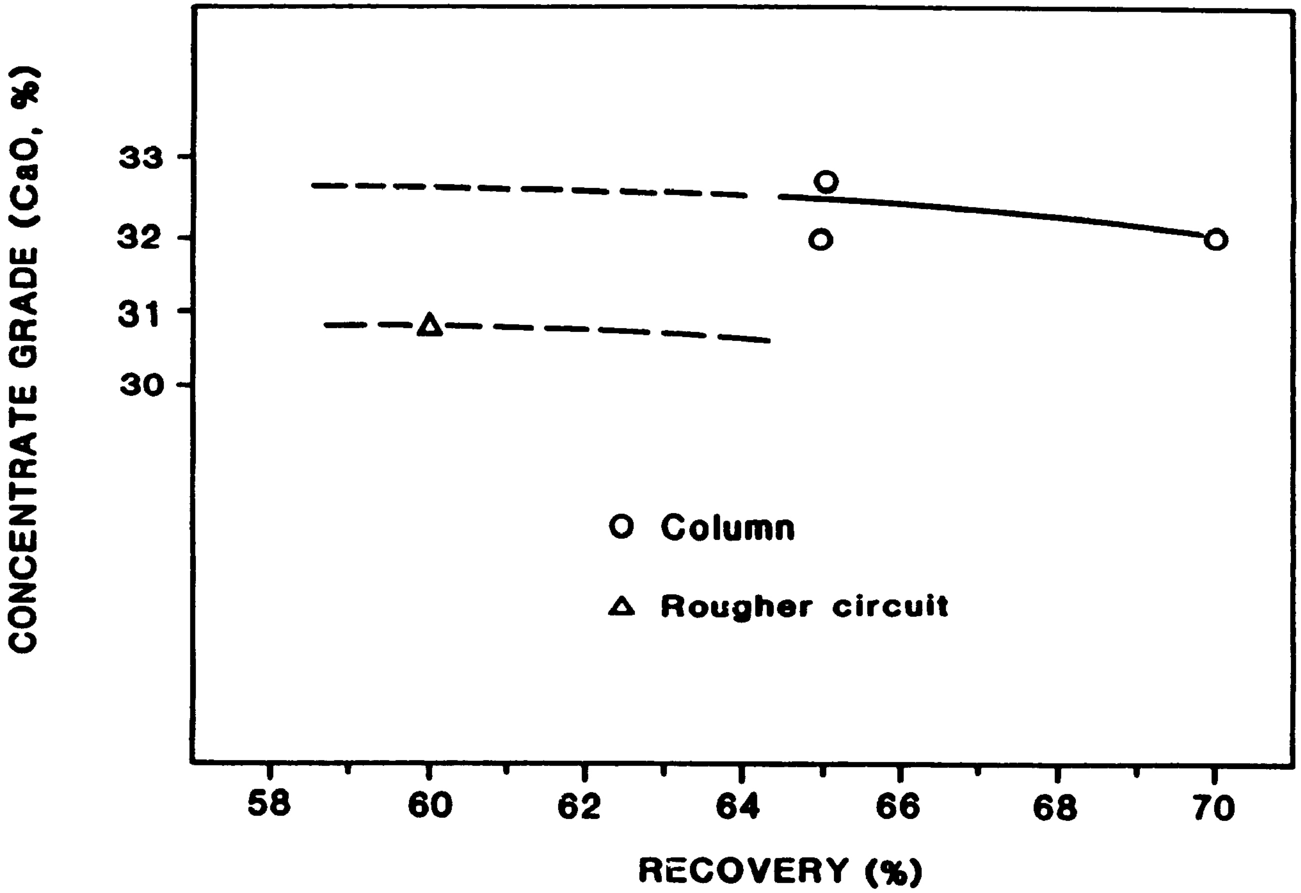


Figure 7 .: Grade-Recovery curves for fines carbonate flotation feed.

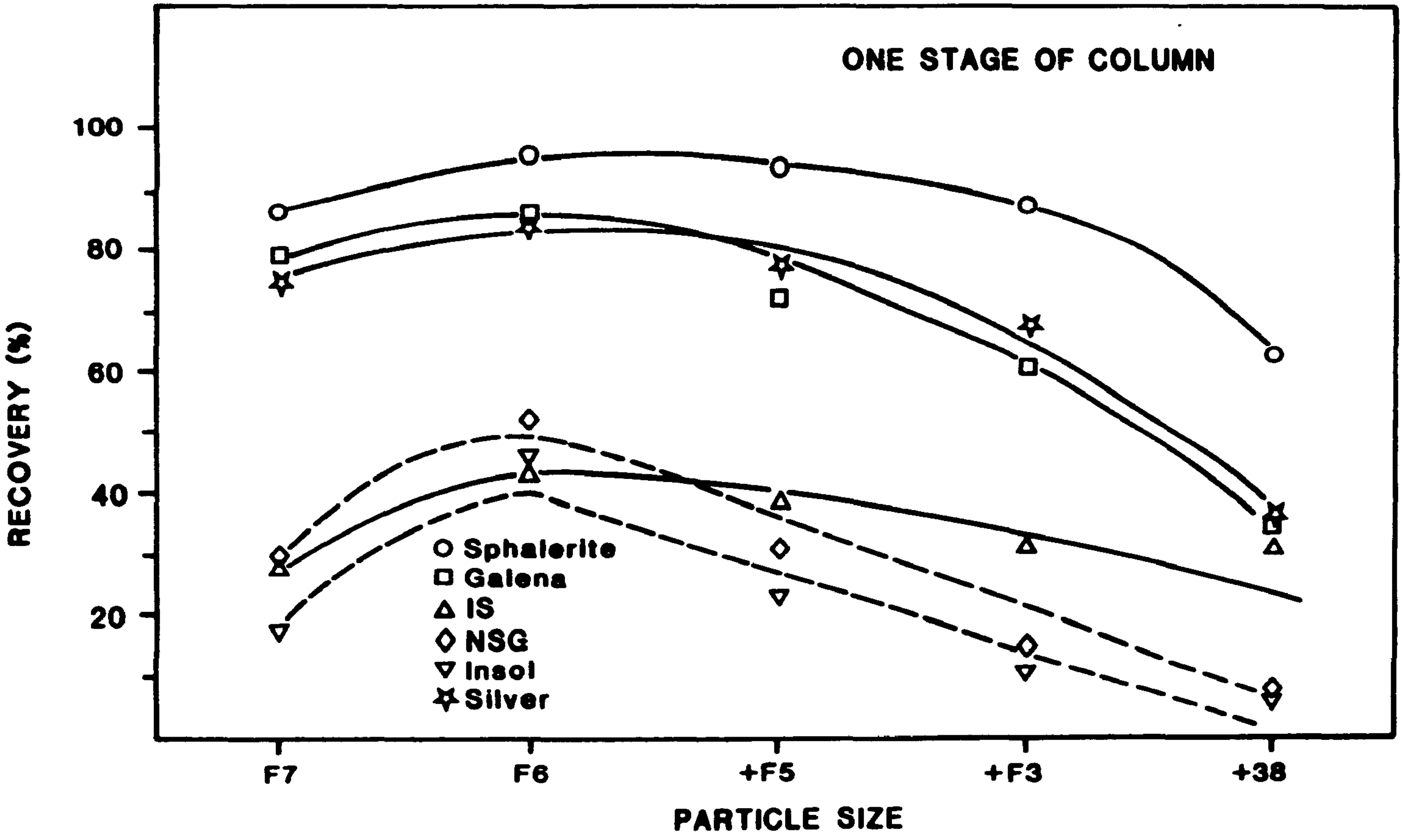


Figure 8.: Recovery size relationship for LGM column results. (OZ206)
(IS: iron sulphides, Insol, silicate gangue)

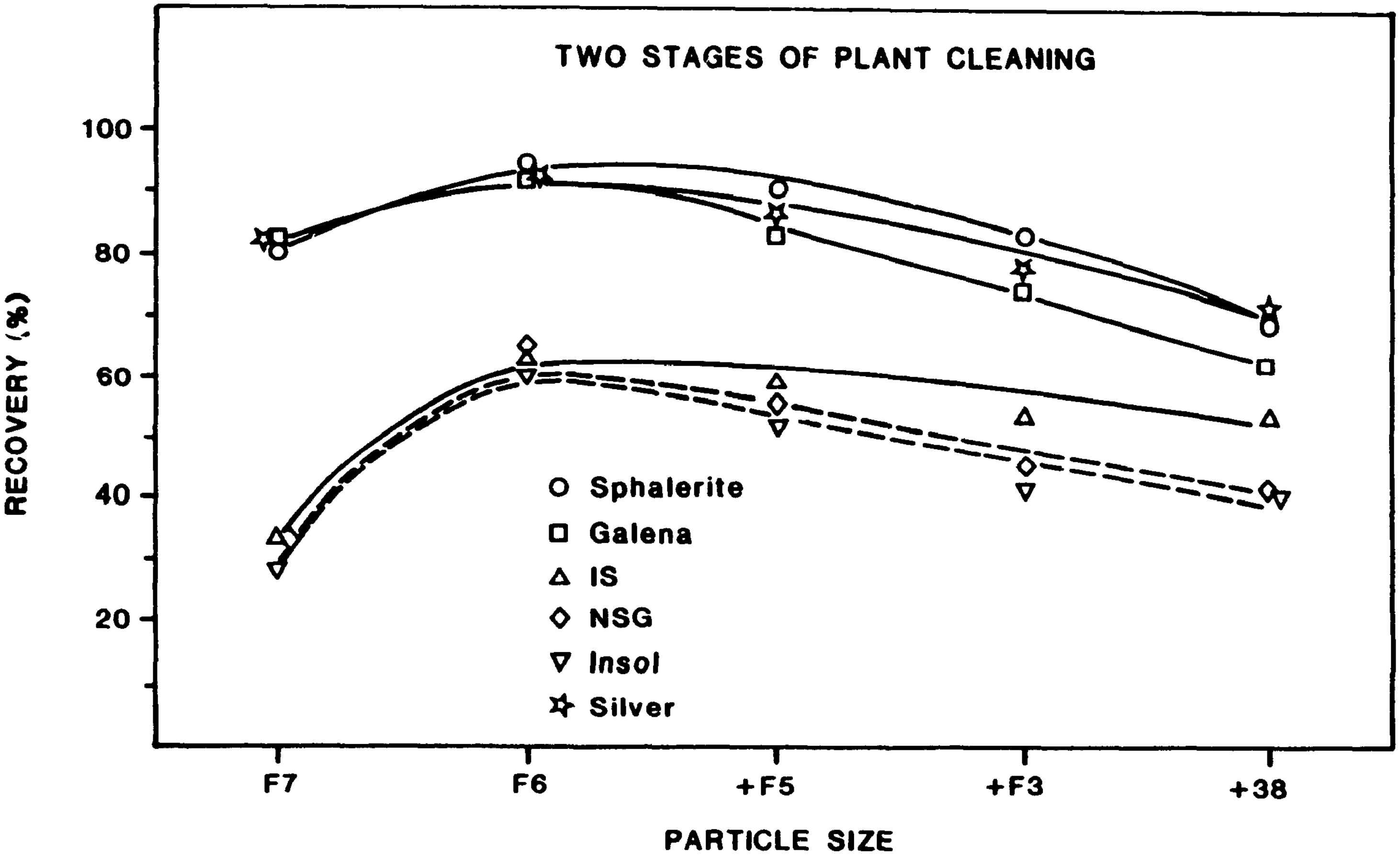


Figure 9.: Recovery-size relationship for LGM (plant results - OZ202)
(IS: iron sulphide; NSG: non-sulphide gangue)

(two stages of conventional cleaning). The shape of the recovery-size curves is similar. Notable differences are: in the plant, higher coarse galena recovery but lower sphalerite recovery; in the column, lower coarse iron sulphide (IS) and gangue (NSG and Insol) recovery. The lower recovery (improved rejection) of the coarser IS and NSG was the main cause of the increased column zinc concentrate grade.

Figures 10 and 11 show the recovery-size relationship for the Zn R/T Conc (the finest stream tested, about 75% -8 μm), for one and two column stages, respectively. The most notable feature is the preferential recovery of the finest sphalerite fraction in the first stage (Figure.10). This may be related to the troublesome medium rate constant sphalerite described in another publication (Johnson, 1987). Recovery of F7 NSG in the first stage was about 3.5%, and less than 1% for the IS. For the two stages, recovery of IS and NSG was generally lowest for the finest fractions, e.g., the NSG recovery in the +F3 fraction was 25% vs only 12% in the -F5 fraction. The fact that IS recovery is consistently lower than the NSG suggests NSG recovery is through composites.

Figures 12 and 13 show the recovery-size relationship for chalcopyrite, pyrite (unlike in the Pb/Zn plant, pyrite is the only iron sulphide in the Cu plant) and NSG, in the Cu R/T Conc (same products as Figure 6), for the one and two stages of operation, respectively. In the first stage, chalcopyrite particles from F3 to F6 were preferentially floated. Pyrite recovery was about 10% for all particle sizes. NSG recovery was less than 4% for all size fractions below +53 μm. For the two stage operation (Figure 13), chalcopyrite recovery was close to 100% for all particle sizes. Pyrite and NSG recoveries increased mostly at +53 μm reflecting the presence of composite particles. The recovery of F7 NSG was

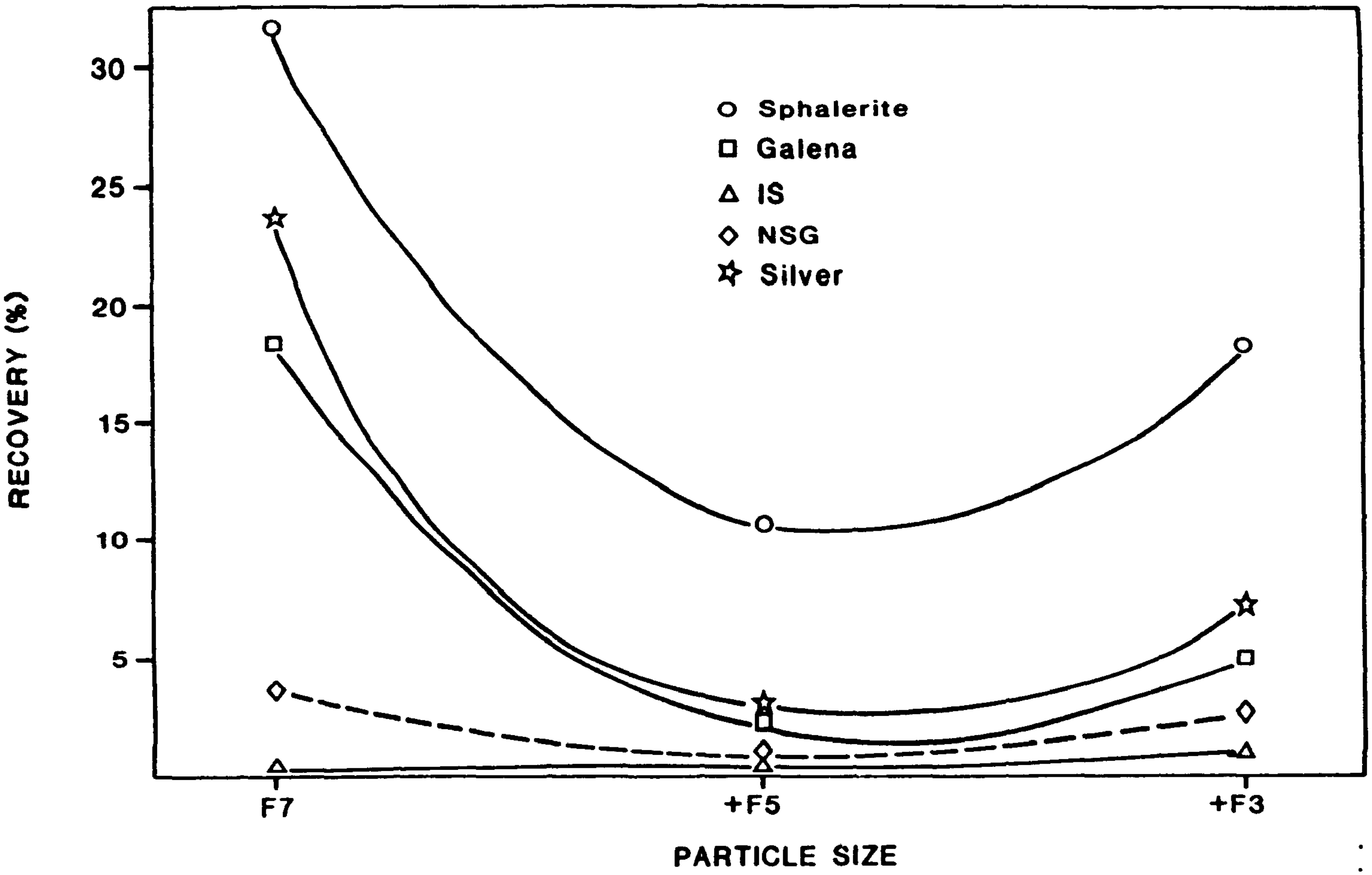


Figure 10.: Recovery-size relationship for Zn retreatment con - one stage (OZ256).

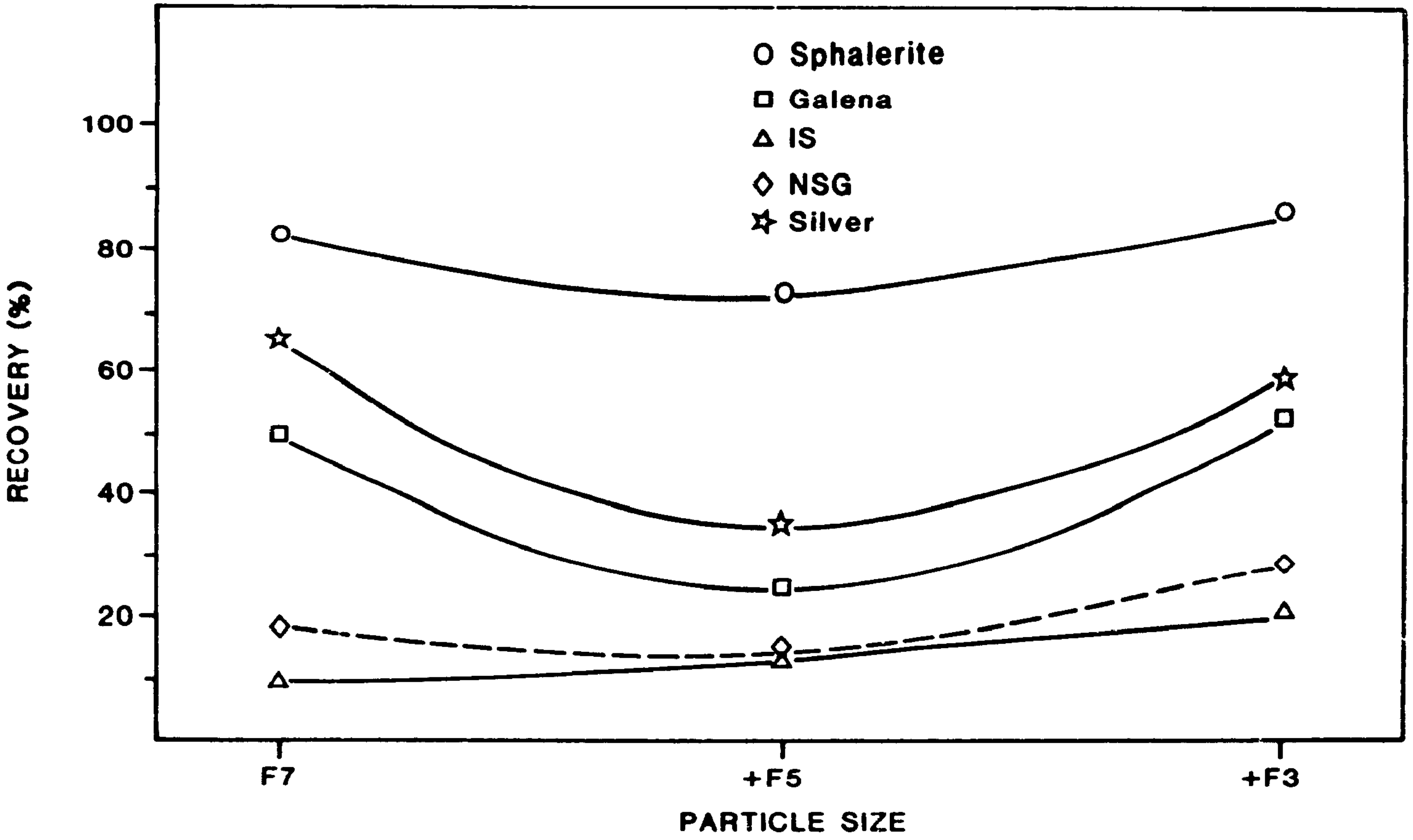


Figure 11.: Recovery-size relationship for Zn retreatment con – Two stages (OZ256 and OZ263)

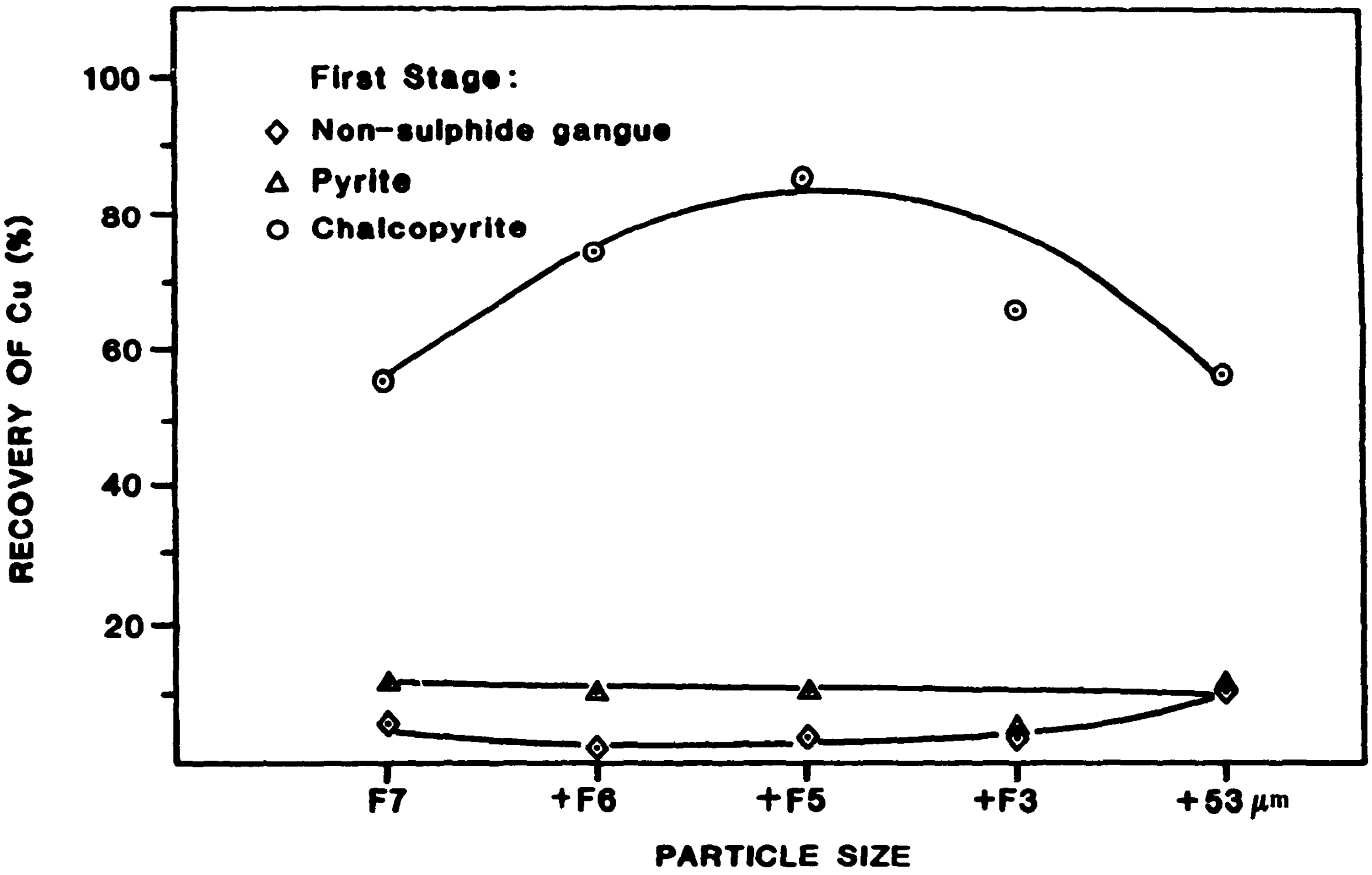


Figure 12.: Recovery-size relationship for chalcopyrite, pyrite and non-sulphide gangue, first flotation stage (copper retreatment concentrate)

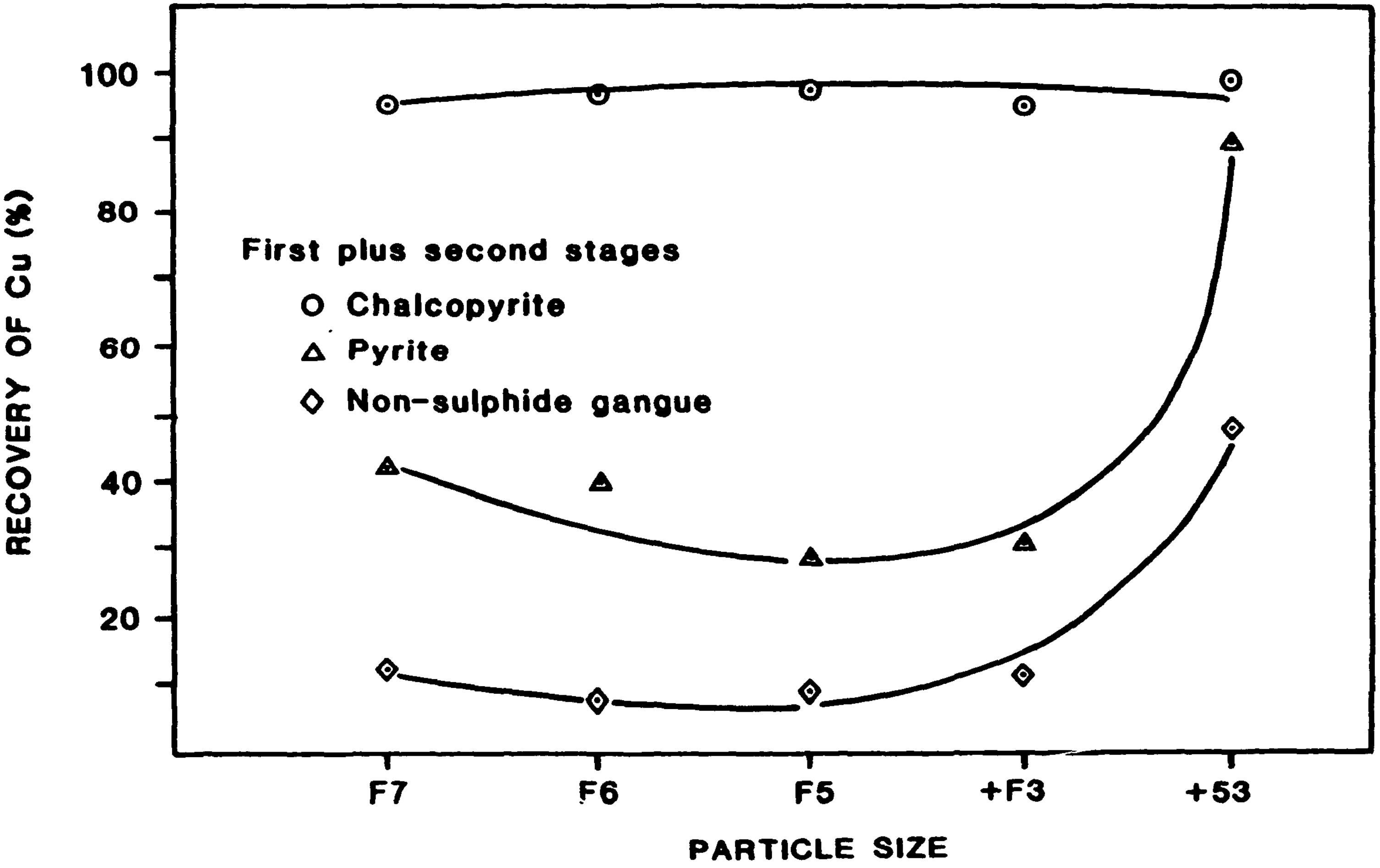


Figure 13.: Recovery-size relationship for chalcopyrite, pyrite and non-sulphide gangue, first and second flotation stages (copper retreatment concentrate)

about 12%, only slightly higher than for the intermediate size fractions. This illustrates that the column maintains good separation into the fine size range.

The flat shape of the IS and NSG recovery-size curves is quite different from the experience at MIM in conventional cells where the gangue recovery in the F7 fraction is generally the highest (Johnson and Jowett, 1982).

Table 7 shows the expected NSG recovery by entrainment in a single stage of conventional cell vs observed column gangue recovery in the F7 fraction, for the LGM, the Zn R/T Conc, and the Cu R/T Conc. Table 8 shows the calculations performed to obtain the expected plant values for a single stage of conventional cells. In general, the recovery of F7 NSG was much lower in the column than that expected by entrainment in the plant.

Size by size mineral analysis was not performed on the fine carbonate flotation at Niobec. However, sizing was done and it was found that the plant rougher concentrate was finer than the column concentrate, which was similar to the final concentrate. This is consistent with the interpretation that the column (and conventional cleaning stages) reject the fine hydrophilic components.

TRACER TESTS

Figure 14 shows typical residence time distributions (RTD) for the concentrate and tailings. Recovery of tracer (and, therefore, of water) is essentially nil. Comparing this to the measured recovery of F7 NSG in the column implies recovery is by mechanisms other than entrainment.

Table 9 summarizes the tracer test conditions and RTD parameters obtained. The low dispersion coefficients indicate close to plug flow

TABLE 7. EXPECTED GANGUE RECOVERY BY ENTRAINMENT IN A CONVENTIONAL CELL VS COLUMN GANGUE RECOVERY IN THE F7 FRACTION

STREAM	EXPECTED RECOVERY IN A CONVENTIONAL CELL (%) (*)	RECOVERY OF F7 NSG IN THE COLUMN (%) (**)	FEED SIZE (% F7)
Cu R/T CONC	30	12	62
Zn R/T CONC	35	16(**)	75
LGM	56	24(**)	50

(*) SEE TABLE 8
(**) AVERAGE OF NSG AND INSOL RECOVERIES

TABLE 8. ESTIMATION OF THE PERCENT F7 NSG RECOVERED IN THE CONCENTRATE BY ENTRAINMENT FOR A SINGLE STAGE OF CONVENTIONAL CELLS (11) WITH THE SAME FEED AS FOR THE COLUMN

STREAM	RECOVERY OF SOLIDS (%)	%SOLIDS FEED	%SOLIDS CONC	RECOVERY OF WATER (%)	RECOVERY OF F7 NSG (%) (*)
Cu R/T CONC	47	27	32	37	30
Zn R/T CONC	56	34	40	43	35
LGM	70	38	38	70	56

(*) (RECOVERY OF WATER) 0.8

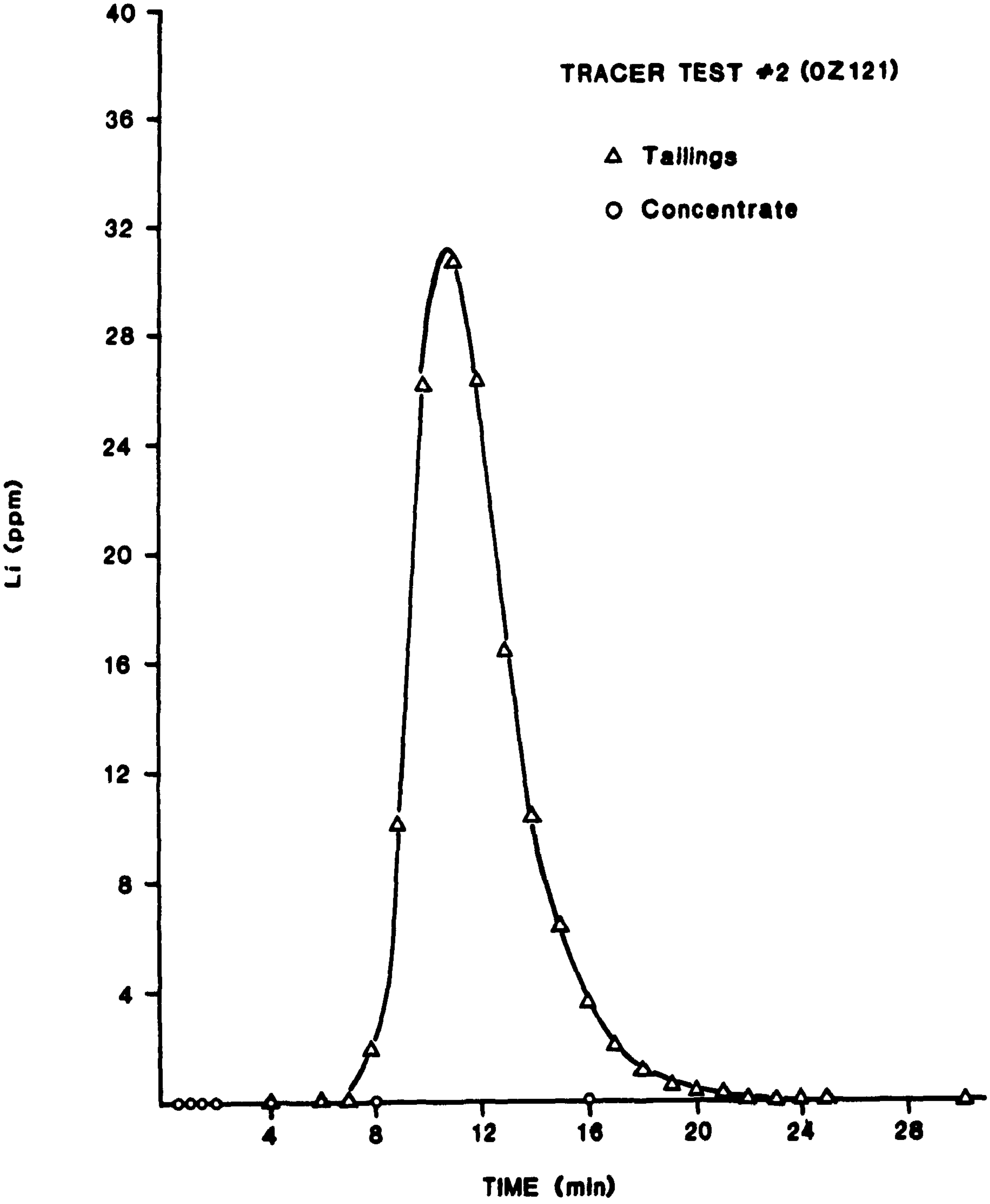


Figure 14. Residence time distribution, impulse tracer in feed.

TABLE 9. SUMMARY OF TRACER TEST CONDITIONS AND RTD PARAMETERS

TEST (*) #	LOCATION	SOLIDS PRESENT	SUP. VELOCITY (cm/s)			FD (cm)	RTD OF TAILINGS		
							RESIDENCE TIME (mins)		DISPERSION COEFFICIENT (**) m^2/s
			Jt	Jg	Jww		NOMINAL	MEAN	
1	MIM	YES	1.8	2.8	0.9	120	8.4	5.5	0.0007
2	MIM	YES	0.8	2.8	0.8	120	18.5	11.8	0.0014
3	NIOBEC	NO	1.0	1.6	0.5	95	15.4	14.6	0.0050

(*) IN ALL CASES RECOVERY OF FEED WATER TO CONCENTRATE WAS NIL

(**) ESTIMATED FROM THE RELATIVE VARIANCE OF THE RTD conditions (as does the shape of the tailings RTD in Figure 14). There is a marked difference in the nominal and mean residence time in the case where solids are present. The difference is related to the volume occupied by gas and solids. It is possible that the volume of gas in the column is increased in the presence of solids because the bubble/particle aggregate has a reduced rise velocity.

COLUMN OPERATING VARIABLES

Gas Rate: The superficial gas velocity generally used at MIM was from 2.5 to 3.0 cm/s; however, when the column was run at values as low as 1.6 cm/s, no difference was observed in grade or recovery. At Niobec, Jg varied from 1.0 to 2.0 cm/s with no appreciable differences in grades or recoveries.

Bias Rate: The superficial bias rates (Jb) used at MIM and Niobec varied from -0.05 to 0.9 cm/s. In all cases the experimental points lay on the same grade-recovery curve.

Froth Depth: The column was generally operated with a froth depth varying from 80 to 150cm. Some experiments with froth depths as shallow as 40cm did not show any difference in concentrate grade or recovery.

Feed Densities: The column was run at feed percent solids from 15% to 50%, with no difference in the position of the grade recovery curve. This characteristic is quite different from conventional cells were dilution of the pulp improves the position of the grade recovery curve by lowering the recovery of entrained solids.

DISCUSSION

METALLURGICAL PERFORMANCE

The column gave superior metallurgy compared to the plant and compared to carefully controlled laboratory tests. The origin of this improved performance lies either in improved rejection of entrained minerals or improved selectivity between two floating species.

Entrainment: The tracer studies and Table 9 show that the column effectively suppresses transfer of feed water into the concentrate. Feed water recovery approaches zero compared to 10% to 40% typical in mechanical cells. The F7 NSG recovery recorded in the column being higher than the water recovery may reflect composite particles, or agglomeration with floatable species.

The ability of the column to suppress entrainment means long flotation times can be employed to recover the slow floating fines without undue loss of grade. In this manner the column can give high recovery of fine particles. (Note: this has nothing to do with improved collection efficiency of fine particles in columns, which may or may not be a factor).

Selectivity: Improved selectivity between hydrophobic species (e.g. strongly hydrophobic species vs weakly hydrophobic liberated sulphide minerals and weakly hydrophobic composite particles) in a column compared with conventional cells is not so clear, but there is some evidence. For example the success of columns in the separation of Cu/Mo (Coffin and Miszczak, 1982; Miszczak, 1986), Cu/Fe (Amelunxen and Redfern, 1985; Dobby et al, 1985), and Cu/Ni (Feeley et al, 1987; Miszczak, 1986) does not seem entirely attributable to suppression of entrainment alone. Selectivity may be enhanced by the very deep froths possible in column flotation (Yianatos et al, 1987c). In this work the LGM data give the best evidence of improved selectivity. Figure 9 shows that the plant recovers more coarse IS and coarse galena than the column. This recovery is thought to reflect composite particles, in which case the column has shown superior rejection of composites.

These differences of performance between the column and conventional cell (the column showing superior rejection of entrained particles and possibly superior selectivity) suggests caution should be exercised if columns and conventional cells are to be intermixed, especially if circulating loads move from one cell type to another.

EFFECT OF COLUMN OPERATING VARIABLES

Gas Rate, Froth Depth, Bias Rate and Feed Density: Over the ranges tested there was no significant change in the grade recovery curve. This lack of dependance on operating variables appears to be a growing conclusion (Clingan and McGregor, 1987). It has ramifications in column control, making it a fairly forgiving device to bring under stabilizing control. For optimizing control gas rate is currently the favoured variable (Clingan and McGregor, 1987; Dobby et al, 1985). If the gas rate is kept high, then performance will vary with gas rate, for example further increases in gas rate will reduce recovery and grade (Yianatos et al, 1987d). At high gas rate, therefore, the position of the grade-recovery curve does become sensitive to gas rate.

The lack of effect of froth depth does suggest froth depth be kept to a minimum. Entrained solid is known to be rejected within a few centimetres of the interface when the column is operated in the correct range of gas and bias rates (Yianatos et al, 1987d). Thus so long as entrainment of liberated unwanted solid is the only problem, a shallow froth should suffice. This has the advantage of increased collection zone volume and retention time. However, if secondary upgrading (rejection of composites or free unwanted hydrophobic minerals) is a factor in reaching the target grade, deep (100cm) froths may be necessary. Deep froths do provide a buffer against poor control of the interface, commonly measured by pressure and thus subjected to error (Moys and Finch, 1987; Chaddock, 1987). Deep froths also help damp out recovery of feed water if high gas rates are being used.

Low bias rates offer the advantage of increased retention time. It is not known how low a bias can be and still yield acceptable cleaning, although bias rates lower than 0.1 cm/s have been suggested (Moys and Finch, 1987). The problem is on-line measurement of bias. Current techniques measure slurry rather than the required water which should ideally be controlled (Finch, 1987).

The ability of the columns to work with very high feed densities (e.g. 50%), while maintaining good cleaning action, makes them very attractive, as it means high capacity (Espinosa-Gomez et al, 1988).

SUMMARY AND CONCLUSIONS

Several flotation streams of very fine particles (d80, 8-23 μm) were studied by column flotation. Column metallurgical performance was compared to the plant where possible and to carefully controlled laboratory tests. Column results were better than plant and laboratory results in all the streams tested giving, in general, for the same metal recoveries, concentrate grades about 6% higher (in absolute terms). Entrainment was measured in the column by tracer tests and by the recovery of the NSG F7 fraction in the concentrate. Entrainment was estimated for conventional cells in the plant. For a given feed, the entraiment of the NSG F7 fraction in the column was much lower than expected for a single stage of conventional cells. With two stages of conventional cleaning at lower feed %solids than the column, a single stage of column cleaning was still superior. Evidence for improved rejection of relatively coarse composite particles in the column compared with conventional cells was also obtained.

5.4 SUMMARY AND CONCLUSIONS

The fine stream at Niobec and other streams of very fine particles (d80, 8-23 μm) were studied by column flotation. Column metallurgical performance was compared to the plant where possible and to carefully controlled laboratory tests. Entrainment was measured in the column by tracer tests and by the recovery of the finest size fraction. Entrainment was estimated for mechanical cells in the plant.

The main findings were:

1) Column flotation performance with the fines carbonates showed that carbonate-pyrochlore separation was superior to the plant rougher circuit, and the column floats equalled the circuit floats Nb_2O_5 grade after two cleaning stages.

2) The performance of the column on the fine stream in comparison with the slimes discard was by far superior. For about the same carbonate recovery to floats (about 60% CaO recovery) the pyrochlore recovery is 10% vs 40% for the slimes. This poor performance on the slimes is attributed to inadequate dispersion. It is considered that with the chemistry established, the column will prove the ideal device for slimes processing.

3) The conclusion that column flotation provides superior metallurgy of fines is supported by tests on six streams in both oxide and sulphide mineral systems. In general, for the same metal recoveries, the column produced concentrate grades about 6% higher (in absolute terms).

4) Elimination of feed water recovery and hence elimination of particle recovery by entrainment in a flotation column has been demonstrated. This is a major reason for the superior metallurgy in the column.

5) Some evidence is presented of superior selectivity in the column, perhaps related to the large froth depths (greater than 100 cm) used.

SUMMARY OF CONCLUSIONS
(for the whole thesis)

Water Quality

- Recycle water decreases the electrophoretic mobility of the particles and the surface potential of the particles becomes nearly indistinguishable. Thus the use of collectors which exploit chemical rather than electrostatic-type interactions is required.
- In general recycle water decreased selectivity and increased reagent consumption.
- Dispersion becomes particularly difficult with recycle water, requiring high concentration of dispersants which have a detrimental effect on flotation.

Selective Flotation Options

- Four options are contemplated to selectively float the fine pyrochlore from the slimes: using BPA at pH 3.9; using sulfosuccinate with tartaric acid at pH 9; using amine acetate with polyacrylamide (as at Niobec); and using BHA (at pH 6). This last option is technically feasible, but not economically attractive. Several reagents which may be as selective and more economically attractive still remain to be tested. They were not tested due to the problems of dispersion and froth collapse/coalescence encountered.

Dispersion and Carbonate Flotation

- Selective carbonate flotation (the stage prior to pyrochlore flotation) could not be achieved using the Niobec reagent combinations, due to lack of dispersion.

- To disperse the slimes, high dispersant concentrations are required which induce depression of carbonates.

Bubble Coalescence, Froth Collapse

- Fatty acids promote bubble coalescence, and froth collapse, probably due to coating of the bubble giving a hydrophobic surface and promoting water drainage. These problems are further enhanced with dispersed solids e.g. with sodium silicate and other dispersants, possibly by promoting the rupture of the thin films between bubbles.
- Bubble coalescence and froth collapse can be avoided working at high pulp densities (e.g. greater than 10% solids) which is best achieved at the plant site. This higher %solids is required to provide sufficient concentration of fatty acid emulsifier to promote a stable froth.

Column Flotation Tests

- Column flotation was found to be ideal for the selective flotation of fine particles, due to the reduction of entrainment and, to a lesser extent, improved selectivity.

CONTRIBUTIONS TO ORIGINAL KNOWLEDGE

1) Selective flotation of pyrochlore from silicates (microcline, biotite, chlorite), using two hydroxamate collectors: benzohydroxamic acid and N-benzoyl-N-phenyl-hydroxylamine, at pH 6 and 3.5, respectively was demonstrated in microflotation tests.

2) Selective flotation of pyrochlore from silicates (microcline, biotite and chlorite) using sulfosuccinate (collector) with tartaric acid (depressant) at pH 9 was demonstrated in microflotation tests.

3) A new technique was developed to evaluate and quantify dispersion. The technique uses a particle size analyzer, which gives a measure of apparent particle size.

4) A new approach was developed to quantify bubble coalescence along the column collection zone. The method is based on measuring changes in local gas holdup.

5) It was shown that the column is more efficient than conventional cells in the selective flotation of very fine particles (less than 20 μm). Entrainment is greatly reduced and increased selectively can be achieved in some cases.

SUGGESTIONS FOR FUTURE WORK

1) In the development of new flotation reagents it is necessary always to work with plant water or water of a quality similar to the plant, in order to closely simulate conditions.

2) Some reagents were found to be potentially selective for pyrochlore and deserve to be tested. These can be classified as selective pyrochlore collectors and selective pyrochlore depressants:

I) Collectors

- potassium octyl hydroxamate
- cinnamyl hydroxamic acid
- iso-nonyl imino-bis-methylene phosphonic acid
- dodecyl imino-bis-methylene phosphonic acid
- methyl-benzyl arsonic acid

II) Depressants

- pyrocatechol violet
- o-dihydroxy chromenols (7,8-dihydroxy-2, 4-dimethybenzopyrylium chloride)

3) To recover the pyrochlore from the slimes by column flotation (or by any other physical separation technique) it is essential to achieve better water quality to make dispersion possible at lower dispersant concentrations. Only when adequate dispersion can be achieved at lower dispersant concentration will selective pyrochlore flotation be possible.

4) There is sufficient circumstantial evidence that desliming at a size well below that currently practised at Niobec will give a deslimed material which can be treated. Desliming at about 4 μm and processing in columns is worh evaluating.

5) Any future column testing involving the fatty acid system should be performed at higher pulp densities (e.g. greater than 10%), to avoid froth collapse.

6) Column flotation should be considered whenever the treatment of very fine particles is required.

7) Column flotation in conjuction with fine particle separation techniques, such as carrier flotation, should be considered.

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APPENDIX 1. Reports 3,4 and 5 of Series Prepared for the Centre de Recherches Minerals, Quebec; and Manuscript Entitled: Effects of the Water Chemistry on Pyrochlore and Silicates Flotation (to be published in Minerals Engineering)

PROGRESS REPORT-PYROCHLORE PROJECT

REPORT # 3

R. ESPINOSA-GOMEZ

The present report summarizes the work done at McGill University on the pyrochlore project, for the period of March to mid-July, 1984. The report is divided in three main sections:

1) Microprobe study on two types of pyrochlore : a) high magnetic susceptibility (black pyrochlore), and b) low magnetic suscentibility.

2)Electrokinetic study on pyrochlore (low and high magnetic susceptibility), black pyrochlore, feldspars and biotite, in the presence of three modifiers, hydrochloric acid (HCl), oxalic acid ($C_2H_2O_4$) and fluosilicic acid (H_2SiF_6).

3) Procedure for selecting reagents for pyrochlore flotation, in order to attempt a direct pyrochlore flotation.

1) MICROPROBE STUDY

A microprobe study was performed on high and low magnetic susceptibility pyrochlore samples isolated from a pyrochlore concentrate. The isolation procedure is described in Report # 2. The low magnetic susceptibility fraction represented 95% of the total mass, and the remaining 5% was the high magnetic susceptibility fraction (black pyrochlore).

Table 1 summarizes the results. A total of 53 grains were assayed; 34 of low magnetic susceptibility, and 19 of high magnetic susceptibility. The main difference between the two samples was their Fe-content. It averaged 0.04% FeO for the former, and 0.38% FeO for the latter. There was no significant difference between the other assays: TiO2 (2.68 vs. 2.58), Na2O (8.35 vs. 8.31), CaO (16.31 vs. 16.16) and Nb2O5 (67.33 vs. 66.24). The standard deviations of the assays were not large: FeO (.04 +/- .04), TiO2 (2.68 +/- .57), Na2O (8.35 +/- .47), CaO (16.31 +/- .53), and Nb2O5 (67.33 +/- 1.37). Variations may be due to the different grain compositions and to a lesser extent to the precision of the instrument.

Table 2 shows that the low magnetic susceptibility sample is made up not only of low Fe-content pyrochlore grains, but possibly also of other pyrochlore subgroup minerals and some other Nb minerals. For example, point number 33 has 13.23% FeO, 71.61% Nb2O5, but very low content of Na2O (0.76% vs. 8.35%) and CaO (2.52% vs. 16.31%). Point number 24, has 0.16% Fe, but very low Na2O content (4.91% vs. 8.31%). Point number 27, has 0.38% Fe and also very low Nb2O5 content (48.5% vs. 67.3%). The proportion of these different Nb species, is quite high, about 27% of the mass relative to the low magnetic fraction, and 25% relative to the total. Certainly, this heterogeneous chemical composition may have an efffect on surface properties, e.g., electrokinetic mobility (zeta potential), flotation, coagulation.

Table 3 shows that 2 out of 19 grains assayed in the high magnetics fractions are not pyrochlore, but possibly hematite. They represent 10% of the mass relative to this fraction, but only 0.5% of the total.

2. ELECTROKINETIC STUDY

Samples of pyrochlore (low and high magnetic susceptibility), high susceptibility pyrochlore (black pyrochlore), feldspars and biotite were carefully prepared to measure their electrophoretic mobility values (and zeta potential).

2.1 Preparation of samples.

a) Pyrochlore. The method used to isolate pure pyrochlore samples has been already explained in Report # 2.

b) Feldspars. A pure sample of feldspars was obtained from Niobec. To increase its purity, it was carefully passed on the Frantz many times set at its maximum current, and the low susceptibility fraction retained. After the Frantz, the mineralogical species were identified by X-ray diffraction.

The sample was made up of two species, microline ($K(AlSi_3O_8)$) and quartz (SiO_2).

c)Biotite. A pure sample of biotite was obtained from Niobec. To increase its purity, it was carefully passed on the Frantz many times set first at 350 mA and second at 650 mA (350/650). The 350/650 mA fraction was retained. After the separation the sample was carefully observed under the microscope. Biotite, was the only mineral observed in the sample.

The particle size required for electrophoretic mobility measurements is from 1 to 3 um. Samples weighing about 2 g were used to obtain this particle size range. Each sample was first ground to powder in a mortar grinder and then transfered to a 500 ml glass cylinder (after 20 minutes of sample dispersion in an ultrasound bath). The sample was allowed to settle and the -3 um fraction was recovered at the top of the cylinder. The suspension recovered was transfered to a 200 ml flask and stored for futur use . Deionized water was used for these steps. To measure electrophoretic mobility, about 2 ml of suspension were transfered from the flask to a beaker with about 50 ml of the type of water used in that particular measurement. The pH was adjusted and allowed to equilibrate for about 4 hrs. After this time, the pH was measured again and 5 ml of the suspension used to measure electrophoretic mobility. The remaining 45 ml were used to rinse many times the flat cell and wash the electrodes.

2.2 Types of water used.

Three types of water were used on the study: organic free water, distilled water and potable water from Niobec. Organic free water was used for comparison, organics in the water can modify the electrokinetic mobility of the particles. The use of potable water from Niobec was considered of primary importance to simulate the real electrokinetic mobilities of the particles in the plant. Potable water is used in the plant for pyrochlore-silicate flotation at Niobec.

2.3 Apparatus.

Electrokinetic mobility of the particles was measured by a Mark II micro-electrophoresis apparatus (Rank Brothers). It consists essentially of a transparent chamber comprising a flat type quartz cell in which observations of moving particles are made under an applied voltage through platinum electrodes. Movement of the particles was observed through the eye-piece micrometers, which were located on each side of a timer accurate to 1/10 of a second. One of the eye-pieces is fitted with a scale of 104 um/division, the other 36um/division.

The water bath temperature of the micro-electrophoreses apparatus was determined during the observations with a total immersion thermometer with graduations of 0.1 C. The temperature used was 25 C.

2.4 Calculation of electrophoretic mobility.

The electrophoretic mobility was calculated using the following relation:

$$u = \frac{\text{particle velocity}}{\text{potential gradient}} = \frac{d/t}{E/l} \quad \frac{(\text{cm/s})}{(\text{volt/cm})}$$

where:

d: distance travelled by the particle (cm)

t: average time required to travel distance d, (s)

E: strength of the applied field (volts)

l: inter-electrode distance (cm)

u: electrophoretic mobility (cm/s/volt/cm).

2.5 Results

A total of 13 electrophoretic mobility vs. pH curves were obtained. Five curves for pyrochlore (low and high magnetic susceptibility), 4 curves for black (high susceptibility) pyrochlore, 2 curves for feldspars and 2 curves for biotite.

Figure 1 presents the electrophoretic mobility vs. pH curves for pyrochlore in the presence of three types of water, organic free, distilled and potable water (from Niobec). The pH modifiers used were HCl and KOH. The mobility of the particles and the PZC changed with the type of water. The

PZC were 2.5, 3.5 and 2.7 for free organic, distilled and potable waters respectively. A very significant decrease in electrophoretic mobility was experienced when potable water was used. A complete analysis of the anions and cations presents on the potable water has not yet been performed. Some of the cations present are Ca: 23 ppm; Mg: 1.3 ppm; Pb: 0.3 ppm; Fe: 0 ppm; and Cu: 0 ppm. The natural pH of the water is 8.2, so it is possible that it contains a high concentration of carbonates. Other anions such as sulfates and phosphates may also be present. The presence of cations such as Ca and Mg may be the main reason for the lower values of electrophoretic mobility.

Figure 2. Shows the electrophoretic mobility vs. pH for pyrochlore with potable water using three types of modifiers, HCl, C2H2O4 and H2SiF6. The HCl and H2SiF6 tend to reduce the electrophoretic mobility to a larger extent . However, there is no significant difference especially below pH 3. The PZC were 2.7, 2.5 and 2.4 for HCl, C2H2O4 and H2SiF6 respectively.

Figure 3. Shows the electrophoretic mobility vs. pH curves for pyrochlore, feldspars and biotite using potable water and C2H2O4 as modifier. The PZC were 2.5, 2.0 and 1.5 for pyrochlore, feldspars and biotite, respectively. The electrophoretic mobility for both feldspars and biotite were about the same. For pyrochlore the mobility was less, but not to a significant extent.

Figure 4 Shows the electrophoretic mobility vs. pH curves for pyrochlore, feldspars and biotite using potable water and H_2SiF_6 as modifier. The PZC were 2.4, 2.0 and 1.9 respectively. As with the $C_2H_2O_4$, the mobility of feldspars and biotite were about the same. The mobility of the pyrochlore was reduced more than with the $C_2H_2O_4$ but again not to a statistical significant extent.

Figure 5 Shows the mobility vs. pH curves for pyrochlore (basically low magnetic susceptibility), and black pyrochlore, using distilled water and HCl/KOH as pH modifiers. The PZC were 3.5 and 4.6 for pyrochlore and black pyrochlore respectively. This difference is significant and may be related with the higher Fe-content in the black pyrochlore.

Figure 6 Shows the mobility vs. pH curves for pyrochlore and black pyrochlore using potable water and HCl as pH modifier. The PZC were 2.7 and 2.3 for pyrochlore and black pyrochlore respectively. In general the mobility of the black pyrochlore was higher than that of the pyrochlore.

Figure 7. Shows the mobility vs. pH curves for pyrochlore and black pyrochlore using potable water and $C_2H_2O_4$ as pH modifier. The PZC were 2.5 and 2.2 for pyrochlore and black pyrochlore respectively. For values above pH 6.5, the mobility of pyrochlore is higher than for the black pyrochlore; for values of pH below 6.5, the opposite is true.

Figure 8. Shows the mobility vs. pH curves for pyrochlore and black pyrochlore using potable water and H2SiF6 as pH modifier. The PZC were 2.4 and 2.3 for pyrochlore and black pyrochlore respectively. As in Figure 7, for values of pH above 6.5, the mobility of pyrochlore is higher than for the black pyrochlore, and vice versa for pH $\leq$ 6.5.

Figures 9 and 10, present the confidence intervals of the electrophoretic mobility values on pyrochlore and black pyrochlore. Each point is the main value of 20 mobility measurements and the bars point out +/- 1 standard deviation of such mean value.

Figures 11 and 12, present the confidence intervals of the electrophoretic mobility values on feldspars and biotite.

Table 4 summarizes all the PZC values obtained during the electrokinetic study. In general, the gangue minerals have PZC values below pH 2 and the pyrochlore, above 2.3.

2.6 Discussion of results.

a) Electrokinetic properties of the pyroclore.

As shown in Figures 1 and 2, the electrokinetic properties of the pyrochlore change according with the type of water and modifier used. In general the PZC was found at a pH ranging from 2.4 - 2.7. Only one curve, using distilled water, gave a significantly different value, 3.5. This high value may possibly be related to metalic trace metals present in the distilled water used which usually tend to increase the PZC. With the present information no satisfactory explanation can be offered to justify such an increase.

As mentioned for the other curves, the differences in PZC are small, 2.4 - 2.7. Differences may be related to the strength of the particular acid to leach out the metal impurities present in the crystal lattice of the pyrochlore, and the ionic species that are formed. However, such differences may not be real, since it is quite difficult to detect the real pH for the PZC especially at low values of pH. The limitation is that at low pH values it is not possible to increase the strength of the applied field required to increase the mobility of the particles, without having a strong risk of polarization. This fact limits the exact determination of the PZC at low pH values.

Curve 1 from Figure 1 (organic free water), shows a sudden increase to a peak in the mobility of the particles at pH values between 3 and 4. One possible explanation can be the leaching of some impurities present in the crystal lattice, without further readsorption on the particle surface. This curve will be repeated to determine if there is some water contamination or if the peak is real.

Curve 3 from Figure 3, shows a significant decrease in the mobility of the particles in the presence of potable water. This water contains considerable cationic and anionic contaminants which strongly affect particle mobility. To ascertain which ions are mainly affecting the mobility , a complete analysis of the water is required. At present only some of the contaminants are known, e.g. Ca: 23 ppm, Mg: about 1.3 ppm, and Pb: less than 1 ppm; these cations are known to reduce the mobility of the negative particles.

The values of PZC obtained in this study (2.4-2.7) agreed with previous results obtained at Ecole Polytechnique (2.6-2.8), although, it is important to stress that the type of water used in both studies were different, potable at McGill and distilled at Ecole Polytechnique.

b) Electrokinetic properties of pyrochlore, feldspars and biotite.

Figures 3 and 4 present the mobility of the pyrochlore, feldspar and biotite particles. In general, both C2H2O4, and H2SiF6 have more effect on the mobility of the pyrochlore than either feldspar or biotite, but not to a significant extent.

These results suggest that the flotation mechanisms which govern pyrochlore flotation at Niobec (using a cationic collector), are not purely electrostatic. Unless a silicate (feldspars and biotite) depressant is used which can reduce the mobility of the silicates.

Since the PZC of the silicates is in all the cases below 2.0 and for the pyrochlore, above 2.3, there is only a narrow margin (2.0 - 2.2) in which cationic collectors can selectively separate the silicates. Thus, it may be convenient to remove the silicates in two steps: a) direct pyrochlore flotation at pH of about 3.8 (as is presently done); and b) inverse flotation of pyrochlore at pH 2-2.2 using acetic acid as pH modifier, and a strong cationic collector to float off the silicates. Apparently, it should be possible. This requires further attention.

c) Electrokinetic properties of pyrochlore and black pyrochlore.

The black pyrochlore is the fraction of the pyrochlore in the ore which has a relatively high magnetic susceptibility. It can be recovered on the Frantz at currents of 250 mA, and represent about 5% of the total mass of the pyrochlore sample studied.

Figures 5-8 show the electrophoretic mobility of the pyrochlore particles (i.e. basically low magnetic susceptibility particles), under different conditions. The curves in Figures 6-8 were obtained using potable water and 3 different pH modifiers, HCl, $C_2H_2O_4$ and H_2SiF_6. In general, the black pyrochlore presented a higher mobility, with the exception of values of pH above 6.5 when using $C_2H_2O_4$ and H_2SiF_6.

The PZC for black pyrochlore and pyrochlore were 2.7 vs. 2.3, 2.5 vs. 2.2, and 2.4 vs. 2.3 for HCl, $C_2H_2O_4$ and H_2SiF_6, respectively.

From this discussion it is noted that electrokinetic properties differ according to the pyrochlore composition. Since the pyrochlore from

Niobec is made up of several chemical compositions, as suggested in the microprobe study, it can be expected that each particular composition has its own electrophoretic mobility. This is pursued later.

d) Confidence intervals of the electrophoretic mobility values.

Figures 9 and 10 present the confidence intervals of the electrophoretic mobility values on pyrochlore and black pyrochlore. Obviously there is a wide spread in the mobility measurements. One contributing factor is the differences due to the different chemical composition of the pyrochlores. The variations in chemical composition were clearly shown in Tables 2 and 3.

This heterogeneity may play a significant role in the collection mechanisms. Heterogeneity in the pyrochlore may also contribute to pyrochlore flotation losses, especially when flotation is performed at low values of pH, about 3. At these pH values the mobility of the particles is very low and consequently also surface potential is less negative. This leads to a weaker interaction with cationic collector. One possible way to solve this problem, is the inverse flotation of pyrochlore at pH values of about 2.1, using a strong cationic collector.

Figures 11 and 12 present the confidence intervals of the electrophoretic mobility values for feldspars and biotite. As mentioned in section 2.1, the feldspars are made up of microcline and quartz. This mixture of silicates may be one reason for the range in mobility values. In the case of mica it is well known that the biotite has a hetereogeneous composition, which gives it different electrokinetic properties.

3. DIRECT FLOTATION OF PYROCHLORE.

This section explains a procedure to look for selective reagents for pyrochlore flotation, in order to attempt a direct pyrochlore flotation.

A microflotation cell specially designed for fine particles, will be used. The particle size range to be tested is about -16 + 3 um, which is quite close to the size distribution of the Niobec slimes. In all the tests potable water from Niobec will be used.

The best reagents selected in this study will be tested by column flotation on the slimes from Niobec.

The direct flotation of pyrochlore has been attempted on many occasions all around the world, (1)-(9). Only one research team has been successful in developing a procedure which gave very good results; this was on Niobec ore (6)-(8). However, the flowsheet of the process is complex due to the nature of the ore .

Table 5, presents the ore composition and some literature values of the point of zero charge (PZC) of the minerals in Niobec ore. Notice that the pyrochlore represents only about 1% of the mass . The most abundant contaminants are carbonates (principally dolomite, and calcite), silicates and to a lesser extent apatite and silicates. Direct pyrochlore flotation has proved difficult due to the similar flotation characteristics of the pyrochlore and the two main contaminants, carbonates and silicates. In particular the silicates present a serious problem due to their similar electrokinetic properties in an aqueous media. Notice from Table 5 that the PZC of pyrochlore is about 2.6, biotite about 1.5, chlorite about 4.5, and Na,K feldspars about 1.8. These values are quite close, limiting selective cationic flotation; it is difficult to achieve selective depression of the silicates. To effect a selective anionic flotation it would be necessary to avoid the activation of the silicates by Fe(+3) and Al(+3) cations. Direct pyrochlore flotation would require a very selective Nb flotation reagent.

The search for selective reagents for pyrochlore has been already attempted without any great succes (10)-(12). However, there are many other reagents that can be tested.

3.1 Selective Nb Flotation Reagents

In order to search for available Nb organic flotation reagents a systematic procedure was followed. A literature survey in analytical chemistry of Nb was carried out to look for the more selective reagents (13)-(17),(21). Also considered were flotation reagents already tested for Nb and Ta, (1)(4)(5)(8)(18), and flotation reagents suggested by some manufacturers, e.g. Hoechst Canada Inc. and Allied Colloids. For each reagent, the optimum selectivity conditions were examined. During the search two basic groups of reagents were found, one suited to the flotation of pyrochlore (flotation collector), the second suitable to the selective depression of pyrochlore (flotation depressant). Some ideas on the selection of flotation reagents have been given elsewhere, (19)(20).

Tables 6 and 7, summarize the organic reagents found during the search. Table 6 shows the reagents suitable as collectors. Some interesting points are noted: 1) none of the reagents is totally selective for Nb; 2) for most of the reagents, Fe(+3) cations cause chemical interference; 3) some reagents have apparently good selectivity but only within a very narrow pH range, for example, the BPA can react selectively with the pyrochlore but only at pH 3.5-3.6 and using tartarate and EDTA to mask Al, Ca, Mg and Fe(111). Table 7 presents some organic depressants that apparently are selective for Nb, allowing a possible successful inverse pyrochlore flotation. Among them are: pyrocatechol violet, tannin (at pH about 4.5) and bromopyrogallol red. Also shown is a reagent that can complex Fe cations, citric acid.

3.2 Selection of simple flowsheets for the direct flotation of pyrochlore at Niobec

Most of the flotation reagents potentially suitable for the direct flotation of pyrochlore are summarized on Tables 6 and 7. However, they have to be tested under several conditions to look for optimum results. The approaches to be used are the following:

A) Direct flotation of pyrochlore (with prior carbonate and apatite flotation).

B) Inverse flotation of pyrochlore (with prior carbonate and apatite flotation), i.e. floating the silicates.

C) Direct flotation of pyrochlore (without prior carbonate and apatite flotation).

A) Direct flotation of pyrochlore (with prior carbonate and apatite flotation)

One of the most important points noted from Table 6 is the need to go to low pH to achieve any selectivity. About 70% of the Niobec ore (Table 5) is carbonates and about 7% apatite, which are both soluble in acid. Thus it may be advisable to float these minerals prior to lowering the pH. After the removal of as much of the carbonates and apatite as possible, the main problem would be the selectivity between pyrochlore and the silicates. This separation will be attempted using the following routes:

1) pyrochlore flotation using citric acid as silicate depressant, and also to mask Fe(+3), and Nb collectors at various pH and collector concentrations.The use of citric acid as an Fe complexing agent to produce a hydrophilic compound, is suggested on Table 7. In addition citric acid acts as a depressant for quartz, mica, carbonates and iron oxides (22). It is also necessary to test the effect of pH. For example, Table 6 suggests optimum pH conditions for alkyl hydroxamates, phenyl arsonic acid and 8-hydroxyquinoline, as 7, 4.8 and 4.0 respectively.

2) Pyrochlore flotation with tartarate and ethylenediamine tetracetic acid (EDTA) as silicate depressant and N-benzoyl-N-phenyl-hydroxylamine (BPA) as Nb collector.

3) Depression of silicates at high acid concentration (pH ` 1.8), and further pyrochlore flotation at optimum pH of the Nb collectors. For example, pyrochlore flotation can be attempted at pH of about 3 using sodium alkyl sulphate. Citric acid may be required to help silicate depression.

B) Inverse flotation of pyrochlore (with prior carbonate and apatite

flotation).

The inverse flotation of pyrochlore from silicates also looks attractive. At all pH above 4.5 the silicates present in the ore bear a negative charge. They can then be floated using cationic collectors while depressing the pyrochlore. This approach will be attempted using the following routes:

1) depression of pyrochlore using the more selective Nb depressants, as listed in Table 3, such as tannin, pyrocatechol violet and bromopyrogallol red; followed by cationic flotation of silicates. The pH of the tests has to be always above 4.5;

2) depression of pyrochlore using less selective Nb depressants, such as tartaric acid, followed by cationic flotation of silicates at pH above 4.5.

C) Direct flotation of pyrochlore (without prior carbonate and apatite flotation).

This approach will be attempted using two types of depressants; sodium hexametaphosphate to depress carbonates and apatite, and citric acid to depress silicates. Pyrochlore flotation will be achieved by hydroxamic acid and suitable Nb collectors at pH ¨ 5.

Several other possibilities may exist with other reagents and conditions. They will be explored as information becomes available.

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TABLE 1. SUMMARY OF THE MICROPROBE STUDY ON PYROCHLORE OF LOW AND HIGH MAGNETIC SUSCEPTIBILITY.

CONCENTRATION (%)		
COMPOUND (%) (C)	[1] LOW MAGNETIC SUSCEPTIBILITY	[2] HIGH MAGNETIC SUSCEPIBILITY
	(%C +/- 1σ)	(%C +/- 1σ)
FeO	.04 +/- .04	.38 +/- .18
TiO_2	2.68 +/- .57	2.58 +/- .49
Na_2O	8.35 +/- .47	8.31 +/- .16
CaO	16.31 +/- .53	16.16 +/- .47
Nb_2O_5	67.33 +/- 1.37	66.24 +/- 1.19
	94.71 +/- 1.22	93.68 +/- 1.34

[1] A total of 34 grains were assayed.
[2] A total of 19 grains were assayed.

	CONCENTRATION (%)							
COMPOUND	POINT NUMBER							
	1	*2	3	4	5	6	7	8
FeO	.05	.33	.05	0	.24	0	0	.09
TiO_2	2.63	2.28	2.48	2.47	3.16	3.02	2.62	2.35
Na_2O	6.72	4.37	8.68	8.24	8.05	8.58	8.49	9.33
CaO	16.88	15.19	16.19	16.57	16.94	16.47	16.35	15.56
Nb_2O_5	68.65	64.11	67.83	68.11	65.7	67.67	67.46	68.13
	94.93	86.28	95.22	95.4	94.1	95.74	94.92	95.46

* Most likely other pyrochlore subgroup minerals.

TABLE 2. MICROPROBE STUDY OF THE LOW MAGNETIC SUSCEPTIBILITY PYROCHLORE (-250 mA, FRANTZ), CONTINUATION.

	CONCENTRATION (%)							
COMPOUND	POINT NUMBER							
	*9	10	11	12	*13	14	15	16
FeO	2.06	0	.19	.02	.45	.01	.05	0
TiO2	3.49	1.53	2.52	1.92	.94	2.21	3.45	2.19
Na2O	4.21	8.98	8.37	8.61	3.95	8.41	7.96	8.64
CaO	14.[illegible]	16.1	15.66	15.91	15.29	16.44	17.11	15.63
Nb2O5	[illegible]	9.67	67.24	68.4	69.81	68.82	67	67.16
			93.98	94.85	90.45	95.89	95.58	93.52

* Most likely other pyrochlore subgroup minerals.

TABLE 2. MICROPROBE STUDY OF THE LOW MAGNETIC SUSCEPTIBILITY PYROCHLORE (-250 mA, FRANTZ), CONTINUATION.

COMPOUND	CONCENTRATION (%)							
	POINT NUMBER							
	17	18	*19	20	21	*22	23	*24
FeO	0	.01	5.34	.03	.06	1.4	0	.16
TiO_2	2.82	1.73	2.56	2.28	3.36	4.72	2.63	3.61
Na_2O	8.15	8.75	.15	8.77	7.86	4.31	8.54	4.91
CaO	16.58	15.43	10.65	16.33	17.6	13.86	15.9	17.73
Nb_2O_5	68.2	65.57	73.97	69.86	66.84	57.03	68.28	67.5
	95.74	93.5	92.67	97.27	95.28	81.33	95.35	93.29

* Most likely other pyrochlore subgroup minerals.

TABLE 2. MICROPROBE STUDY OF THE LOW MAGNETIC SUSCEPTIBILITY PYROCHLORE (-250 mA, FRANTZ), CONTINUATION.

COMPOUND	CONCENTRATION (%)							
	POINT NUMBER							
	25	26	*27	*28	29	30	31	32
FeO	0	.05	.38	.4	0	.05	0	.03
TiO_2	3.29	2.58	1.75	1.65	2.49	4.21	2.96	3.17
Na_2O	8.05	8.21	5.01	4.93	8.53	8	8.2	8.28
CaO	17.05	16.47	10.21	10.05	16.33	16.34	16.42	16.63
Nb_2O_5	66.65	67.8	48.51	49.44	66.89	64.58	66.23	66.29
	95.04	95.11	65.85	66.46	94.24	93.18	93.81	94.41

* Most likely other pyrochlore subgroup minerals.

TABLE 2. MICROPROBE STUDY OF THE LOW MAGNETIC SUSCEPTIBILITY PYROCHLORE (-250 mA, FRANTZ), CONTINUATION.

CONCENTRATION (%)			
COMPOUND	POINT NUMBER		
	*33	34	average
FeO	13.23	.09	.04
TiO2	2.77	2.96	2.68
Na2O	.76	8.35	8.35
CaO	2.52	15.31	16.31
Nb2O5	71.61	64.24	67.33
	90.9	90.94	94.71

*Most likely other pyrochlore subgroup minerals.

TABLE 3. MICROPROBE STUDY OF THE HIGH MAGNETIC SUSCEPTIBILITY PYROCHLORE (+250 mA, FRANTZ)

COMPOUND	CONCENTRATION (%)							
	POINT NUMBER							
	1	2	3	4	5	6	7	*8
FeO	.5	.2	.2	.4	.2	.6	.6	87.4
TiO2	2.4	3.5	2.8	2.8	2.3	3.7	2.8	.3
Na2O	8.4	8.1	8.3	8.1	8.4	8.1	8.1	.1
CaO	16.2	16.8	16.1	14.7	16.1	16.8	16.5	.1
Nb2O5	66.2	66.3	67.6	62.9	66.6	63.8	66.1	.1
	93.8	94.9	95	89	93.6	93	94	88

* Possibly, hematite grains.

TABLE 3. MICROPROBE STUDY OF THE HIGH MAGNETIC SUSCEPTIBILITY PYROCHLORE (+250 mA, FRANTZ), CONTINUATION.

COMPOUND	CONCENTRATION (%)							
	POINT NUMBER							
	9	10	11	12	13	*14	15	16
FeO	.1	.6	0	.4	.6	83.3	.5	.3
TiO2	2.9	1.8	2.6	2.3	2.1	3.6	1.9	2.4
Na2O	8.4	8.5	8.4	8.3	8.4	.1	8.6	8.2
CaO	16.1	15.8	16.6	16.2	15.9	.1	15.9	16.5
Nb2O5	67.1	66.4	68	66.8	66.7	.6	66.7	66.5
	94.5	93.1	95.6	94	93.8	87.8	93.6	94

* Possibly, hematite grains.

TABLE 3. MICROPROBE STUDY OF THE HIGH MAGNETIC SUSCEPTIBILITY PYROCHLORE (+250 mA, FRANTZ), CONTINUATION.

CONCENTRATION (%)				
COMPOUND	POINT NUMBER			
	17	18	19	AVERAGE
FeO	.4	.5	.4	.39
TiO2	2.8	2.7	2.1	2.58
Na2O	8.1	8.5	8.3	8.31
CaO	16.1	16.3	16.1	16.16
Nb2O5	66	65.7	66.6	66.24
	93.5	93.6	93.6	93.68

TABLE 4. SUMMARY OF THE POINT OF ZERO CHARGES OBTAINED ON THE ELECTROKINETIC STUDY.

MINERAL	*PZC			
	pH MODIFIER	TYPE OF WATER		
		ORGANIC FREE	DISTILLED	POTABLE
PYROCHLORE	HCl	2.5	3.5	2.7
PYROCHLORE	C2H2O4			2.5
PYROCHLORE	H2SiF6			2.4
FELDSPARS	C2H2O4			2.0
FELDSPARS	H2SiO6			2.0
BIOTITE	C2H2O4			1.5
BIOTITE	H2SiO6			1.9
PYROCHLORE (BLACK)	HCl		4.6	2.3
PYROCHLORE (BLACK)	C2H2O4			2.2
PYROCHLORE (BLACK)	H2SiO6			2.3

*PZC, point of zero charge, or rather, PZM, point of zero mobility.

TABLE 5. MINERALOGICAL COMPOSITION OF THE NIOBEC ORE.

MINERAL	Wt. (%)	PZC
CARBONATES	65	
DOLOMITE		7
CALCITE		8.2
ANKERITE		
SULPHIDES	1	
PYRITE		1.5
PYRRHOTITE		
OXIDES		
PYROCHLORE (10:1)	1	2.6
COLUMBITE		
MAGNETITE, HEMATITE AND ILMENITE.	2	6
APATITE	7	5
ZIRCON	.2	5.8
SILICATES		
BIOTITE		1.5
CHLORITE		4.5
Na, K, FELDSPARS		1.8

TABLE 6. ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS.

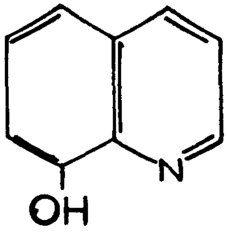
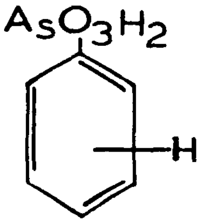
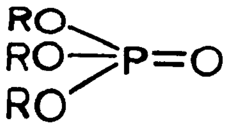
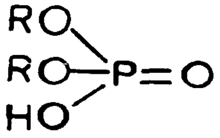
ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS			
NAME	STRUCTURE	SELECTIVITY	REMARKS
8-Hydroxyquinoline. Ref. (13),(14),(15),(21).		Poor	In acetate solutions react with Al,Fe,Nb,Ta,Th,U,V,Cu,Ag.
Benzenearsonic acid (phenylarsonic acid). Ref. (13),(14),(15),(21).		Good	In acid medium, Nb ions are precipitated above pH,4.8 Fe is also precipitated. Very stable Nb compounds.
Trialkyl phosphate (TBP). Ref. (21).		Poor	Fe and Nb are extracted. It is convenient to work at very low pH.
Dialkyl phosphate (HDEHP). Ref. (21).		Poor	Fe and Nb are extracted. It is convenient to work at very low pH.

TABLE 6. ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS. (CONTINUATION).

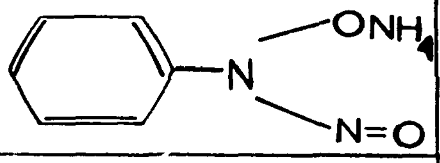
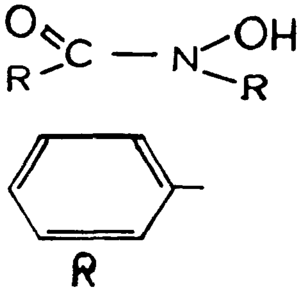
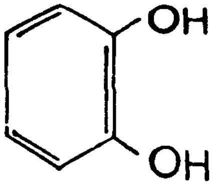
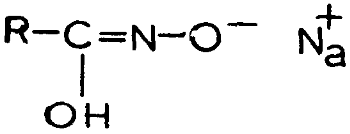
ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS			
NAME	STRUCTURE	SELECTIVITY	REMARKS
Cupferron (N-Nitrosophenyl hydroxylamine, ammonium salt). Ref. (13),(14),(15),(21).		Poor	Under strong acid conditions Fe(III),Nb,Sn,Ta,Ti,V and Zr precipitate.
N-Benzoyl-N-phenyl-hydroxylamine (N-phenylbenzohydroxamic acid, BPA, NBPHA, BPHA). Ref. (13),(14),(15),(21).		Good,in a very narrow pH range.	Nb (V) can be selectively separated from Al,Ca,Fe(III),Mg,Ta,Th,and U(VI) at pH 3.5 - 3.6, with tartarate, EDTA.
Catechol (Pyrocatechol 1,2-dihydroxybenzene). Ref. (13),(14),(15),(21).		Poor	It forms colored chelates with Fe(III), Mo(VI), Ti,V,Nb and Ta,but most of the reactions are of little practical importance.
Alkyl hydroxamates. Ref. (5),(10).	 $\mathrm{R{-}C(OH){=}N{-}O^{-}\ Na^{+}}$	Poor	Optimum flotation conditions at pH-7.

TABLE 6. ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS. (CONTINUATION).

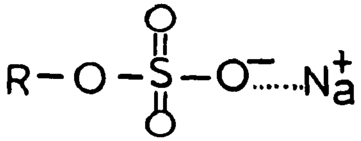
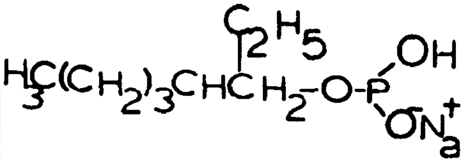
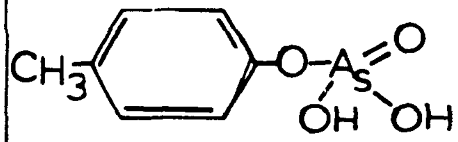
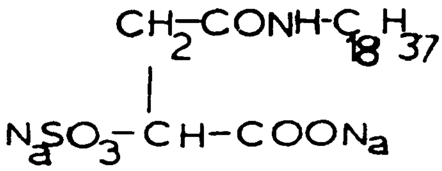
ORGANIC REAGENTS MORE SELECTIVE AS Nb FLOTATION COLLECTORS			
NAME	**STRUCTURE**	**SELECTIVITY**	**REMARKS**
Sodium alkyl sulphate. Ref. (4),(7).	 $R-O-\overset{O}{\underset{O}{S}}-O^{-}\cdots\cdots Na^{+}$	Good	Good selectivity in the pH range of 2.5-3.5.
Alkyl phosphonic acids; e.g. 2-ethylhexyl phosphonic acid. Ref. (18).		Poor	Strongly depress by Fe(III).
Alkyl (aryl) arsonic (or antimonic) acids, RAsO4H2 (RSbO4H2), such as p-tolylarsonic acid. Ref. (18).		Good	Precipitate very easy with Fe(III), Nb(V) and Ca(II).
Sulfosuccinamate: N-octadecyl sulfosuccinamate (Aerosol 18). Ref. (18).	 $\begin{array}{l} \quad\quad CH_2-CONH-C_{18}H_{37} \\ \quad\quad\ \ \vert \\ NaSO_3-CH-COONa \end{array}$	Good if iron can be complex.	Optimum pH for Ta at 2.4. Fe(III) can be complex with citric acid.

TABLE 7. ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE.

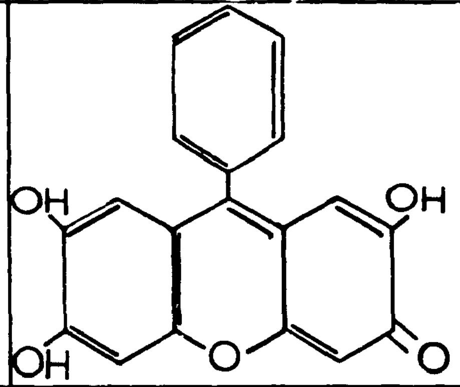
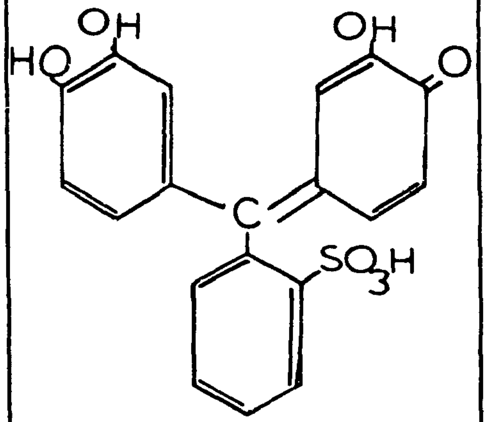
ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE			
NAME	STRUCTURE	SELECTIVITY	REMARKS
Phenylfluorone (2,3,7-Trihydroxy-9-phenyl-fluorone,2,6,7-thrihydroxy-9-phenylisoxanthene-3-one. Ref. (13),(14),(15),(21).		Poor	Selective for Nb(V) and Fe(III). Works better with 0.8% H3PO4.
Pyrocatechol violet (3,3',4-Trihydroxyfuchsone-2"-sulfonic acid). Ref. (13),(14),(15),(21).		Good	Selective for Nb(III) at pH 4-5 Selective for Al(III) at pH -6 Selective for Fe(III) at pH 5.7-6.4.

TABLE 7. ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE. (CONTINUATION).

ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE.

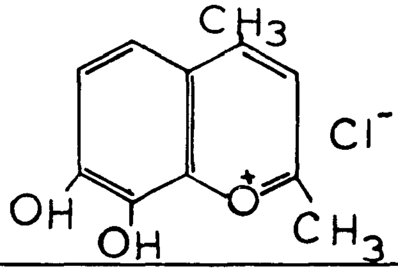
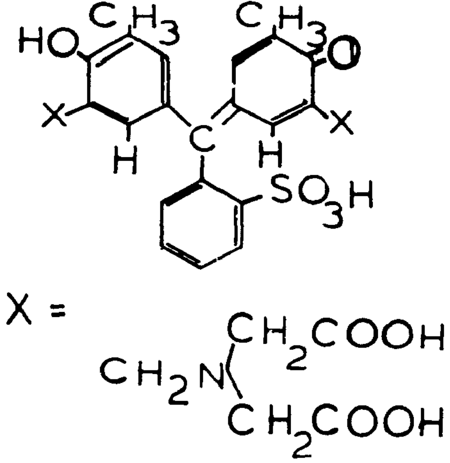
NAME	STRUCTURE	SELECTIVITY	REMARKS
o-Dihydroxy chromenols (7,8-dihydroxy-2,4 -dimethybenzopyrylium chloride). Ref. (13),(14),(15),(21).		Good	Selective depressant for Ge,Zr,Ti,Th,Mo,W,V,Ta,Nb,Sn.
Xylenol Orange Ref. (21)		Poor	Reacts with xylenol orange at pH 2-3.

TABLE 7 . ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE. (CONTINUATION).

ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE

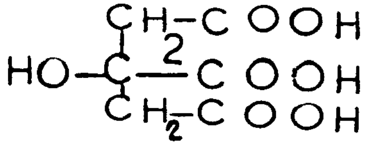
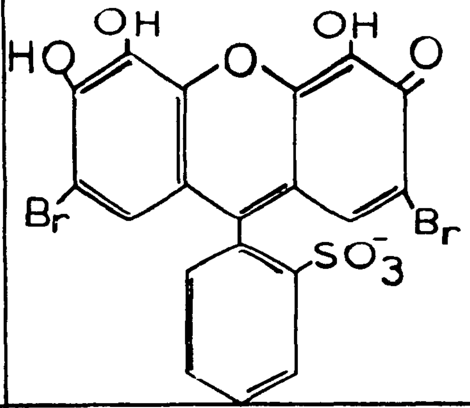
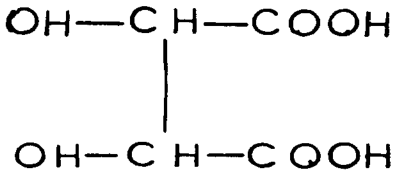
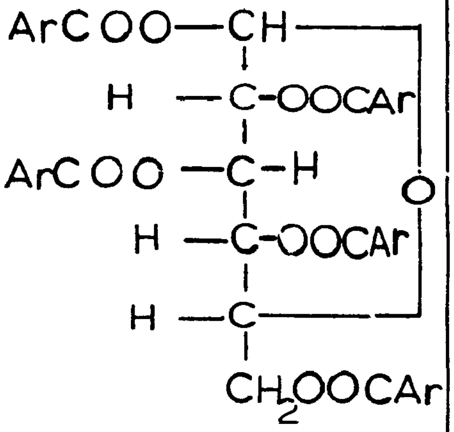
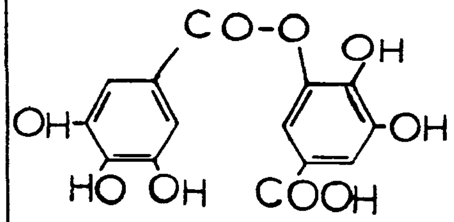
NAME	STRUCTURE	SELECTIVITY	REMARKS
Citric acid (2-hydroxypropane-1,2,3-tricarboxylic acid). Ref. (18),(22).	 $HO-\overset{CH_2-COOH}{\underset{CH_2-COOH}{C}}-COOH$	Good selectivity with iron ions.	Citric acid can complex Fe(II) and Fe(III) cations. It is used as a depressant, for quartz, mica, carbonates and iron oxide.
Bromopyrogallol Red (5,5'-Dibromopyrogallol sulfonepthalein, BPR). Ref. (13),(14),(15),(21).		Good	This reagent in combination with Di-n-octylmethylamine can complex Nb at pH 5.
Tartaric acid (1,2-Dihydroxyethane-1,2-dicarboxylic acid). Ref. (13),(14),(15),(21).	 $\begin{matrix}OH-CH-COOH\\ \vert\\ OH-CH-COOH\end{matrix}$	Poor	Tartaric acids forms the most stable complex with Nb.

TABLE 7 . ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE. (CONTINUATION).

ORGANIC REAGENTS MORE SELECTIVE AS Nb DEPRESSANTS IN THE INVERSE FLOTATION OF PYROCHLORE			
NAME	STRUCTURE	SELECTIVITY	REMARKS
Tannin (Rose Tannin). Ref. (13),(14),(15),(21).	 Ar, indicates the residue of the metadigallic acid, which has the following formula: 	Good	Ta,Ti(IV) and Nb are quantitatively precipitated by tannin from weakly acid oxalate solutions at pH 4.5. Al and Fe are not precipitated. Separation is even better in presence of complexone III

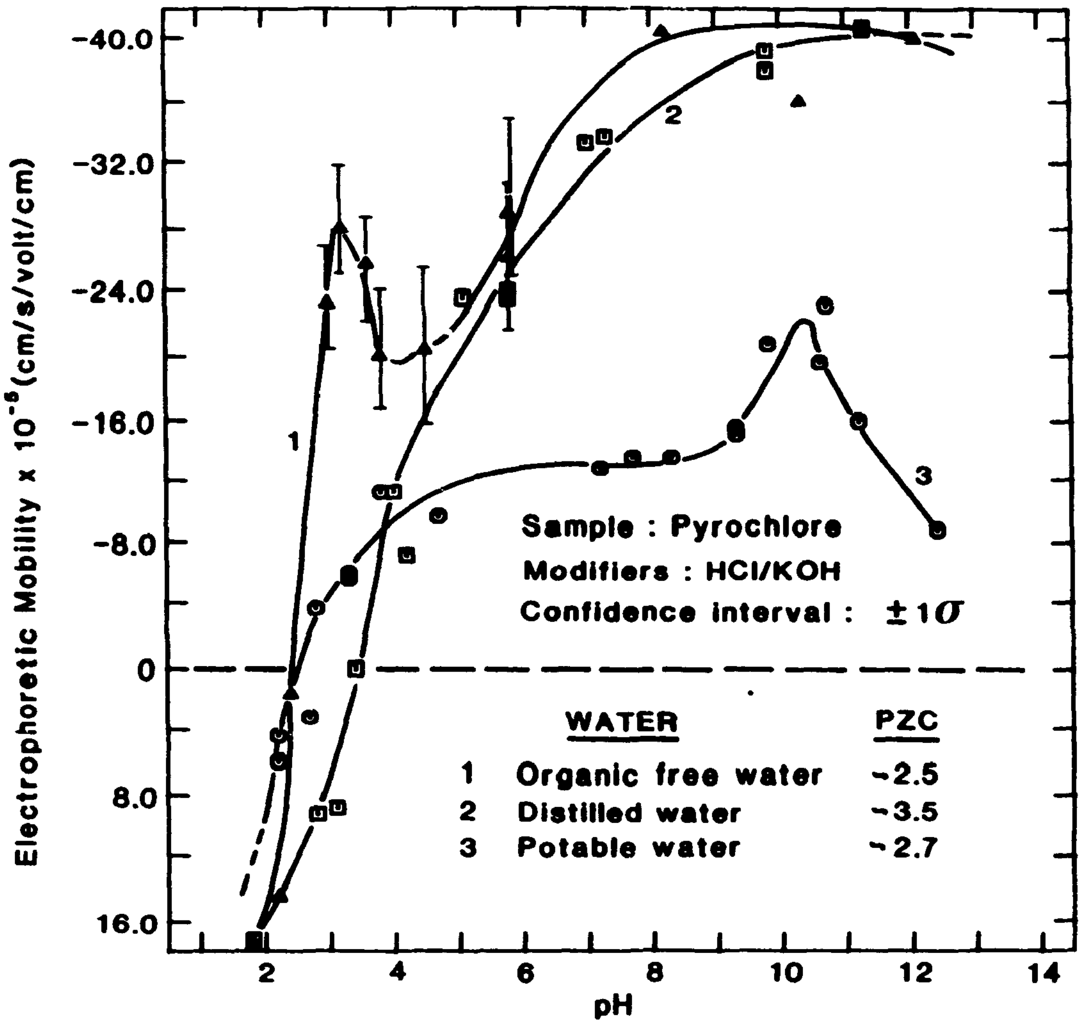


Figure 1. Electrophoretic mobility vs pH curves for pyrochlore in the presence of three types of water

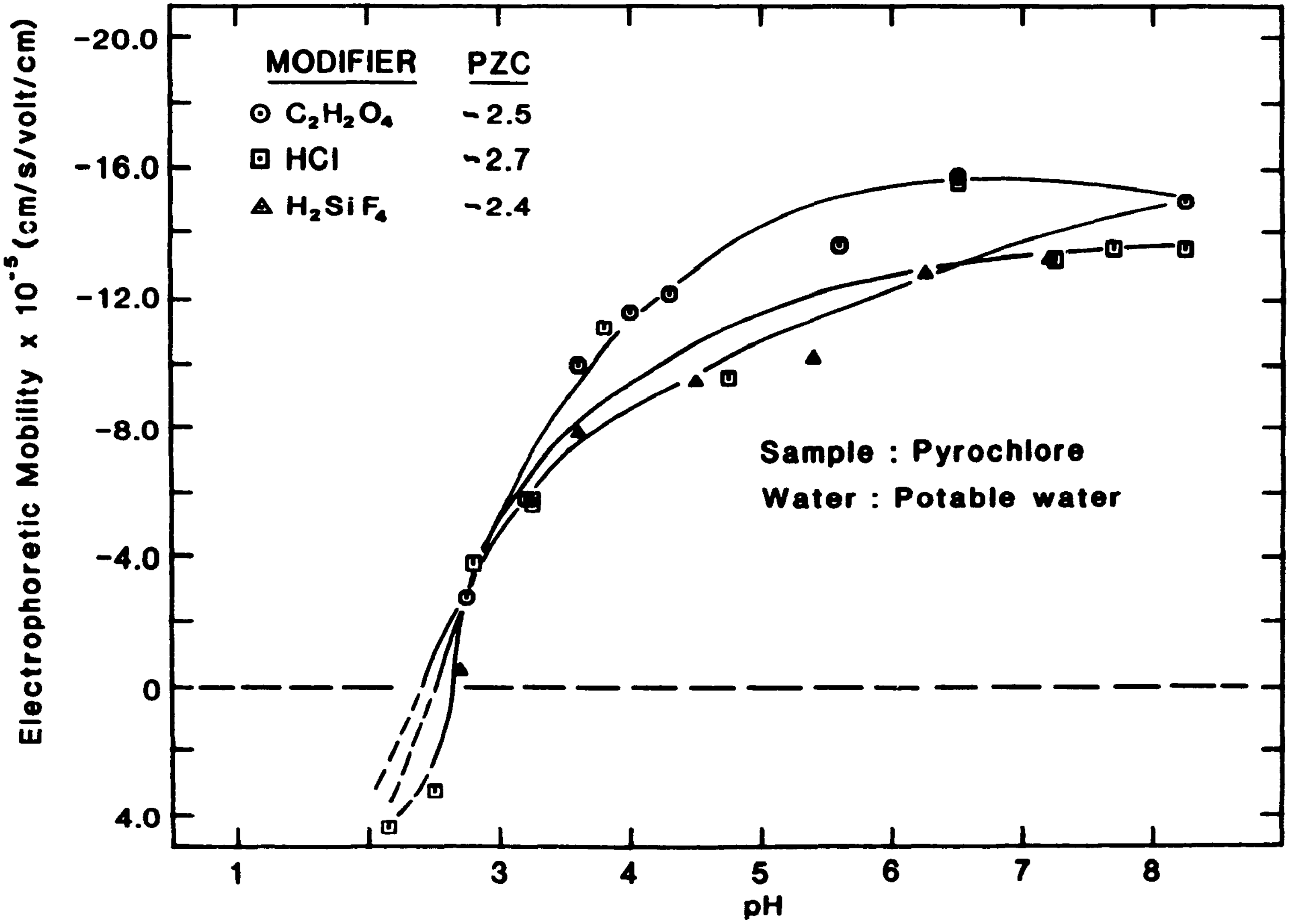


Figure 2. Electrophoretic mobility vs pH curves for pyrochlore with potable water and three pH modifiers

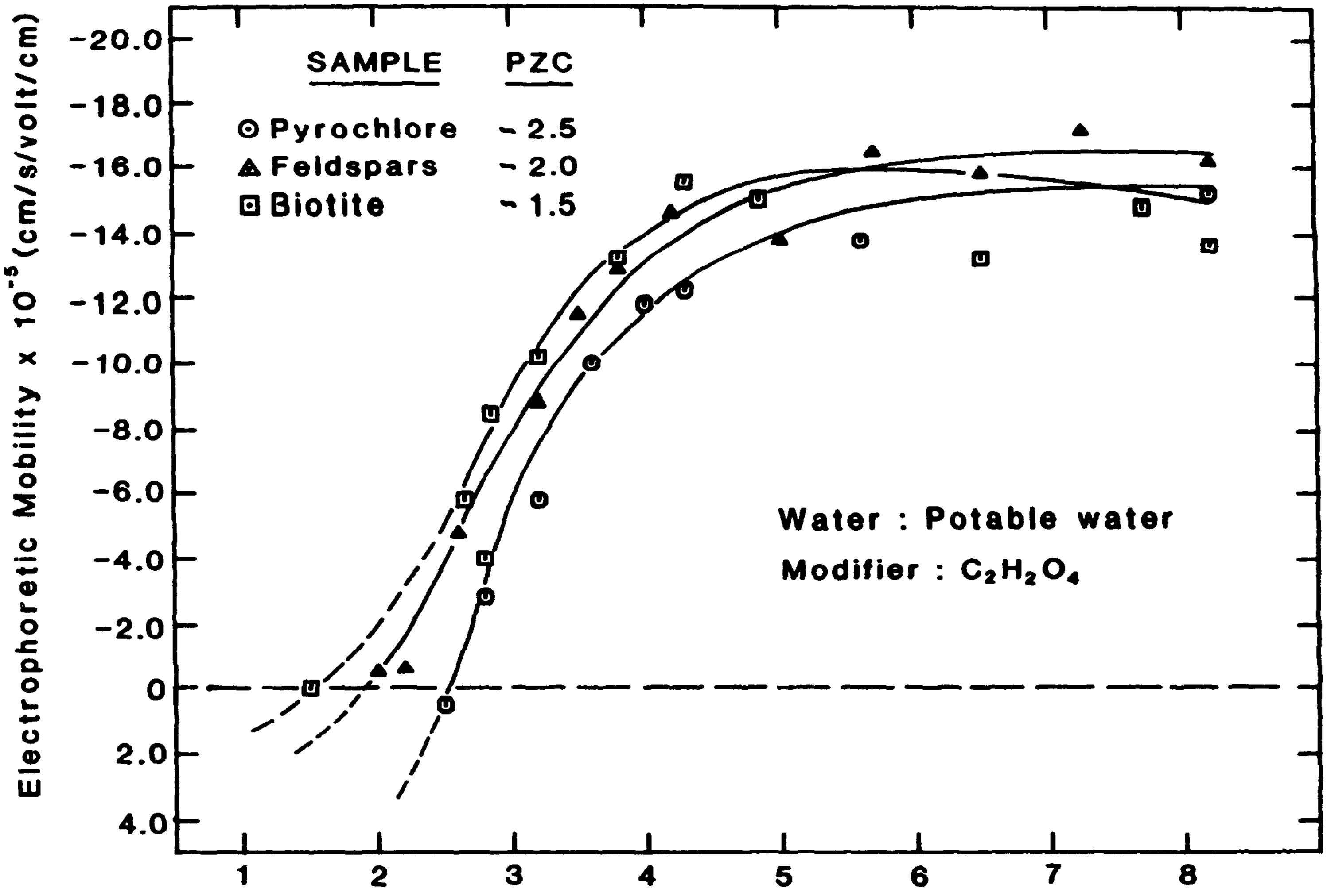


Figure 3. Electrophoretic mobility vs pH curves for pyrochlore feldspars and biotite using potable water and oxalic acid

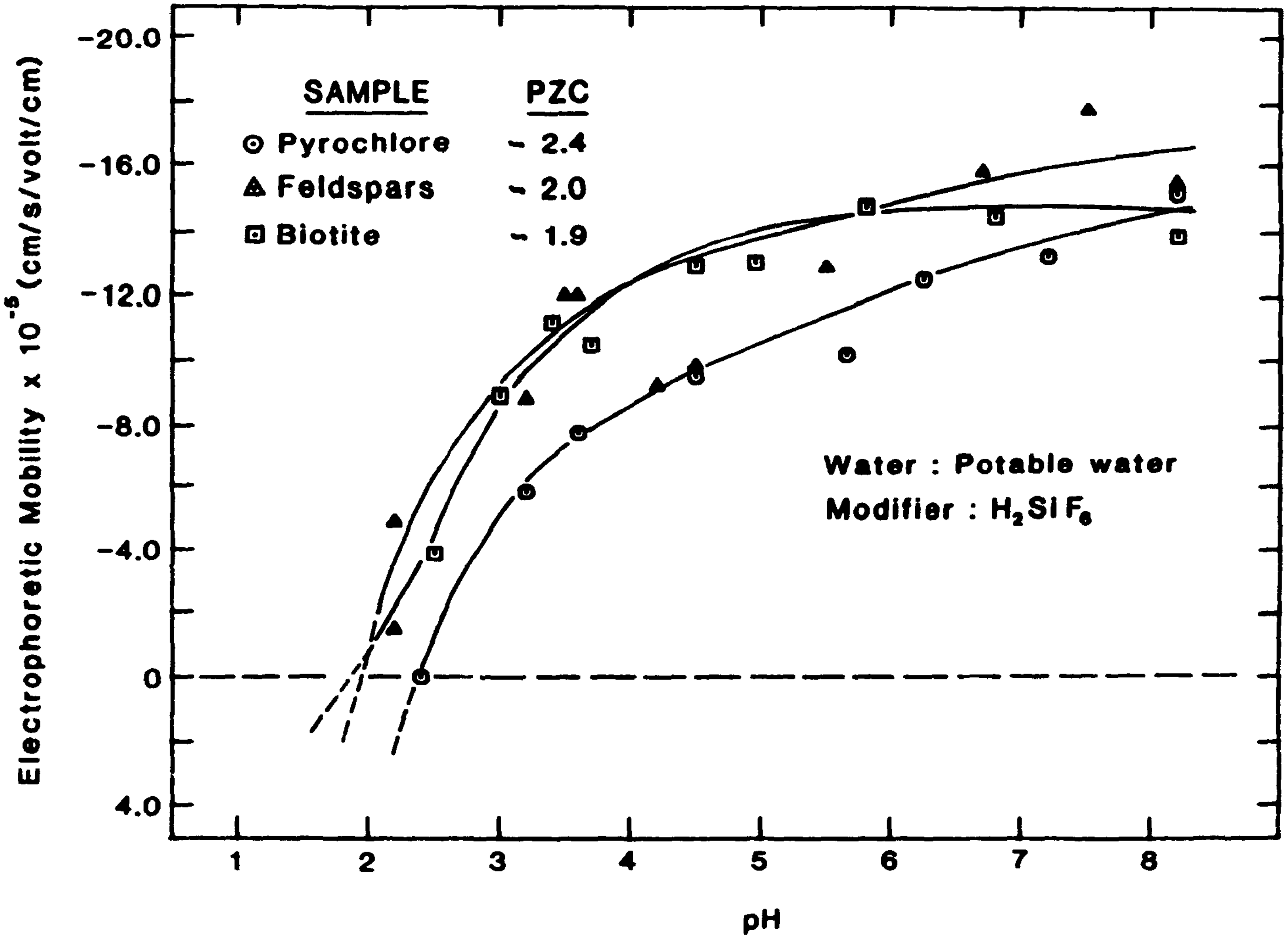


Figure 4. Electrophoretic mobility vs pH curves for pyrochlore feldspars and biotite using potable water and fluosilicic acid

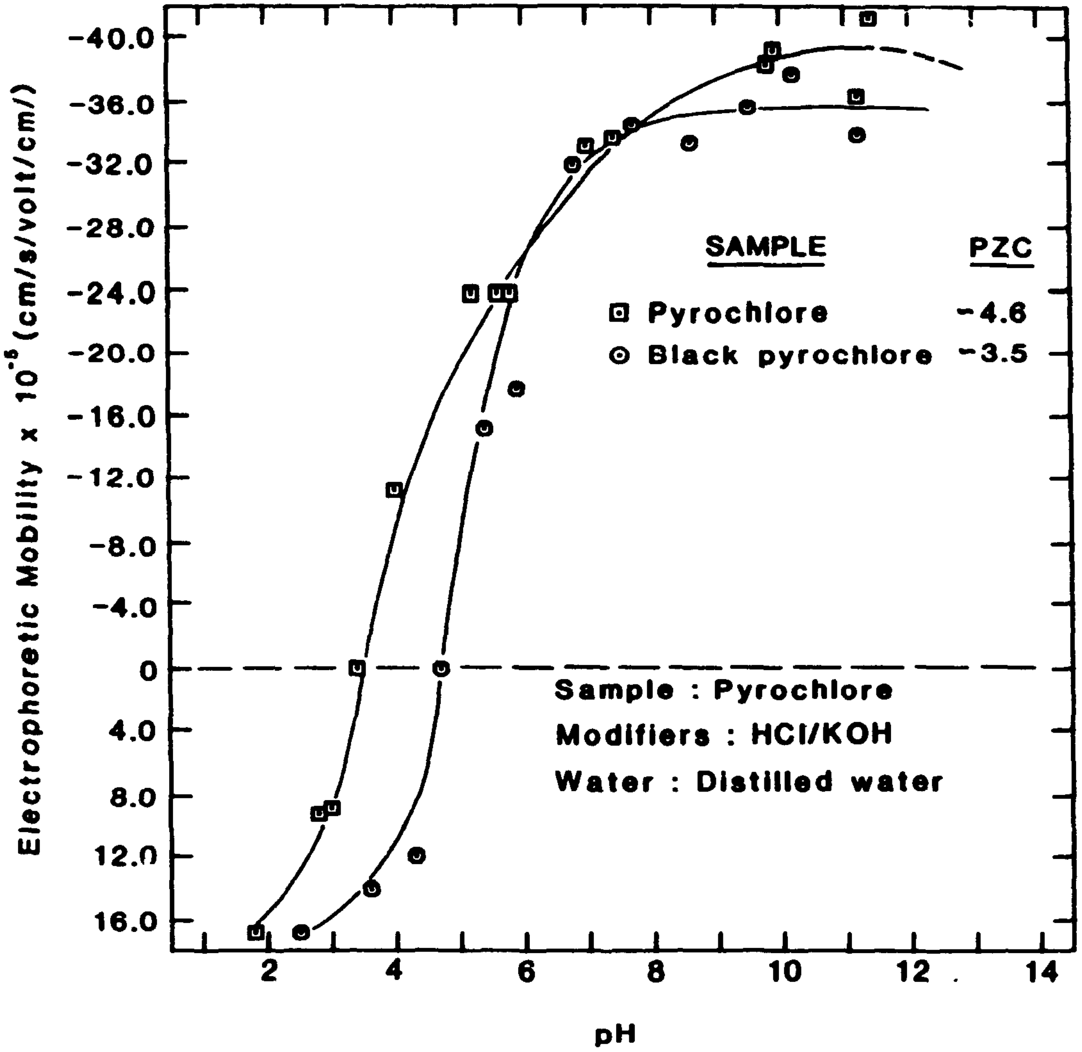


Figure 5. Electrophoretic mobility vs pH curves for two types of pyrochlore, using distilled water and two pH modifiers

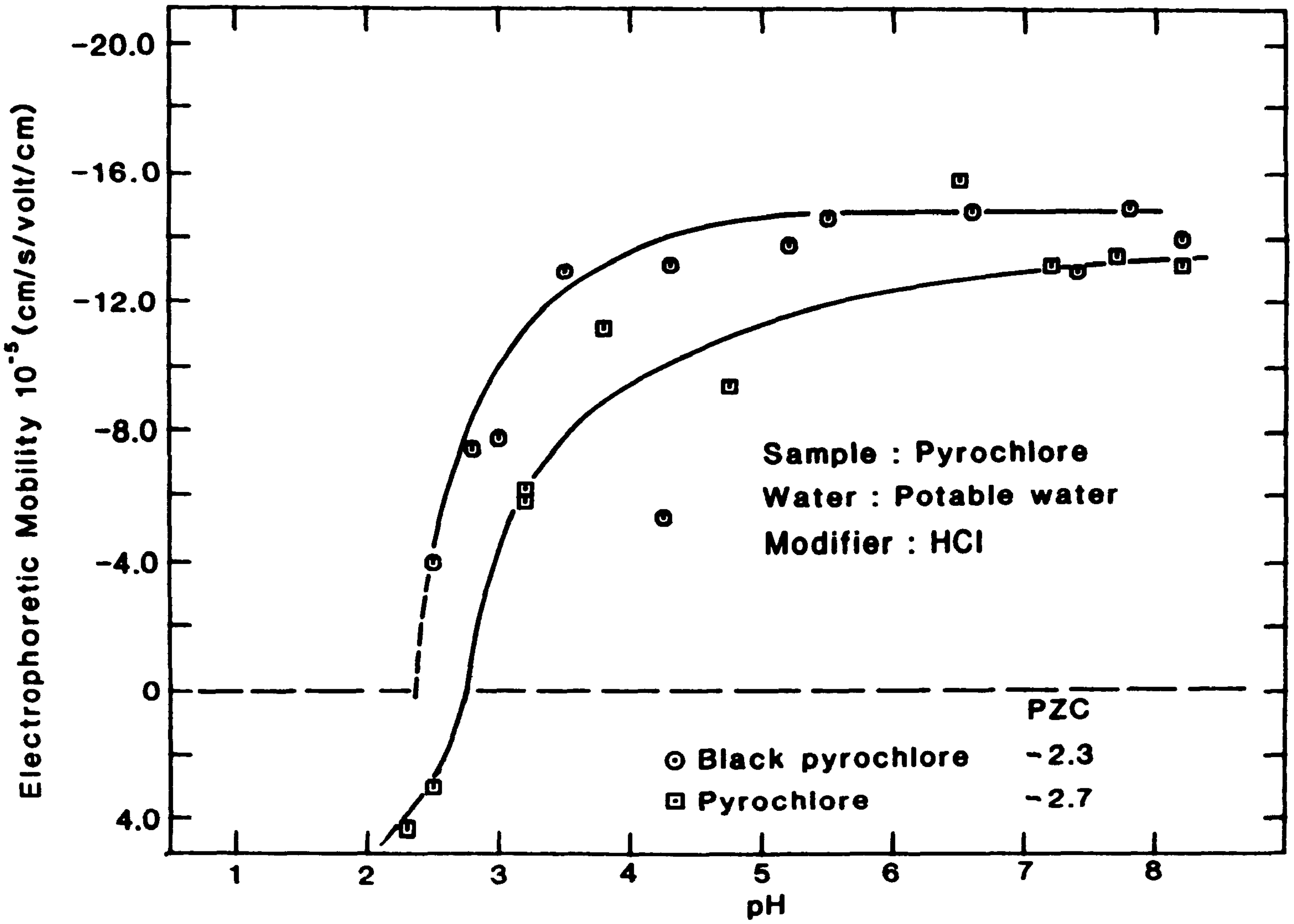


Figure 6. Electrophoretic mobility vs pH curves for two types of pyrochlore using potable water and HCl

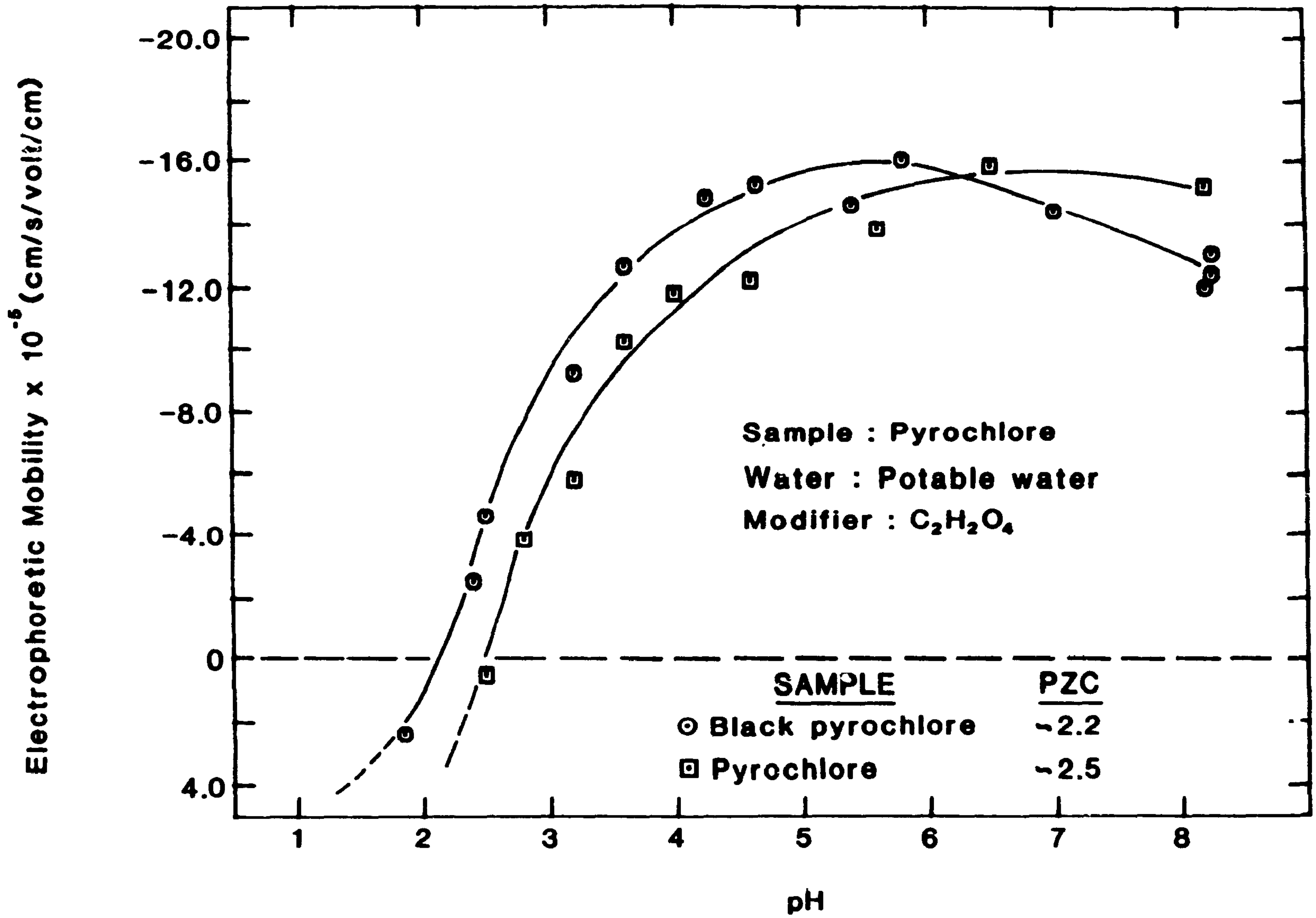


Figure 7. Electrophoretic mobility vs pH curves for two types of pyrochlore using potable water and oxalic acid

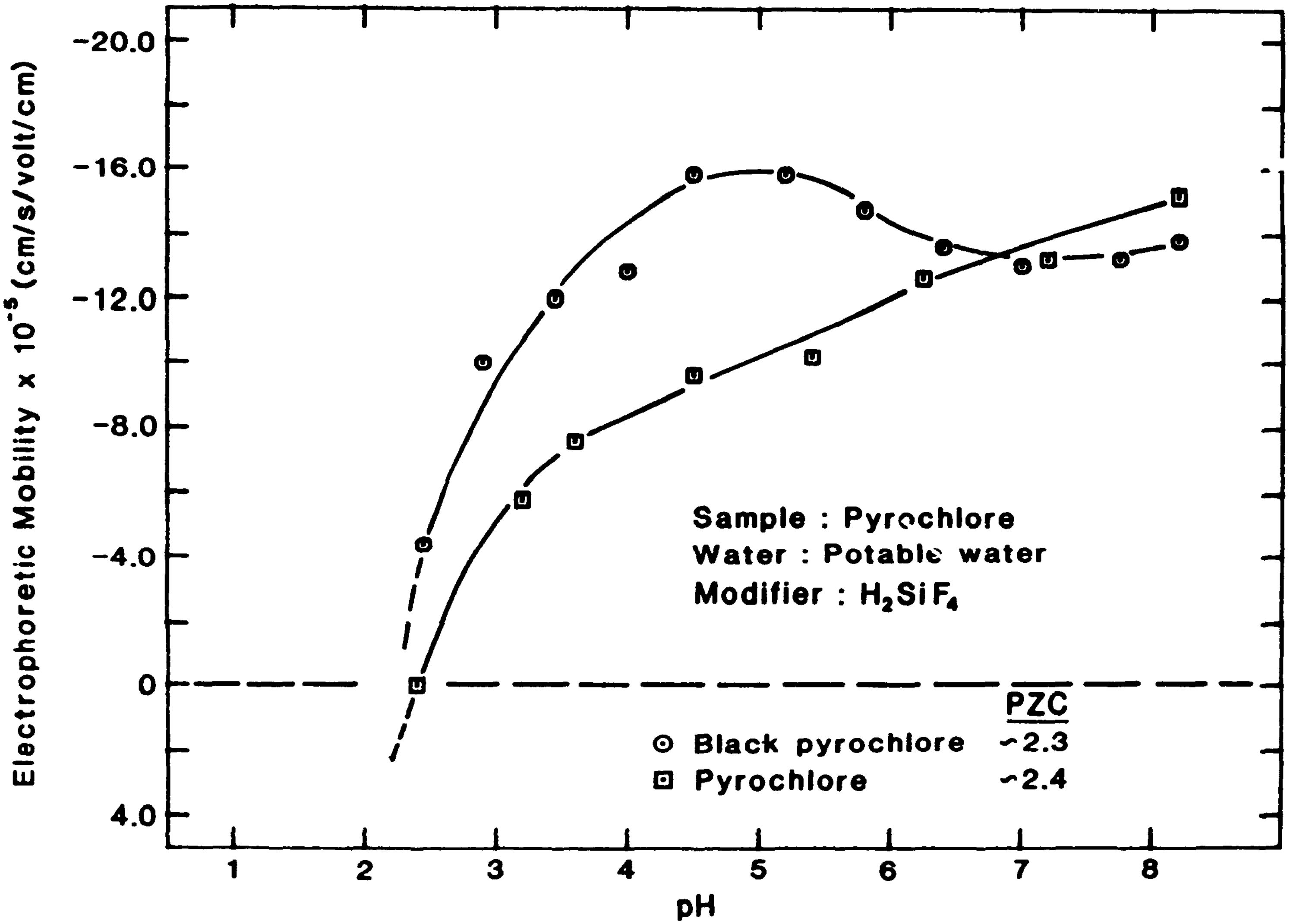


Figure 8. Electrophoretic mobility vs pH curves for two types of pyrochlore using potable water and fluosilicic acid

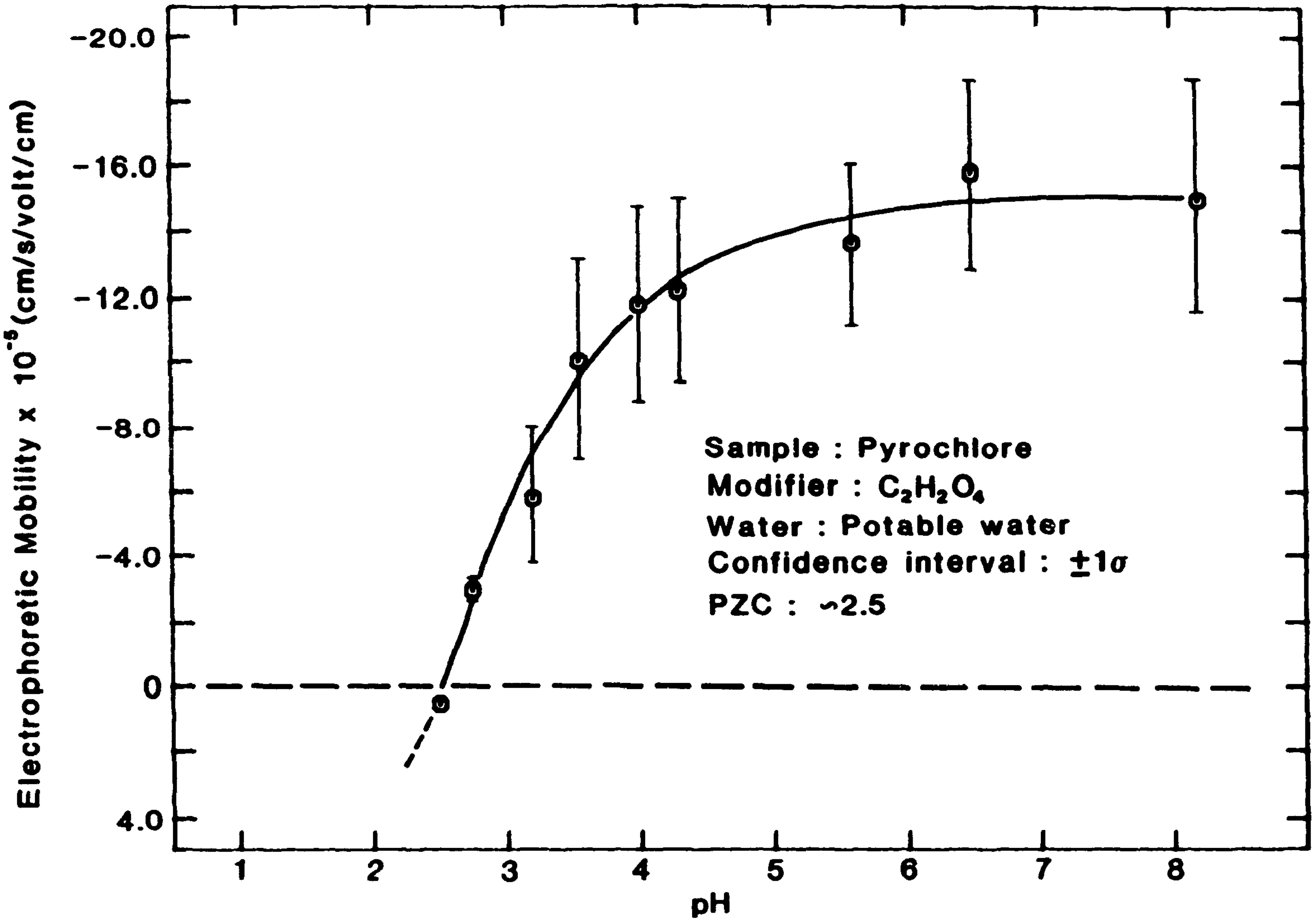


Figure 9. Confidence intervals in the electrophoretic mobility curves for pyrochlore

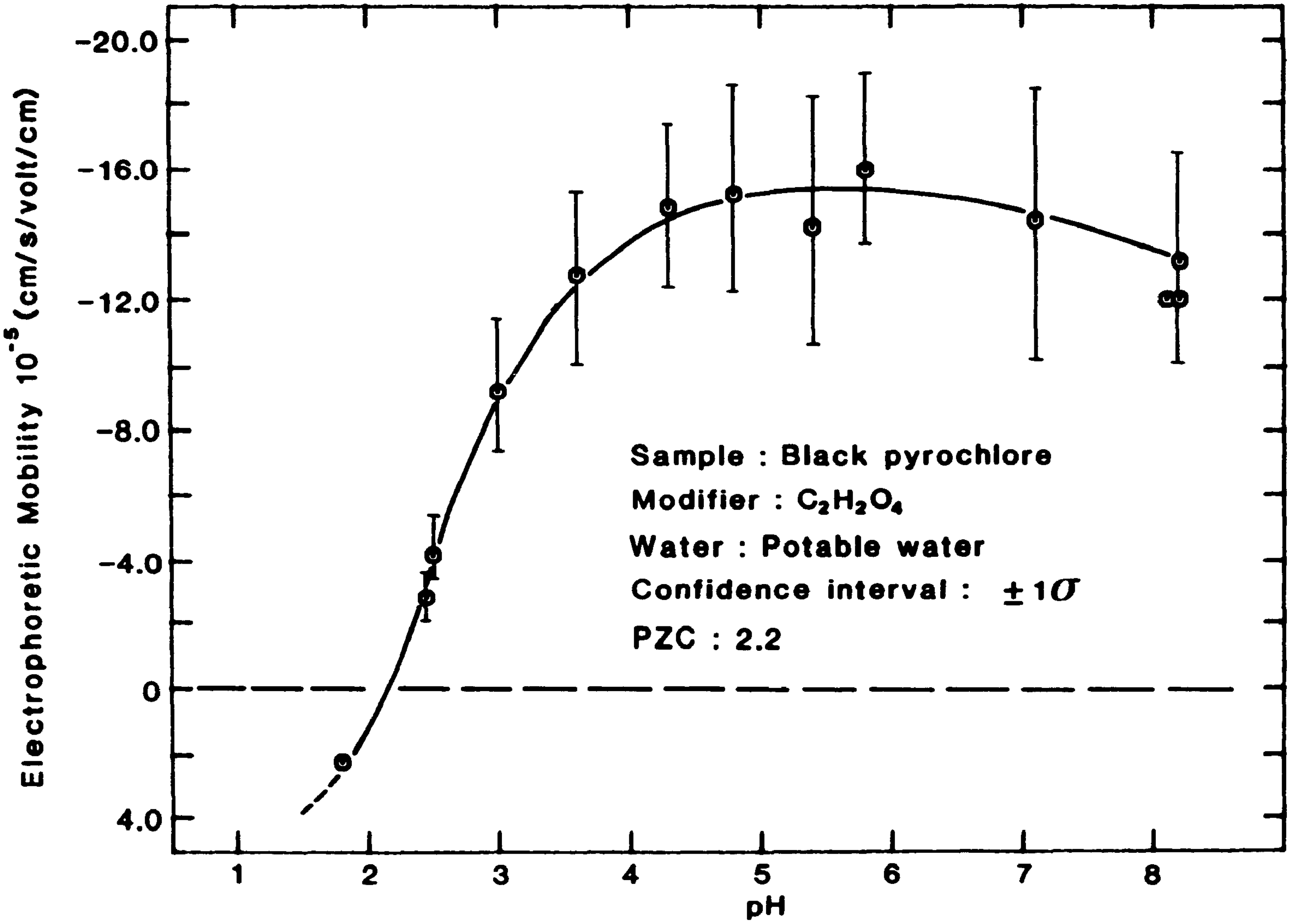


Figure 10 Confidence intervals in the electrophoretic mobility curves for pyrochlore

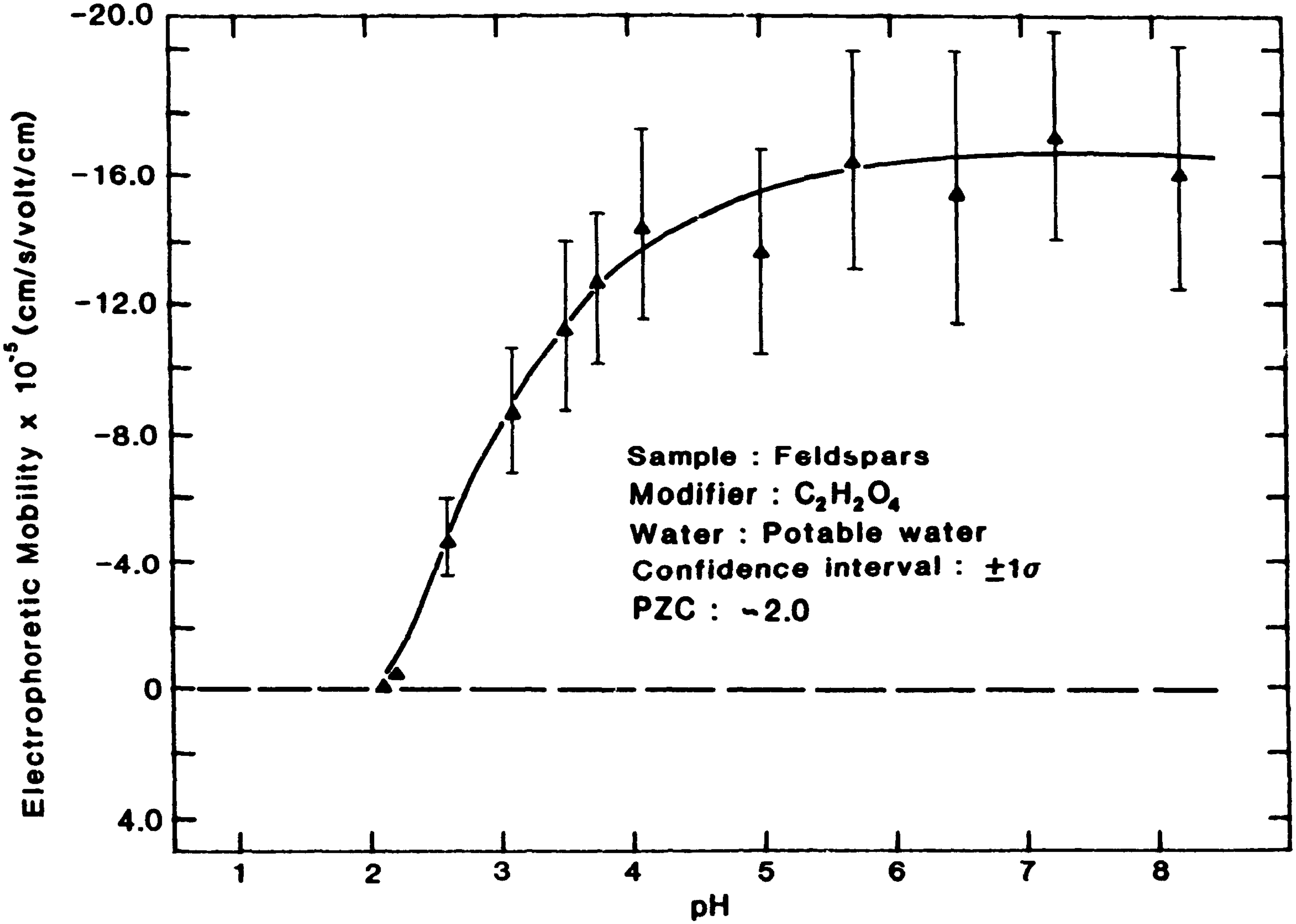


Figure 11 Confidence interval in the electrophoretic mobility curves, for feldspar

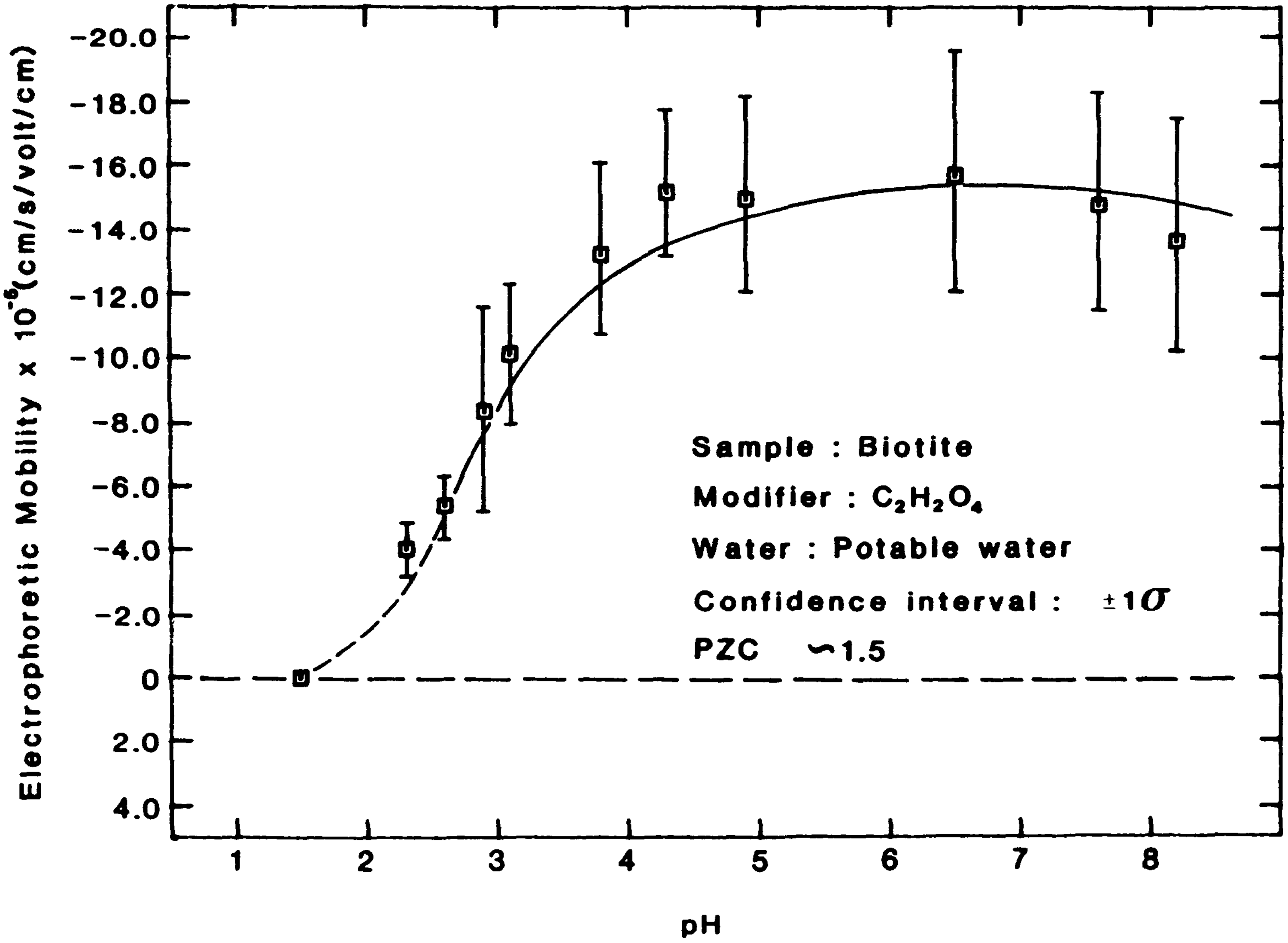


Figure 12 Confidence interval in the electrophoretic mobility curves, for biotite

PROGRESS REPORT-PYROCHLORE PROJECT

REPORT # 4

R. ESPINOSA GOMEZ

The present report summarizes the work done at McGill University on the pyrochlore project, for the period of August to December 1984. This report deals with two main points:

1) Electrokinetic study on pyrochlore, feldspars and biotite in the presence of fluosilicic acid (H_2SiF_6) using recycling plant water from Niobec.

2) Flotation results using some selective reagents.

1) ELECTROKINETIC STUDY

1.1 Results

Samples of pyrochlore, feldspars and biotite were carefully prepared to measure their electrophoretic mobility; details on the preparation were given in Report # 3. Recycle water from Niobec was used during these tests to compare with the results using potable water (report # 3). The recycle water simulates more closely the conditions at Niobec (1). The recycle water has the following cations in solution: Ca 174 ppm (vs. 23ppm for the potable water), 110 ppm Mg (vs. 1.3 ppm for the potable water) and Fe ` 0.5 ppm (same value for potable water). The concentration of other cations and anions has not been determined yet. The pH modifier used during these tests was H2SiF6.

Figures 1 to 4 summarize the results of the electrokinetic study. Figure 1 shows a comparison of electrophoretic mobility values of pyrochlore in the presence of recycle water and potable water (For more details on electrophoretic mobility values using potable water, see Report # 3). Several observations are made from this figure: a) the absolute elecrophoretic mobility values for potable water are in general higher than using recycle water, b) recycle water reverses the zeta potential from negative to positive between pH 4.2 to 3.2. Below pH 3.2 the zeta potential remains negative until a pH of about 1.9, c) recycle water produced precipitates at pH values greater than 8.6 and in the pH range 3 to 6.5. Note that the lowering in electrophoretic mobility and sign reversal corresponds to the pH range in which precipitates are formed.

Figures 2 and 3 show the mobility values for microcline (feldspar) and biotite respectively. In general, the recycle water caused similar effects for these minerals as for pyrochlore. The zeta potential for feldspar is reversed to positive from 4.1 to 3.2, and finally below pH 1.8 the zeta potential again returns to positive. For biotite the zeta potential is reversed to positive from 4.2 to 2.9 and finally below pH 2.0 the zeta potential again returns to positive.

Figure 4 summarizes the mobility values for pyrochlore, feldspar and biotite using recycle water. The following observations are made: a) for the three minerals the reversal in zeta potential occurs in the pH range 3 to 4.2, b) using recycle water the three minerals become electrokinetically similar, and, c) the lowering and reversal in the zeta potential is closely related to the formation of precipitates .

1.2 Discussion

The higher concentration of cations in the recycling water , Ca 174 ppm vs. 23 ppm for potable water and Mg: 110 ppm vs. 1.3 ppm for potable water, probably plays a major role in the lowering of the electromobility of the particles. The reversal in the zeta potential is probably also related to the high concentration of these cations. However more details concerning the contribution of each cation is required; a more complete analysis of the water composition is being performed.

The fact that recycle water causes the three minerals to become electrokinetically similar is detrimental to selective flotation. If the three minerals bear the same charge and about the same zeta potential a cationic collector has equal chance of being adsorbed on any of the three surfaces. However, this effect may be reduced using selective depressants for the silicates, or using very selective pyrochlore collectors, which exploit chemical rather than electrostatic-type interactions.

The reversal in the zeta potential from negative to positive, could be a source of pyrochlore losses at Niobec, if the collection mechanisms are purely electrostatic. Even if these are not the only one to produce flotation, a reduction of their contribution to flotation could also cause a decrease in recovery.

The presence of precipitates is another factor which directly affects the zeta potential. These precipitates may adsorb on the mineral surface converting the zeta potential to that of the precipitate substance. More studies on the origin and type of precipitates will be performed.

In general it may be said that the better the quality of the flotation water, the more readily can selective separation be achieved.

2) FLOTATION RESULTS AND DISCUSSION

A microflotation cell specially designed for fine particles was used, (Figure 5). The design is as the one originally proposed elsewhere (2) but with some modifications, especially in the location of the feed . Pure minerals samples were used during the tests e.g., pyrochlore (from Niobec), biotite, chlorite and microcline (the most abundant silicate mineral at Niobec). The particle size range of the minerals used in flotation is about -14 + 1 um, which is quite close to the size range to be used in the flotation of the slimes from Niobec. It is convenient to work with the real particle size distribution because in this way it is possible to ascertain the order of collector concentration required and also to become aware of the relative flotation rates of the particles. Before using the cell with the selected flotation reagents (for more information about the reagents selected, see Report # 3) the best cell operating conditions were determined, e.g., the optimum sample size (1 g), air flow rate (74 c.c./min.) and froth level (4 cm). In most of the tests, potable water from Niobec was used. Recycle plant water was also used in some tests. Test conditions were as follow: Preparation of the pulp (1 g of mineral and 40 ml of water), and pH adjustment with HCl. Addition of the reagent (to the same pH of flotation test) and pH adjustment again. Conditioning time 15 minutes, 5 minutes of dispersion in an ultrasound bath and then the pulp was transferred to the microflotation cell with the air flow already on. Level was adjusted with the test water (potable or recycle water) containing some frother. During the flotation (flotation time: 8 minutes) the froth level was continuously adjusted. Normally 5 concentrates were collected in each test, at 1, 2, 4 and 8 minutes of flotation; and non floats were also recovered. The results are reported as mass retained in the cell (%) vs. volume of liquid recovered to floats, instead of flotation time, as better correlation was observed on a volume basis than on a time basis.

At present 3 of the potential collector (Nb-reagents) have been tested; a) phenylarsonic acid, b) 8- hydroxyquinoline (oxine) and c) N-benzoyl- N phenyl - hydroxylamine (BPA). (See structures in Figure 6).

a) Phenylarsonic acid.

This reagent was tested under the suggested literature conditions (3) at several concentrations. The flotation response was poor. This behavior may be related to the hydrogen radical in the benzene ring. It may have been hydrolyzed at the pH tested, causing the complex to be hydrophilic.

b) 8- Hydroxyquinoline (oxine)

Since oxine is not soluble in water, it was dissolved with acetic acid and its pH was further adjusted with ammonium acetate. Figure 7 to 10 summarize the flotation results using oxine.

Figure 7 shows that recovery improves as the pH is increased from 4 to 6.5.

Figure 8, shows the performance of oxine at different concentrations, and using 0.1 % oil (volume basis on the pulp) to help fines flotation. These flotation tests were carried out at pH 6. Notice that at 0 collector addition there is already 23 % of flotation. This is due to physical entrainment in the microflotation cell. This entrainment is expected to be eliminated when using the flotation column. Figure 8, shows that using 20 ml of a solution 1 * 10(E-3) of oxine the pyrochlore recovery is over 90 %. Maximum recovery is limited to about 92 % because of material being retained in the cell due to its design.

Figure 9 shows the effect of oil addition on the flotation recovery. Oil was found to increase the recovery and to decrease the collector consumption. The oil is added as an emulsion and the minute droplets readily adsorb on the hydrophobic surfaces of the minerals. The oil-coated particles agglomerate increasing the flotation rate due to a particle size effect. Optimum oil addition was found to be 0.1 %.

Figure 10 shows the selectivity of pyrochlore relative to the silicates (biotite, chlorite and microcline) using potable water, pH 6.0, 0.1 % oil addition and 5 ml of 1 * 10(E-3) M solution of oxine. With 5 ml of this solution only about 60 % recovery is obtained. However this low collector addition was used as recovery is then sensitive to any disturbance in the system. Notice from Figure 10 that : a) pyrochlore is more selective to this reagent than the silicates; b) the flotation efficiency follows the order: pyrochlore ¨ microcline ¨ chlorite ¨ biotite.

Figure 11 shows the selectivity of pyrochlore relative to the silicates using recycle water (instead of potable water). The rest of the flotation conditions are exactly the same as those used in the tests using potable water. Note that recycle water produces a lower selectivity than potable water. Microcline is now floated as well as the pyrochlore. However, both chlorite and biotite are not floated.

Oxine precipitates Nb and Al at pH 6, (4). Consequently it is possible that oxine induces flotation of the pyrochlore and microcline due to chemisorption at their Nb and Al sites respectively. Ca and Mg the main cations of the recycle water, (Mg is also present in chlorite and biotite) do not precipitate with oxine to any extent below pH 9 (4). Thus, in some way recycle water is promoting futher oxine-Al interactions. At present no clear explanation can be given. However it is possible that the reduction of negative zeta potential (reduction in the electrophoretic mobility) of the microcline caused by the recycle water promotes adsorption of the anionic oxine.

To increase pyrochlore selectivity it is necessary to add silicate depressants, especially when working with recycle water. So far depressants have not been studied but it is planned to test several, among others citric acid, ethylenediaminetetraacetic acid (EDTA) and sodium silicate. Another way to increase selectivity is working with a better quality of water (e.g. potable water). That may be another solution to be considered.

c) N-Benzoyl- N- Phenyl - Hydroxylamine (BPA).

Since BPA is not soluble in water it was dissolved in ethyl alcohol and further diluted with water.

Figures 12 to 16 summarize the results obtained with the BPA (so far only potable water has been used). Figure 12 shows the flotation response of pyrochlore at a pH of 3.55 and the effect of oil addition on the flotation recovery. The following points are noted: a) it is possible to float pyrochlore using BPA; b) recoveries as high as 90 % were obtained using 10 ml of a 1 * 10(E-2) M BPA solution, and, c) a 0.1 % oil addition caused a significant increase in pyrochlore recovery; consequently the BPA consumption can be reduced considerably.

Figure 13 shows the effect of pH on pyrochlore recovery using 5 ml of a 1* 10(E-2) M solution of BPA, 0.1 % oil and 150 ppm of depressant (EDTA). Notice that from pH 3 to 6.5 the same recoveries are attained. This behavior agrees with the precipitation range in which BPA precipitates Nb ions (5). Thus a chemisorption mechanism must be responsible for the pyrochlore flotation.

Figure 14 shows the effects of EDTA depressant concentration,(150 and 730 ppm), on pyrochlore and microcline flotation with BPA. Notice that, a) 150 ppm does not materially affect pyrochlore flotation; b) pyrochlore flotation ceases at depressant additions of 730 ppm, and, c) microcline flotation is equally suppressed at 150 ppm and 730 ppm.

EDTA was used because it is a very effective Al complexing agent, and therefore a potential microcline depressant (6); this appears to be verified. However if added in excess it can also complex the Nb ions, causing pyrochlore depression.

Figure 15 shows the selectivity of BPA against biotite, chlorite and microcline, at pH 3 and using 150 ppm of EDTA. Notice that: a) selectivity of pyrochlore is good compared with biotite and microcline, and b) there is no selectivity between chlorite and pyrochlore. Figure 16 shows the same tests as Figure 15, for a pH of 4.55. In general the same behavior is observed at both pH values. The only difference is that biotite and microcline are slightly more depressed at a pH of 4.55. However, the use of a selective depressant is still required to help selectivity.

More reagents and depressants will be used in the following tests. A special emphasis will be given to the selection of good silicate depressants, and the testing of commercially available pyrochlore flotation reagents, e.g. sulfosuccinates (PROCOL CA 540), and, phosphonic acids (FLOTINOR P-195, FLOTINOR P-184). The best conditions will be tested by column flotation.

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(1) Biss R., Private communication.

(2) Siwek, B., Zembala, M. and Pomianowski, A. 'A method for determination of fine-particle flotability'. Int. Journal of Min. Proc. 8 (1981) 85-88.

(3) Cheng, K.L., Keihei Veno and Toshiaki Imamura. ' Handbook of organic analytical reagents'. CRC Press, Inc., pp. 445-448, 1982.

(4) Same as (2) pp 256-257.

(5) Majumdar A.K. and Bijoli K.P., 'Separation of niobium and tantalum by N-benzoyl -N-phenylhydroxylamine'. Anal. Chim. Acta, 24 (1961) 497-498.

(6) Same as (2) p. 218.

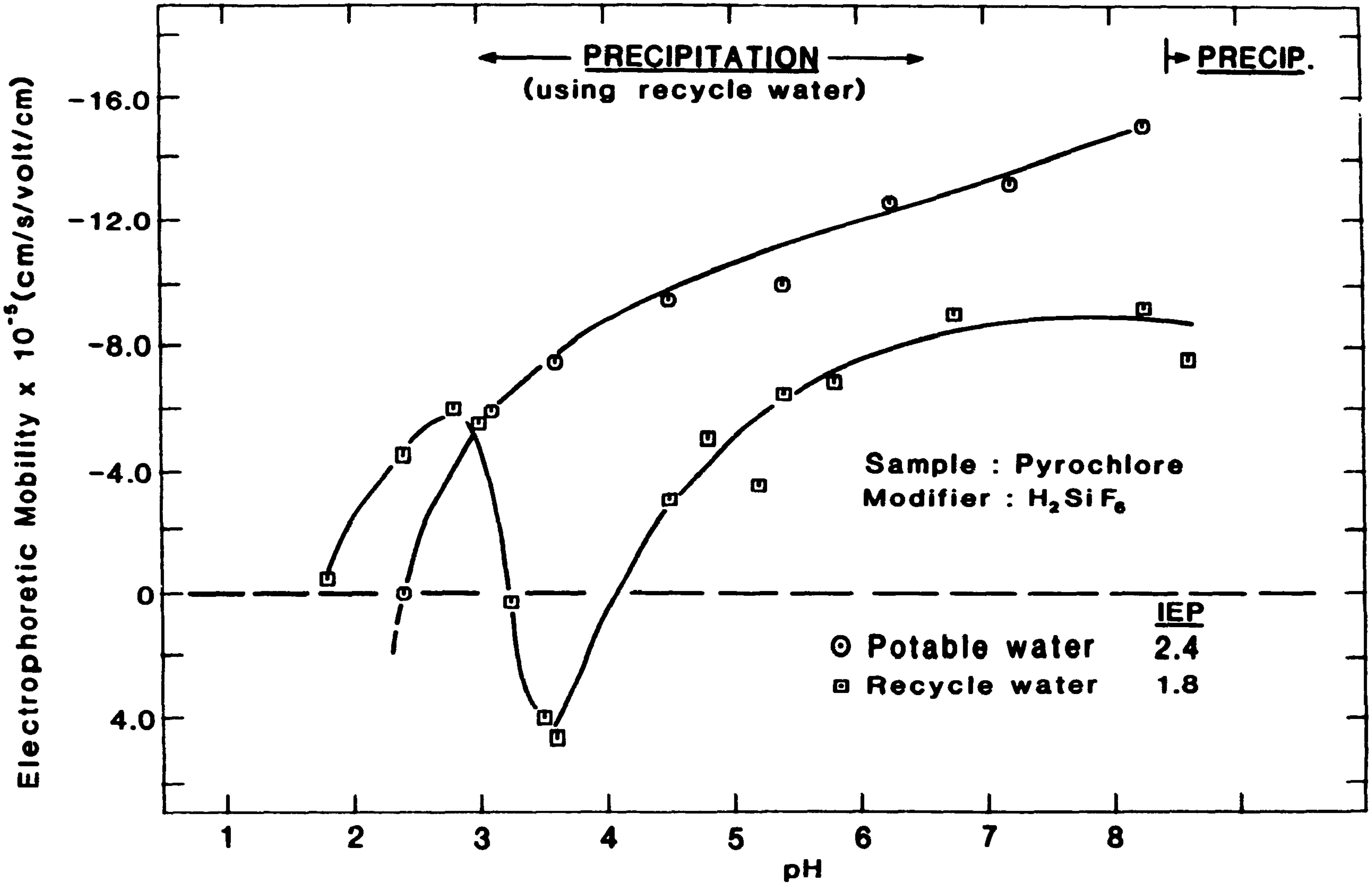


Figure 1. Electrophoretic mobility curves for pyrochlore using recycle water

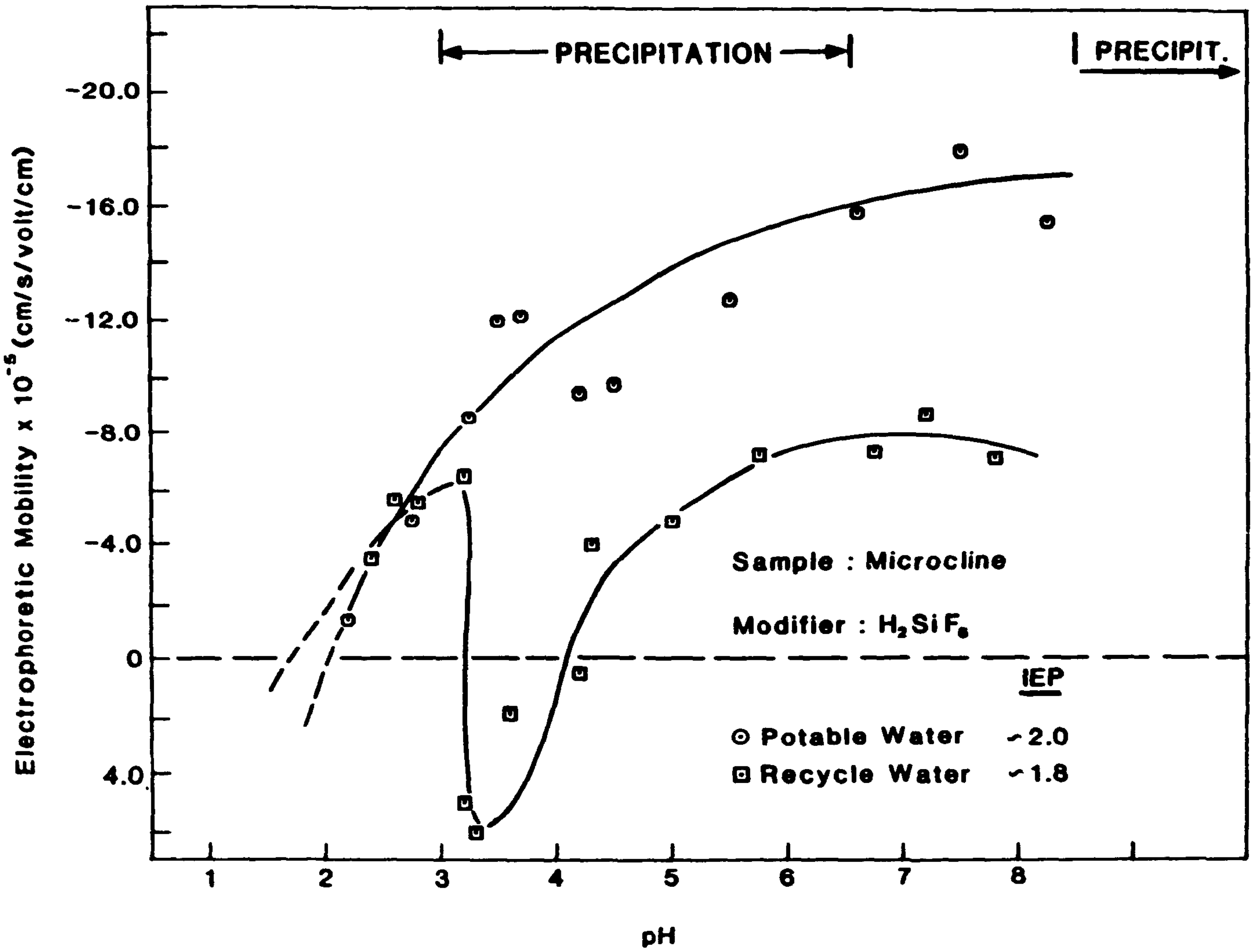


Figure 2. Electrophoretic mobility curves for microcline using recycle water

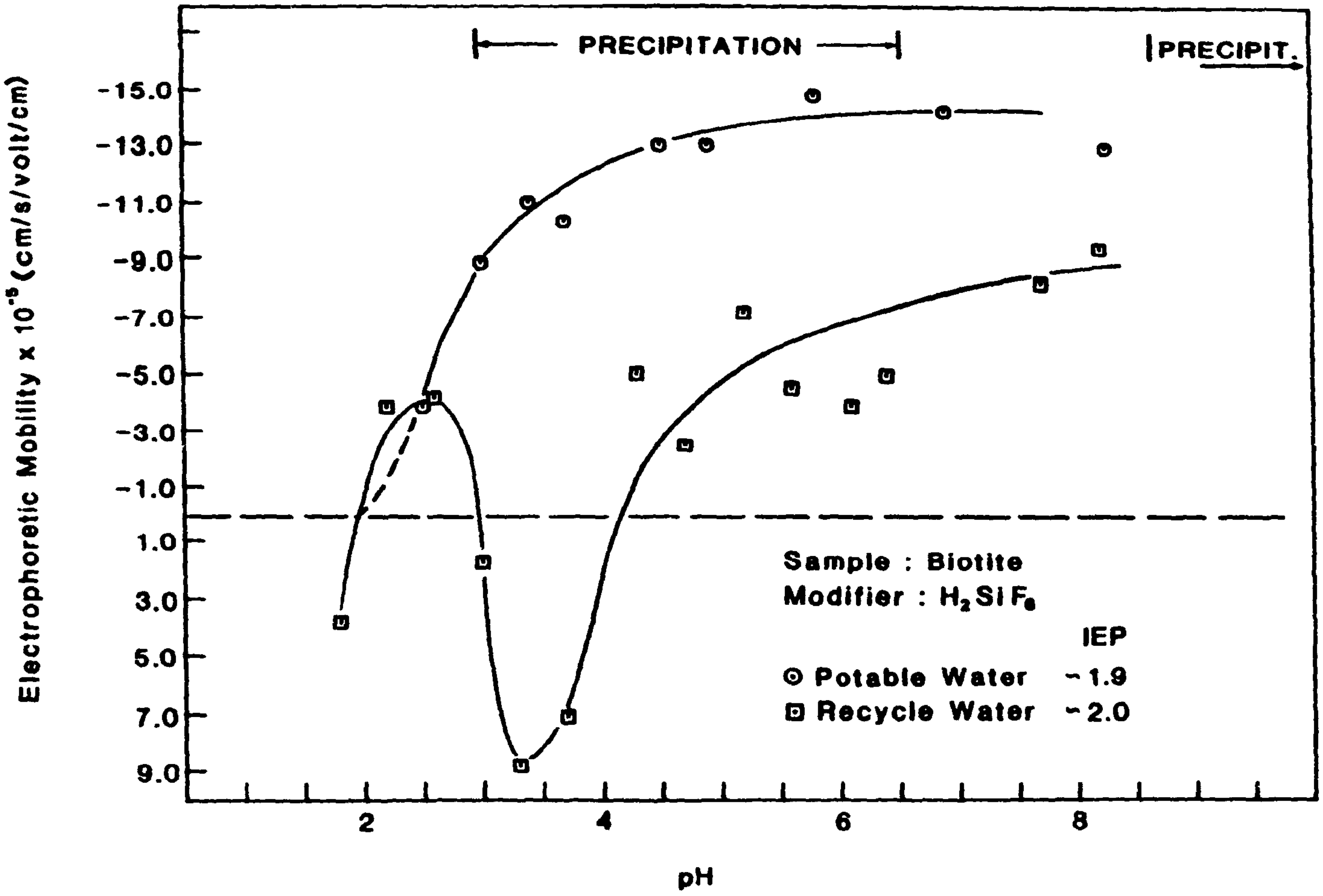


Figure 3. Electrophoretic mobility curves for biotite using recycle water

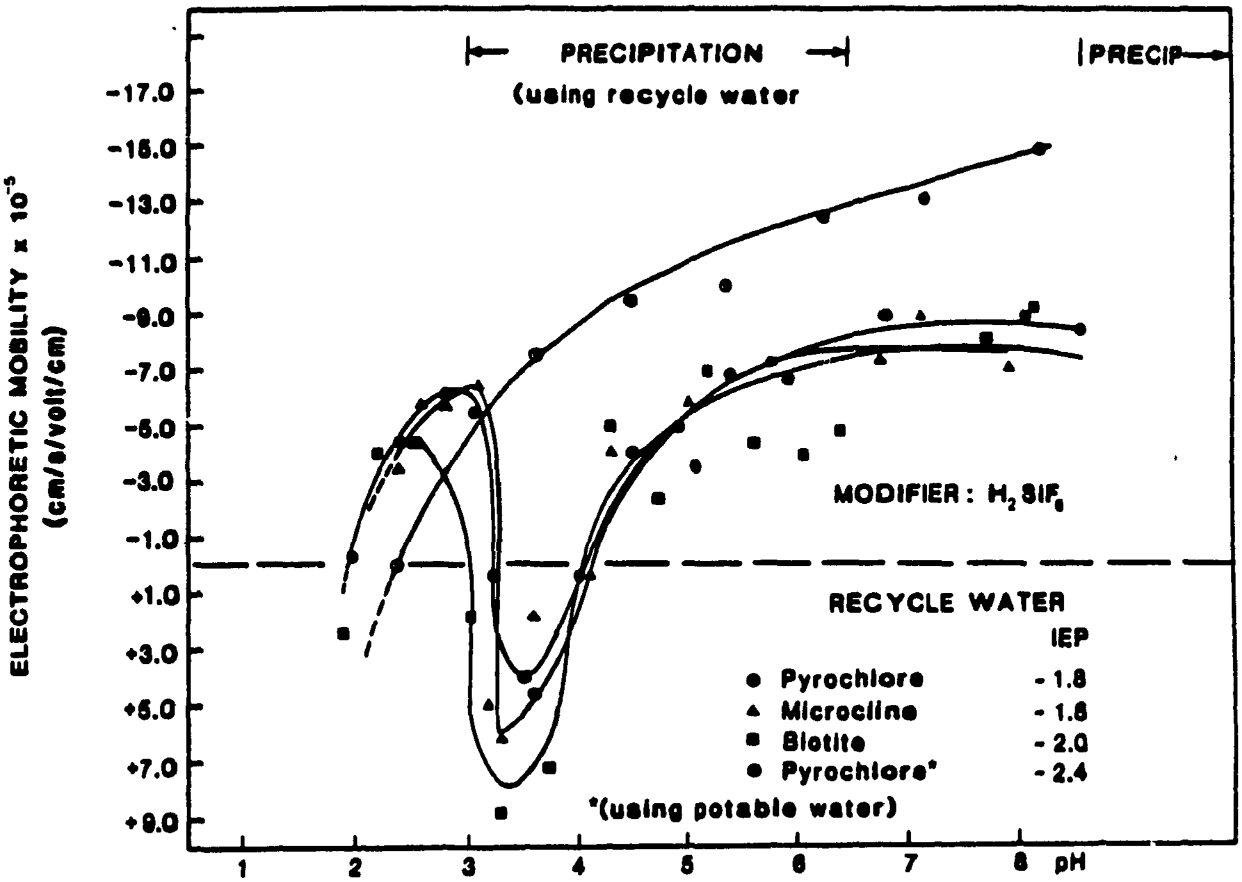


Figure 4. Electrophoretic mobility using recycle water.

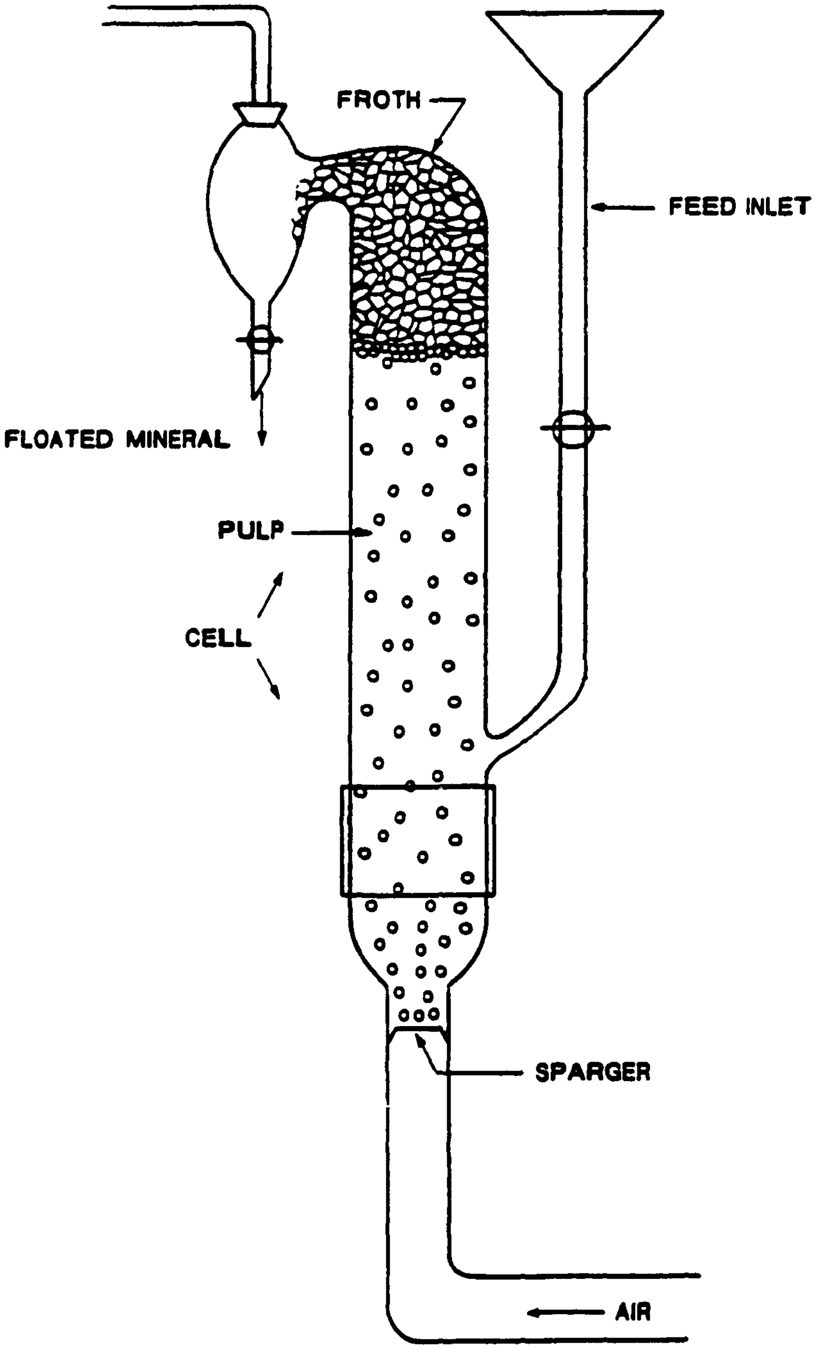
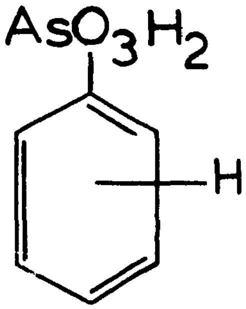
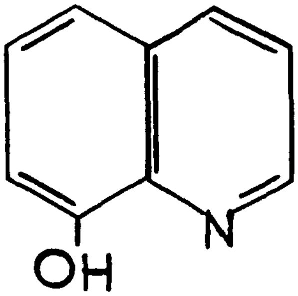


Figure 5. Microflotation cell

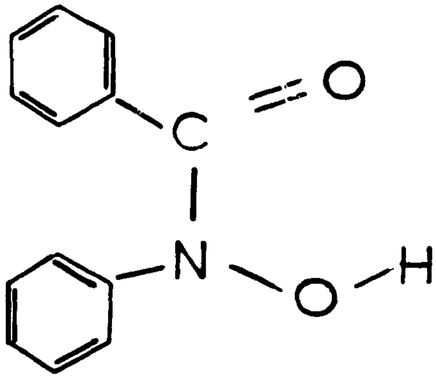
FIGURE 6. STRUCTURAL FORMULAS OF THE REAGENTS.



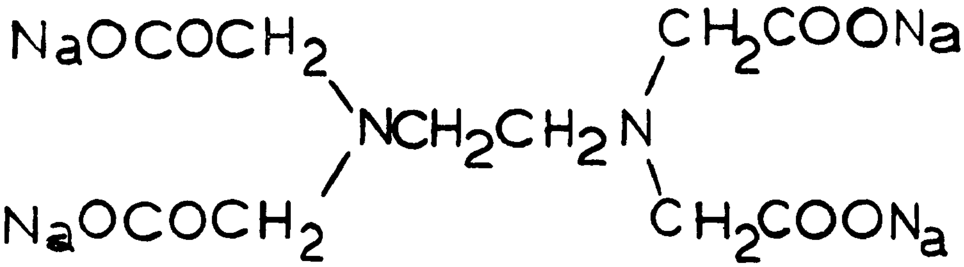
a) PHENYLARSONIC ACID



b) 8-HYDROXYQUINOLINE (OXINE)



c) N-BENZOYL-N-PHENYL-HYDROXYLAMINE (BPA)



d) ETHYLENEDIAMINE-TETRAACETIC ACID (EDTA)

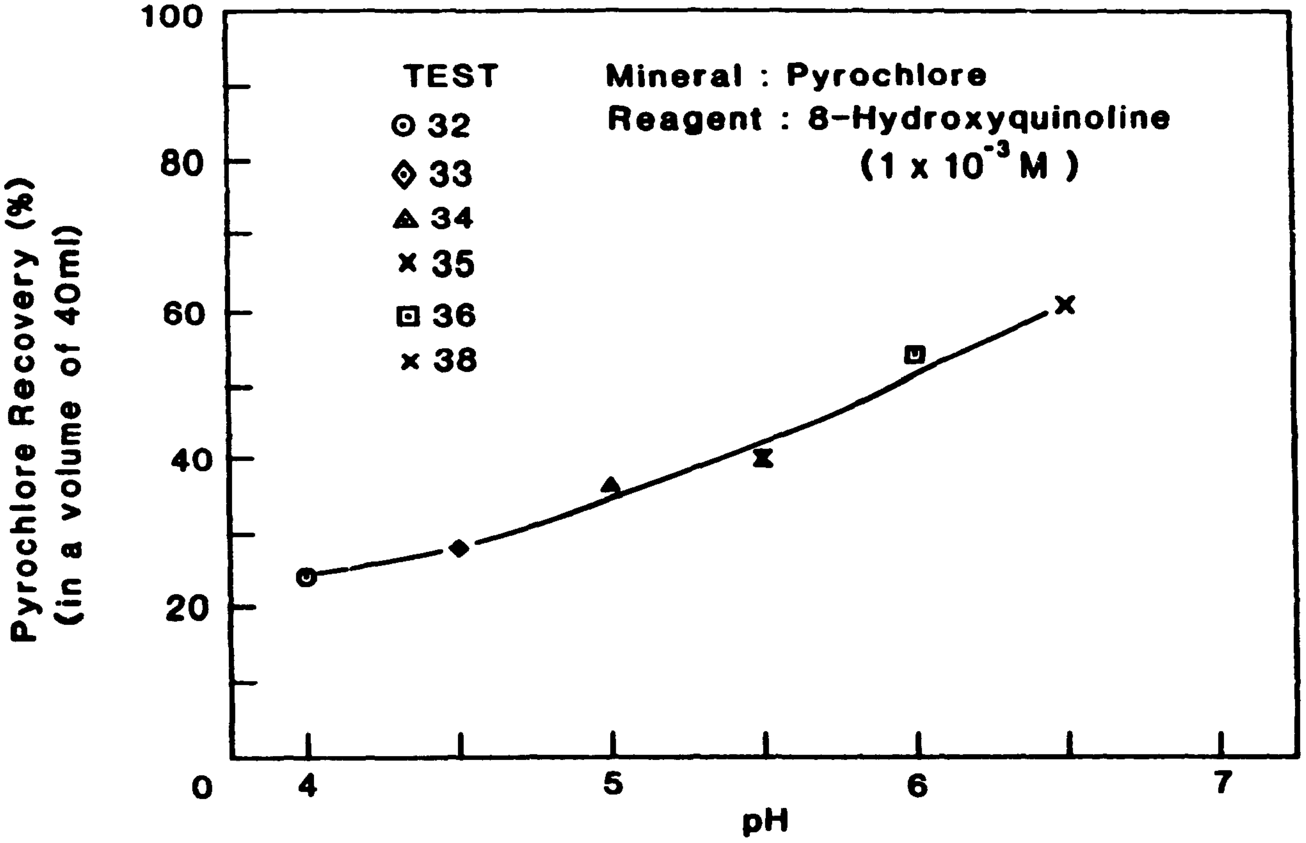


Figure 7. Pyrochlore recovery vs 8-Hydroxyquinoline at 10-3 M

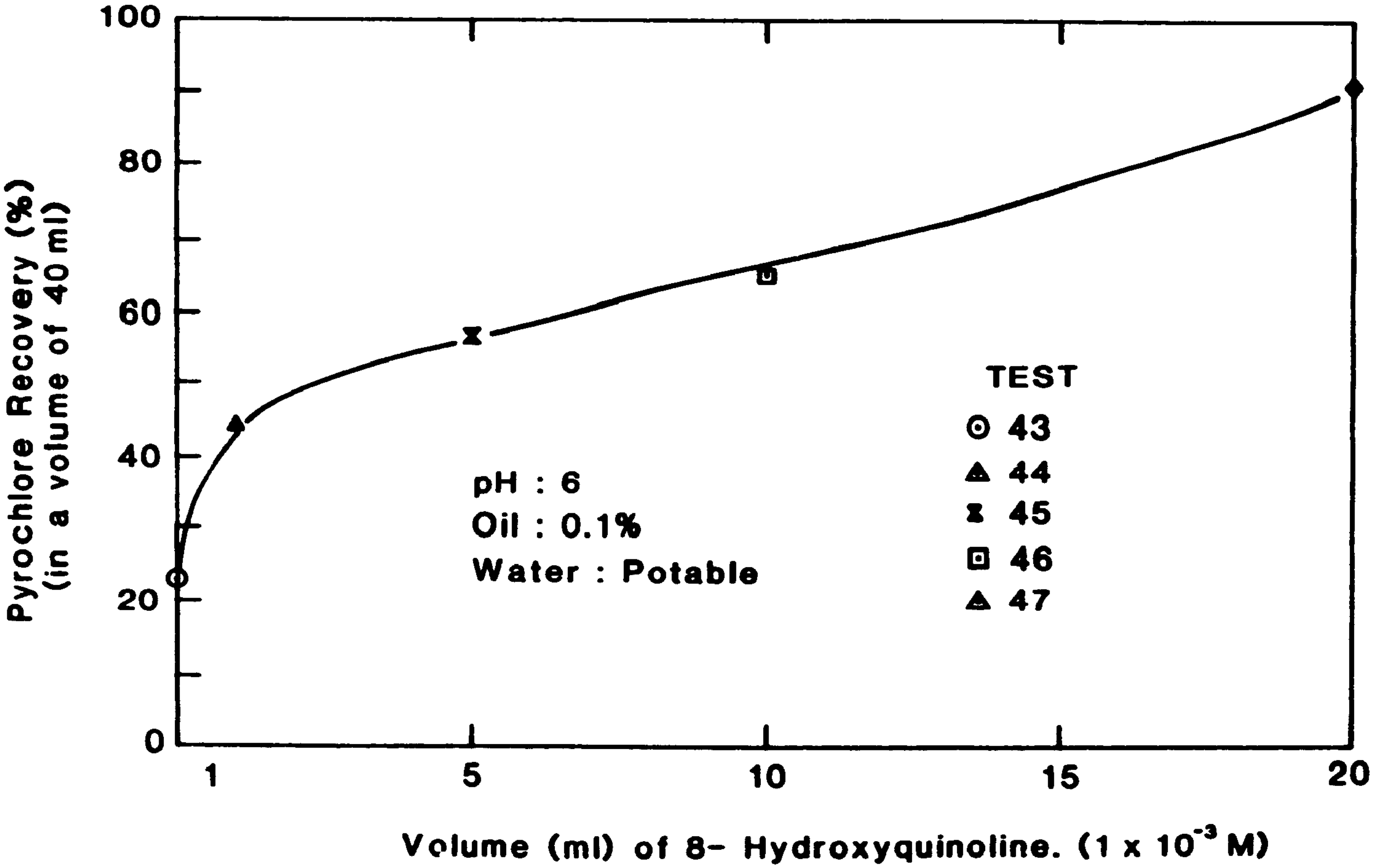


Figure 8 Pyrochlore recovery vs 8-Hydroxyquinoline concentration.

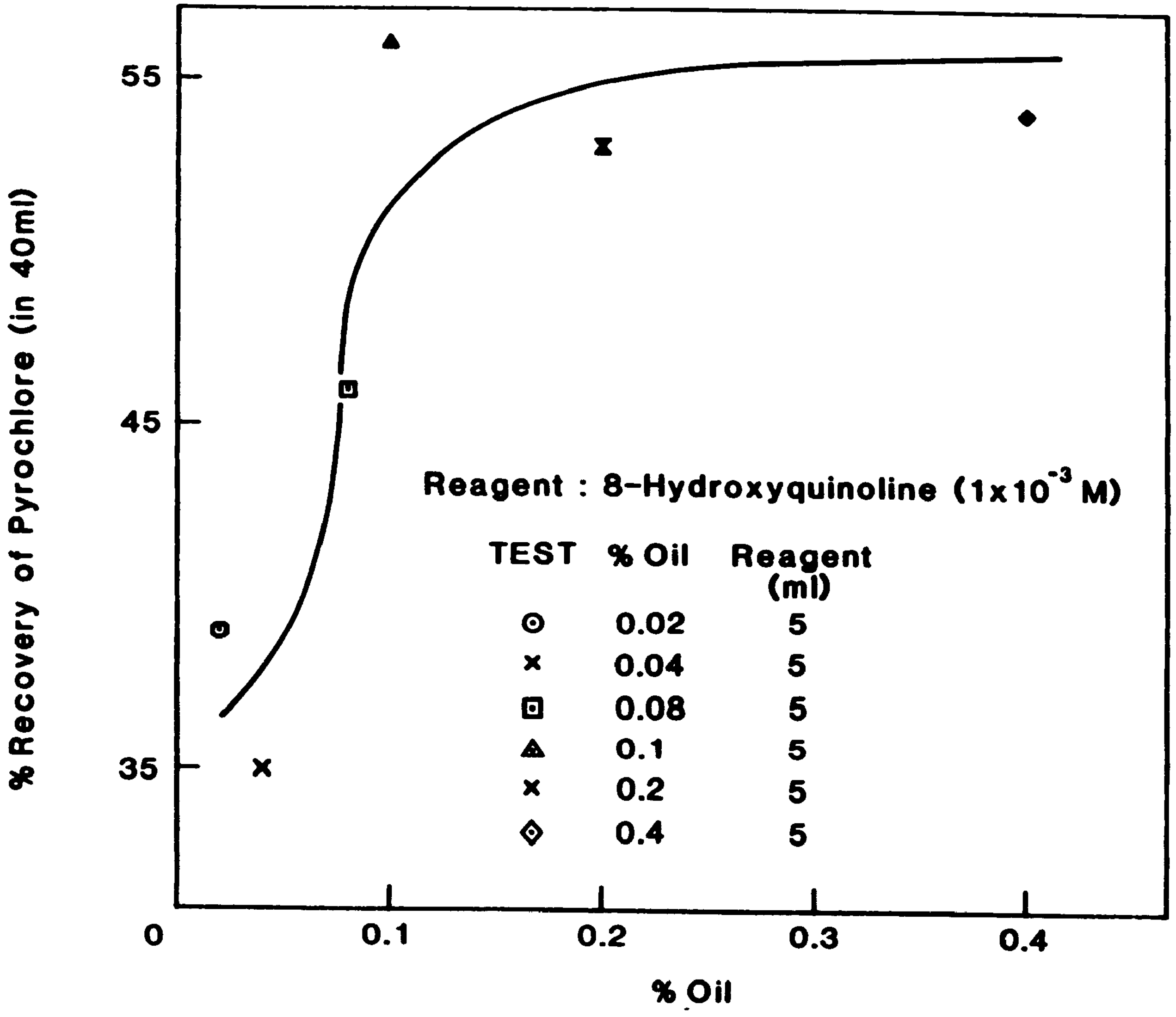


Figure 9 Effect of oil addition concentration in flotation recovery.

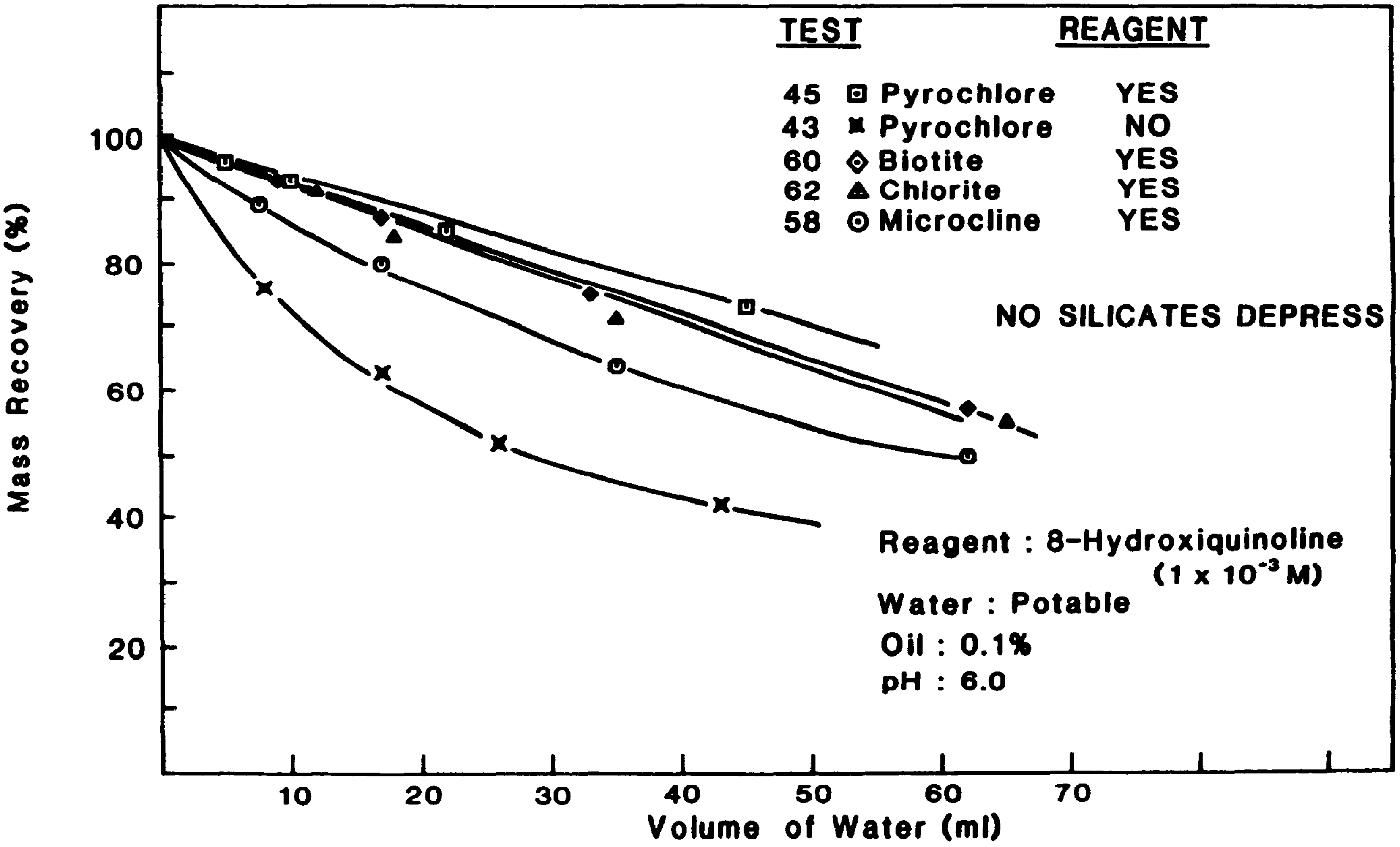


Figure 10 Flotation selectivity of 8-hydroxiquinoline using potable water.

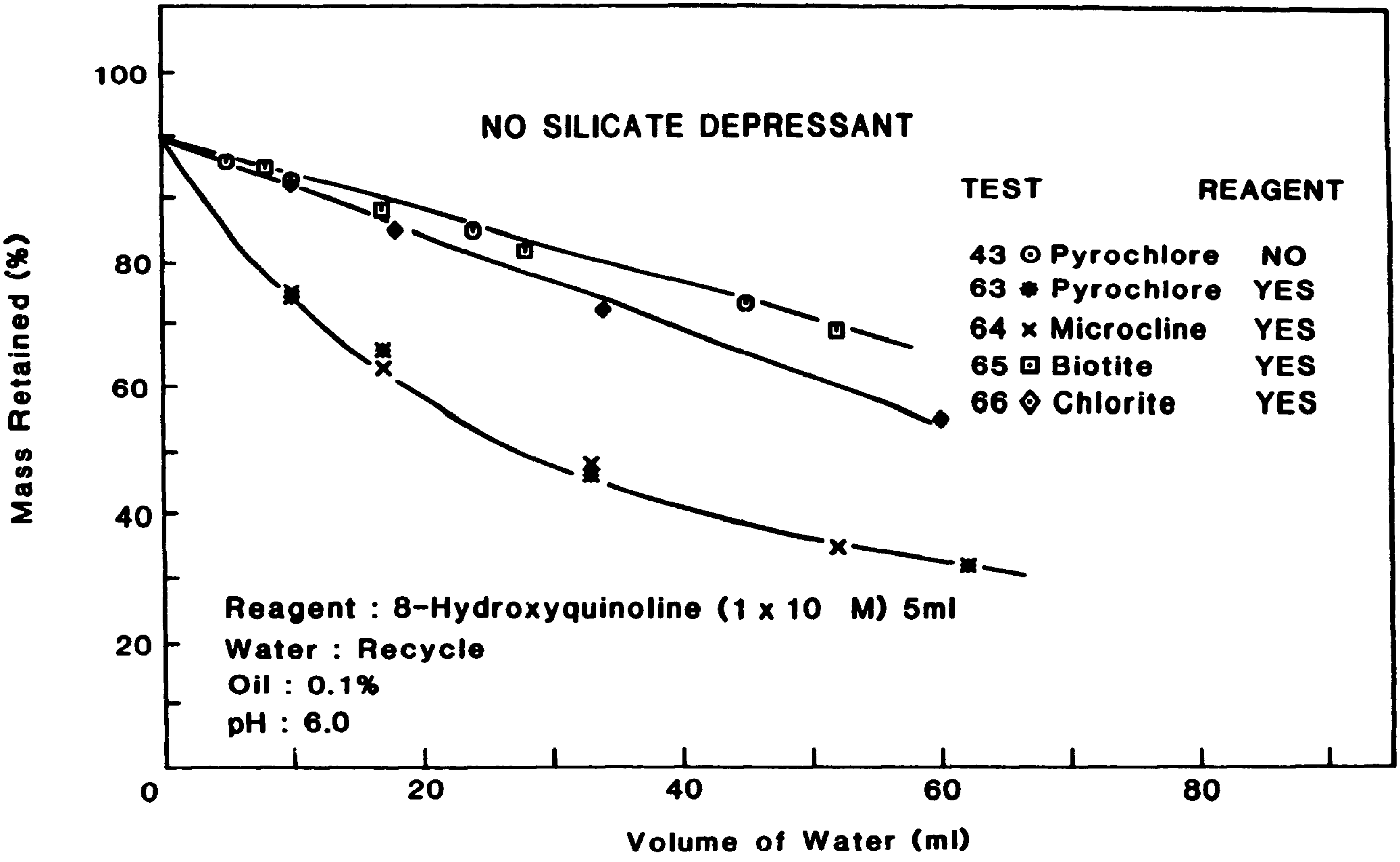


Figure 11 Flotation selectivity of 8-Hydroxyquinoline, using recycle water.

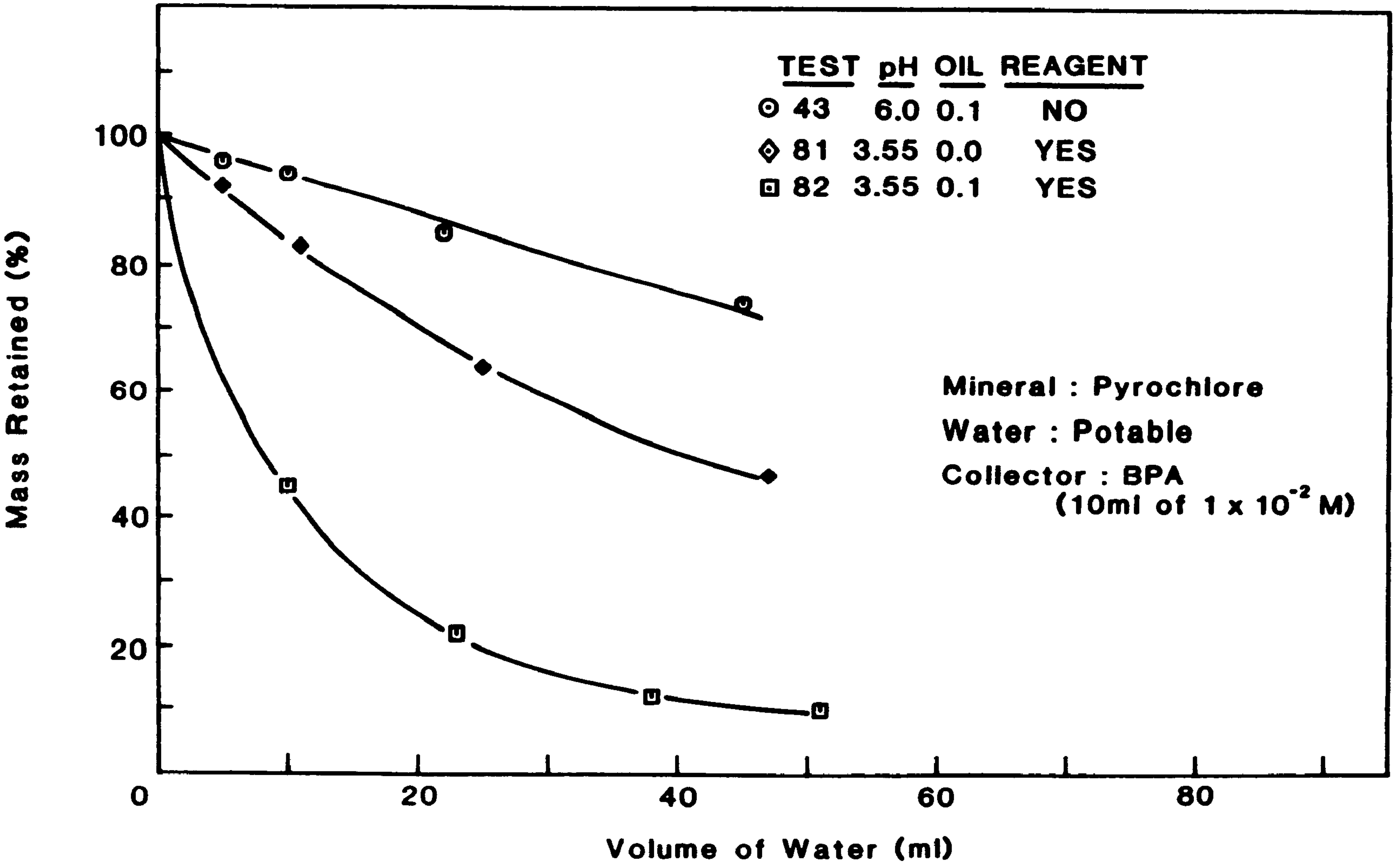


Figure 12 Flotation recovery of pyrochlore using BPA and BPA + oil addition.

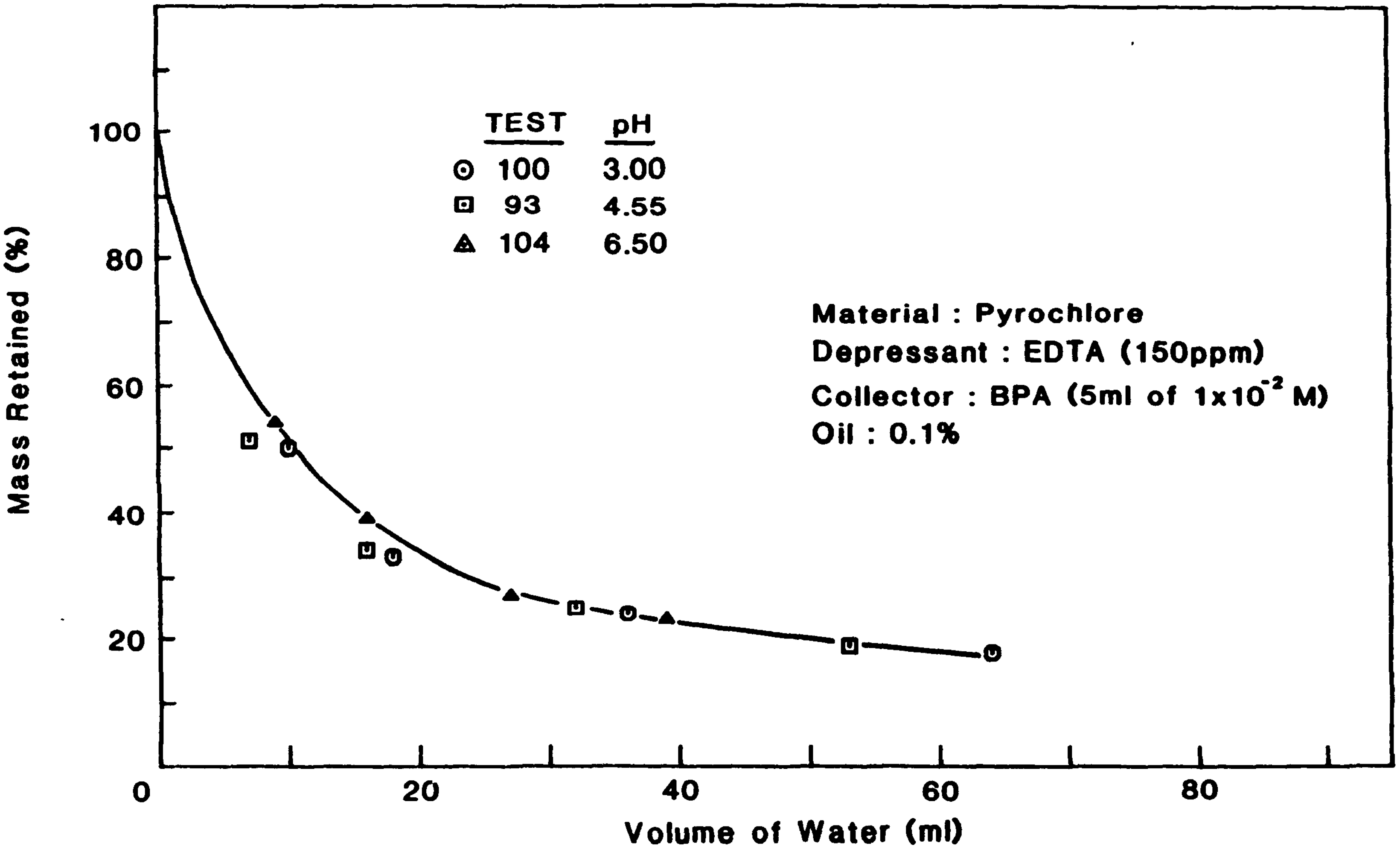


Figure 13 Flotation selectivity of BPA at different pH values

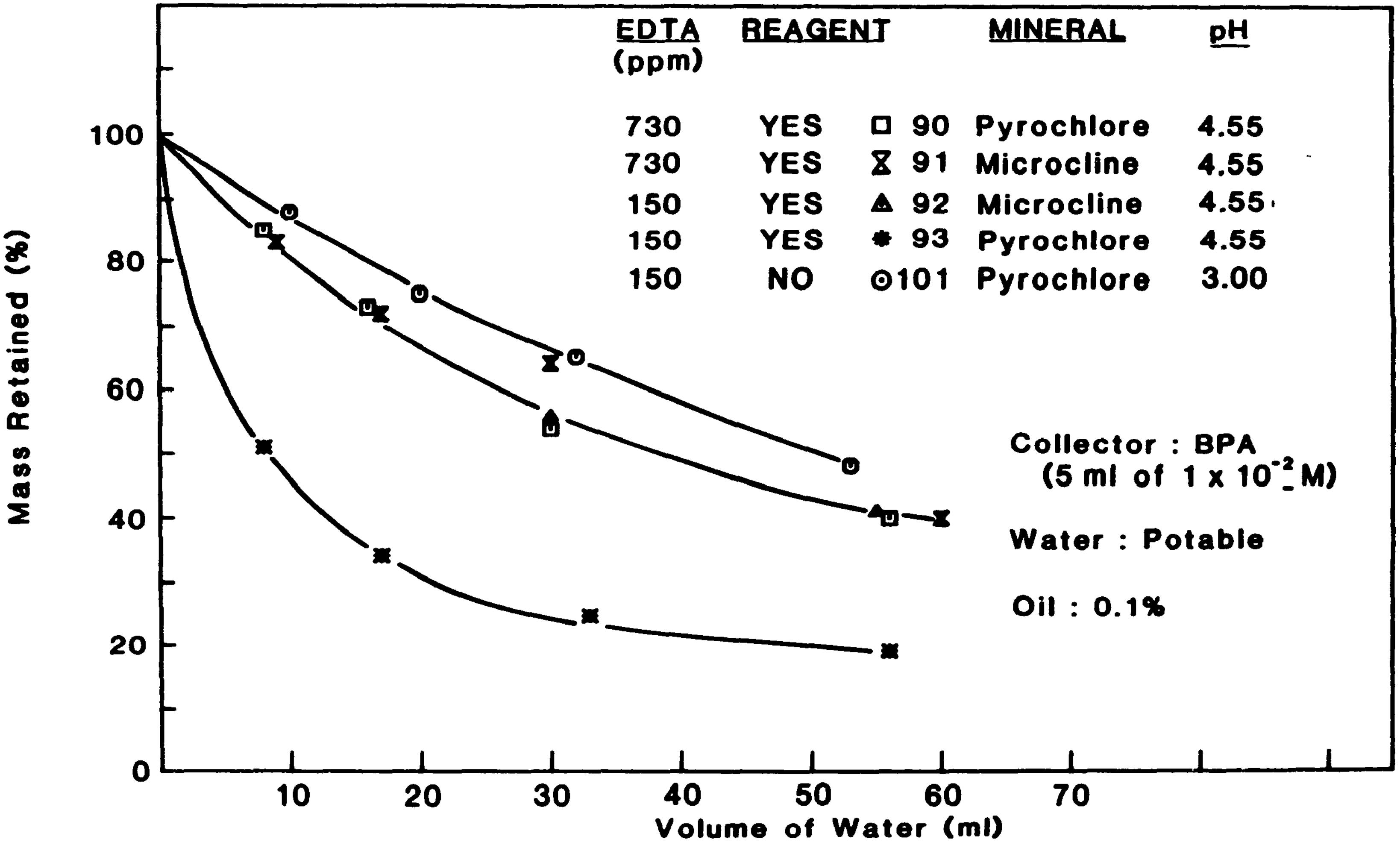


Figure 14 Effect of depressant concentration, EDTA, on pyrochlore and microcline flotation.

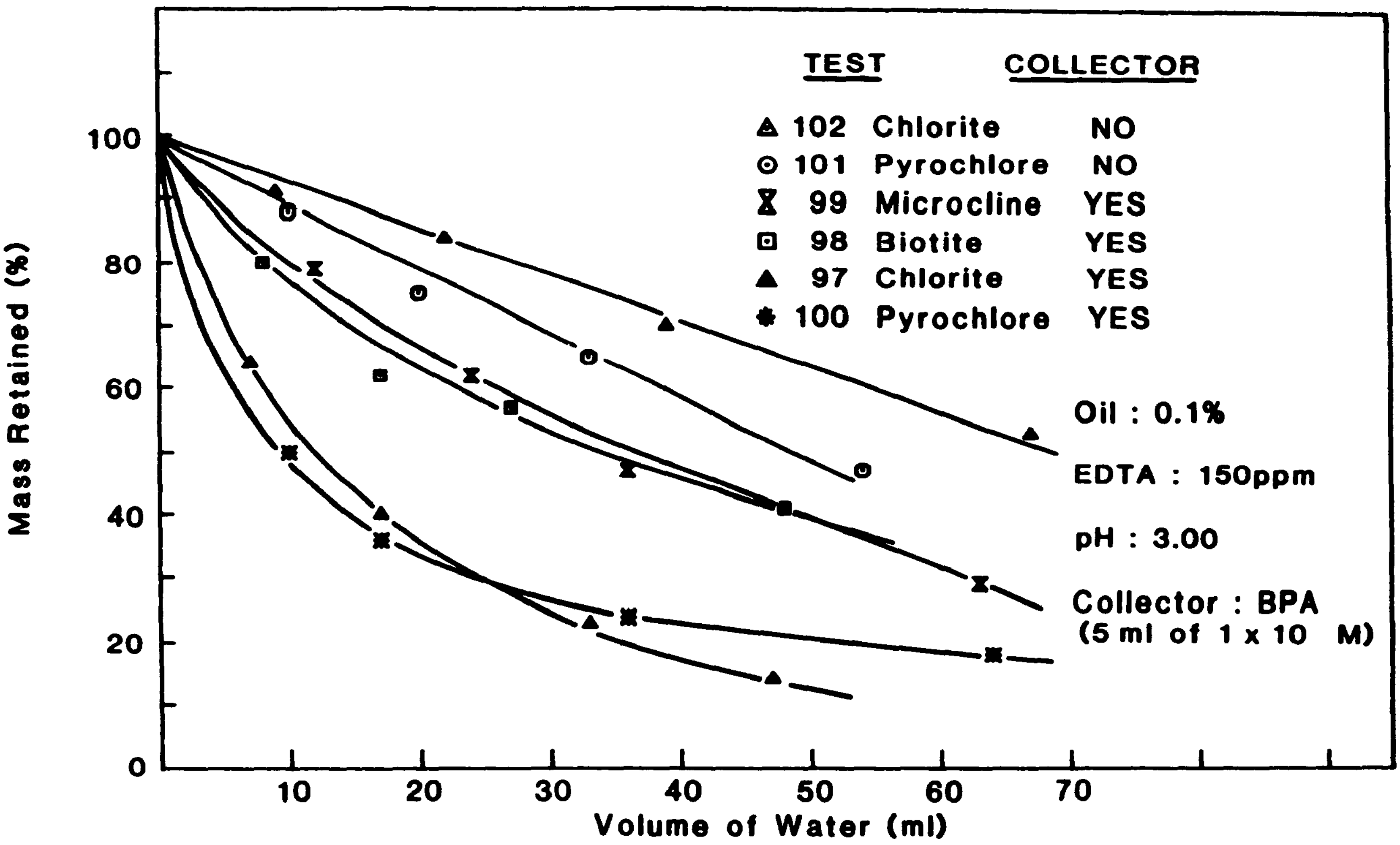


Figure 15 Flotation selectivity of BPA using potable water and 150 ppm EDTA.

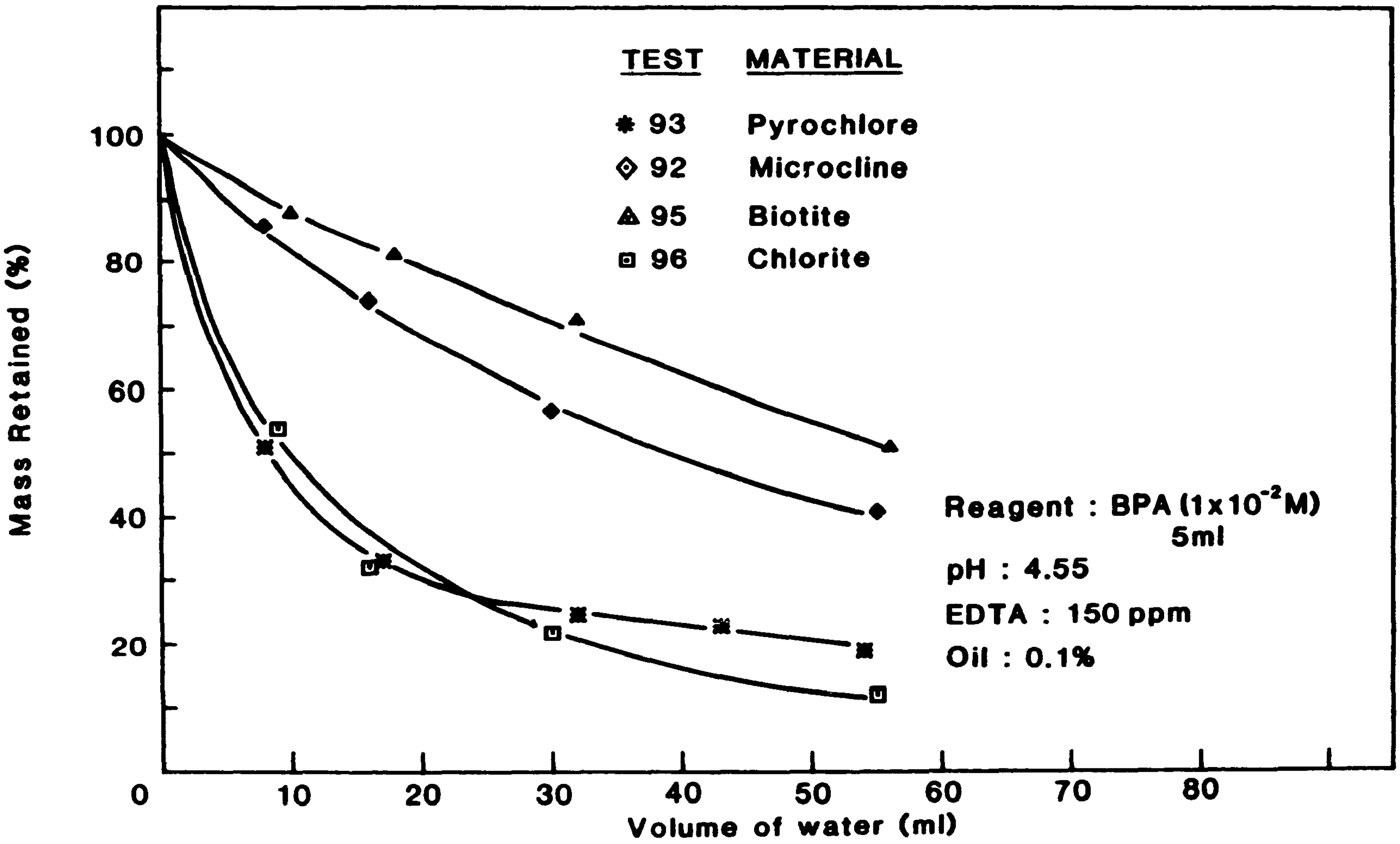


Figure 16 Flotation selectivity of BPA using potable water and 150 ppm EDTA.

PROGRESS REPORT- PYROCHLORE PROJET

REPORT # 5

R. ESPINOSA-GOMEZ

J.A. FINCH

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The present report summarizes the work done at McGill University on the pyrochlore project, for the period January to March, 1985. The report is divided in two parts:

A) Microflotation results using two types of collectors: a) phenyl ethylene phosphonic acid (P-184,PEP) and b) an imidazoline collector (F-2642). Several depressants were tested with each collector.

B) Application of a fundamental model of particle capture process as a guide in fine particles flotation.

A. MICROFLOTATION

Two flotation reagents, phenyl ethylene phosphonic acid (PEP) and an imidazoline collector (F-2642), both produced by Hoechst Canada Inc., were tested with pyrochlore and the silicate contaminants (microcline, chlorite and biotite). The microflotation procedure described in Report # 4 was used in these tests.

1. RESULTS

1.1 Phenyl Ethylene Phosphonic Acid (P-184, PEP)

Two phosphonic acids (phenyl ethylene phosphonic acid (PEP) and aliphatic phosphonic acid (APA)) were provided by Hoechst Canada Inc. Only one of them, PEP, has been tested. This was dissolved in distilled water to a concentration of 0.05 %. In all the tests 0.1 % oil (volume basis on the pulp), HCl and potable water from Niobec were used.

Figures 1 and 2 show the range of optimum pH (4-5) and collector addition (above 4 kg/ton) respectively. Figure 1 also shows that at pH values above 6 even using a large collector addition (10 kg/ton) the pyrochlore flotation is very limited.

In the subsequent tests a pH value of 4.5 and a collector addition of 1 kg/ton were used. The purpose of the low PEP dosage was to have a system more sensitive to any effect of depressants. Table 1 shows the selectivity of pyrochlore vs the silicates with and without various depressants. Values less than 1 indicate selectivity is being achieved; however, the Table shows satisfactory selectivity was not attained with any depressant.

1.2 Imidazoline collector (F-2642)

Special attention was given to this collector because this is the one currently used at Niobec. The pH modifier used in these tests was $C_2H_2O_4$. Comparing Figures 3 and 4 shows the advantage of using $C_2H_2O_4$; the

H2Si06 tends to depress pyrochlore flotation more than the silicates. Figure 5 shows that H2Si06 alone does not cause the depression, (see curve 1).

Figure 6 shows the flotation with F-2642 in the pH range 1 to 7. Pyrochlore flotation starts to decrease only below pH 2. However, even at pH 1, good recovery is still obtained.

The first depressant used was a polyacrylamide copolymer (DK-813) which is currently in use at Niobec; it induces agglomeration in the system. The collector and depressant additions were 1 kg/ton and 5 kg/ton respectively. (No oil was used in the rest of the tests with F-2642). Figure 7 shows the recovery by entrainment in the microflotation cell, using no depressant, DK-813 , and tannic acid. The magnitud of entrainment is in the order DK-813ˋnoneˋtannic acid. In all cases, however, entrainment can be considered negligible.

Four types of water were used in the DK-813 depressant tests: a) potable water; b) recycle water; c) recycle water treated at pH 3.6, and d) recycle water treated at pH 1.9. The recycle water treatment was simply to adjust to pH 3.6 or pH 1.9 and remove the precipitates produced. Figure 8 shows the results for potable water and treated recycle water at pH 1.9, and Figure 9 the results for recycle water and treated recycle water at pH 3.6. With potable water pyrochlore selectivity vs microcline increases as the pH is reduced from 5 to 2. At values above 5, selectivity is poor. Using treated recycle water (at pH 1.9), the selectivity below pH 5 approaches that with potable water, at pH 10 selectivity is between that in potable and recycle water. With recycle water (Figure 9) the selectivity remains almost constant above pH 4, and appears to increase below pH 4; Using treated recycle water (at pH 3.6) the selectivity is about the same.

Figures 10 and 11 show the flotation results for pyrochlore vs microcline, chlorite and biotite at the more selective pH values using potable water. At pH 2 (Figure 10) there is good selectivity of pyrochlore vs biotite and microcline; at pH 8 to 10 (Figure 11) selective flotation of pyrochlore cannot be attained. Figure 12 shows that at pH 2 using recycle water selectivity vs biotite and microcline is still achieved.

Another depressant used was Na2SiO3 (10 kg/ton). Figures 13 and 14 show the results using potable and recycle water respectively. With the former, only selectivity vs biotite is achieved; with the later a marginal selectivity vs chlorite and biotite is achieved.

Tannic acid (2.5 kg/ton) was also tested. Figure 15 shows good pyrochlore selectivity vs all the silicates at pH 6 using potable water. Using recycle water at pH 6, Figure 16, selectivity is only achieved with respect to the chlorite and to a lesser extent with respect to the microcline. This depressant is a good dispersant.

The depressant 9582, supplied by Niobec, Figure 17, was selective below pH 4 vs biotite, using potable water, and at pH 2 vs all the silicates tested. This depressant was not tested using recycle water because with potable water the results were not encouraging. However, further testing might be worthwhile.

Other depressants such as EDTA, Calgon, DA-811 and starch where also tested. The first three did not depress either the pyrochlore or the silicates, even using large dosages (more than 25 kg/ton). Starch depressed both pyrochlore and silicates at pH 4 but only when using more than 15 kg/ton.

2. DISCUSSION

2.1 Phenyl Ethylene Phosphonic Acid (PEP)

This anionic collector did not yield any selectivity (Table 1). However, some points revealed by these tests were: a) best depressants for microcline are DK-813, DA-811 and EDTA, all anionic depressants; b) best depressant for chlorite was EDTA; c) in general the best depressants were EDTA and DK-813; and, d) according with Figure 1, the adsorption of the phosphonic acid on the pyrochlore surface cannot be explained as a physical adsorption by electrostatic interaction. In all the pHs tested pyrochlore and PEP have negative charge. Consequently a chemisorption interaction is suggested.

2.2 Imidazoline Collector (F-2642)

a) $C_2H_2O_4$ vs H_2SiF_6

Figures 3 and 4 showed that using potable water at pH 4 H_2SiF_6 depresses the pyrochlore better than $C_2H_2O_4$. This is not due to the H_2SiF_6 alone, but rather it is a synergistic effect between H_2SiF_6 and the depressant DK-813. This is shown in curve 1 of Figure 5. The effects of both acids were not studied using recycle water, thus, it is difficult to extrapolate these results directly to the plant.

b) Recovery by Entrainment

The recovery by entrainment, Figure 7, changed accordingly with the type of depressant used. DK-813 gave the lowest entrainment. This is possibly due to the agglomeration induced by this depressant reducing the mass of fines. Using no depressant the entrainment increased but not to any significant extent. With tannic acid entrainment increased steadily with time. It is possible that tanning induces weak hydrophobicity to the particles. However, in any case entrainment was insignificant.

c) Pyrochlore Recovery vs pH

Figure 6 suggests that the adsorption mechanism for pyrochlore is not only electrostatic but also of a chemical-type. Otherwise flotation would stop below the pH corresponding to the iep (2.5). This observation is also supported by the results in Figure 9. If adsorption were electrostatic, flotation should cease at pH values from 3 to 4, and below 2, (1).

d) DK-813

Figures 8 and 9 showed the importance of the type of water in controlling flotation performance.

With potable water, flotation selectivity is achieved at acid pH , below 3. At pH 7 to 8 both pyrochlore and microcline are depressed; at pH 10 both pyrochlore and microcline float well but without selectivity. The depression at pH 7 to 8 may be related to the degree of ionization of the depressant. At these pHs, the same behavior was obtained with all the types of water used.

Recycle water induces the same flotation response for all the minerals. This is illustrated in Figure 9 and was observed before in the electrokinetic study (1). However, there is some selectivity, the pyrochlore being more floatable under all the conditions tested. Again, the acid pH range is the one showing best selectivity. At pH 10 both pyrochlore and microcline flotation is suppressed. This is probably due to the large amount of Ca and Mg cations in the recycle water vs the potable water, 199 ppm vs 26 ppm Ca, and 98 ppm vs 1 ppm Mg. These cations are specially active above pH 10 reducing the negative charge and consequently the collector electrostatic interaction with the mineral surfaces (2).

The purpose of partially treating the recycle water was to try to improve selectivity, especially at acid pH values. Recycle water treated at pH 3.6 gave little improvement, Figure 9. However, Figure 8 shows that treating the water at pH 1.9, shifts the response to one similar to that for potable water. The amount of precipitates removed at pH 3.6 and 1.9 was 260 and 625 ppm respectively. Another way to remove Ca and Mg is using Zeolites (3). An application of this method will be done shortly.

Figures 10 and 12 showed that using both potable and recycle water it is possible to achieve pyrochlore selectivity vs microcline and biotite at acid pH values, especially below pH 4. These results agree with plant practice. However, a chlorite depressant is also required.

e) $Na2SiO3$

Figures 13 and 14 showed that sodium silicate depressed biotite using potable water, and depressed chlorite and biotite, but only marginally, using recycle water. This reagent could be added with the DK-813 to increase chlorite depression. However, the additional benefits are apparently limited. This combination is used at Niobec.

f) Tannic acid

Tannic acid, Figure 15 produced good silicate depression and dispersion in a pH range from 4 to 7. This range is convenient for flotation, especially when some of the gangue contaminants are carbonates and apatite. Tannic acid has the advantage of being a strong depressant for carbonates, apatite, hematite and pyrite (4).

Three possible mechanisms of tannin depression have been postulated (5): a) physical adsorption by electrostatic interaction, b) adsorption by hydrogen bonding and , c) chemisorption. Depending on the pH value, each of these mechanisms, is capable of silicate depression. Specifically below pH 7, formation of hydrogen bonds between the active groups of the tannin and the hydroxyls present on the silicate surfaces, may cause tannin adsorption and depression. Interestingly this mechanism does not affect so strongly the pyrochlore. More studies are required to understand better these adsorption mechanisms.

g) Other depressants

EDTA. This anionic depressant did not give good results in the presence of the cationic collector F-2642. It is possible that due to its small number of OH-sites, it cannot impart enough hydrophilicity to the particles. Because of these results other short chain anionic depressants such as citric acid and tartaric acid were not tested.

Calgon (Sodium Hexametaphosphate). This anionic depressant was not able to depress the silicates despite its large number of active sites. However it did produce excellent dispersion without affecting flotation at all. The addition of a dispersant will be advantageous in ultrafines flotation.

DA-811 This depressant showed the same behavior as calgon.

Starch. This depressant was not selective. Large amounts were required to initiate depression. This must be related to its low degree of dissociation at acid pH. In addition, it induced flocculation, even at very low additions. Depressants that induce flocculation, should be avoided in fines flotation.

2.3 Flotation Routes.

So far two flowsheets are comtemplated to float the pyrochlore from the slimes:

a) Direct pyrochlore flotation

b) Direct pyrochlore flotation with prior carbonate flotation.

a) Direct pyrochlore flotation. This flowsheet is contemplated using F-2642 as pyrochlore collector and tannic acid as gangue depressant, with possibly calgon as a dispersant. Figure 15 showed that F-2642 can be selective vs the silicates at pH 5 to 7. Although the use of tannic acid to depress carbonates, apatite, hematite and pyrite, has not been tested yet, studies show that its depression power is even stronger with these gangue minerals than with silicates (4). Also the flotation response of F-2642 with carbonates and apatite has not been tested here yet, but a previous study showed that their flotability is much less than pyrochlore (6).

The disadvantage of this route is that it is feasible only using potable water. With recycle water there are problems to depress biotite and microcline. These limitations encourage further studies on treating the recycle water.

b) Direct pyrochlore flotation with prior carbonate flotation.

This route is feasible using both potable and recycle water. It involves first, a carbonate flotation (as is current practice at Niobec); second, pyrochlore flotation at pH 6 with F-2642 as collector, tannic acid as depressant and possibly calgon as dispersant; and third, a further pyrochlore cleaning stage at pH 2 to 3 using F-2642 as collector and DK 813 as depressant. Although both waters can be used potable water is preferred.

Future work will involve two selective chelating agents: 8-Hydroxyquinoline and N-Benzoyl-N-Phenyl-Hydroxylamine to be tested with several silicate depressants. Also four other possible pyrochlore collectors will be tested: a) sulfosuccinate (PROCOL CA-540), b) benzohydroxamic acid, c) sodium alkyl sulphate, and, d) aliphatic phosphonic acid. Some preliminary flotation column tests will be performed with the flowsheets described.

B. APPLICATION OF A FUNDAMENTAL MODEL OF PARTICLE CAPTURE PROCESS AS A GUIDE IN FINE PARTICLES FLOTATION. (7).

The basic equation of this fundamental model is :

$$Ek = Ec \cdot Ea \qquad (1)$$

where Ek is the collection efficiency, Ec the collision efficiency and Ea the attachment efficiency. Ec is defined as the rate at which particles collide with the bubble divided by the rate at which particles flow across the projected area of the bubble. Ea is defined as the fraction of all colliding particles that reside on the bubble for a time greater than the induction time. This model has provided a convenient explanation of recovery vs size curves based on three parameters: a) induction time, b) bubble diameter, and, c) particle density. The model predicts that collection efficiencies become similar (lack of selectivity) for particles less than 5-10 um. This is because of the long contact time of the smaller particles with the bubbles, thus almost regardless of induction time, attachment efficiency approaches 100 %. Finally, the model also predicts that smaller bubbles do not improve selectivity, but do improve the rate of flotation.

Since in this work the particle size of concern is -15 um, the model can be used to guide selection of flotation conditions. One way to increase selectivity is by working with larger particle sizes. An increase in particle size can be induced by adding oil. The minute droplets (i.e. emulsion) readily adsorb on the hydrophobic surfaces of the minerals, increasing surface hydrophobicity and inducing agglomeration.

Another way to increase selectivity is by increasing the difference in induction time (Ti) between valuable and gangue minerals. This is the objective of searching for selective reagents. A more subtle route suggested by the model, however, is to reduce the absolute value of the induction time i.e. reduce the flotation rate constant. This can be effected by keeping reagent dosage low ('starvation' dosage). The model was run to study selectivity between mineral A (more floatable) and mineral B (less floatable) as a function of induction time while maintaining a relative induction time of 2.5. The results are given in Table 2.

Note at short induction times (Ti(A) = 10 ms) that selectivity of 50 um particles poses no problem while at sizes 14 um and less selectivity is poor (collection efficiency of 14 um A is only twice that of B). As the absolute induction time is increased the size for maximum selectivity shifts to smaller values. For example for Ti(A) = 50 ms particles 14 - 16 um give good selectivity. (At Ti(A) = 50 ms attachment efficiency of 50 um particles is essentially 0 because particles of such size cannot have a contact time greater than 50 ms regardless where on the bubble they first collide).

Increasing Ti means reducing collector dosage. Clearly there is a trade-off between selectivity and flotation rate (which will decrease as Ti increases). However flotation rate is amenable to some physical control e.g. decreasing bubble size will increase rate without decreasing selectivity. Column flotation tests will be conducted with 'starvation' collector dosage in mind.

CONCLUSIONS

1) Phenyl ethylene phosphonic acid did not present good selectivity for pyrochlore flotation vs silicates.

2) $C_2H_2O_4$ was found to be a more suitable pH modifier than H_2SiF_6 when DK-813 is used as depressant.

3) The type of water used was found to be very important for selective flotation. Potable water is better than recycle water. Treatment of recycle water offers some possibility of retrieving selectivity.

4) Two flotation routes have been identified for selective pyrochlore flotation:

a) direct pyrochlore flotation at pH 6 using F-2642 as collector and tannic acid as gangue depressant, and

b) direct pyrochlore flotation with prior carbonate flotation, involving three steps: 1) carbonate flotation as is currently practice at Niobec, 2) pyrochlore flotation at pH 6 using F-2642 as collector and tannic acid as gangue depressant, 3) pyrochlore flotation at pH 2 to 3 using F-2642 and DK-813 as gangue depressant.

5) Column flotation tests should be run with starvation collector dosage.

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TABLE 1. FLOTATION RESULTS USING 1 Kg/Ton PHENYL ETHYLENE PHOSPHONIC ACID AS COLLECTOR

DEPRESSANT	* SELECTIVITY INDEX		
(Kg/Ton)	MICROCLINE	CHLORITE	BIOTITE
NONE	.52	1.43	.98
DK-813 (A) (11.0)	.78	.9	1.15
CALGON (B) (0.7)	1.2	1.4	1.57
Na2SiO3 (2.0)	1.39	1.29	1.42
DA-811 (C) (1.0)	.48	1.05	1.29
EDTA (D) (0.6)	.8	.8	1.63
9582 (E) (0.2)	1.64	1.52	1.72

* R (%) of silicate / R (%) of pyrochlore
(A) Polyacrylamide copolymer (from Allied Colloids)
(B) Calgon (Mainly sodium hexametaphosphate)
(C) Silicate depressant (from Allied Colloids)
(D) Ethylenediamine tetraacetic acid
(E) Silicate depressant

TABLE 2. COLLECTION EFFICIENCY RATIOS FOR SEVERAL INDUCTION TIMES AND PARTICLE SIZES.

MINERAL	* Ti (ms)	Ek (A) / Ek (B)				
		PARTICLE DIAMETER (um)				
		50	14	10	6	2
A B	10 25	>1500	2	2	1	1
A B	50 125	0	>10000	>30000	32	2
A B	150 375	0	3	* * V. L	V.L.	31
A B	400 1000	0	0	0	0	24

* Ti (B) / Ti (A) = 2.5

* * VERY LARGE

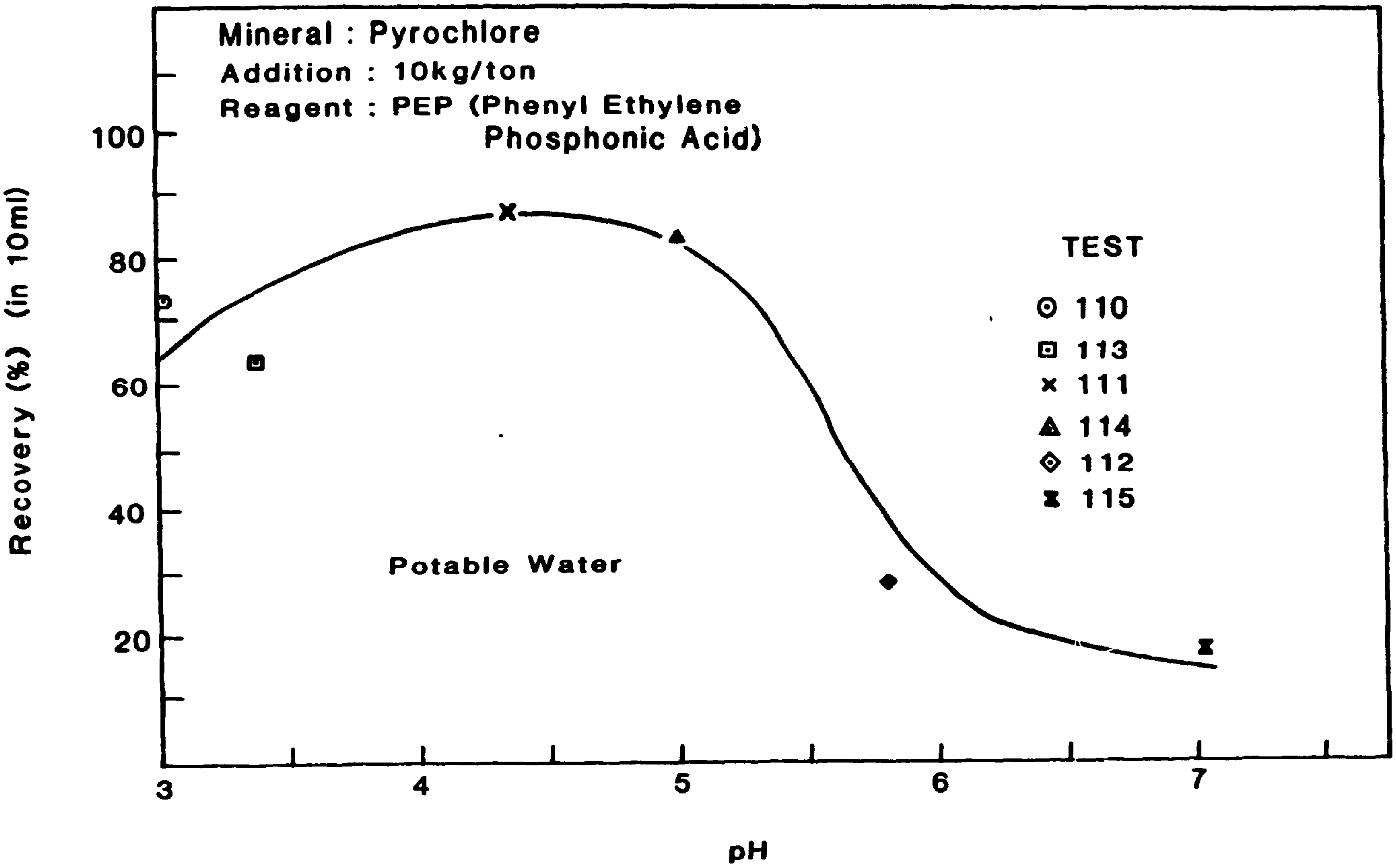

Figure 1. Flotation selectivity of pyrochlore with phenyl ethylene phosphonic acid. Selection of optimum pH

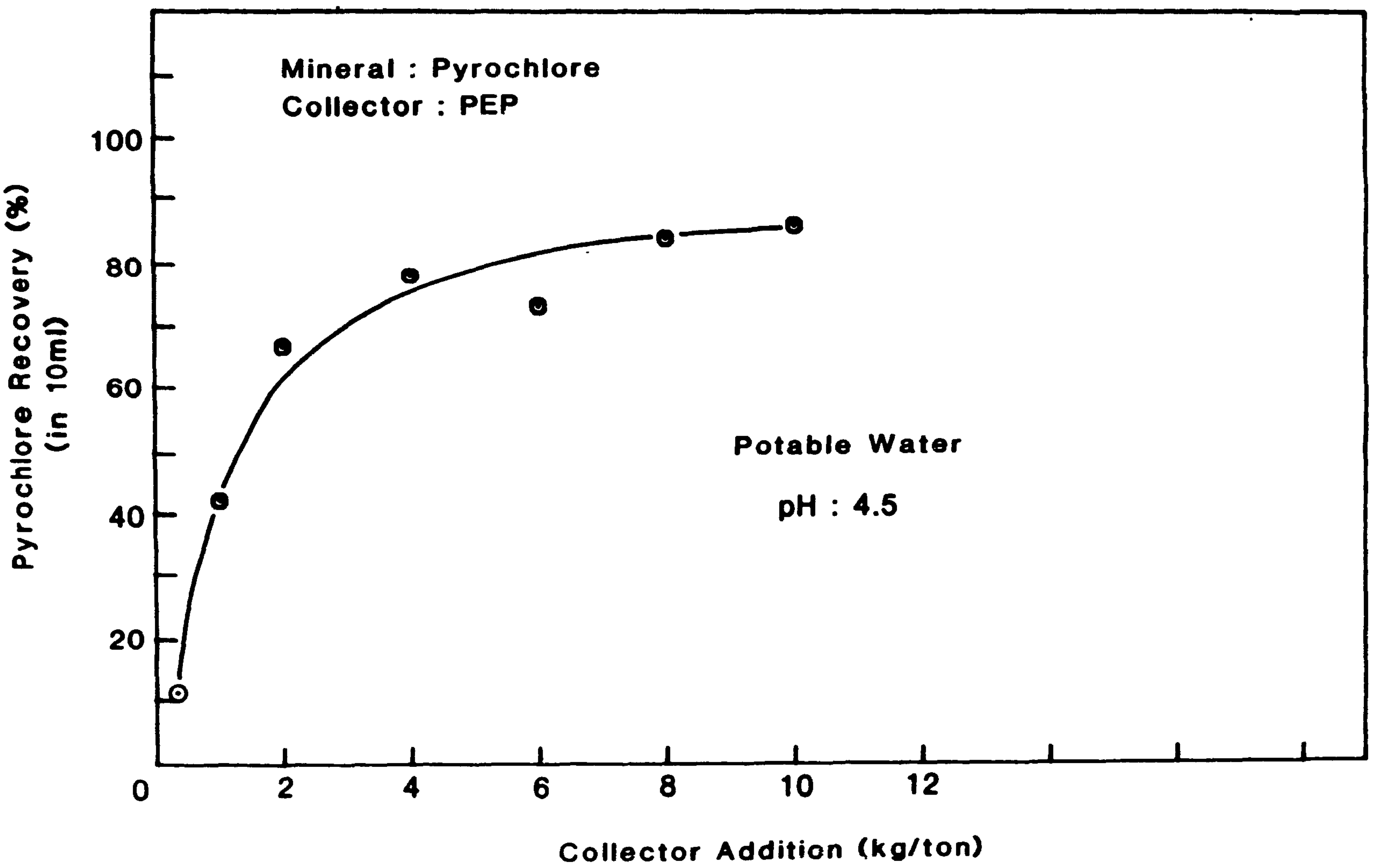

Figure 2. Flotation selectivity of pyrochlore with phenyl ethylene phosphonic acid. Selection of optimum reagent concentration

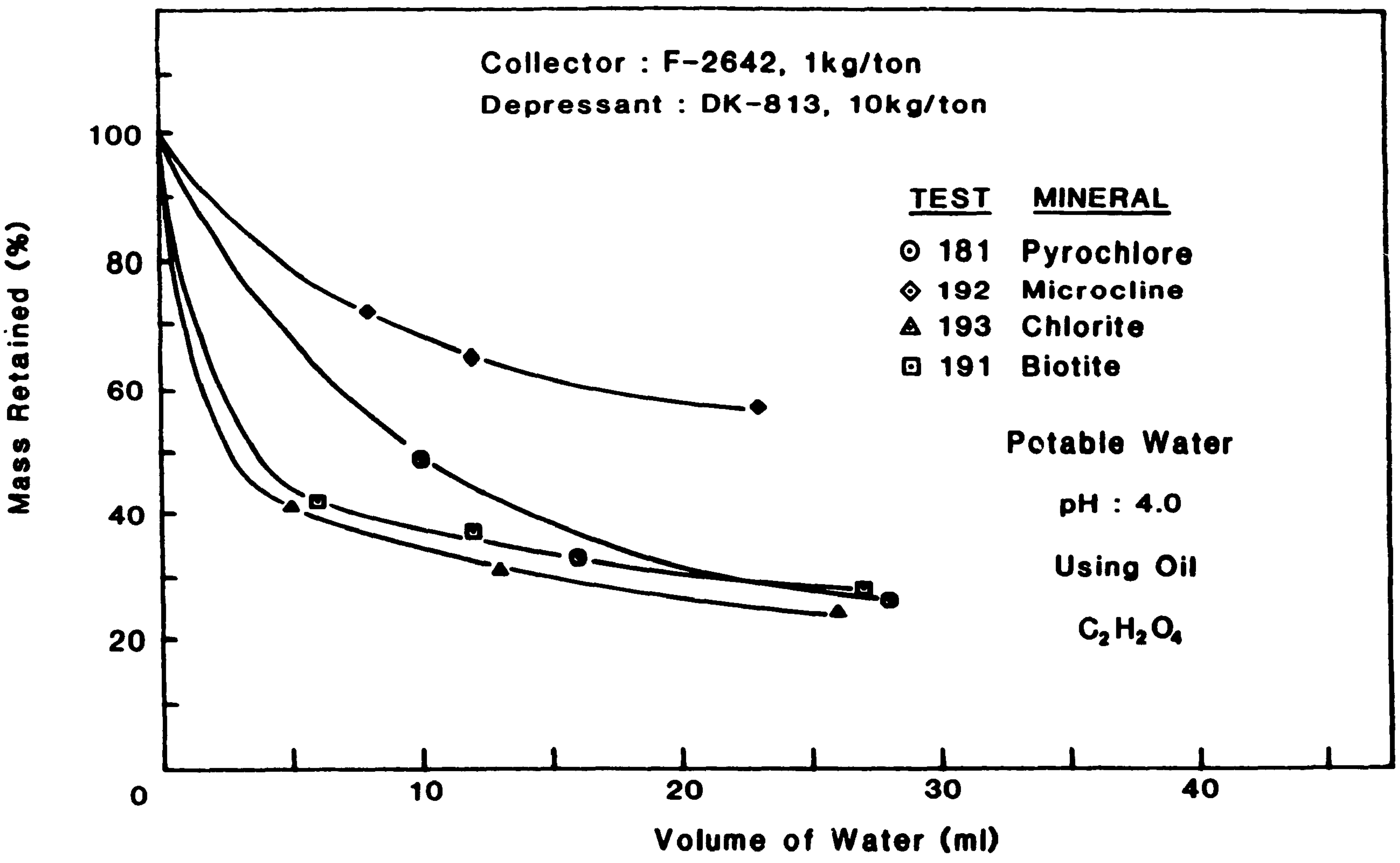

Figure 3. Flotation selectivity of pyrochlore and silicates

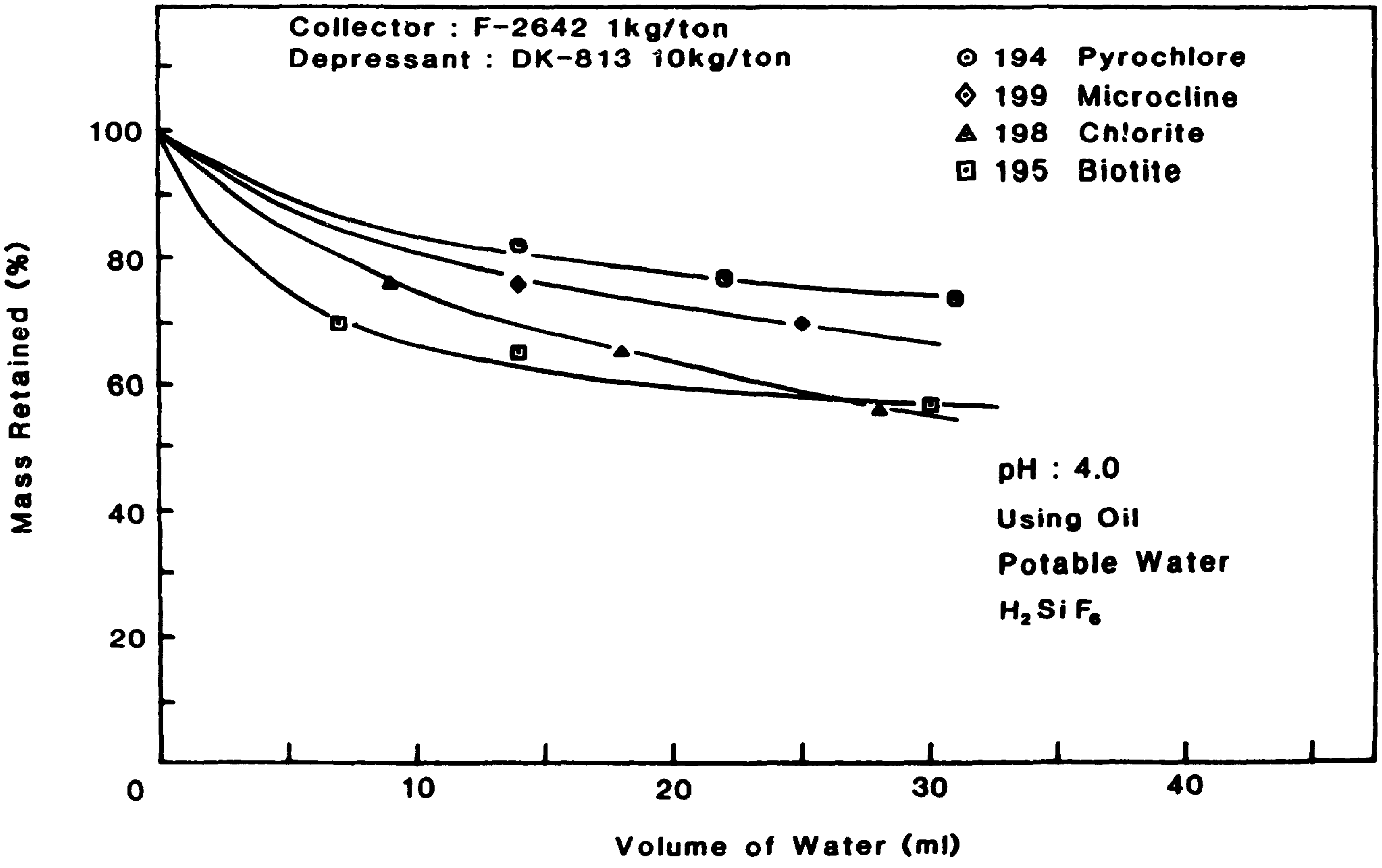

Figure 4. Flotation selectivity of pyrochlore and silicates with F2642, using fluosilic acid and oil as emulsifier

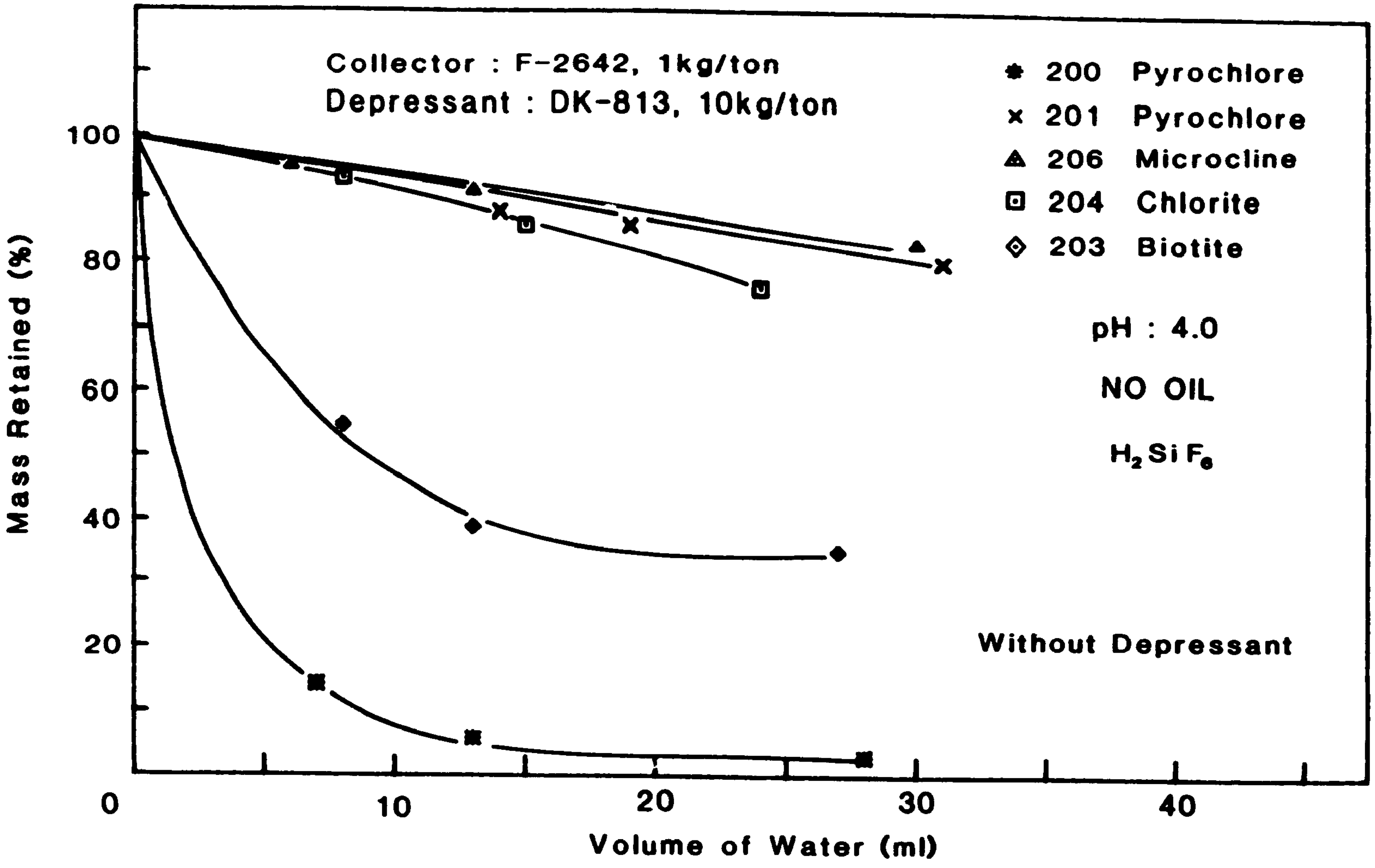

Figure 5. Flotation selectivity of pyrochlore and silicates with F2642, using fluosilic acid. No oil

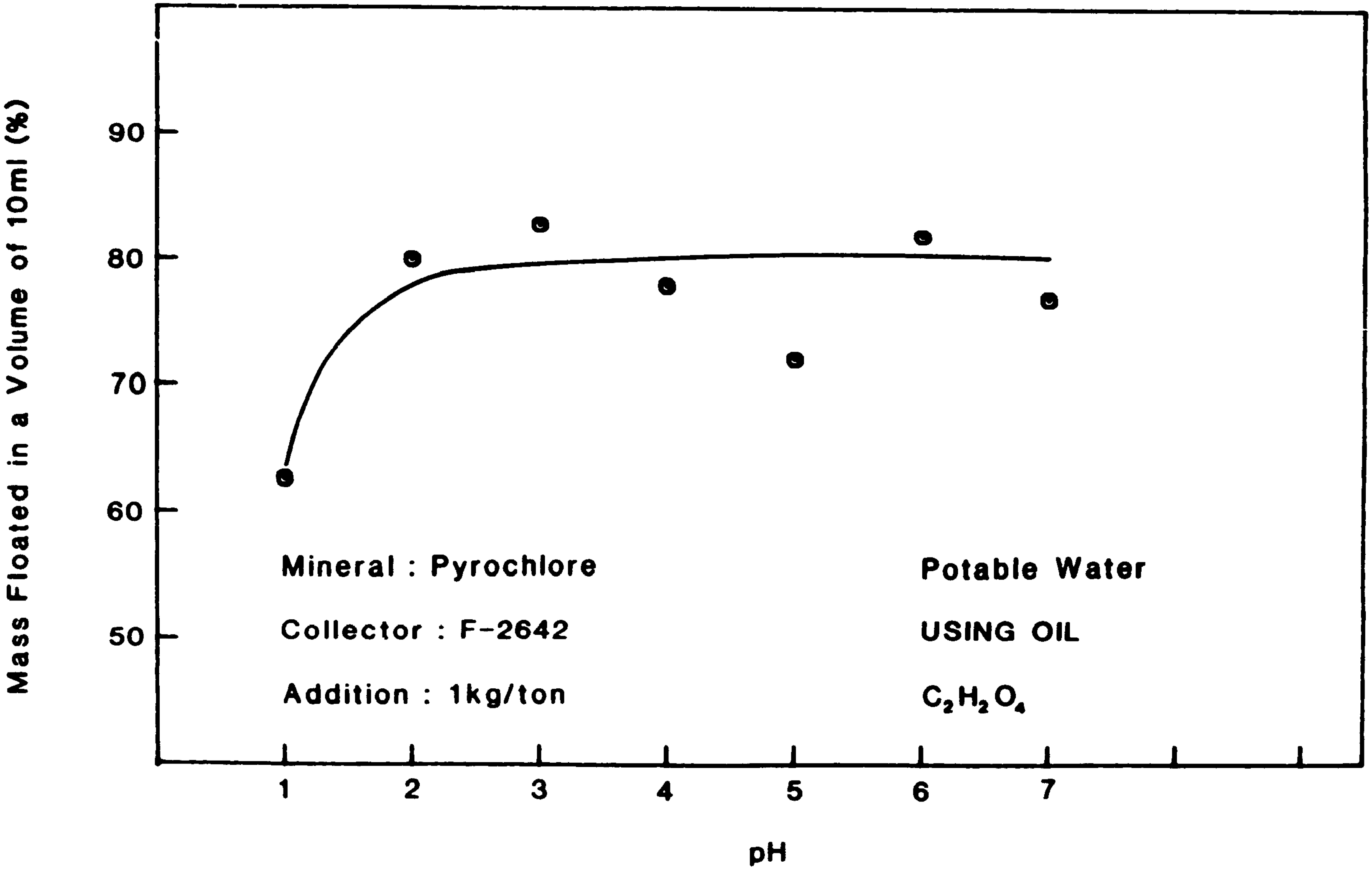

Figure 6. Flotation selectivity of pyrochlore and silicates with F2642, usinf oxalic acid and oil

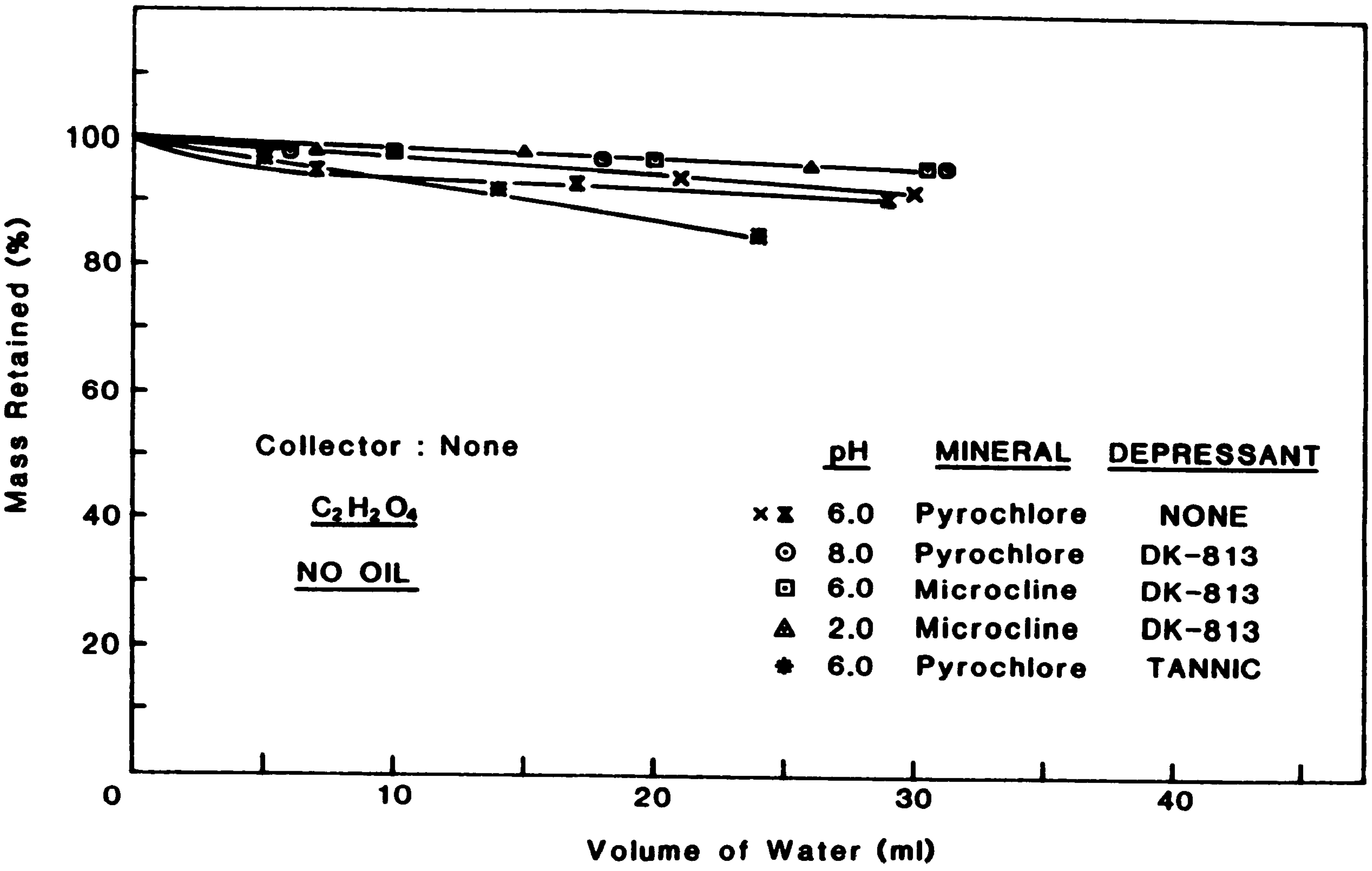

Figure 7. Flotation selectivity of pyrochlore and microcline with F2642 and two types of depressants

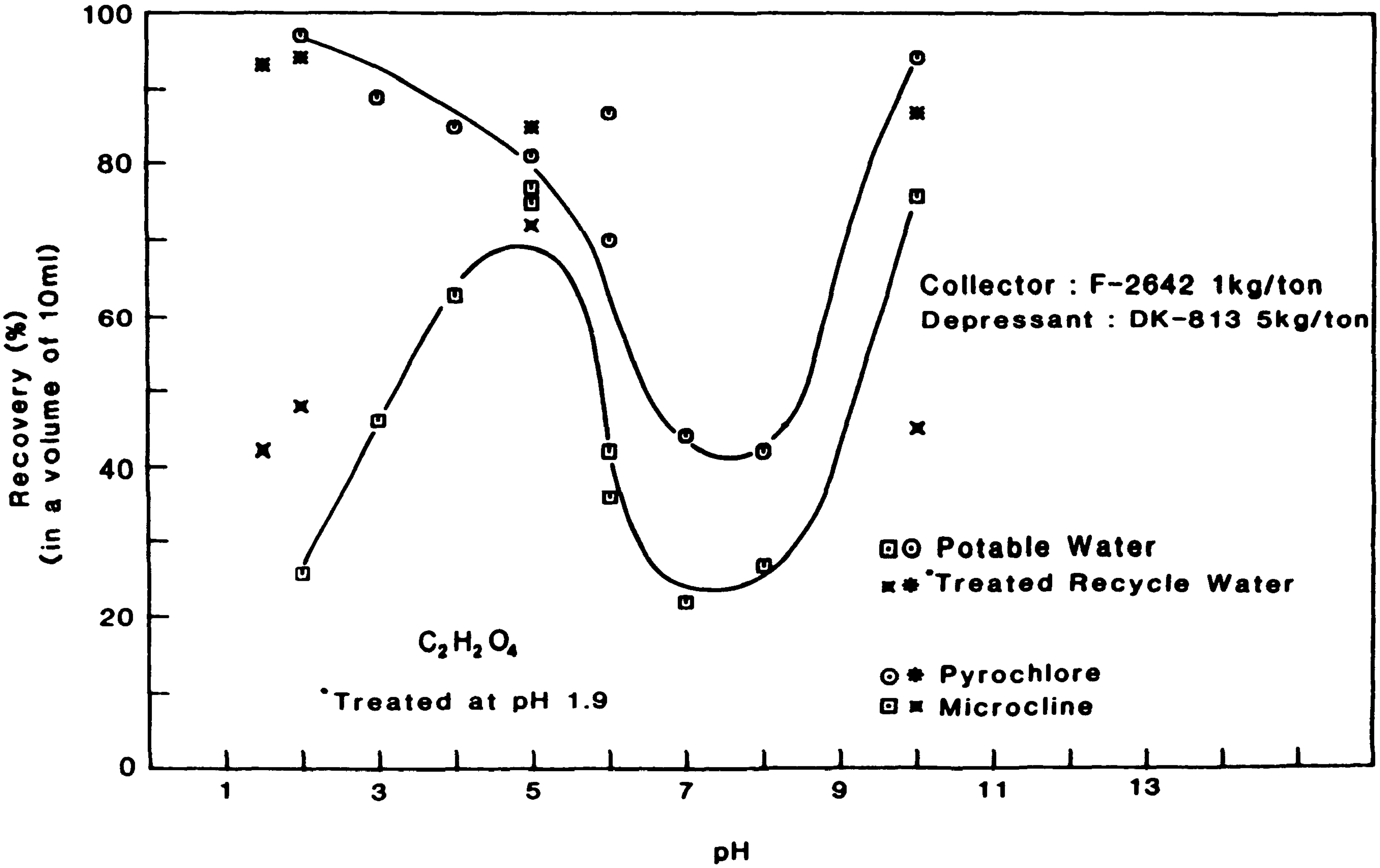

Figure 8. Electrophoretic mobility curves for pyrochlore and microcline using axolic acid and two types of water, potable and treated recycle

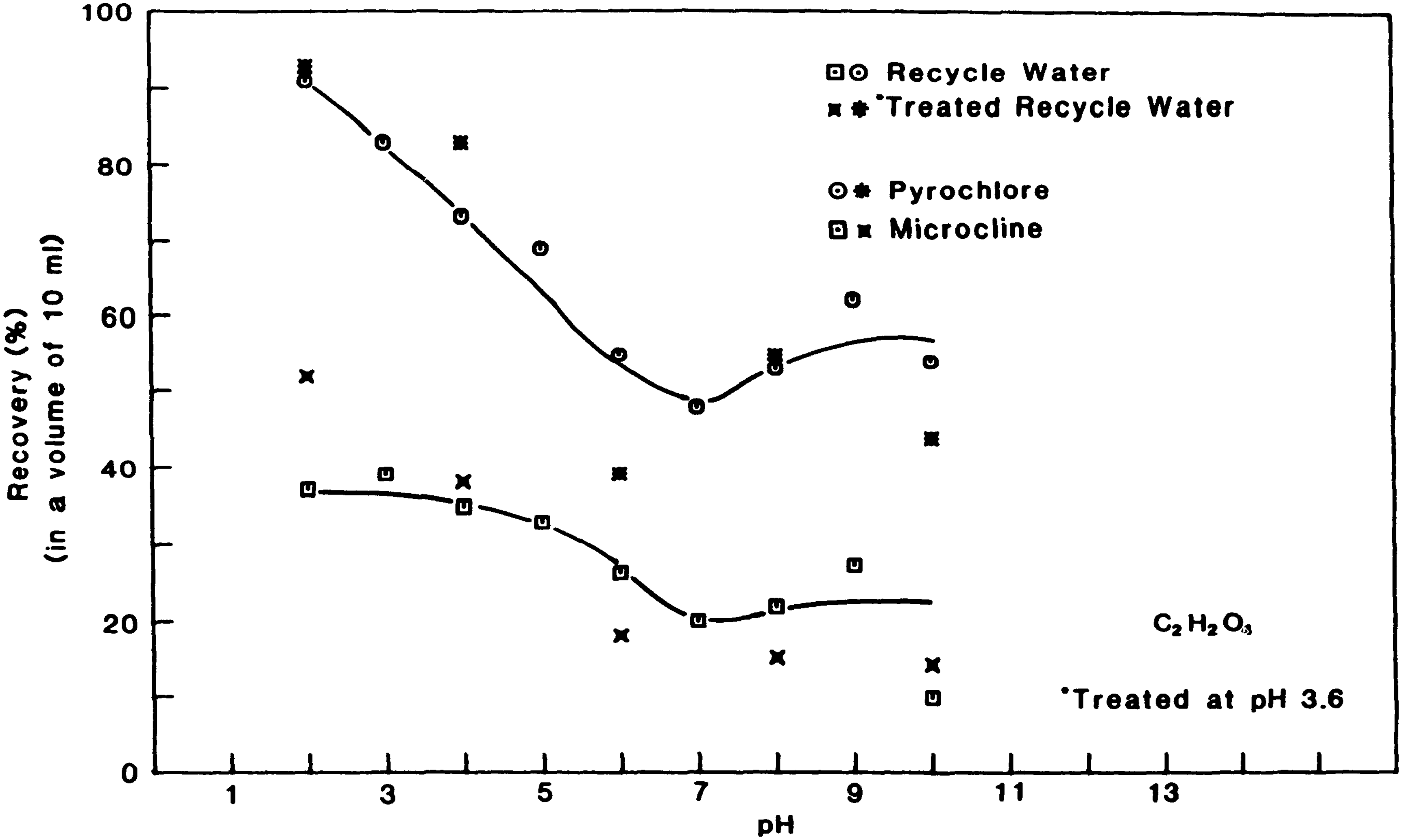

Figure 9. Electrophoretic mobility curves for pyrochlore and microcline using oxalic acid and two types of water: recycle and treated recycle

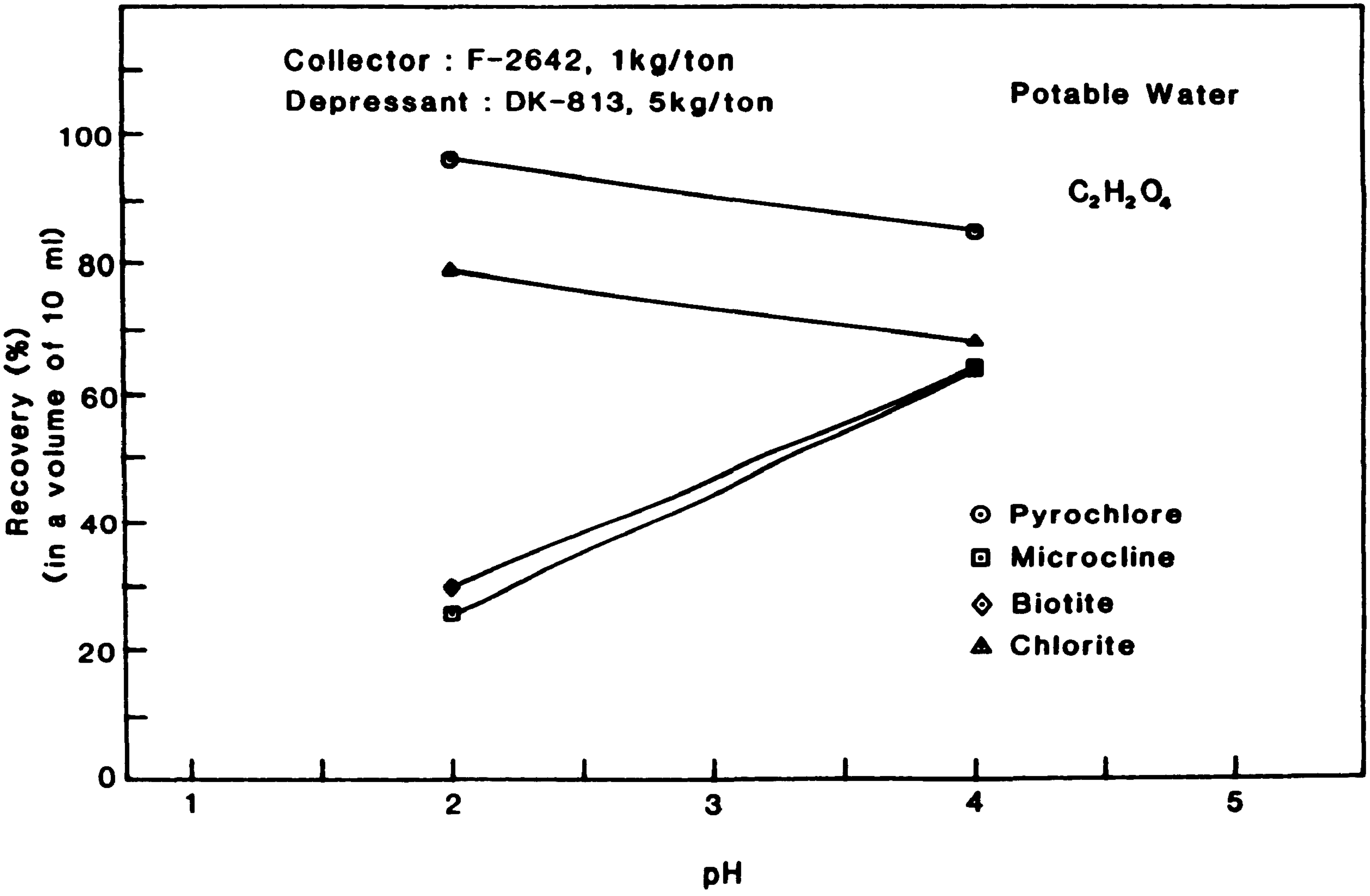

Figure 10 Flotation selectivity of pyrochlore and silicates with optimum 2 additions of F2642 and depressant DK 813. Selection of best pH. Using potable water

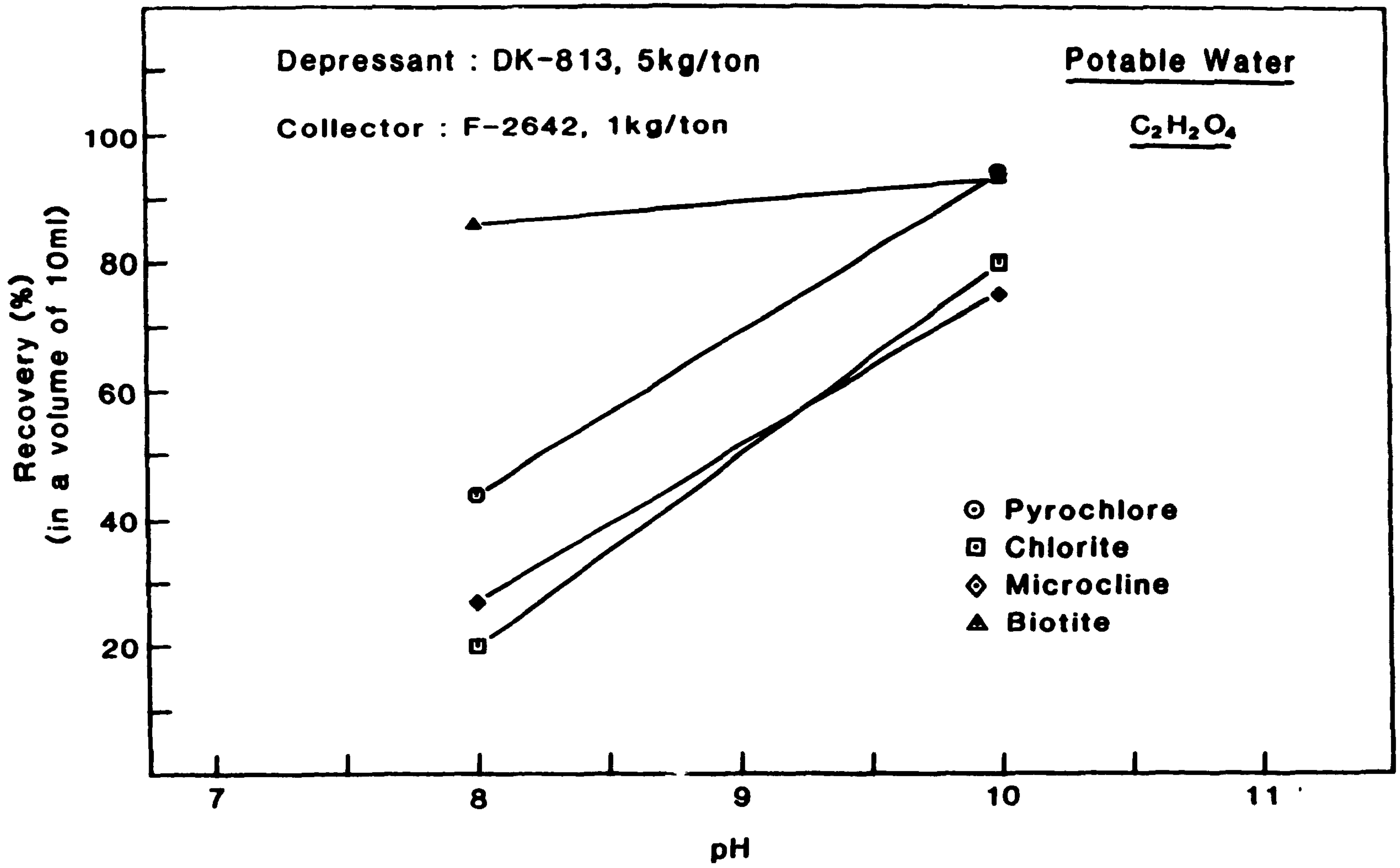

Figure 11 Flotation selectivity of pyrochlore and silicates with optimum 2 additions of F2642 and depressant DK 813. Selection of best pH. Using potable water

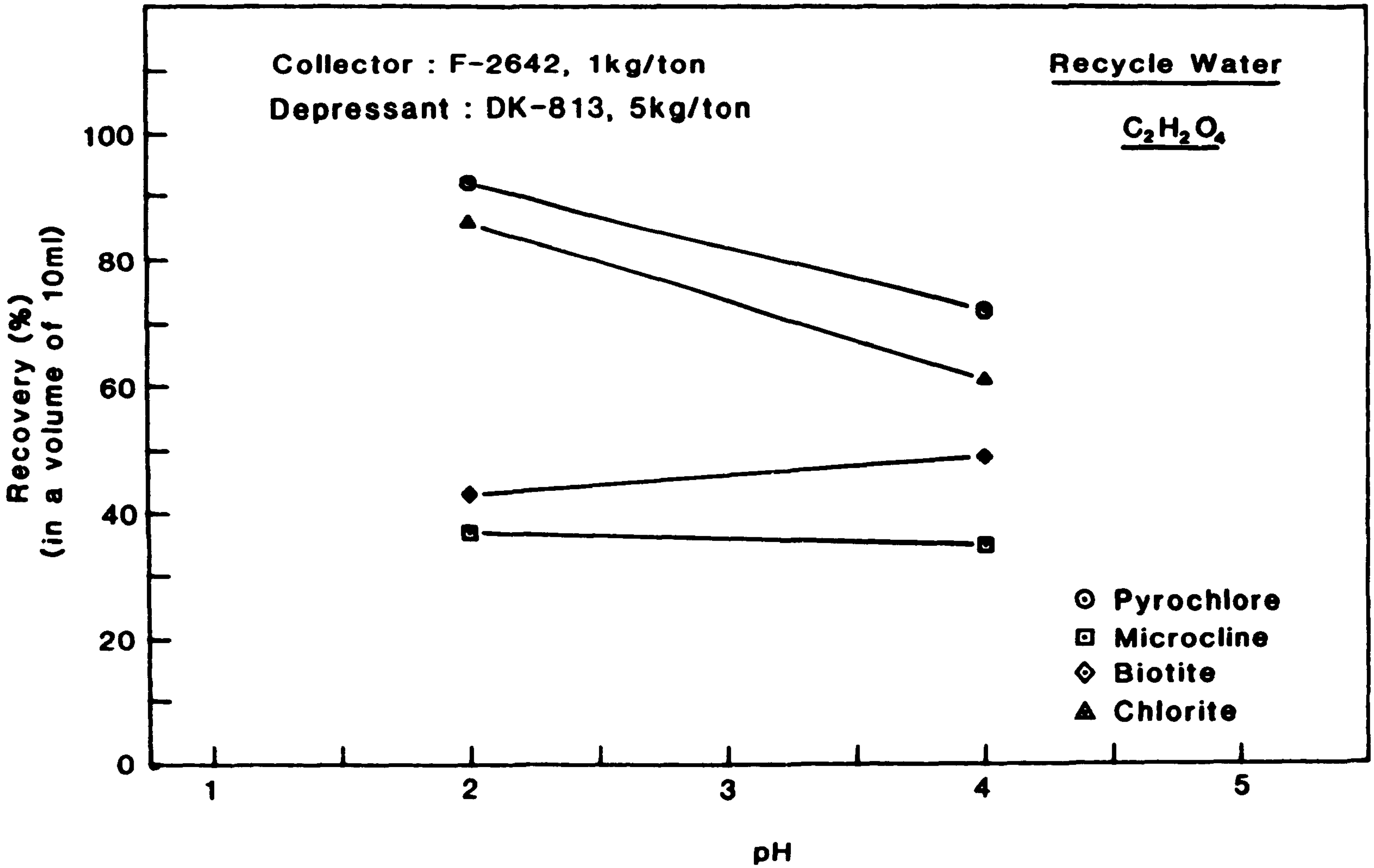

Figure 12 Flotation selectivity of pyrochlore and silicates with optimum additions of F2642 and depressant DK 813. Selection of best pH. Using recycle water

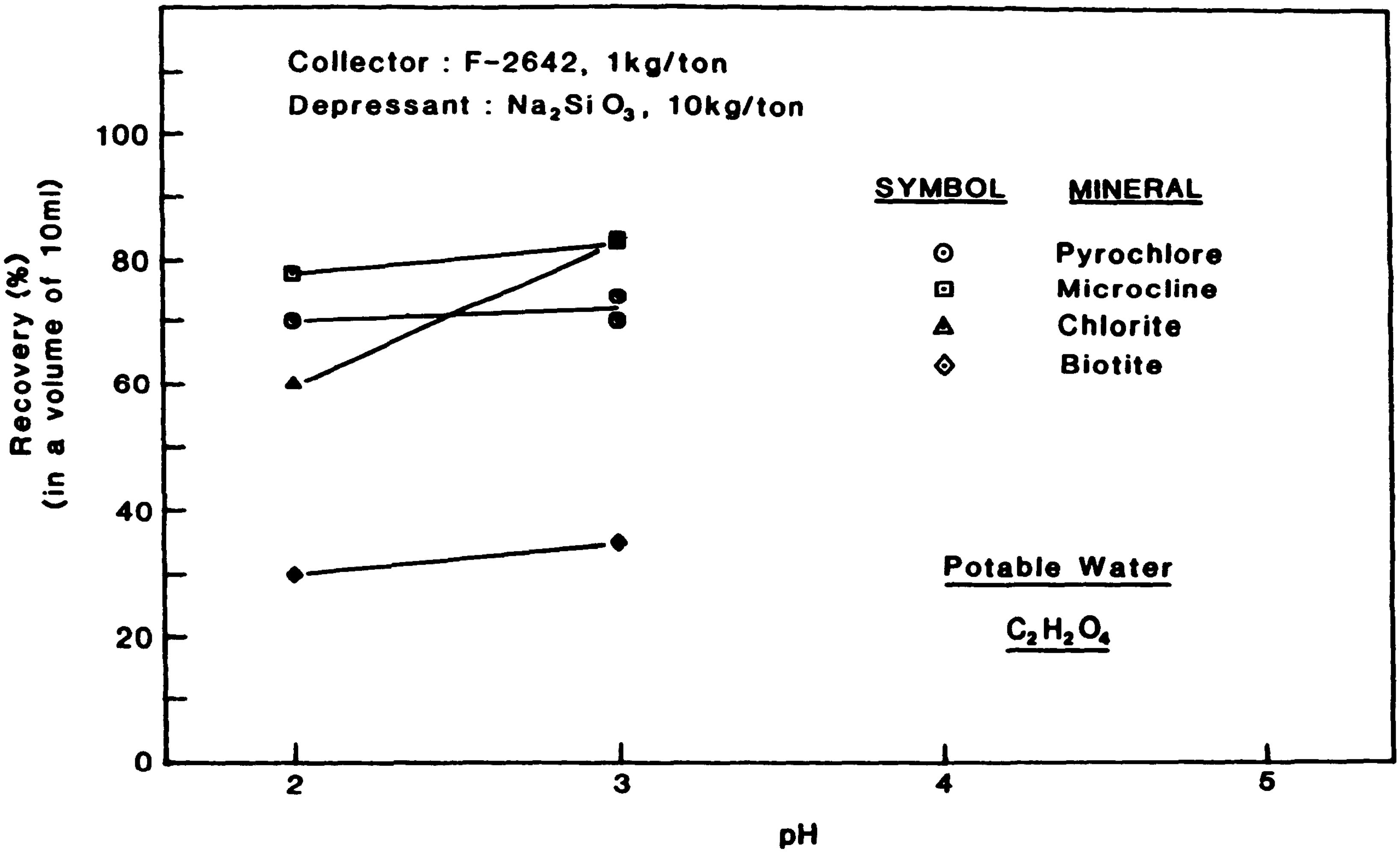

Figure 13 Flotation selectivity of pyrochlore and silicates with F2642 and sodium silicate as depressant. Potable water

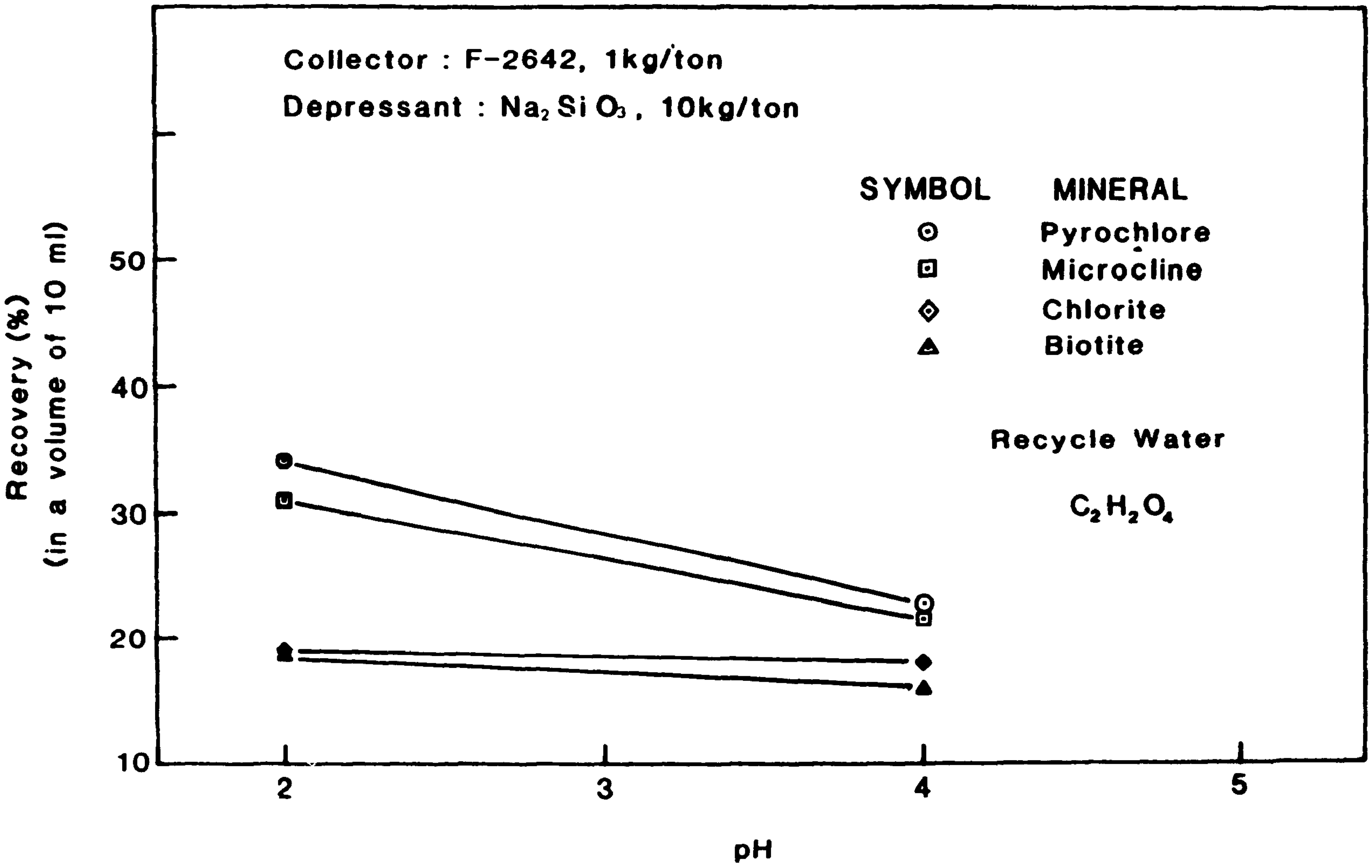

Figure 14 Flotation selectivity of pyrochlore and silicates with F2642 sodium silicate as depressant. Recycle water

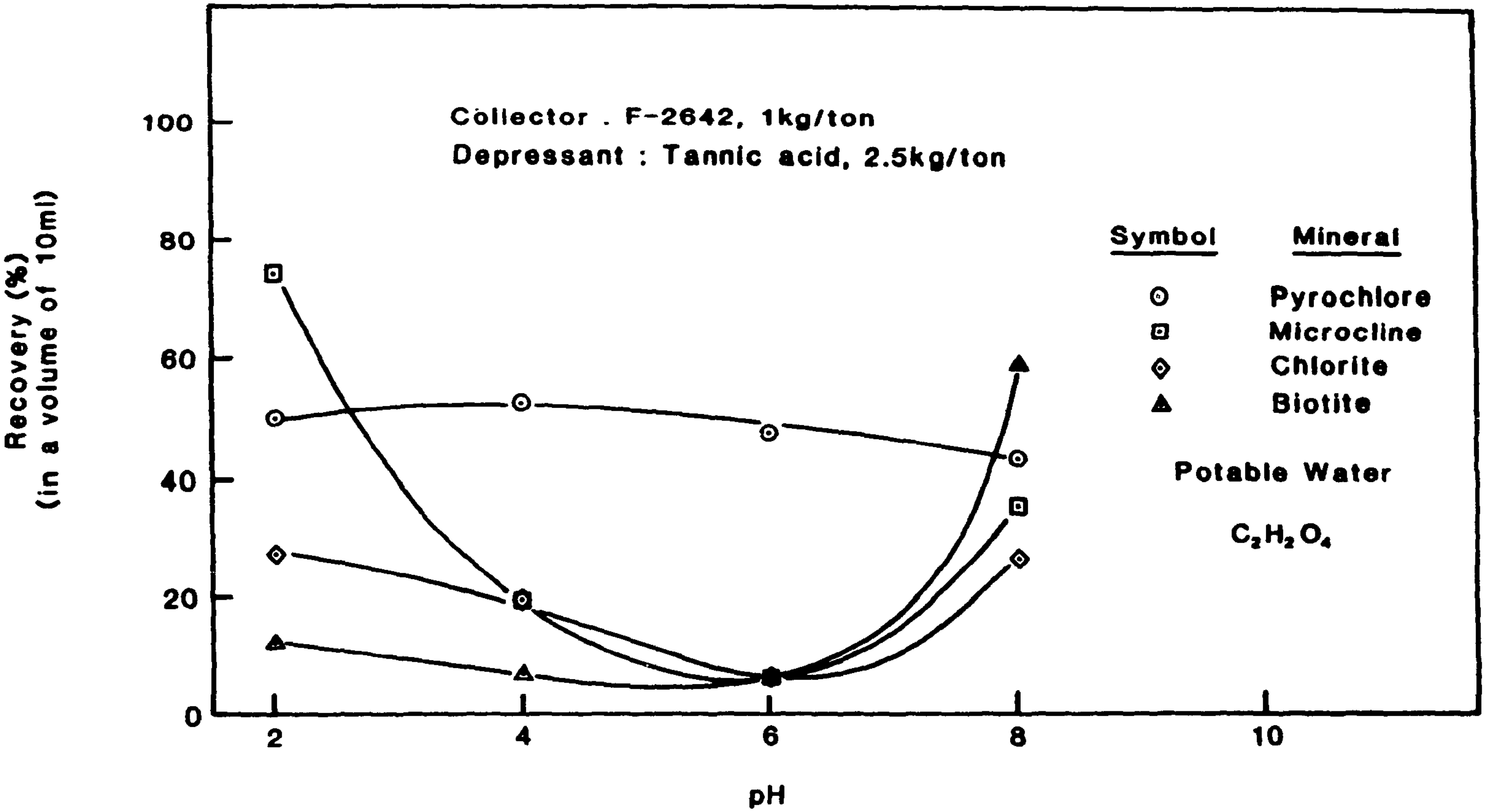

Figure 15 Flotation selectivity of pyrochlore and silicates using F2642 and tannic acid as depressant. Potable water

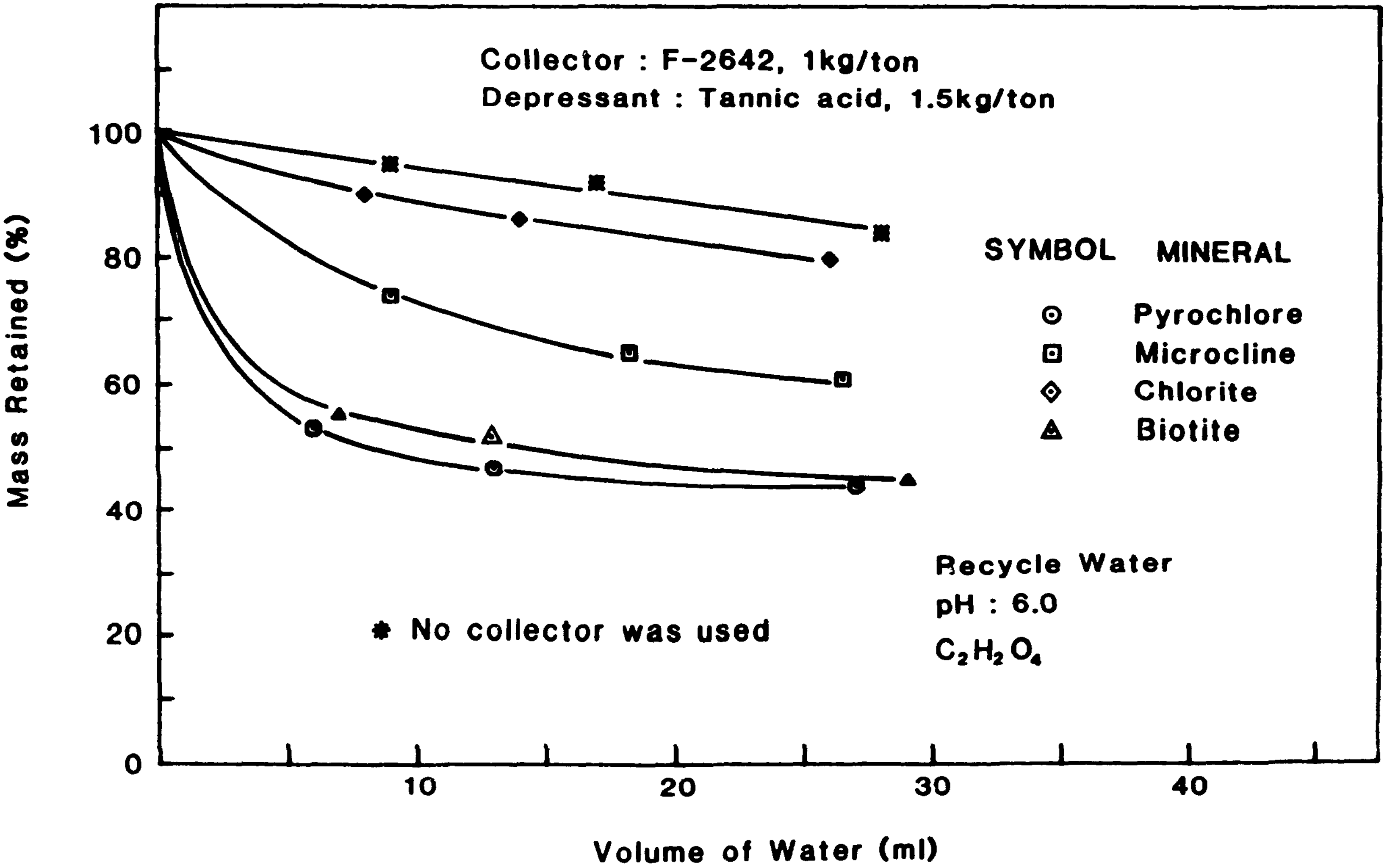

Figure 16 Flotation selectivity of pyrochlore and silicates using F2642 and tannic acid as depressant. Recycle water

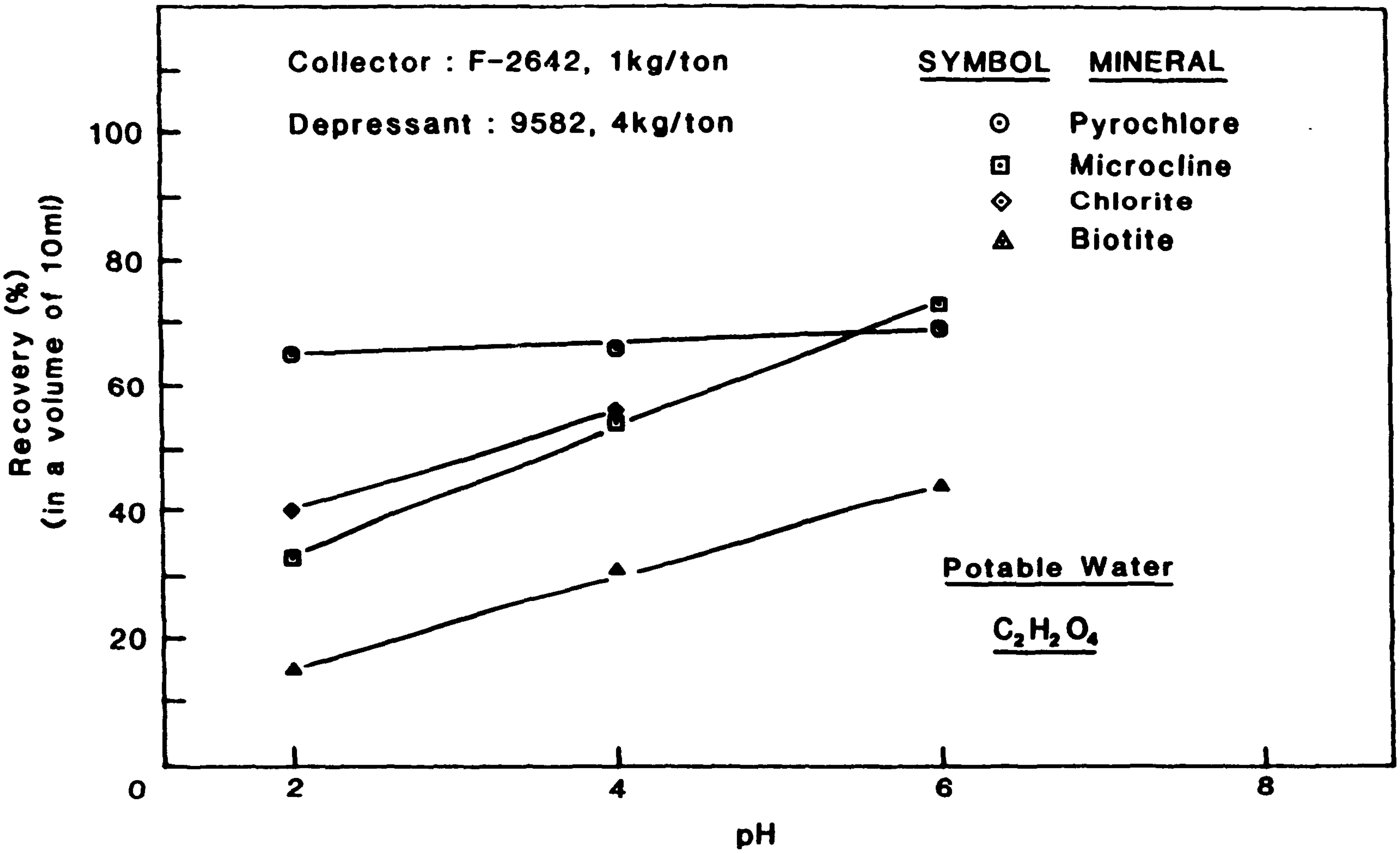

Figure 17 Flotation selectivity of pyrochlore and silicate using F2642 and depressant 9582

EFFECTS OF WATER CHEMISTRY ON THE FLOTATION OF PYROCHLORE AND SILICATE MINERALS

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Abstract

Flotation of pyrochlore and silicate minerals (albite, biotite, chlorite and microcline) in distilled water, Montreal tap water and plant recycle water from La Mine Niobec has been investigated. The recovery of pyrochlore with a secondary amine type reagent is much lower in recycle water than in the other waters. Treatment with oxalic acid (to pH~2) improves the recovery in recycle water. No significant improvement is observed when the pH is lowered by HCl or H_2SiF_6. This suggests that the precipitation of Ca^{++} present in recycle water leads to improved recovery. This is confirmed by treatment of the recycle water with a cationic exchange resin which resulted in excellent flotation of pyrochlore.

Electrokinetic measurements show that Ca^{++} and Mg^{++} cations strongly reduce the negative charge at the pyrochlore surface. This hinders adsorption of the cationic collector. A possible mechanism of collector action is discussed.

Treatment of recycle water with active carbon removes the organic matter. This improves pyrochlore selectivity by reducing the flotation of silicate minerals.

INTRODUCTION

Use of recycle water in flotation plants is becoming increasingly common. This can have significant effects on flotation recovery and selectivity since the recycle water contains many dissolved compounds, both inorganic and organic which radically alter the chemistry of the system. Specific effects have been described by many investigators [1-6]. It has been generally found that improvement of water quality leads to significantly better flotation performance.

The composition of plant recycle water depends upon its origin, the minerals being processed and the reagents used for the flotation process. For example, recycling water used for the flotation of salt-type minerals like calcite and dolomite leads to a gradual build up of Ca^{++} and Mg^{++} ions in the recycle water.

The purpose of the present work is to investigate the effects of water chemistry on the flotation of pyrochlore and silicate minerals including albite, biotite, chlorite and microcline. These minerals are important in the flotation of pyrochlore (a Nb-mineral) from associated silicate minerals at La Mine Niobec, Quebec. A simplified flowsheet of Niobec plant is shown in Figure 1. The water plant recycle water carries much higher concentrations of Ca^{++} and Mg^{++} ions and residual organics than the fresh water (referred to locally as potable water). In a previous publication [7] effects of the type of water on the selective flotation of pyrochlore from Niobec were described. The results were interpreted in terms of electrokinetic effects, hydrolysis of cations, and mineral-collector interactions. In the present work these studies have been extended to understand the nature of the chemical species present in recycle water which affect flotation. Techniques to reduce the concentration of compounds and the subsequent flotation of pyrochlore and silicate minerals are described.

Experimental Methods and Materials

Microflotation

A microflotation cell especially designed for fine particles was used. The details of construction are described in the previous publication [7]. The mineral pulp was prepared by mixing 0.5 g mineral in 50 ml water, pH was adjusted to the desired value and the flotation collector in solution was added. After conditioning for 10 minutes 0.1 ml of 10% Dowfroth 250 was added and transferred to the microflotation cell with the air already on at 74 ml/min. The froth depth was adjusted to 4 cm with the test water containing frother Dowfroth 250 (0.2 ml of 10% solution per 100 ml water). Flotation was conducted until the froth was barren, usually after 3-4 minutes. The floated product was collected, dried and weighed.

Electrokinetic measurements

Electrophoretic mobilities of pyrochlore in water in presence of Ca^{++} and Mg^{++} cations and the flotation agent HOECHST HOE-F-2642 were measured using a Mark II electrophoresis apparatus (Rank Brothers) [7].

Minerals

Pure samples of silicate minerals, albite, biotite, chlorite and microcline were obtained from Ward's Natural Science Research Establishment. The materials were ground in a laboratory ball mill. Two size fractions were isolated, 149-53 μm by screening and 14-20μm by Cyclosizing (cone 3).

Three samples of pyrochlore obtained from Niobec were investigated. Sample no. 1 was separated from the ore by tabling, and electrostatic separation. Sample no. 2 was originating in weathered brownish ore bodies and separated by tabling, magnetic separation and leaching with HCl at 60° C. Sample no. 3 was Niobec production concentrate which was leached with HCl and washed with acetone to remove adsorbed flotation agents. The chemical composition of the three samples was as follows.

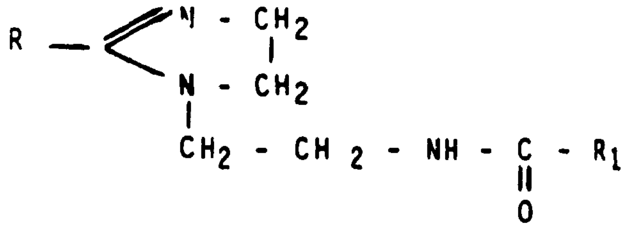
Table 1

Percent	Nb_2O_5	SiO_2	P_2O_5	CaO	Fe
Sample no. 1	48.3	1.9	1.7	12.5	5.8
Sample no. 2	60.0	0.9	0.3	8.7	4.2
Sample no. 3	67.8	0.9	0.2	10.2	1.4

Each of the samples was tested in the microflotation cell adding only frother and no collecting agent. There was no perceptible flotation which confirmed that the samples did not carry any surface activity.

Reagents

The collecting agents used for the flotation of pyrochlore at Niobec is an amine type compound, [1- alkylamidoethyl 2-alkylimidazoline], manufactured by Hoechst and sold under the Trade name HOE-F-2642 [8]. It has the following chemical composition.



R = 16 - 18 C
R_1 = 4 - 6 C

Two examples of active carbon, one high porosity (trade name WPH) and the other low porosity (trade name WPL) manufactured by Calgon Corporation were investigated for water treatment. Both were supplied in powder form.

An ion exchange resin was used specifically to remove Ca^{++} and Mg^{++}. It was a hydrogen type cationic resin manufactured by Rohm and Haas (trade name Amberlite IR-120 Plus). This is a styrene based resin produced in the form of beads. It is a high capacity resin and can be regenerated by treatment with 2% H_2SO_4 solution.

Water treatment

Water from three sources were used in the flotation studies, laboratory distilled water, Montreal tap water and plant recycle water from Niobec. Each sample was analyzed by atomic absorption spectroscopy to determine the concentrations of Ca^{++} and Mg^{++} ions. The results are in Table 2.

Table 2

Concentration of Mg^{++} and Ca^{++} in the water samples used for flotation. (ppm)

	Ca^{++}	Mg^{++}
Distilled water	<1	<1
Montreal tap water	38	2
Niobec recycle water	290	100

The plant recycle water was further analyzed to determine the concentration of organic matter. The results showed that the recycle water samples used in this investigation had a TCC (total organic carbon) value of 8.0 ppm, COD (chemical oxygen demand) 279 ppm and BOD (biochemical oxygen demand) < 5 ppm.

Hydrogen Peroxide Treatment.

Recycle water was treated with H_2O_2 to determine the effect of oxidation of organic matter. The pH of the water was lowered from its natural pH (7.8) to 6, and 2 ml 30% H_2O_2 added per 100 ml water. The water was agitated for 10 - 15 minutes. As a result of this treatment the value of COD dropped to 159 ppm.

Treatment with Active Carbon.

The recycle water was treated with active carbon to determine the possible removal of organic matter. The quantity of carbon added was in the range 1-8 g per litre of water. The weighed amount of carbon was added to 500 ml batches of recycle water and stirred for 1 hour. After the stirring was stopped the carbon was found to settle within about 20 minutes. The water was however filtered to exclude any possibility of the adsorption of flotation agent by residual carbon.

The water treated with active carbon had TOC value reduced to < 0.4 ppm and COD value to 31 ppm. There was no significant difference in results between high porosity and low porosity carbon.

Treatment with Ion Exchange Resins

Amberlite IR-120 Plus is a high capacity cationic resin which adsorbs Ca^{++} and Mg^{++} ions releasing an equivalent quantity of H^+ ion. About 100 g resin was packed in a laboratory ion exchange column (25 cm height, 3 cm diameter). The recycle water was allowed to percolate through the resin bed. The water thus treated had negligible concentrations (< 1 ppm) of Ca^{++} and Mg^{++} ions. The pH of the water dropped to < 1.8.

The ion exchange resin could be used repeatedly. When saturated with Ca^{++} and Mg^{++} ions the resin was regenerated by treatment with 2% H_2SO_4.

Results

Flotation of pyrochlore. Effect of reagent concentration.

Effect of the quantity of collector on the flotation of pyrochlore is shown in Figure 2. The results were better with distilled water. Maximum recovery of about 55 - 60 percent was achieved with 1.0 mg collector per g mineral in tap water while in distilled water only 0.5 mg collector per g mineral was required.

Flotation was very poor in plant recycle water under similar condition, e.g. with 1.0 mg collector per g mineral, only 20% pyrochlore was floated. A much larger quantity of reagent was required to obtain recovery similar to that in distilled water.

In general, the flotation recovery of iron pyrochlore (Sample 2) is lower than that of sodium pyrochlore (Sample 1). Samples 1 and 3, both sodium pyrochlore showed virtually same recoveries. Poor flotation of iron pyrochlore is also noted in the plant.

Flotation of pyrochlore. Effect of pH.

The effect of pH on the flotation of pyrochlore is shown in Figure 3 for distilled and tap water and in Figure 4 for recycle water. With tap water and distilled water the effect of pH in the range 2 - 6 on the recovery of pyrochlore is negligible. However, with recycle water lowering of pH increases the recovery of pyrochlore, the increase being greater when the pH is lowered by oxalic acid.

At Niobec, the flotation pH is progressively decreased from 6.5 to ≃ 2.5 in the cleaning circuit, using fluosilicic and oxalic acids.

Flotation of Silicate Minerals.

Flotation recoveries of the four silicate minerals, albite, biotite, chlorite and microcline as a function of reagent concentration in tap water and in distilled water are recorded in Figure 5. Flotation recoveries in recycle water are shown in Figure 6. Recoveries are higher in recycle water. Small differences are observed between tap and distilled water.

The effect of pH on the flotation of silicate minerals in tap water and distilled water is shown in Figure 7. The results for flotation in recycle water are shown in Figure 8. In all cases the recovery of silicates decrease with decrease in pH except biotite which shows minimum recovery at pH 4. Recovery with oxalic acid is slightly lower than with HCl, the opposite to the finding for pyrochlore (Figure 4). Again, the removal of Ca^{++} by precipitation with oxalic acid was considered the cause.

Effect of Electrolytes.

In order to test the hypothesis regarding the role of Ca^{++}, sodium carbonate was added in slight excess and $CaCO_3$ and $MgCO_3$ precipitates removed. This treatment lowered the Ca^{++} and Mg^{++} concentrations to 6 and 15 ppm respectively. But the recovery of pyrochlore was still low (Table 3). This was thought to be due to the introduction of Na^+ ions.

To explore this Na_2CO_3 was dissolved in distilled water (0.15g g/100 ml) and the pH adjusted to 6 with HCl or H_2SO_4. This treatment resulted in the release of Na^+ equivalent amounts to (Ca^+, Mg^{++}) in the recycle water. Flotation results with this water are recorded in Table 3. The recovery of pyrochlore was poor and is comparable to that obtained with untreated recycle water.

Table 3

Flotation of pyrochlore in distilled water with dissolved chloride and sulphate at pH6.

Water	Percent Recovery	
	Sodium Pyrochlore	Iron Pyrochlore
Distilled Water (no salt added)	86	26
Recycle water (untreated)	26	21
Recycle water + Na_2CO_3	28	20
Distilled water + chloride	28	15
Distilled water + sulphate	38	18

These results indicate the deleterious effect on pyrochlore flotation of electrolytes in general (rather than just Ca^{++}).

Effect of Treatment with Active Carbon.

Figure 9 shows the results of flotation of pyrochlore and silicate minerals in recycle water at pH 6 treated with increasing quantities of active carbon. The results show that there is no significant improvement in the flotation of pyrochlore with up to 8 g active carbon per litre of water. However, there is a reduction in the recovery of silicate minerals by up to 40% (absolute) in the case of chlorite. The quantity of active carbon required to get maximum depression of silicates appears to be about 6 g per litre of water.

Figure 10 shows the effect of pH on the flotation of pyrochlore and silicate minerals in recycle water treated with active carbon (6 g per litre of water). The reduced recovery of silicates is maintained (compare with Figure 8) while that of pyrochlore is largely unaltered (compare witn Figure 4). Biotite again shows a minimum recovery at pH 4.

Effect of Treatment with Ion Exchange Resin.

The ion exchange treated water showed a pH of 1.8 due to replacement of Ca^{++}, Mg^{++} with H^{+}. Flotation of pyrochlore was very poor (< 5% recovery) in such strongly acidic water. The pH of this water was raised by NaOH. The results obtained for pyrochlore with ion exchange treated water are compared with those with untreated water in Table 4.

Table 4

Comparison of the results of pyrochlore flotation in untreated recycle water and the recycle water treated with ion exchange resin.

pH	Percent Recovery	
	Untreated Water	Water treated with ion exchange resin
2	76	84
4	26	74
6	15	66

Recovery in treated water is significantly higher. Maximum recovery is still at pH 2. It drops by 10 - 15% when the pH is raised above 2; this is possibly related to the "electrolyte" effect mentioned before.

The results obtained with the ion exchange treated water for various silicate minerals are shown in Figure 11. The pH effect follows the same trend as with untreated water (Figure 8) but now pyrochlore recovery is constantly higher than silicate recovery.

Electrokinetic measurements.

The results of the electrokinetic measurement represented by the variation of zeta potential with ionic or reagent concentration are shown in Figures 12 and 13. Figure 12 shows the variation of zeta potential of pyrochlore with increasing concentraions of Ca^{++} and Mg^{++} ions. Increasing Ca^{++} and Mg^{++} ion concentration makes the zeta potential less negative which indicates adsorption of Ca^{++} and Mg^{++} cations.

Figure 13 shows the effect of the reagent HOECHST F 2642 on the zeta potential of pyrochlore at pH 6. The initially negative zeta potential of pyrochlore is sharply changed to a positive values with only 5 ppm of reagent.

DISCUSSION

The flotation of pyrochlore is very poor in recycle water at its natural pH ($\sim$7.5). The recovery increases when the pH is lowered by oxalic acid used at Niobec, but no significant improvement is observed if the pH is lowered by HCl or by H_2SiF_6. This suggests that the precipitation of Ca^{++} ions in the recycle water leads to better flotation of pyrochlore.

In the tests conducted to precipitate Ca^{++} by Na_2CO_3 the recovery of pyrochlore did not improve significantly. The release of an equivalent quantity of Na^+ was thought to be responsible for this. The results obtained with distilled water with dissolved NaCl and Na_2SO_4 showing low recovery of pyrochlore confirmed this and suggest an 'electrolyte effect' which hinders the flotation of pyrochlore.

Removal of Ca^{++} and Mg^{++} ions by ion exchange resin gave excellent recovery of pyrochlore at pH 2 - 2.5. The recovery drops when the pH is raised. This is consistent with the negative influence of 'electrolyte effect' as Na^+ is introduced from the NaOH used to increase pH.

The reagent HOECHST F 2642 has a secondary amine group. This would make it a cationic collector functioning over a pH range 2 - 6 in water free of electrolytes.

The electrokinetic measurements show pyrochlore is negatively charged, and it changes to positive on reaction with the collector. This change is consistent with the cationic nature of the reagent and an electrostatic mechanism of adsorption. Adsorption would be hindered if the pyrochlore surface charge is increased by some other agent, and the electrokinetic measurements show Ca^{++} and Mg^{++} to increase the charge.

It seems to be a little surprising that the cationic collector functions at pH down to nearly 2 (in the absence of electrolytes) since increasing H^+ ion concentration would increase positive charge on the surface. It is suggested that the secondary amine group interacts with the H^+ ions at the mineral/solution interface forming aminium species. The chemical interaction energy overcomes electrostatic repulsion at pH down to nearly 2. Only at extremely low pH (<< 2), as occurs with the ion exchange treated recycle water does the positive surface charge become sufficiently high that adsorption of the cationic reagent is hindered.

Effect of Organics.

Treatment with active carbon significantly lowers the concentration of organic matter in recycle water. This does not lead to significant improvement in the flotation recovery of pyrochlore but it is beneficial in reducing the flotation of silicate minerals. This means improved selectivity.

The exact composition of the organic matter is not known. It would probably consist of degradation products of reagents used in different flotation stages and generated in tailing ponds. Their presence could have a negative effect on flotation performance by promoting the flotation of silicate minerals with pyrochlore thus reducing selectivity. Treatment with active carbon leads to a large removal of these organics resulting in improved selectivity.

Water treatment, implication for metallurgy.

A combination of active carbon treatment to remove organics and reduce silicate flotation and ion exchange to remove Ca^{++}, Mg^{++} and increase pyrochlore recovery, appears to be means of improving selective flotation of pyrochlore. This is seen from Table 5.

Table 5

Flotation of Pyrochlore and Silicate Minerals in Active Carbon - and Ion Exchange Treated Water after Exposure to Untreated Recycle Water.

Mineral	pH 2.2 Percent Recovery		pH 4 Percent Recovery	
	After exposure to untreated recycle water	Without exposure to untreated water	After exposure to untreated recycle water	Without exposure to untreated water
Pyrochlore	80	84	71	74
Albite	46	34	65	58
Biotite	55	50	34	21
Chlorite	44	36	32	26
Microcline	38	26	60	52

In practice this will probably mean active carbon treatment of the recycle water before contact with the minerals; (stage CF in Figure 1) once adsorbed the organics causing some silicate flotation will be difficult to remove. Ion exchange treatment, on the other hand, could be conducted in the pyrochlore flotation circuit (stage PF in Figure 1). Since carbonates are present in the ore, control of Ca^{++}, Mg^{++} concentration will be difficult unless most of the carbonates have been previously removed.

Pyrochlore/silicate selectivity will still require a progressive decrease in pH. At pH 6, biotite can be removed so that its high recovery at pH 2 is not a problem. As pH is reduced recovery of the other silicates decreases while that of pyrochlore increases. Progressive pH decrease is also required to minimise solubilizing Ca^{++}, Mg^{++}. The ion exchange resin also acts as a pH modifier; it would be necessary to balance its pH and Ca removal effects.

Summary and Conclusions

The present study on the effect of water chemistry on the flotation of pyrochlore and silicate minerals leads to the following conclusions.

1. Flotation of pyrochlore by 1-alkylamidoethyl-2-alkylimidazoline which has an -NH group is adversely affected by the presence of Ca^{++} and Mg^{++} ions. An 'electrolyte effect' which causes low recovery in presence of any strong electrolyte (e.g., NaCl or Na_2SO_4) is also observed.

2. The presence of relatively high concentrations of Ca^{++} and Mg^{++} in plant recycle water causes low recovery of pyrochlore. The recovery is improved by lowering the pH of the recycle water from 6 to nearly 2 by oxalic acid which precipitates most of the Ca^{++} ions. Lowering of pH by HCl does not lead to significantly improved recovery since Ca^{++} are not precipitated.

3. The adverse effect on pyrochlore flotation due to Ca^{++} ions is related to the surface charge at the mineral becoming less negative. This hinders the adsorption of the secondary amine which functions as a cationic collector.

4. The removal of Ca^{++} and Mg^{++} by ion exchange resin leads to exellent flotation of pyrochlore at pH 2 - 3.

5. Removal of organic matter present in the recycle water helps pyrochlore selectivity (lowering the flotation of silicate minerals). [This could be useful in improving the selectivity of pyrochlore flotation.]

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Captions of Figures

Figure 1	Simplified flowsheet of processing of Niobec ore.
Figure 2	Flotation of pyrochlore in different water types. Effect of collector concentration at pH 6.
Figure 3	Effect of pH on the flotation of pyrochlore at collector concentration 0.5 mg/g.
Figure 4	Effect of pH on the flotation of pyrochlore in Niobec recycle water. Collector concentration 1.0 mg/g.
Figure 5	Effect of reagent concentration on the flotation of silicate minerals at pH 6.
Figure 6	Flotation of silicate minerals in recycle water at pH 6. Effect of collector concentration.
Figure 7	Flotation of silicate minerals in tap water and in distilled water. Collector concentration 1.0 mg/g.
Figure 8	Flotation of silicate minerals in recycle water. Effect of pH. Collector concentration 1.0 mg/g.
Figure 9	Flotation of pyrochlore and silicate minerals in recycle water (pH 6) treated with active carbon (Collector, HOECHST F 2642, 1 mg/g).
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Figure 11	Effect of pH on the flotation of pyrochlore and silicate minerals in recycle water treated with ion exchange resin.
Figure 12	Effect of Ca^{++} and Mg^{++} on surface charge of pyrochlore (pH 6).
Figure 13	Effect of HOECHST F 2642 on surface charge of pyrochlore (pH 6).

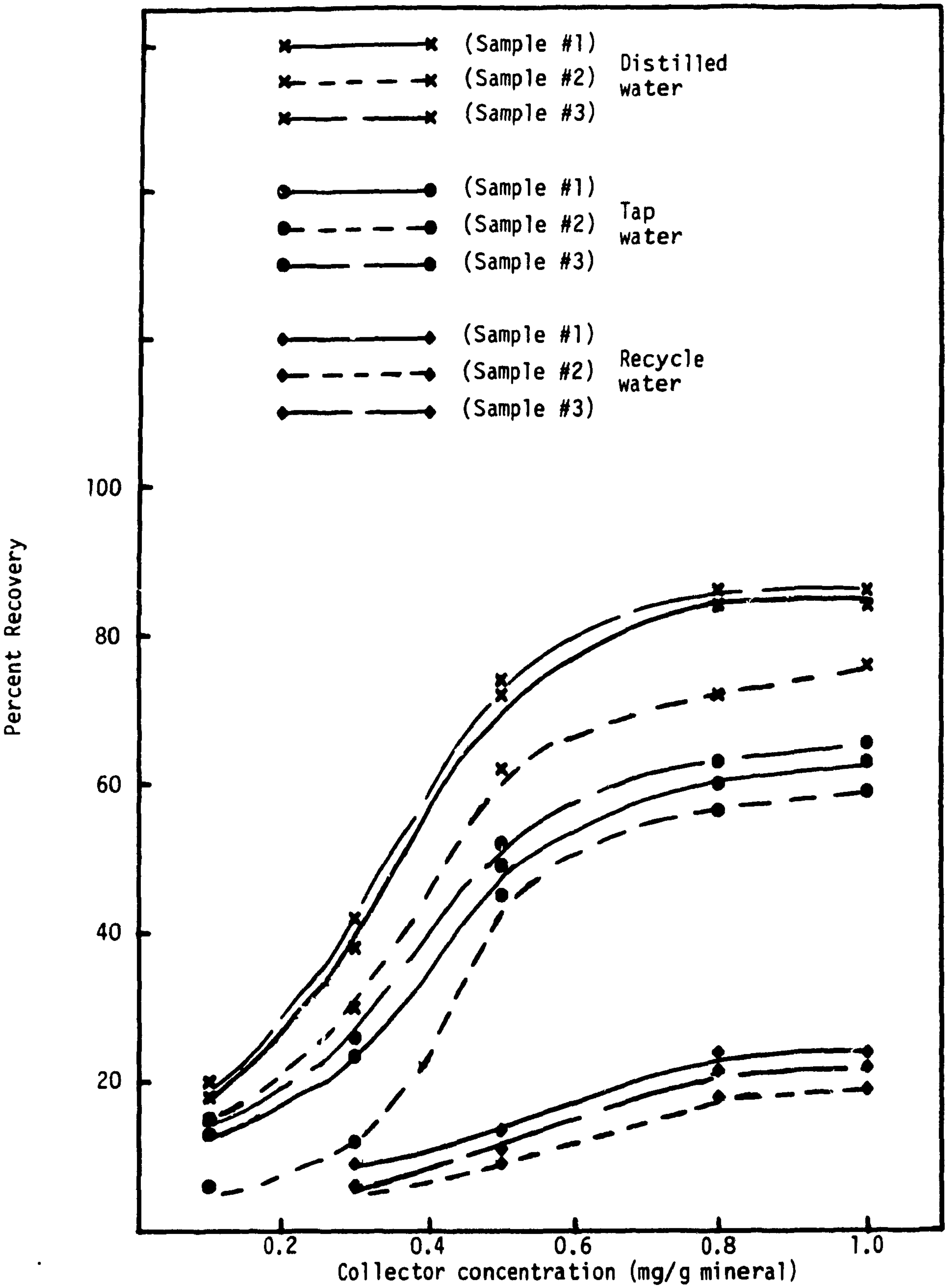


Fig. 2 Flotation of pyrochlore in different water types. Effect of collector concentration at pH 6.

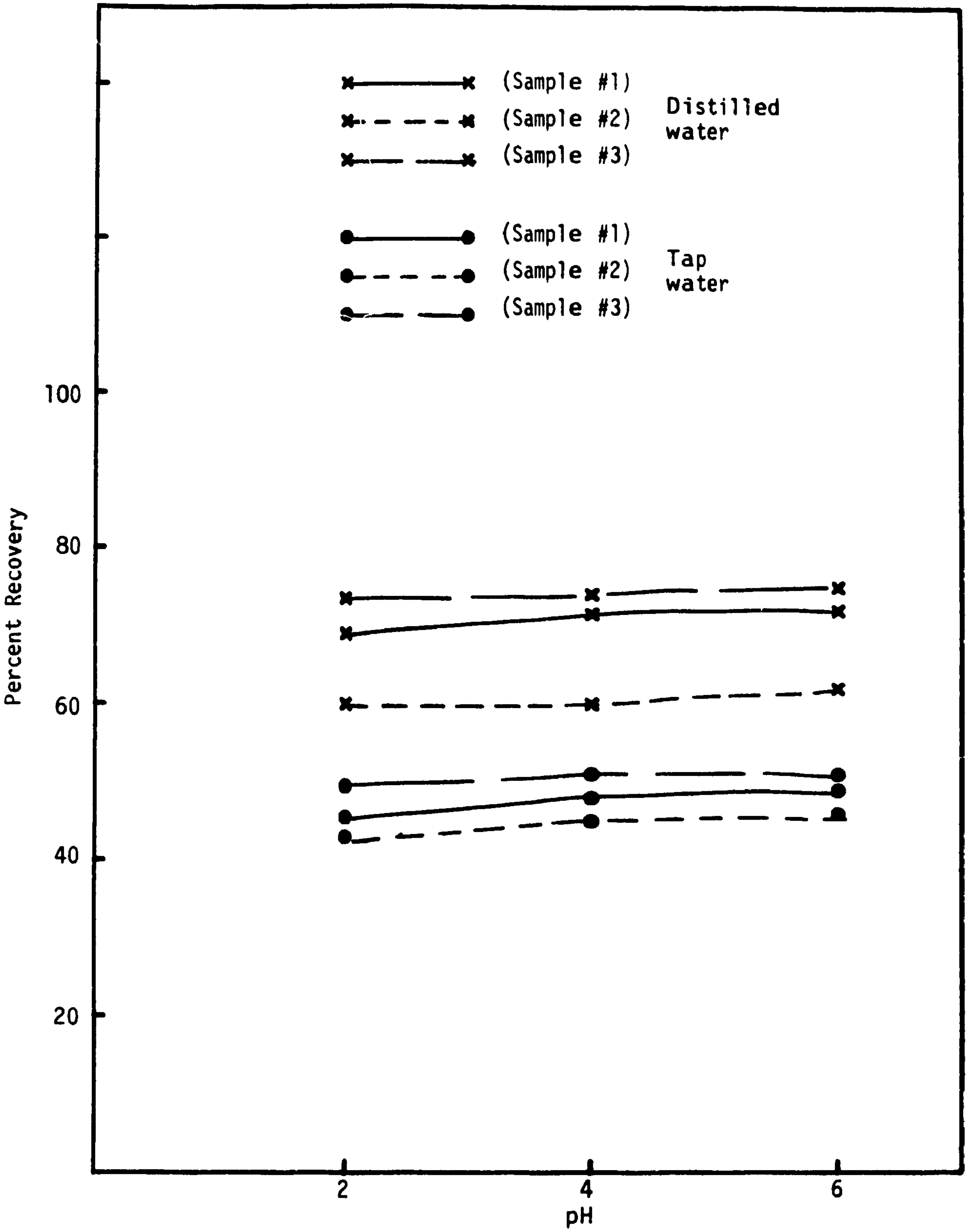


Fig. 3 Effect of pH on the flotation of pyrochlore at collector concentration 0.5 mg/g

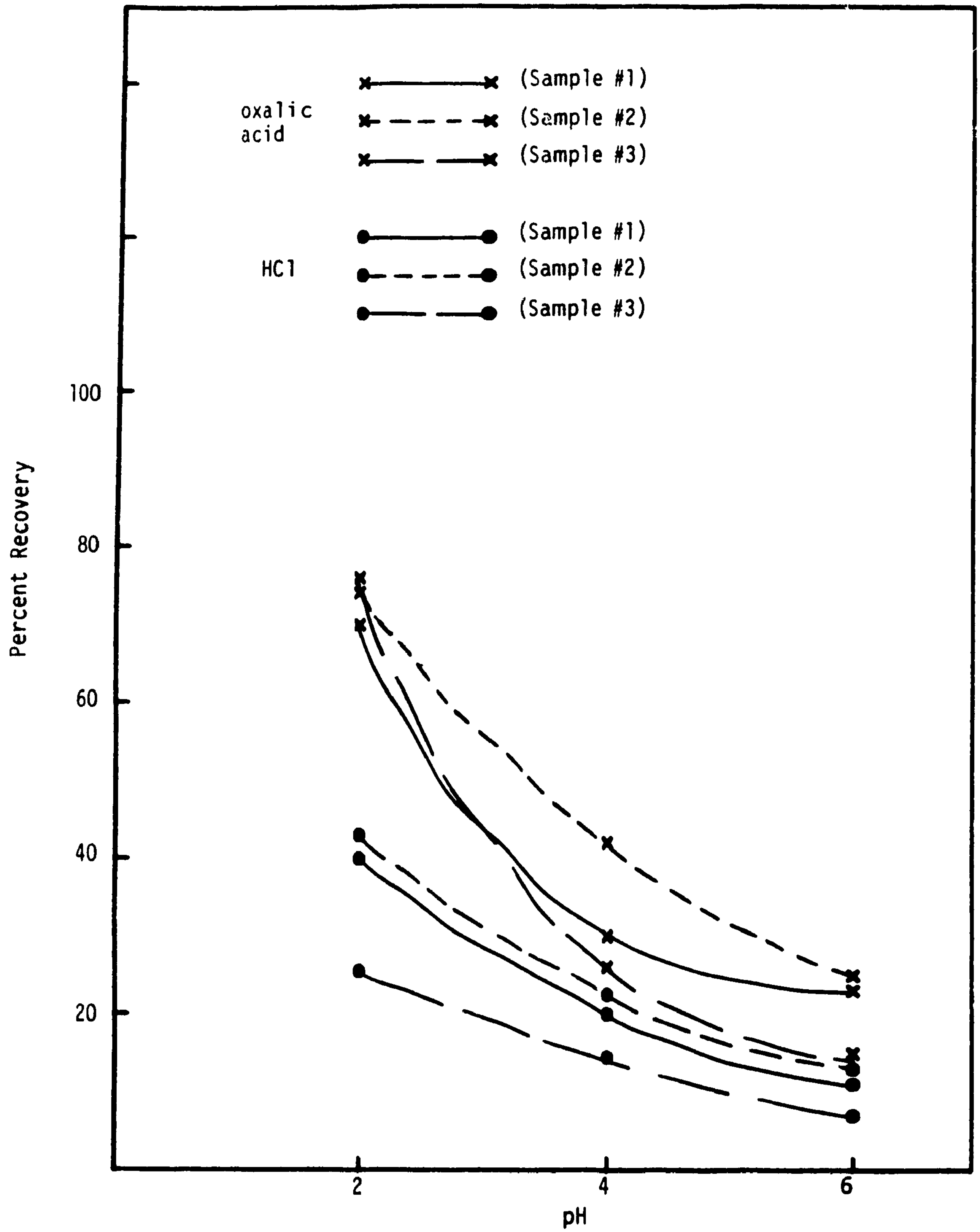


Fig. 4 Effect of pH on the flotation of pyrochlore in Niobec recycle water. Collector concentration 1.0 mg/g.

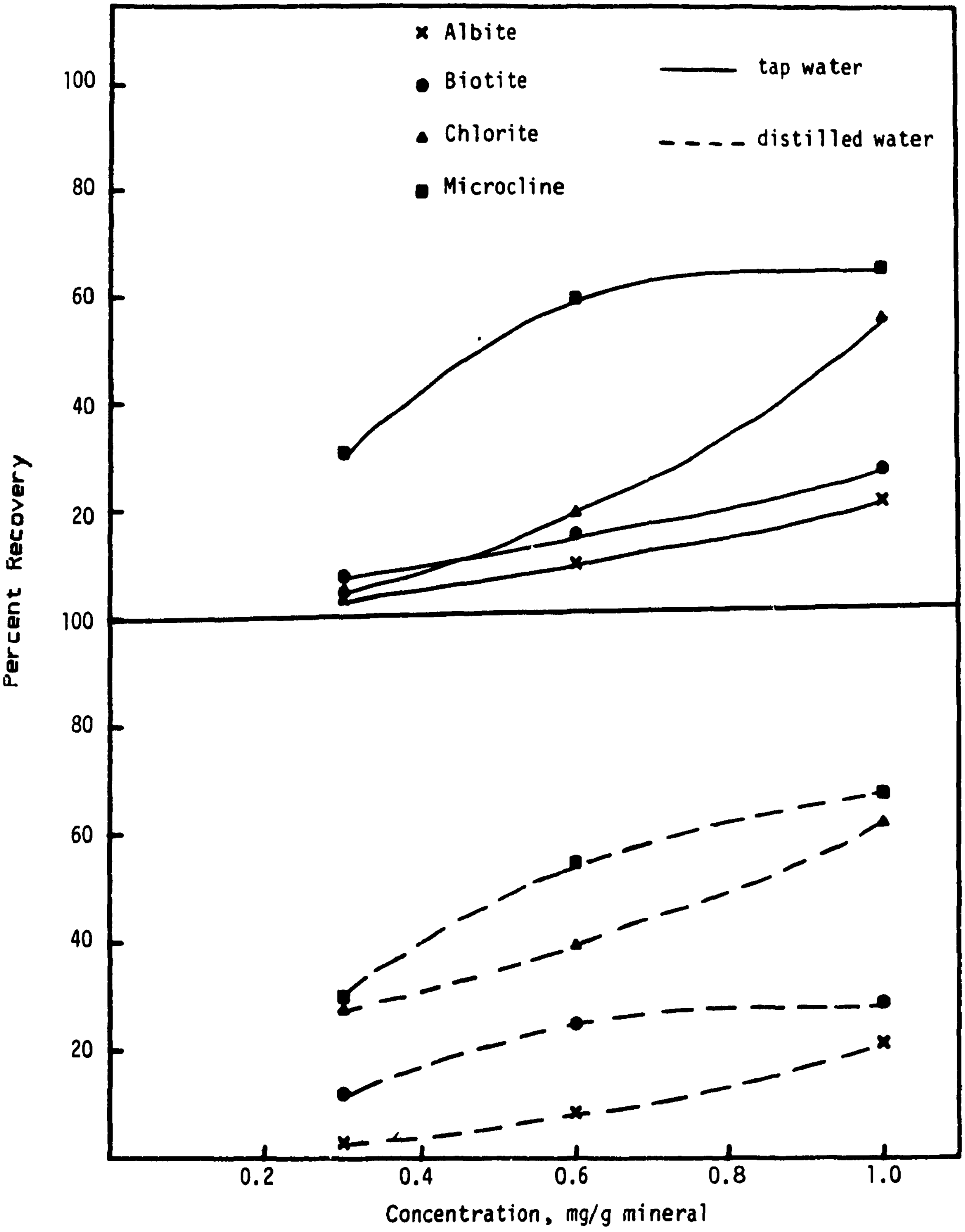


Fig. 5 Effect of reagent concentration on the flotation of silicate minerals at pH 6

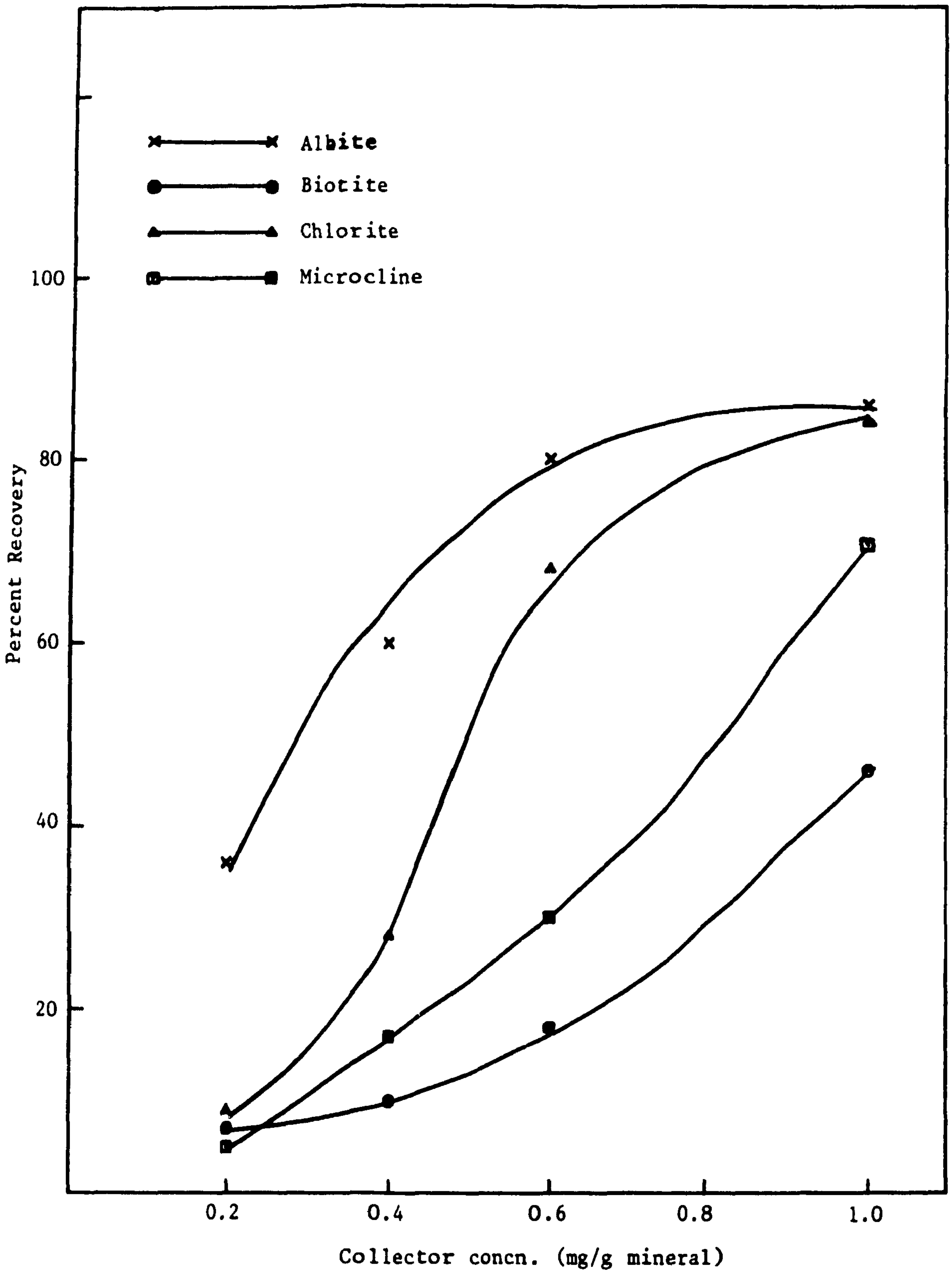


Figure 6 Flotation of silicate minerals in recycle water at pH 6. Effect of collector concentration.

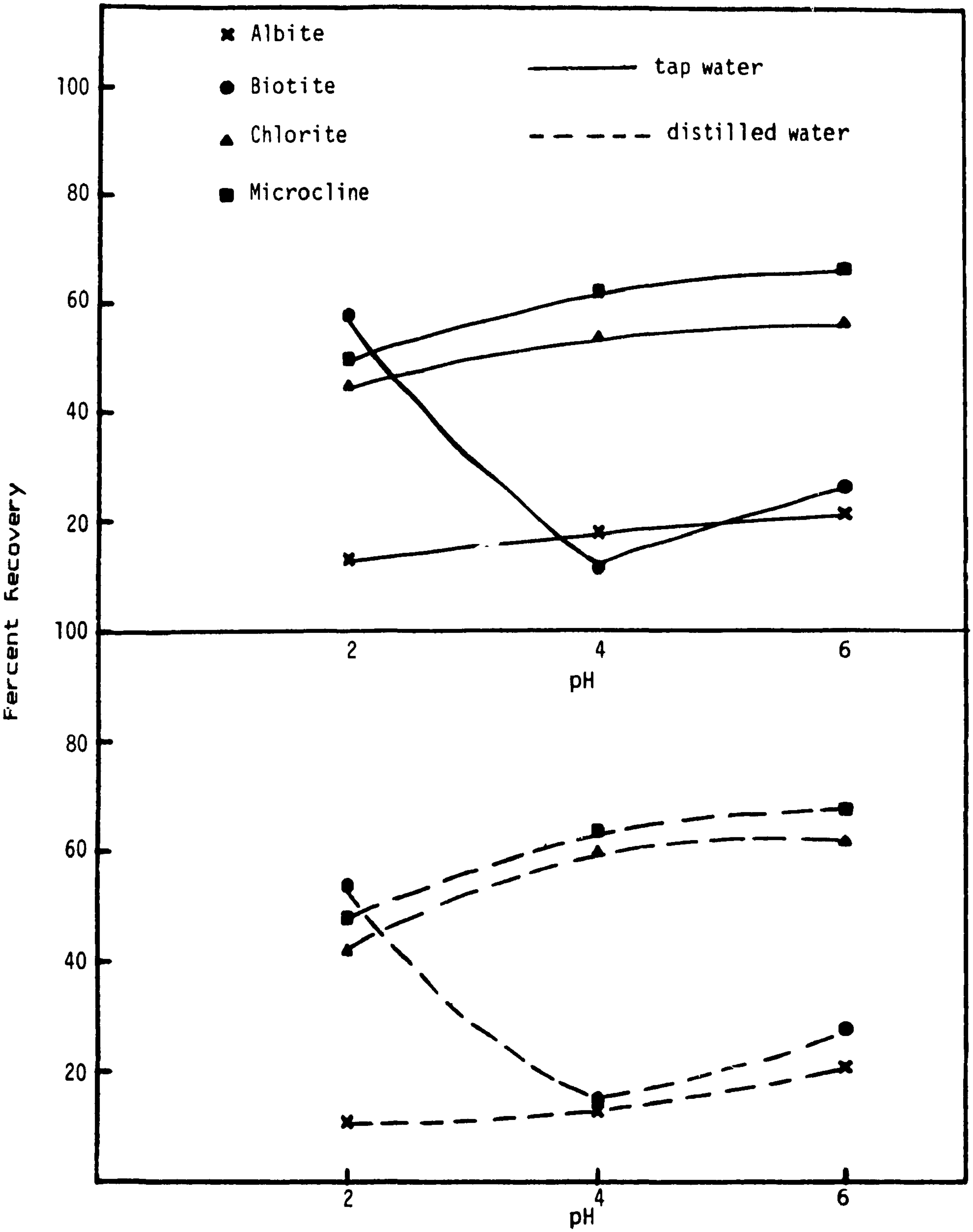


Fig. 7 Flotation of silicate minerals in tap water and in distilled water. Collector concentration 1.0 mg/g.

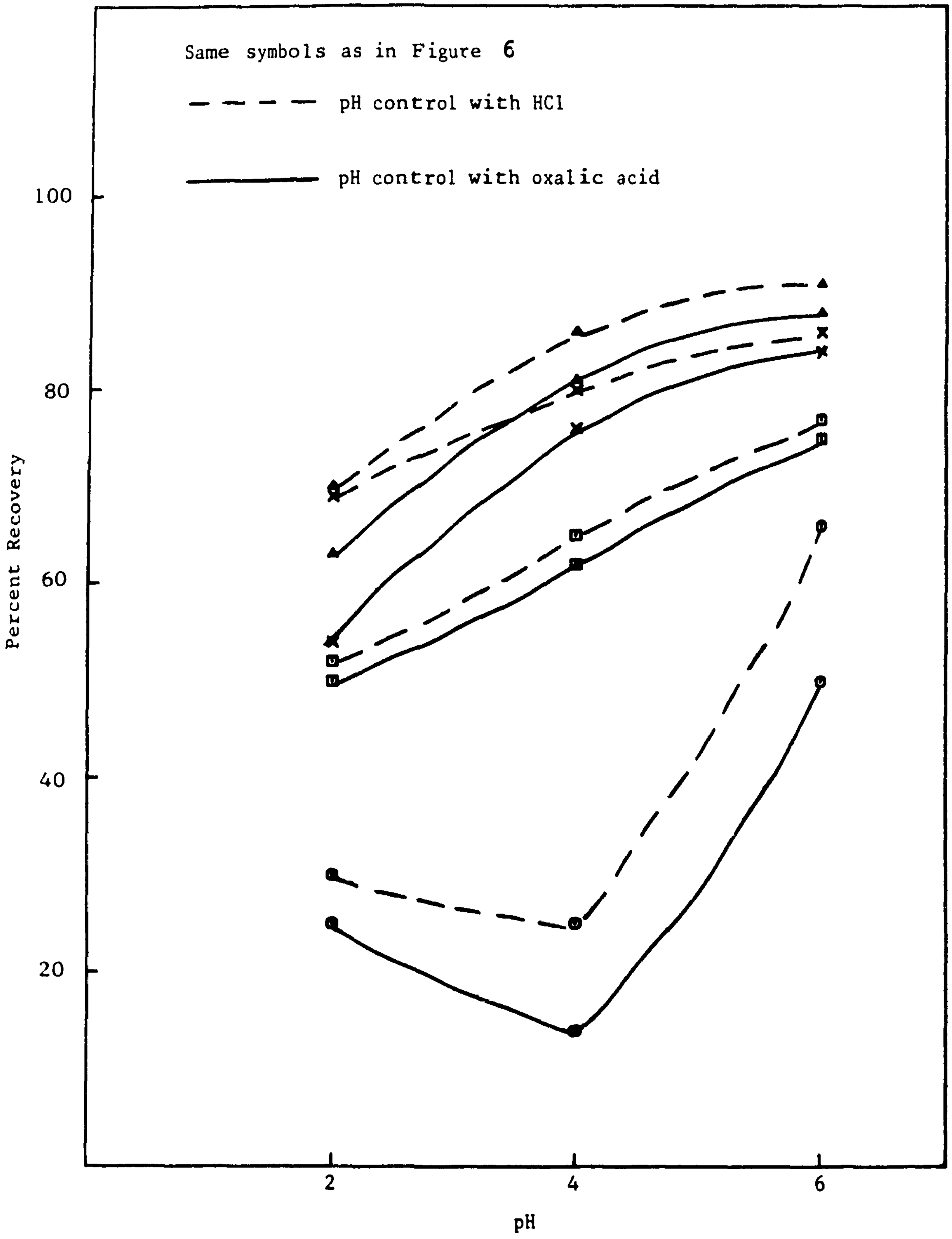


Figure 8 Flotation of Silicate minerals in recycle water. Effect of pH. Collector concentration 1.0 mg/g.

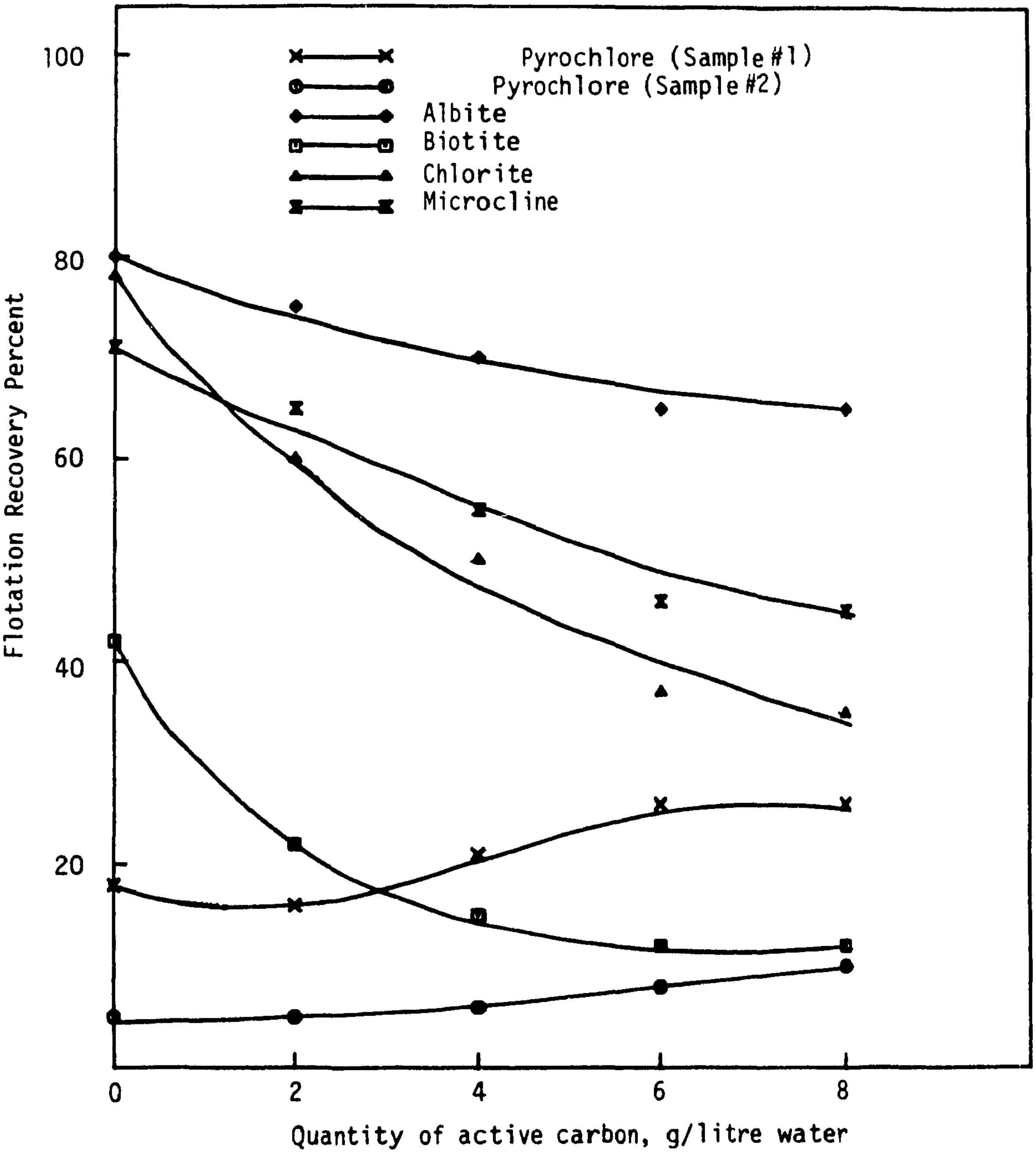


Figure 9. Flotation of pyrochlore and silicate minerals in recycle water (pH 6) treated with active carbon (Collector, HOECHST F 2642, 1 mg/g).

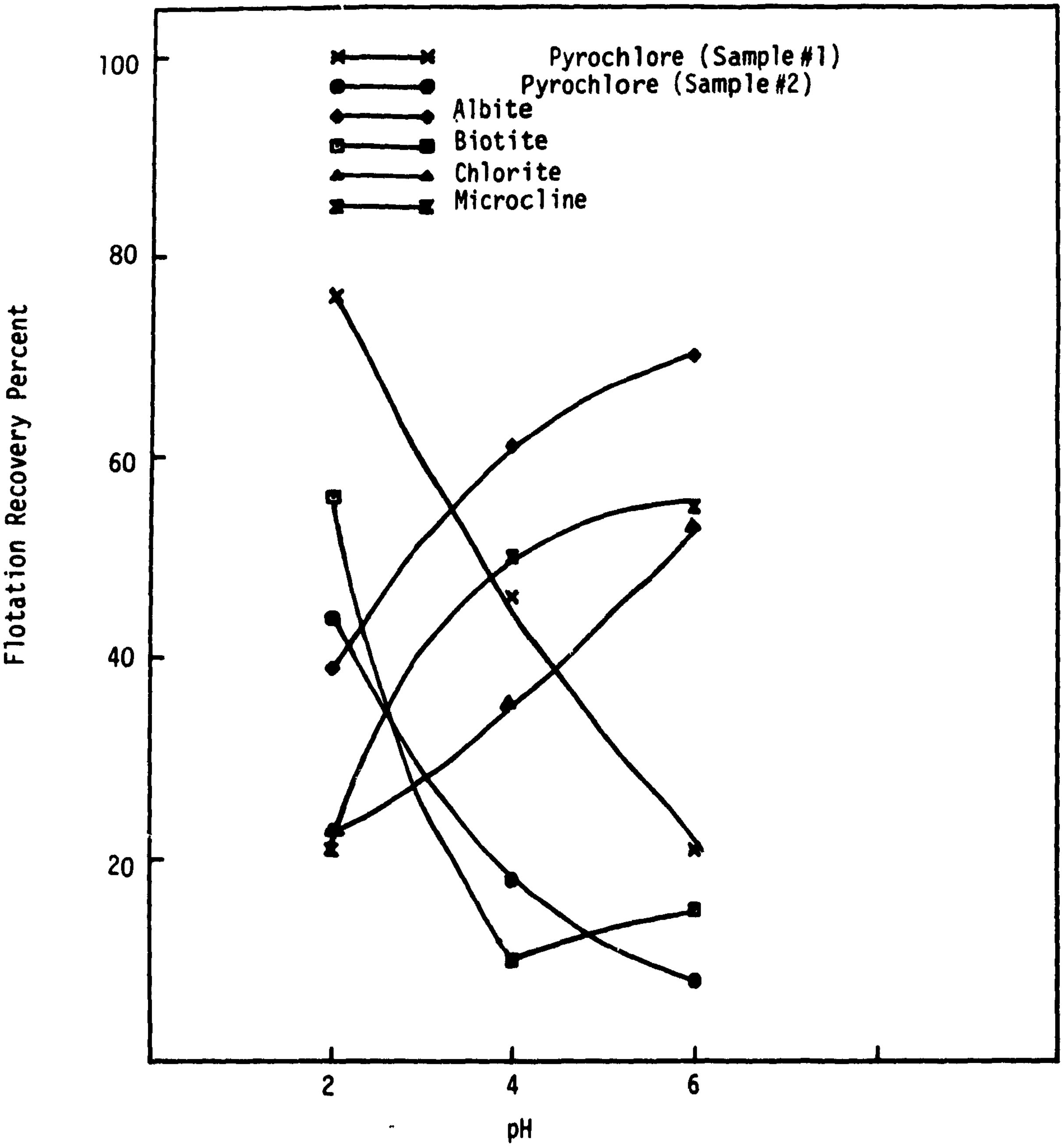


Figure 10 Effect of pH on the flotation of pyrochlore and silicate minerals in recycle water treated with active carbon (6g/litre water). (Collector, HOECHST F 2642, 1 mg/g).

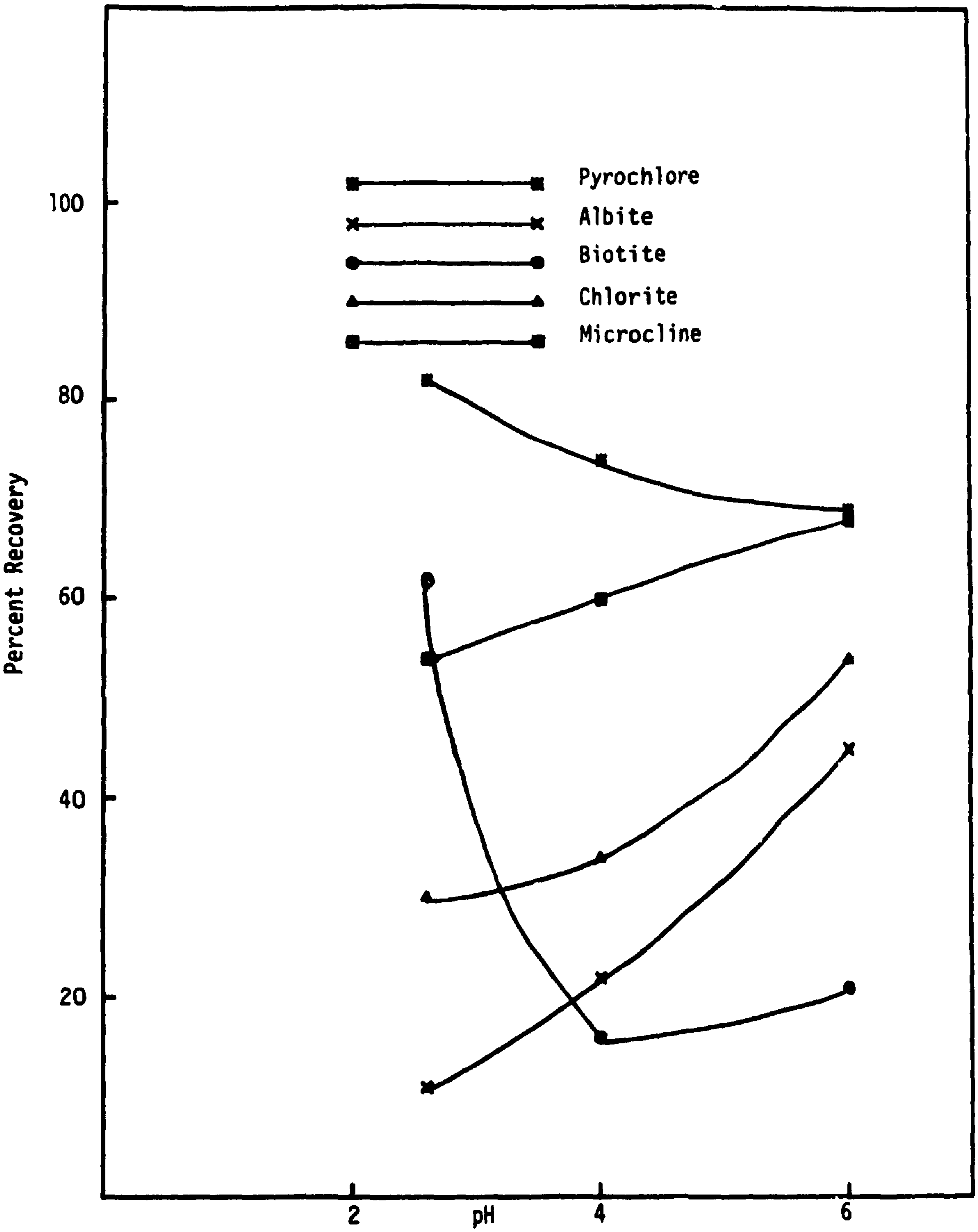


Figure 11

Effect of pH on the flotation of pyrochlore and silicate minerals in recycle water treated with ion exchange resin.

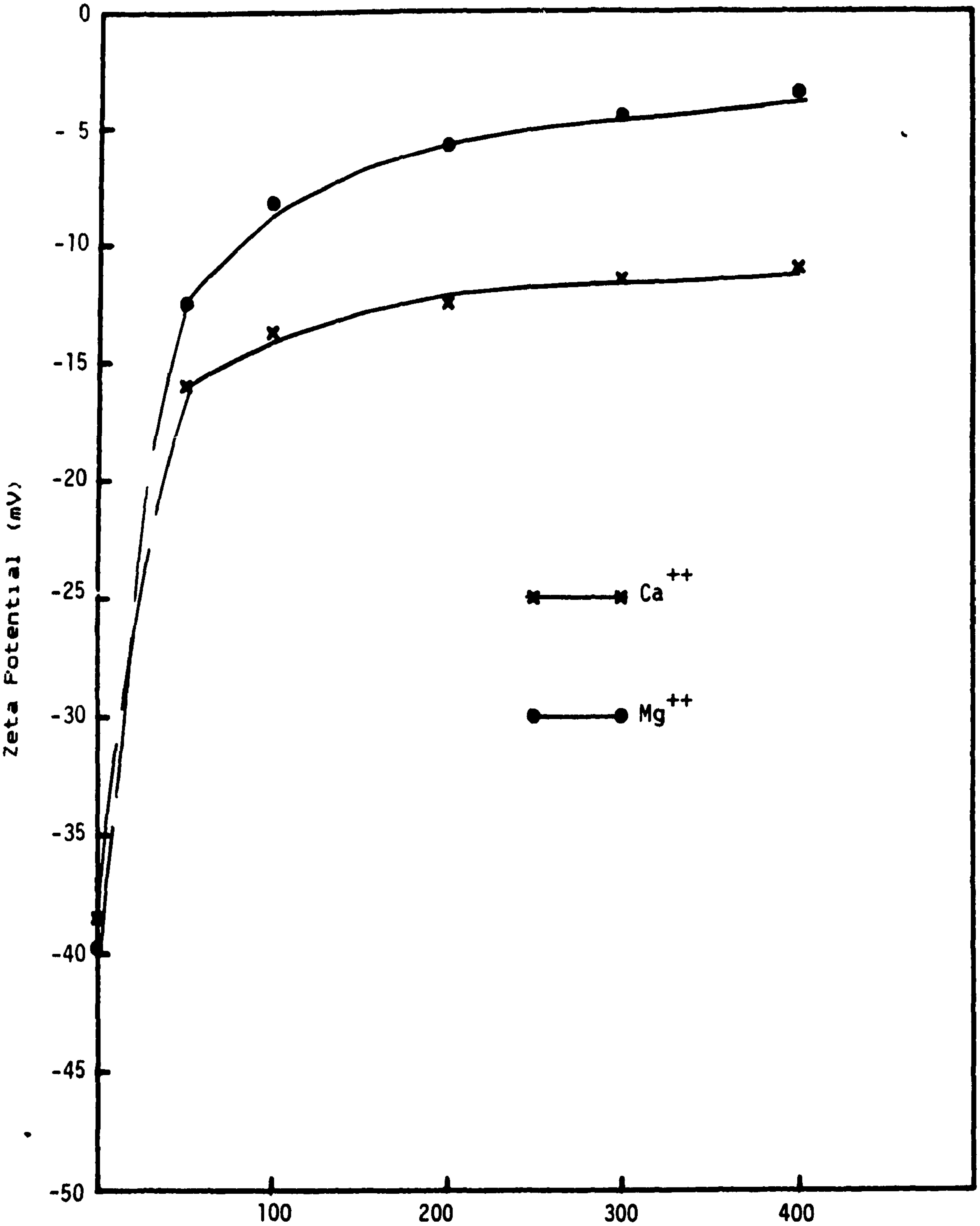


Fig. 12 Effect of Ca^{++} and Mg^{++} on surface charge of pyrochlore (pH 6).

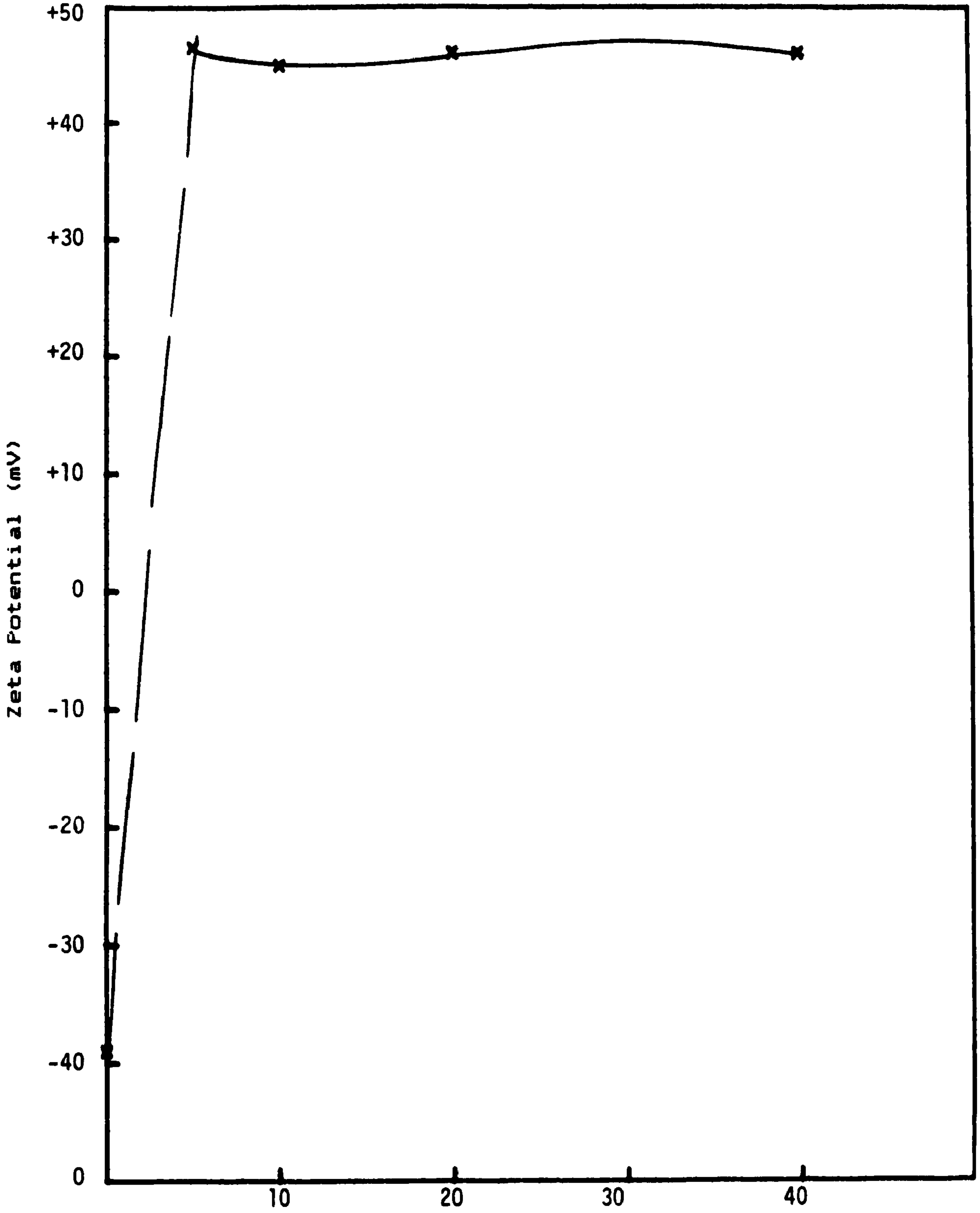


Fig. 13 Effect of HOECHST F 2642 on surface charge of pyrochlore (pH 6).

APPENDIX 2. The Use of Stoke's Law to Calculate Settling Times to Recover -3 um Particles for Electrophoretic Mobility Measurements.

The rate of fall of a small sphere in a viscous fluid can be calculated using stokes' Law:

$$V = \frac{2\ g\ a^2 (d1 - d2)}{9\ \eta}$$

Where,

V = terminal velocity (cm/s)
g = acceleration due to gravity (cm/s^2)
a = radius of the particle (cm)
d1 = density of the particle (g/cm^3)
d2 = density of the medium (g/cm^3)
η = viscocity of the medium (poise)

Assuming the following conditions (e.g. for a pyrochlore particle):

$a = 1.5 \times 10^{-4}$
$\eta = 0.01002$ poise
d1 = 4.2
d2 = 0.9982

thus,

$$V = \frac{2 \times 980\ (1.5 \times 10^{-4})^2 (4.2 - 0.9982)}{9 \times 0.01002}$$

$$V = 0.001566 \text{ cm/s}$$

thus, if the top 5 cm in the graduate cylinder are to be recovered, the settling time will be.

$$t = \frac{5 \text{ cm}}{0.0001566 \text{ cm/s}} \times \frac{1 \text{ min}}{60}$$

$t = 53.2$ min.

Therefore after 53.2 min of settling, the pyrochlore particles which would remain in the top 5 cm would be about 3 μm in diameter or smaller.

APPENDIX 3. Particle Size Distributions of Samples Used in the Microflotation Tests.

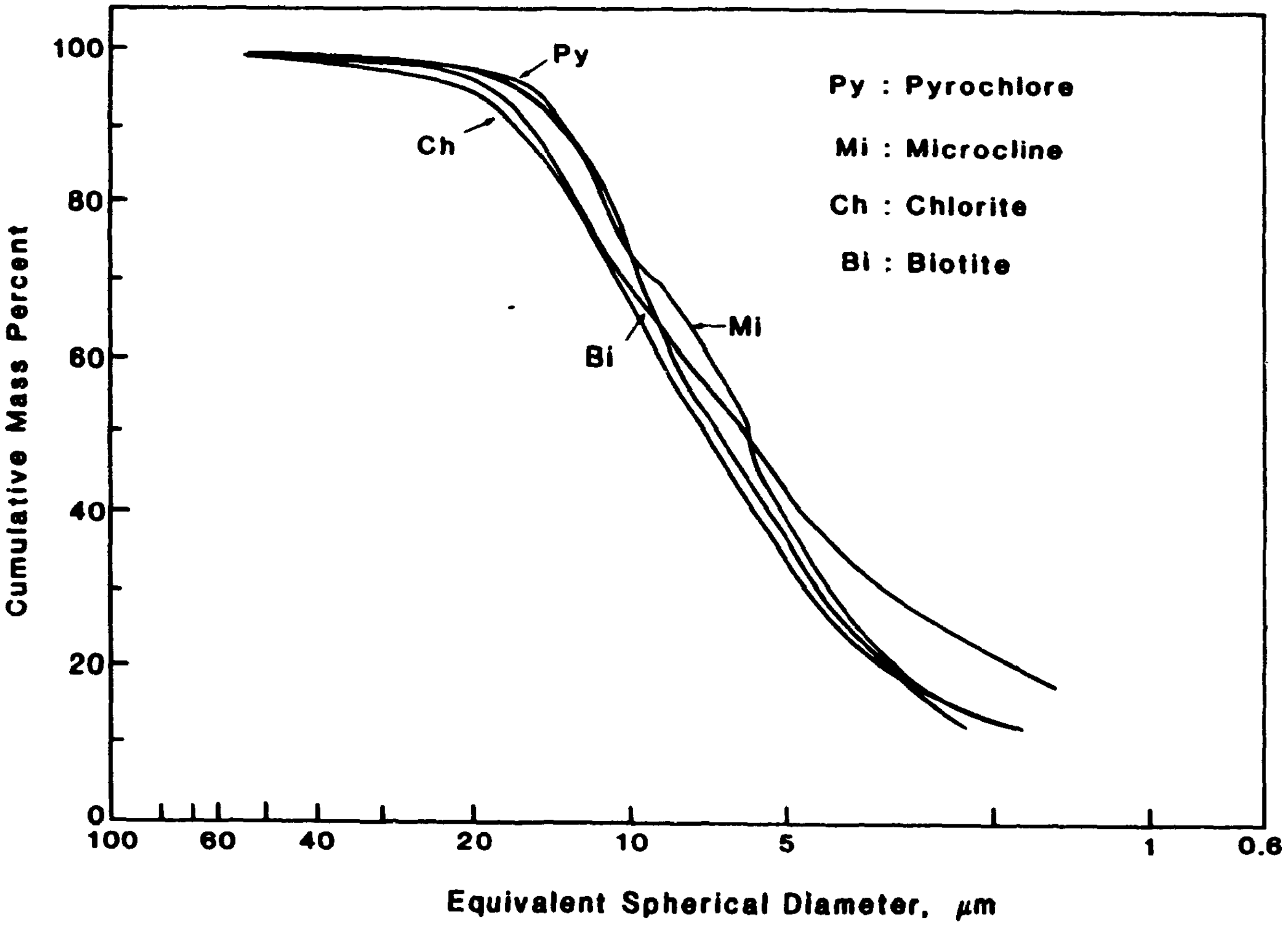


Figure A-3.1 Particle size distributions of samples used in microflotation tests.

APPENDIX 4. Electrokinetic Properties of Synthetic Samples Used in the Electrokinetic Study.

TABLE A.4.1. COMPARISON OF ELECTROKINETIC PROPERTIES OF SYNTHETIC SAMPLES VS NIOBEC SAMPLES (*)

MICROCLINE				BIOTITE			
NIOBEC		SYNTHETIC		NIOBEC		SYNTHETIC	
pH	u (**)	pH	u (**)	pH	u (**)	pH	u (**)
8.2	1.547	8.1	1.80	8.2	1.38	8.1	1.53
7.5	1.77			6.9	1.44	7.3	1.38
6.6	1.57	6.9	1.50	5.85	1.48		
5.5	1.29	5.2	1.35	4.95	1.29	5.8	1.49
4.5	0.972			4.5	1.291		
4.1	0.944	3.8	1.085	3.7	1.043	3.7	0.927
3.6	1.218			3.45	1.119		
3.5	1.213			3.0	0.890	2.9	0.910
3.2	0.868			2.5	0.392	2.3	0.283
2.66	0.495	2.7	0.761	1.72	(***)	1.97	(***)
2.1	0.140	1.9	(***)				

(*) Conditions: Potable water and H2SiF6 as pH modifier
(**) Eletrophoretic mobility, expressed as (cm/s/V/cm)* 10-4
(***) Close to IEP. Polarization occured

APPENDIX 5. Column Feed Preparation. Selection of Cyclone Conditions (Operating Conditions and Number of Stages). Particle Size Distribution of the Products.

A 5.08 cm (2 inch) hydrocyclone (cyclone) was used to perform the preparation of the column feed. Plitt's equation (1976) was used to guide selection of desliming conditions. The Sedigraph 5000D (Sedigraph) was used to monitor the particle size distribution (PSD), particularly the -1 um fraction in the cyclone underflows. Plitt's equation is expressed as follows.

$$d_{50} = \frac{50.5\ Dc^{0.46}\ Di^{0.60}\ Do^{1.21}\ \mu^{0.5} \exp\ (0.063\ \phi\ )}{Du^{0.71}\ h^{0.38}\ Q^{0.46} (\rho_s - \rho\ )^{0.5}}$$

d_{50} = cut size (μm)

Dc, Di, Do, Du, = cyclone, inside, overflow, and underflow diameters, respectively, cm.

h = apex-vortex distance, cm.

Q = pulp flowrate, L/min

ρ_s, ρ = solid and liquid densities, g/cm^3

μ = viscocity, cp

ϕ = solids percent volume in feed slurry.

Table A.5.1 summarizes the experimental cyclone conditions during column feed preparation and the % passing -1 um in each of the cyclone stages.

Figure A.5.1 shows the particle size distributions generated during an entire step to select cyclone conditions for column feed. (The cyclone feed to these tests are the slimes overflow from stage 1 in Figure 3.1.a, Chapter 3). Curves 1-5 are the cyclone underflows of tests 1 to 5. And curve 66 is the column feed (Test 6) generated with the flowsheet shown in Figure 3.1.a, Chapter 3.

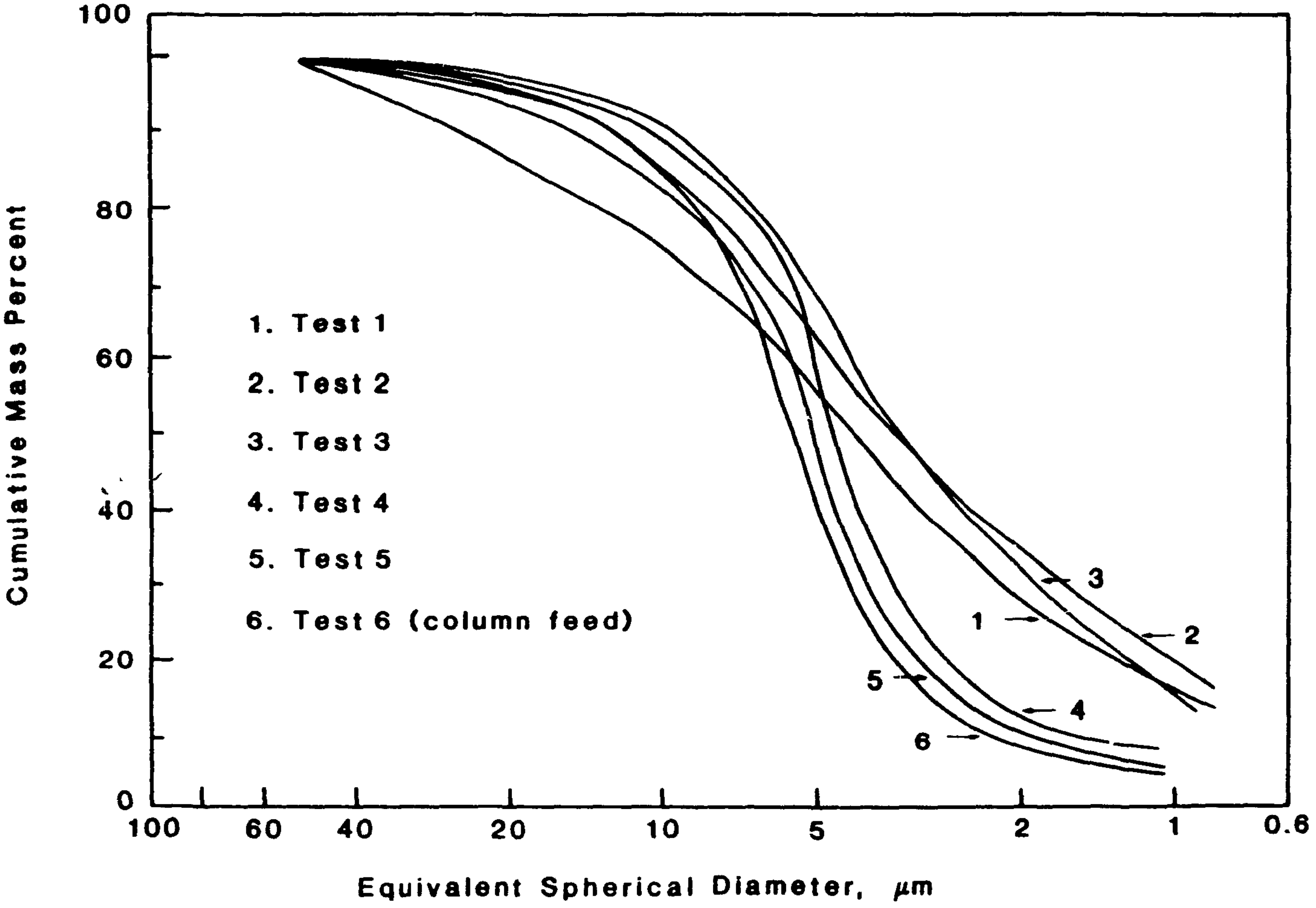


Figure A.5.1 Particle size distributions generated during the process of selection of cyclone conditions for column feed.

TABLE A.5.1 EXPERIMENTAL CYCLON CONDITIONS DURING COLUMN FEED PREPARATION

TEST #	STAGE #	CYCLONE DIMEN.			P kPa	ϕ (%)	Q L/min	d50 (*) (um)	(-1um) (%)
		Di cm	Do cm	Du cm					
ORE									
1	1	1.06	1.43	0.32	103.46	3.33	25.80	17.59	30
	2	0.80	0.79	0.87	413.40	0.33	31.15	3.24	15
2	1	1.06	1.43	0.32	103.46	3.33	25.80	17.59	30
	2	0.80	0.79	0.87	413.40	0.33	31.15	3.24	15
3	1	1.06	1.43	0.32	103.46	3.33	25.80	17.59	30
	2	1.06	1.43	0.32	289.55	3.33	41.91	24.83	14
4	1	1.06	1.43	0.32	103.46	2.33	25.80	16.52	27
	2	1.06	1.43	0.32	413.40	3.33	46.73	27.14	7
5	1	1.06	1.43	0.48	68.95	2.33	25.80	12.53	N.A.
	2	1.06	1.43	0.48	413.82	0.33	46.73	15.66	5
6	1	1.06	1.43	0.48	68.95	2.10	25.80	12.34	N.A.
	2	1.06	1.43	0.32	413.82	1.23	43.90	22.58	6
		1.06	1.43	0.32	413.82	1.00	43.90	22.26	6
		1.06	1.43	0.48	413.82	1.23	46.73	16.58	17
	3	1.06	1.43	0.32	413.82	0.60	43.90	21.71	5
		1.06	1.43	0.32	413.82	0.60	43.90	21.71	5

TABLE A.5.1 EXPERIMENTAL CYCLON CONDITIONS DURING COLUMN FEED PREPARATION (CONTINUATION)

TEST #	STAGE #	CYCLONE DIMEN. cm			P kPa	ϕ (%)	Q L/min	d50 (*) (um)	
		Di	Do	Du					
				SLIMES					
7 (**)	1	0.80	0.79	0.87	413.82	0.67	31.15	3.31	1
8	1	1.06	1.43	0.32	413.82	1.27	43.90	22.64	N.A.
		1.06	1.43	0.32	413.82	0.33	43.90	21.34	N.A.
	2	1.06	1.43	0.48	413.82	0.33	46.30	15.67	N.A.
		1.06	1.43	0.32	413.82	0.33	43.90	21.34	N.A.
	3	1.06	1.43	0.32	413.82	0.33	43.90	21.34	0
		1.06	1.43	0.32	413.82	0.33	43.90	21.34	0

(*) calculated using Plitt's Equation
(**) using a dispersant (calgon, 1%)

APPENDIX 6. Gas Flowmeter Calibration for the Laboratory Flotation Column.

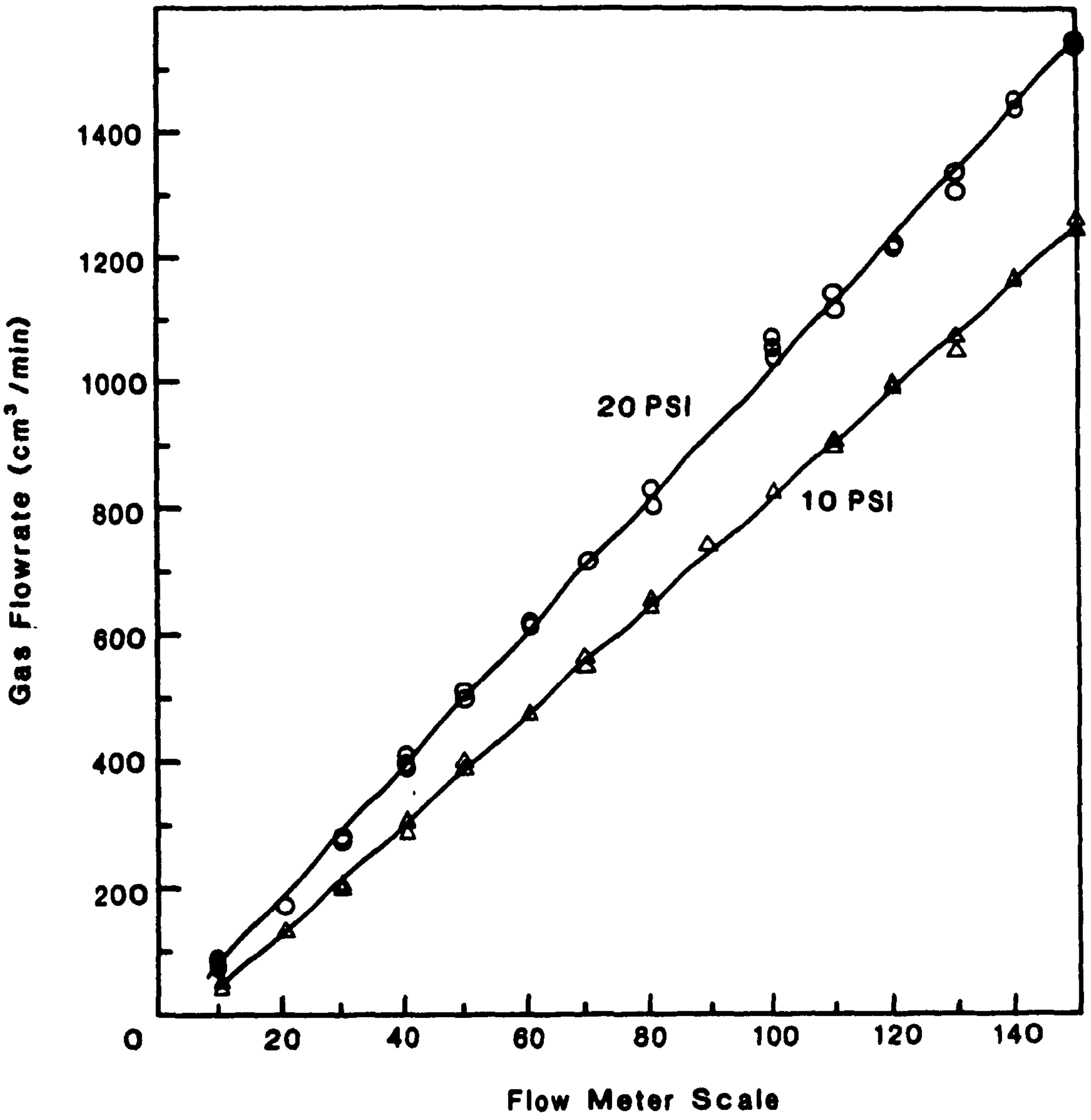


Figure A.6.1 Gas flowmeter calibration

APPENDIX 7. Laboratory Flotation Column Results, Experimental and Adjusted Assays. Metal Distribution.

```
TEST # R1
                              EXPERIMENTAL VALUES
                              *******************

              flowrates                           grades
            solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED       100.00     1.00    13.40    11.00    13.20    22.40     0.49
 CARBS        0.00     1.00    11.70    10.80    13.30    24.90     0.51
 TAILS        0.00     1.00    19.90    14.80    13.40    16.50     0.57

     Cl-W             410.00



                          FLOWRATES AND METAL UNITS
                          *************************

              flowrates                        metal units
            solids     water    SiO2     Fe2O3    MgO      CaO      Nb2O5

   FEED     100.00   9900.00   13.52    11.44    13.27    22.74     0.51
   CARBS     76.85   8018.17    8.92     8.04    10.18    18.94     0.38
   TAILS     23.15   2291.83    4.60     3.40     3.10     3.80     0.13

     Cl-W             410.00



                             METAL RECOVERIES
                             ****************

                   .   SiO2      Fe2O3     MgO      CaO      Nb2O5


         FEED        100.00    100.00   100.00   100.00   100.00
         CARBS        65.98     70.26    76.66    83.28    74.37
         TAILS        34.02     29.74    23.34    16.72    25.63


                               METAL  GRADES
                               *************

                         SiO2      Fe2O3    MgO      CaO      Nb2O5


         FEED            13.52     11.44    13.27    22.74     0.51
         CARBS           11.61     10.46    13.24    24.64     0.49
         TAILS           19.87     14.70    13.38    16.42     0.57
```

TEST # R2

EXPERIMENTAL VALUES

****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	12.60	11.30	13.40	24.90	0.55
TAILS	0.00	1.00	19.30	15.50	13.30	17.30	0.63
C1-W		410.00					

FLOWRATES AND METAL UNITS

****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	13.66	11.63	13.31	23.04	0.53
CARBS	81.51	8479.97	10.10	8.79	10.85	19.87	0.42
TAILS	18.49	1830.04	3.56	2.84	2.45	3.18	0.11
C1-W		410.00					

METAL RECOVERIES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	73.94	75.56	81.55	86.22	78.47
TAILS	26.06	24.44	18.45	13.78	21.53

METAL GRADES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.66	11.63	13.31	23.04	0.53
CARBS	12.39	10.78	13.31	24.37	0.51
TAILS	19.25	15.38	13.28	17.18	0.62

```
TEST # R3
                              EXPERIMENTAL VALUES
                              *******************

                 flowrates                          grades
              solids     % sol.    SiO2     Fe2O3     MgO      CaO      Nb2O5

  FEED        100.00      1.00     13.40    11.00    13.20    22.40     0.49
  CARBS         0.00      1.00      6.16     8.82    13.00    29.60     0.40
  TAILS         0.00      1.00     18.10    13.60    13.50    18.70     0.58

      Cl-W               410.00



                           FLOWRATES AND METAL UNITS
                           *************************

                 flowrates                        metal units
              solids     water     SiO2     Fe2O3     MgO      CaO      Nb2O5

    FEED      100.00   9900.00     13.48    11.50    13.27    22.71     0.50
    CARBS      38.32   4203.33      2.35     3.31     4.97    11.30     0.15
    TAILS      61.68   6106.68     11.13     8.20     8.30    11.42     0.35

      Cl-W               410.00



                              METAL RECOVERIES
                              ****************

                     SiO2      Fe2O3      MgO       CaO       Nb2O5


           FEED     100.00    100.00    100.00    100.00    100.00
           CARBS     17.42     28.74     37.46     49.74     30.02
           TAILS     82.58     71.26     62.54     50.26     69.98


                                METAL  GRADES
                                *************

                        SiO2      Fe2O3      MgO       CaO       Nb2O5


           FEED        13.48     11.50     13.27     22.71      0.50
           CARBS        6.13      8.63     12.97     29.48      0.39
           TAILS       18.05     13.29     13.46     18.51      0.57
```

```
TEST # R4
                         EXPERIMENTAL VALUES
                         *******************

            flowrates                         grades
         solids   % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED    100.00    1.00    13.40    11.00    13.20    22.40     0.49
 CARBS     0.00    1.00     7.71     9.61    13.20    28.60     0.45
 TAILS     0.00    1.00    19.90    13.60    13.30    16.80     0.57

     Cl-W        410.00



                     FLOWRATES AND METAL UNITS
                     *************************

            flowrates                       metal units
         solids    water    SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED   100.00  9900.00   13.57    11.37    13.23    22.70     0.50
  CARBS   51.25  5484.08    3.91     4.83     6.76    14.58     0.23
  TAILS   48.75  4825.92    9.66     6.54     6.48     8.12     0.27

     Cl-W        410.00



                         METAL RECOVERIES
                         ****************

                  SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED    100.00   100.00   100.00   100.00   100.00
         CARBS    28.80    42.47    51.06    64.23    45.27
         TAILS    71.20    57.53    48.94    35.77    54.73



                           METAL  GRADES
                           *************

                     SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED       13.57     11.37    13.23    22.70     0.50
         CARBS       7.62      9.42    13.18    28.45     0.44
         TAILS      19.82     13.42    13.28    16.65     0.56
```

```
TEST # R5
                          EXPERIMENTAL VALUES
                          *******************

              flowrates                          grades
              solids    % sol.   SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED         100.00     1.00    13.40    11.00    13.20    22.40     0.49
 CARBS          0.00     1.00     6.64     9.55    12.90    29.80     0.44
 TAILS          0.00     1.00    16.50    12.70    13.30    19.70     0.55

     C1-W              410.00



                        FLOWRATES AND METAL UNITS
                        *************************

              flowrates                        metal units
              solids    water    SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED        100.00  9900.00    13.48    11.47    13.19    22.62     0.51
  CARBS        30.20  3400.27     2.00     2.84     3.90     8.98     0.13
  TAILS        69.80  6909.73    11.48     8.63     9.29    13.64     0.38

     C1-W              410.00



                          METAL RECOVERIES
                          ****************

                    SiO2     Fe2O3    MgO      CaO      Nb2O5


       FEED        100.00   100.00   100.00   100.00   100.00
       CARBS        14.83    24.76    29.56    39.70    25.91
       TAILS        85.17    75.24    70.44    60.30    74.09



                            METAL  GRADES
                            *************

                       SiO2     Fe2O3    MgO      CaO      Nb2O5


       FEED           13.48    11.47    13.19    22.62     0.51
       CARBS           6.62     9.41    12.90    29.73     0.43
       TAILS          16.45    12.37    13.31    19.54     0.54
```

```
TEST # R6
                          EXPERIMENTAL VALUES
                          *******************

              flowrates                           grades
            solids      % sol.    SiO2      Fe2O3    MgO      CaO      Nb2O5

 FEED       100.00       1.00     13.40     11.00    13.20    22.40     0.49
 CARBS        0.00       1.00      9.30     10.30    13.50    27.30     0.46
 TAILS        0.00       1.00     18.70     13.60    13.40    18.50     0.56

     Cl-W               410.00


                        FLOWRATES AND METAL UNITS
                        *************************

              flowrates                          metal units
            solids     water      SiO2      Fe2O3    MgO      CaO      Nb2O5

   FEED     100.00   9900.00     13.64     11.58    13.37    22.88     0.50
   CARBS     52.54   5611.72      4.82      5.25     7.05    14.21     0.24
   TAILS     47.46   4698.29      8.82      6.32     6.32     8.67     0.26

    Cl-W              410.00


                           METAL RECOVERIES
                           ****************

                     SiO2      Fe2O3     MgO       CaO      Nb2O5


           FEED     100.00    100.00    100.00    100.00    100.00
           CARBS     35.34     45.37     52.71     62.11     47.54
           TAILS     64.66     54.63     47.29     37.89     52.46


                             METAL  GRADES
                             *************

                        SiO2      Fe2O3     MgO       CaO       Nb2O5


           FEED         13.64     11.58     13.37     22.88      0.50
           CARBS         9.17     10.00     13.41     27.05      0.45
           TAILS        18.59     13.33     13.32     18.27      0.55
```

```
TEST # R7
                              EXPERIMENTAL VALUES
                              *******************

               flowrates                           grades
            solids    % sol.    SiO2      Fe2O3    MgO       CaO       Nb2O5

 FEED       100.00      1.00    13.40     11.00    13.20     22.40      0.49
 CARBS        0.00      1.00     9.49     10.50    13.30     27.10      0.49
 TAILS        0.00      1.00    23.80     15.30    13.90     13.60      0.55

     C1-W             410.00



                         FLOWRATES AND METAL UNITS
                         *************************

               flowrates                        metal units
            solids    water     SiO2      Fe2O3    MgO       CaO       Nb2O5

  FEED      100.00  9900.00     13.59     11.58    13.37     22.85      0.50
  CARBS      70.52  7391.50      6.60      7.12     9.29     18.88      0.34
  TAILS      29.48  2918.50      7.00      4.46     4.08      3.97      0.16

     C1-W             410.00



                              METAL RECOVERIES
                              ****************

                        SiO2       Fe2O3     MgO       CaO       Nb2O5


         FEED        100.00     100.00    100.00    100.00    100.00
         CARBS        48.52      61.48     69.48     82.63     67.84
         TAILS        51.48      38.52     30.52     17.37     32.16


                                METAL  GRADES
                                *************

                           SiO2      Fe2O3     MgO       CaO       Nb2O5


         FEED              13.59     11.58     13.37     22.85      0.50
         CARBS              9.35     10.09     13.18     26.78      0.48
         TAILS             23.74     15.13     13.85     13.47      0.55
```

```
TEST # R8
                         EXPERIMENTAL VALUES
                         *******************

              flowrates                             grades
           solids    % sol.    SiO2      Fe2O3    MgO      CaO      Nb2O5

  FEED     100.00     1.00    13.40     11.00    13.20    22.40     0.49
  CARBS      0.00     1.00     8.88     10.20    13.40    27.90     0.47
  TAILS      0.00     1.00    23.90     15.60    14.10    13.90     0.52

      Cl-W           410.00



                     FLOWRATES AND METAL UNITS
                     *************************

              flowrates                          metal units
           solids     water    SiO2      Fe2O3    MgO      CaO      Nb2O5

  FEED     100.00   9900.00   13.69     11.63    13.48    22.96     0.49
  CARBS     66.91   7034.33    5.81      6.54     8.84    18.42     0.32
  TAILS     33.09   3275.67    7.88      5.09     4.64     4.54     0.17

      Cl-W           410.00



                         METAL RECOVERIES
                         ****************

                  SiO2      Fe2O3     MgO       CaO       Nb2O5


       FEED      100.00    100.00    100.00    100.00    100.00
       CARBS      42.46     56.23     65.61     80.23     64.68
       TAILS      57.54     43.77     34.39     19.77     35.32


                          METAL  GRADES
                          *************

                     SiO2      Fe2O3     MgO       CaO       Nb2O5


       FEED         13.69     11.63     13.48     22.96      0.49
       CARBS         8.69      9.78     13.21     27.53      0.47
       TAILS        23.80     15.39     14.01     13.72      0.52
```

```
TEST # R9
                          EXPERIMENTAL VALUES
                          *******************

                flowrates                             grades
              solids    % sol.    SiO2      Fe2O3    MgO       CaO       Nb2O5

 FEED         100.00     1.00    13.40     11.00    13.20     22.40      0.49
 CARBS          0.00     1.00     6.91     11.00    12.91     29.33      0.42
 TAILS          0.00     1.00    14.29     11.99    13.39     22.57      0.53

      Cl-W              410.00



                       FLOWRATES AND METAL UNITS
                       *************************

                flowrates                           metal units
              solids     water    SiO2      Fe2O3    MgO       CaO       Nb2O5

  FEED        100.00   9900.00   13.65     11.49    13.29     22.70      0.51
  CARBS         5.78    982.30    0.40      0.63     0.75      1.69      0.02
  TAILS        94.22   9327.70   13.25     10.86    12.54     21.00      0.48

      Cl-W              410.00



                          METAL RECOVERIES
                          ****************

                      SiO2      Fe2O3     MgO       CaO       Nb2O5


            FEED     100.00    100.00    100.00    100.00    100.00
            CARBS      2.92      5.52      5.62      7.47      4.77
            TAILS     97.08     94.48     94.38     92.53     95.23


                           METAL  GRADES
                           *************

                        SiO2      Fe2O3     MgO       CaO       Nb2O5


            FEED        13.65     11.49     13.29     22.70      0.51
            CARBS        6.90     10.97     12.91     29.31      0.42
            TAILS       14.06     11.53     13.31     22.29      0.51
```

```
TEST # R23
                         EXPERIMENTAL VALUES
                         *******************

              flowrates                        grades
            solids     % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED        100.00      1.00    13.40    11.00    13.20    22.40     0.49
CARBS         0.00      1.00    12.70     9.88    13.30    23.80     0.46
TAILS         0.00      1.00    11.60    10.40    13.60    24.40     0.50

   Cl-W               410.00



                     FLOWRATES AND METAL UNITS
                     *************************

              flowrates                      metal units
            solids     water     SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED       100.00  9900.00     13.04    10.44    13.25    23.11     0.48
 CARBS       98.81 %10192.38     12.90    10.31    13.09    22.82     0.47
 TAILS        1.19   117.62     0.14     0.12     0.16     0.29     0.01

  Cl-W              410.00



                       METAL RECOVERIES
                       ****************

                  SiO2      Fe2O3     MgO       CaO       Nb2O5


      FEED       100.00    100.00    100.00    100.00    100.00
      CARBS       98.94     98.82     98.78     98.75     98.75
      TAILS        1.06      1.18      1.22      1.25      1.25


                        METAL  GRADES
                        *************

                    SiO2      Fe2O3     MgO       CaO       Nb2O5


      FEED         13.04     10.44     13.25     23.11      0.48
      CARBS        13.06     10.44     13.25     23.10      0.47
      TAILS        11.60     10.41     13.60     24.39      0.50
```

TEST # R24

EXPERIMENTAL VALUES

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	11.30	9.27	13.30	24.60	0.48
TAILS	0.00	1.00	13.50	11.00	13.80	20.30	0.49
Cl-W		410.00					

FLOWRATES AND METAL UNITS

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	12.97	10.61	13.48	22.01	0.49
CARBS	34.94	3869.17	4.00	3.29	4.61	8.64	0.17
TAILS	65.06	6440.83	8.97	7.32	8.86	13.37	0.32
Cl-W		410.00					

METAL RECOVERIES

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	30.86	30.98	34.24	39.26	34.44
TAILS	69.14	69.02	65.76	60.74	65.56

METAL GRADES

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	12.97	10.61	13.48	22.01	0.49
CARBS	11.45	9.41	13.20	24.74	0.48
TAILS	13.78	11.25	13.62	20.55	0.49

```
TEST # R25
                              EXPERIMENTAL VALUES
                              *******************

                 flowrates                              grades
               solids     % sol.     SiO2      Fe2O3    MgO       CaO       Nb2O5

FEED           100.00      1.00      13.40     11.00    13.20     22.40      0.49
CARBS            0.00      1.00      11.00     10.30    13.00     25.10      0.48
TAILS            0.00      1.00      13.60     11.20    14.60     20.70      0.42

     C1-W                410.00



                           FLOWRATES AND METAL UNITS
                           *************************

                 flowrates                            metal units
               solids     water      SiO2      Fe2O3    MgO       CaO       Nb2O5

FEED           100.00   9900.00      12.95     10.93    13.75     22.28      0.46
CARBS           34.45   3820.42       3.84      3.56     4.41      8.66      0.17
TAILS           65.55   6489.58       9.11      7.37     9.33     13.62      0.29

     C1-W                410.00



                             METAL RECOVERIES
                             ****************

                    SiO2      Fe2O3     MgO       CaO       Nb2O5


          FEED     100.00    100.00    100.00    100.00    100.00
          CARBS     29.67     32.54     32.10     38.87     36.92
          TAILS     70.33     67.46     67.90     61.13     63.08



                               METAL  GRADES
                               *************

                        SiO2      Fe2O3     MgO       CaO       Nb2O5


          FEED          12.95     10.93     13.75     22.28      0.46
          CARBS         11.15     10.32     12.81     25.14      0.49
          TAILS         13.89     11.25     14.24     20.78      0.44
```

```
TEST # T28E
                          EXPERIMENTAL VALUES
                          *******************

              flowrates                           grades
            solids     % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED       100.00      1.00    13.40    11.00    13.20    22.40     0.49
 CARBS        0.00      1.00    12.10    11.30    12.70    22.40     0.50
 TAILS        0.00      1.00    14.00    11.50    13.10    21.50     0.44

     Cl-W             410.00



                       FLOWRATES AND METAL UNITS
                       *************************

              flowrates                         metal units
            solids     water     SiO2     Fe2O3    MgO      CaO      Nb2O5

   FEED     100.00   9900.00    13.26    11.27    13.02    22.06     0.47
   CARBS     42.42   4609.25     5.16     4.74     5.42     9.56     0.22
   TAILS     57.58   5700.75     8.11     6.53     7.60    12.49     0.26

     Cl-W             410.00



                          METAL RECOVERIES
                          ****************

                      SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED        100.00   100.00   100.00   100.00   100.00
         CARBS        38.88    42.07    41.62    43.36    45.38
         TAILS        61.12    57.93    58.38    56.64    54.62



                            METAL  GRADES
                            *************

                         SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED            13.26    11.27    13.02    22.06     0.47
         CARBS           12.16    11.18    12.78    22.55     0.51
         TAILS           14.08    11.34    13.20    21.70     0.45
```

TEST # T74

EXPERIMENTAL VALUES
****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	8.69	9.60	12.80	25.70	0.40
TAILS	0.00	1.00	17.60	13.00	13.50	17.90	0.46
Cl-W		410.00					

FLOWRATES AND METAL UNITS
****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	13.10	11.15	13.16	22.11	0.45
CARBS	52.12	5570.08	4.61	4.96	6.68	13.47	0.22
TAILS	47.88	4739.93	8.49	6.19	6.47	8.64	0.23
Cl-W		410.00					

METAL RECOVERIES
****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	35.18	44.50	50.80	60.94	48.88
TAILS	64.82	55.50	49.20	39.06	51.12

METAL GRADES
****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.10	11.15	13.16	22.11	0.45
CARBS	8.84	9.52	12.82	25.85	0.42
TAILS	17.74	12.93	13.52	18.04	0.48

```
TEST # T73
                         EXPERIMENTAL VALUES
                         *******************

             flowrates                          grades
           solids    % sol.   SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED      100.00     1.00    13.40    11.00    13.20    22.40     0.49
 CARBS       0.00     1.00     9.00     9.83    12.70    25.50     0.41
 TAILS       0.00     1.00    17.30    12.60    13.40    18.50     0.46

     C1-W            410.00



                      FLOWRATES AND METAL UNITS
                      *************************

             flowrates                        metal units
           solids    water    SiO2     Fe2O3    MgO      CaO      Nb2O5

   FEED    100.00  9900.00    13.19    11.13    13.10    22.17     0.45
   CARBS    50.83  5442.44     4.63     4.96     6.48    13.02     0.22
   TAILS    49.17  4867.56     8.56     6.16     6.61     9.15     0.24

     C1-W            410.00



                         METAL RECOVERIES
                         ****************

                    SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED      100.00   100.00   100.00   100.00   100.00
         CARBS      35.11    44.61    49.50    58.73    48.11
         TAILS      64.89    55.39    50.50    41.27    51.89


                          METAL  GRADES
                          *************

                       SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED          13.19    11.13    13.10    22.17     0.45
         CARBS          9.11     9.76    12.75    25.62     0.43
         TAILS         17.40    12.54    13.45    18.61     0.48
```

```
TEST # R28
                              EXPERIMENTAL VALUES
                              *******************

               flowrates                               grades
              solids      % sol.    SiO2      Fe2O3    MgO       CaO       Nb2O5

FEED          100.00       1.00     13.40     11.00    13.20     22.40      0.49
CARBS           0.00       1.00     11.60     10.90    12.90     23.50      0.58
TAILS           0.00       1.00     13.40     11.00    13.10     22.20      0.43

     Cl-W                410.00


                           FLOWRATES AND METAL UNITS
                           *************************

               flowrates                             metal units
             solids     water       SiO2      Fe2O3     MgO       CaO       Nb2O5

 FEED        100.00    9900.00     13.36     11.00     13.14     22.33      0.46
 CARBS         4.56     861.78      0.53      0.50      0.59      1.07      0.03
 TAILS        95.44    9448.22     12.83     10.50     12.55     21.25      0.44

     Cl-W               410.00


                              METAL RECOVERIES
                              ****************

                       SiO2      Fe2O3     MgO      CaO      Nb2O5


        FEED         100.00    100.00   100.00   100.00   100.00
        CARBS          3.96      4.52     4.48     4.80     5.74
        TAILS         96.04     95.48    95.52    95.20    94.26


                                METAL  GRADES
                                *************

                          SiO2     Fe2O3     MgO      CaO      Nb2O5


        FEED             13.36     11.00    13.14    22.33     0.46
        CARBS            11.60     10.90    12.90    23.50     0.58
        TAILS            13.44     11.00    13.15    22.27     0.46
```

```
TEST # R29
                         EXPERIMENTAL VALUES
                         *******************

          flowrates                        grades
        solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED    100.00     1.00     11.40    11.00    13.20    22.40    0.49
CARBS     0.00     1.00     11.40    10.40    13.10    23.30    0.55
TAILS     0.00     1.00     13.50    11.00    13.10    22.10    0.46

   C1-W          410.00



                       FLOWRATES AND METAL UNITS
                       *************************

          flowrates                      metal units
        solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED   100.00   9900.00    11.62    10.71    13.14    22.80    0.52
 CARBS   82.01   8528.72     9.20     8.72    10.78    18.84    0.43
 TAILS   17.99   1781.28     2.42     1.99     2.36     3.96    0.08

   C1-W          410.00



                         METAL RECOVERIES
                         ****************

                 SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED     100.00    100.00    100.00    100.00    100.00
        CARBS     79.16     81.44     82.05     82.62     84.11
        TAILS     20.84     18.56     17.95     17.38     15.89


                          METAL  GRADES
                          *************

                    SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED        11.62     10.71     13.14     22.80      0.52
        CARBS       11.22     10.64     13.15     22.97      0.53
        TAILS       13.46     11.05     13.11     22.03      0.46
```

```
TEST # R29A
                         EXPERIMENTAL VALUES
                         *******************

              flowrates                              grades
            solids    % sol.    SiO2     Fe2O3     MgO      CaO      Nb2O5

 FEED       100.00     1.00     13.40    11.00     13.20    22.40     0.49
 CARBS        0.00     1.00     12.10    10.80     13.10    23.40     0.50
 TAILS        0.00     1.00     14.20    11.30     13.20    22.00     0.46

     Cl-W            410.00


                       FLOWRATES AND METAL UNITS
                       *************************

              flowrates                            metal units
            solids    water     SiO2     Fe2O3     MgO      CaO      Nb2O5

   FEED     100.00   9900.00    13.43    11.08     13.18    22.47     0.48
   CARBS     36.16   3989.69     4.37     3.89      4.74     8.45     0.18
   TAILS     63.84   6320.31     9.05     7.18      8.44    14.02     0.30

     Cl-W            410.00


                         METAL RECOVERIES
                         ****************

                  SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED    100.00   100.00   100.00   100.00   100.00
         CARBS    32.56    35.16    35.97    37.62    37.95
         TAILS    67.44    64.84    64.03    62.38    62.05


                           METAL  GRADES
                           *************

                    SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED       13.43    11.08    13.18    22.47     0.48
         CARBS      12.09    10.77    13.11    23.38     0.50
         TAILS      14.18    11.25    13.22    21.96     0.47
```

```
TEST # R29B
                                EXPERIMENTAL VALUES
                                *******************

                 flowrates                            grades
               solids    % sol.    SiO2      Fe2O3    MgO      CaO      Nb2O5

   FEED        100.00     1.00     13.40     11.00    13.20    22.40     0.49
   CARBS         0.00     1.00     11.50     10.60    13.20    24.00     0.48
   TAILS         0.00     1.00     15.30     11.20    13.30    21.00     0.42

       Cl-W              410.00



                             FLOWRATES AND METAL UNITS
                             *************************

                 flowrates                          metal units
               solids    water     SiO2      Fe2O3    MgO      CaO      Nb2O5

     FEED      100.00   9900.00    13.43     10.94    13.23    22.44     0.46
     CARBS      48.67   5228.81     5.59      5.17     6.42    11.67     0.24
     TAILS      51.33   5081.19     7.84      5.76     6.82    10.77     0.22

       Cl-W              410.00



                                METAL RECOVERIES
                                ****************

                          SiO2      Fe2O3     MgO      CaO      Nb2O5


              FEED       100.00    100.00    100.00   100.00   100.00
              CARBS       41.61     47.30     48.49    52.02    51.87
              TAILS       58.39     52.70     51.51    47.98    48.13


                                  METAL  GRADES
                                  *************

                             SiO2      Fe2O3     MgO      CaO      Nb2O5


              FEED           13.43     10.94    13.23    22.44     0.46
              CARBS          11.48     10.63    13.18    23.98     0.49
              TAILS          15.28     11.23    13.28    20.98     0.43
```

```
TEST # R32
                      EXPERIMENTAL VALUES
                      *******************

              flowrates                           grades
           solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED       100.00     1.00     13.40    11.00    13.20    22.40     0.49
CARBS        0.00     1.00      6.51    12.80    12.80    28.10     0.34
TAILS        0.00     1.00     16.30    12.10    13.20    19.60     0.53

    Cl-W            410.00



                    FLOWRATES AND METAL UNITS
                    *************************

             flowrates                          metal units
           solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED      100.00   9900.00    13.36    11.83    13.12    22.25     0.48
 CARBS      30.20   3400.27     1.97     3.79     3.87     8.50     0.10
 TAILS      69.80   6909.73    11.39     8.04     9.25    13.75     0.38

    Cl-W            410.00



                       METAL RECOVERIES
                       ****************

                   SiO2     Fe2O3    MgO      CaO      Nb2O5


        FEED      100.00   100.00   100.00   100.00   100.00
        CARBS      14.74    32.04    29.51    38.20    21.65
        TAILS      85.26    67.96    70.49    61.80    78.35



                         METAL  GRADES
                         *************

                    SiO2     Fe2O3    MgO      CaO      Nb2O5


        FEED        13.36    11.83    13.12    22.25     0.48
        CARBS        6.52    12.55    12.82    28.14     0.34
        TAILS       16.33    11.52    13.25    19.70     0.54
```

```
TEST # R33
                      EXPERIMENTAL VALUES
                      *******************

           flowrates                          grades
         solids    % sol.   SiO2    Fe2O3    MgO      CaO      Nb2O5

FEED     100.00     1.00   13.40    11.00   13.20    22.40     0.49
CARBS      0.00     1.00    2.38     6.19   14.70    31.10     0.23
TAILS      0.00     1.00   10.30    11.40   13.90    24.10     0.70

    Cl-W          410.00



                    FLOWRATES AND METAL UNITS
                    *************************

           flowrates                        metal units
         solids     water    SiO2    Fe2O3    MgO      CaO      Nb2O5

  FEED   100.00   9900.00   11.78    11.17   13.56    23.30     0.59
  CARBS    1.19    527.62    0.03     0.07    0.17     0.37     0.00
  TAILS   98.81   9782.38   11.76    11.10   13.38    22.93     0.59

    Cl-W           410.00



                      METAL RECOVERIES
                      ****************

                 SiO2     Fe2O3    MgO      CaO      Nb2O5


       FEED     100.00   100.00   100.00   100.00   100.00
       CARBS      0.24     0.66     1.29     1.59     0.46
       TAILS     99.76    99.34    98.71    98.41    99.54


                        METAL  GRADES
                        *************

                   SiO2     Fe2O3    MgO      CaO      Nb2O5


       FEED        11.78    11.17   13.56    23.30     0.59
       CARBS        2.40     6.19   14.70    31.09     0.23
       TAILS       11.90    11.23   13.55    23.21     0.60
```

```
TEST # R35
                              EXPERIMENTAL VALUES
                              *******************

              flowrates                               grades
           solids    % sol.    SiO2      Fe2O3     MgO       CaO       Nb2O5

 FEED      100.00     1.00     13.40     11.00     13.20     22.40      0.49
 CARBS       0.00     1.00     11.90     10.40     13.30     23.70      0.49
 TAILS       0.00     1.00     16.00     11.70     13.20     19.40      0.42

     Cl-W           410.00



                           FLOWRATES AND METAL UNITS
                           *************************

              flowrates                             metal units
           solids    water     SiO2      Fe2O3     MgO       CaO       Nb2O5

  FEED     100.00  9900.00     13.32     10.90     13.24     22.31      0.47
  CARBS     66.35  6978.47      7.93      6.95      8.81     15.77      0.33
  TAILS     33.65  3331.54      5.39      3.95      4.44      6.54      0.14

     Cl-W           410.00



                              METAL RECOVERIES
                              ****************

                     SiO2      Fe2O3     MgO       CaO       Nb2O5


         FEED      100.00    100.00    100.00    100.00    100.00
         CARBS      59.52     63.75     66.49     70.68     69.87
         TAILS      40.48     36.25     33.51     29.32     30.13


                               METAL  GRADES
                               *************

                       SiO2      Fe2O3     MgO       CaO       Nb2O5


         FEED          13.32     10.90     13.24     22.31      0.47
         CARBS         11.95     10.47     13.27     23.76      0.50
         TAILS         16.03     11.74     13.19     19.43      0.43
```

```
TEST # R36
                            EXPERIMENTAL VALUES
                            *******************

               flowrates                          grades
             solids    % sol.    SiO2      Fe2O3    MgO      CaO      Nb2O5

 FEED        100.00     1.00    13.40     11.00    13.20    22.40     0.49
 CARBS         0.00     1.00     5.11      8.25    14.30    28.90     0.39
 TAILS         0.00     1.00    10.10      9.71    14.00    28.40     0.42

      Cl-W            410.00



                         FLOWRATES AND METAL UNITS
                         *************************

              flowrates                          metal units
            solids    water      SiO2      Fe2O3    MgO      CaO      Nb2O5

  FEED      100.00   9900.00    11.70     10.34    13.61    25.44     0.45
  CARBS       1.19    527.62     0.06      0.10     0.17     0.34     0.00
  TAILS      98.81   9782.38    11.64     10.24    13.44    25.10     0.45

     Cl-W            410.00



                             METAL RECOVERIES
                             ****************

                      SiO2      Fe2O3     MgO       CaO       Nb2O5


       FEED          100.00    100.00    100.00    100.00    100.00
       CARBS           0.52      0.95      1.25      1.35      1.02
       TAILS          99.48     99.05     98.75     98.65     98.98



                               METAL  GRADES
                               *************

                        SiO2      Fe2O3     MgO       CaO       Nb2O5


       FEED             11.70     10.34     13.61     25.44      0.45
       CARBS             5.13      8.26     14.30     28.86      0.39
       TAILS            11.78     10.36     13.60     25.40      0.46
```

TEST # R80A

EXPERIMENTAL VALUES

****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	10.40	9.10	12.90	25.00	0.41
TAILS	0.00	1.00	22.50	15.30	13.50	13.00	0.51
Cl-W		410.00					

FLOWRATES AND METAL UNITS

****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	13.36	10.76	13.11	22.21	0.46
CARBS	75.75	7909.16	7.90	7.03	9.83	19.05	0.33
TAILS	24.25	2400.84	5.46	3.72	3.28	3.16	0.13
Cl-W		410.00					

METAL RECOVERIES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	59.14	65.38	74.98	85.76	72.43
TAILS	40.86	34.63	25.02	14.24	27.57

METAL GRADES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.36	10.76	13.11	22.21	0.46
CARBS	10.43	9.28	12.97	25.14	0.44
TAILS	22.51	15.36	13.52	13.05	0.52

TEST # R80B

EXPERIMENTAL VALUES
****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	10.80	9.49	12.60	24.60	0.46
TAILS	0.00	1.00	16.00	16.00	13.70	11.10	0.53
Cl-W		410.00					

FLOWRATES AND METAL UNITS
****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	12.49	10.90	12.98	22.05	0.48
CARBS	79.36	8266.33	9.15	7.59	10.14	19.74	0.37
TAILS	20.64	2043.67	3.34	3.31	2.84	2.31	0.11
Cl-W		410.00					

METAL RECOVERIES
****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	73.24	69.66	78.13	89.54	77.16
TAILS	26.76	30.34	21.87	10.46	22.84

METAL GRADES
****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	12.49	10.90	12.98	22.05	0.48
CARBS	11.52	9.57	12.78	24.88	0.47
TAILS	16.19	16.02	13.75	11.17	0.53

```
TEST # R80C
                              EXPERIMENTAL VALUES
                              *******************

                  flowrates                                  grades
               solids      % sol.    SiO2      Fe2O3    MgO       CaO       Nb2O5

  FEED         100.00       1.00     13.40     11.00    13.20     22.40      0.49
  CARBS          0.00       1.00     10.70      9.43    12.70     24.00      0.45
  TAILS          0.00       1.00     25.30     16.10    13.60     11.20      0.53

     Cl-W                 410.00



                           FLOWRATES AND METAL UNITS
                           *************************

                  flowrates                                metal units
               solids      water     SiO2      Fe2O3    MgO       CaO       Nb2O5

   FEED        100.00    9900.00     13.25     10.74    13.00     22.09      0.47
   CARBS        83.30    8656.35      9.02      8.04    10.72     20.21      0.39
   TAILS        16.70    1653.65      4.23      2.70     2.28      1.88      0.09

     Cl-W                 410.00



                              METAL RECOVERIES
                              ****************

                       SiO2      Fe2O3     MgO       CaO       Nb2O5


          FEED        100.00    100.00    100.00    100.00    100.00
          CARBS        68.07     74.88     82.48     91.49     81.25
          TAILS        31.93     25.12     17.52      8.51     18.75



                                METAL  GRADES
                                *************

                         SiO2      Fe2O3     MgO       CaO       Nb2O5


          FEED           13.25     10.74     13.00     22.09      0.47
          CARBS          10.83      9.65     12.87     24.26      0.46
          TAILS          25.33     16.14     13.63     11.25      0.53
```

```
TEST # R81
                           EXPERIMENTAL VALUES
                           *******************

              flowrates                      grades
            solids    % sol.   SiO2    Fe2O3    MgO     CaO     Nb2O5

 FEED       100.00     1.00    11.00   11.00   13.20   22.40    0.49
 CARBS        0.00     1.00     7.29    8.02   12.60   27.50    0.42
 TAILS        0.00     1.00    21.40   14.30   13.50   14.40    0.58

     Cl-W            410.00



                        FLOWRATES AND METAL UNITS
                        *************************

              flowrates                    metal units
            solids    water    SiO2    Fe2O3    MgO     CaO     Nb2O5

  FEED      100.00  9900.00    11.66   10.44   13.01   22.85    0.48
  CARBS      66.42  6985.58     4.55    5.57    8.45   18.07    0.28
  TAILS      33.58  3324.42     7.11    4.87    4.55    4.78    0.20

     Cl-W            410.00



                           METAL RECOVERIES
                           ****************

                      SiO2    Fe2O3    MgO     CaO     Nb2O5


         FEED        100.00  100.00  100.00  100.00  100.00
         CARBS        39.02   53.40   64.98   79.06   59.14
         TAILS        60.98   46.60   35.02   20.94   40.86


                             METAL  GRADES
                             *************

                         SiO2    Fe2O3    MgO     CaO     Nb2O5


         FEED            11.66   10.44   13.01   22.85    0.48
         CARBS            6.85    8.39   12.73   27.20    0.43
         TAILS           21.18   14.49   13.56   14.25    0.58
```

```
TEST # R82
                        EXPERIMENTAL VALUES
                        ********************

             flowrates                            grades
          solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED     100.00     1.00     13.40    11.00    13.20    22.40     0.49
 CARBS      0.00     1.00      7.95     8.41    12.60    27.00     0.43
 TAILS      0.00     1.00     19.60    13.40    13.50    16.00     0.56

    Cl-W            410.00



                    FLOWRATES AND METAL UNITS
                    **************************

             flowrates                          metal units
          solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED    100.00   9900.00    13.29    10.78    13.07    22.15     0.49
  CARBS    54.70   5825.35     4.38     4.67     6.93    14.85     0.24
  TAILS    45.30   4484.65     8.90     6.12     6.14     7.30     0.25

    Cl-W            410.00



                       METAL RECOVERIES
                       ****************

                  SiO2     Fe2O3     MgO      CaO      Nb2O5


        FEED     100.00   100.00   100.00   100.00   100.00
        CARBS     32.99    43.28    53.02    67.04    48.12
        TAILS     67.01    56.72    46.98    32.96    51.88


                         METAL  GRADES
                         *************

                     SiO2     Fe2O3     MgO      CaO      Nb2O5


        FEED         13.29    10.78    13.07    22.15     0.49
        CARBS         8.01     8.53    12.67    27.14     0.43
        TAILS        19.65    13.50    13.56    16.12     0.56
```

```
TEST # R83
                              EXPERIMENTAL VALUES
                              *******************

              flowrates                          grades
           solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED       100.00     1.00    13.40     11.00   13.20    22.40     0.49
CARBS        0.00     1.00     7.97      8.81   12.20    27.00     0.43
TAILS        0.00     1.00    14.70     11.20   13.20    20.90     0.49

     C1-W            410.00



                            FLOWRATES AND METAL UNITS
                            *************************

              flowrates                       metal units
           solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED     100.00  9900.00    13.35     10.83   13.08    22.25     0.48
  CARBS     20.57  2446.56     1.64      1.82    2.51     5.56     0.09
  TAILS     79.43  7863.45    11.71      9.01   10.56    16.69     0.39

     C1-W            410.00



                              METAL RECOVERIES
                              ****************

                   SiO2     Fe2O3    MgO      CaO      Nb2O5


        FEED     100.00    100.00   100.00   100.00   100.00
        CARBS     12.30     16.81    19.23    24.99    18.39
        TAILS     87.70     83.19    80.77    75.01    81.61



                                METAL  GRADES
                                *************

                      SiO2     Fe2O3    MgO      CaO      Nb2O5


        FEED         13.35     10.83   13.08    22.25     0.48
        CARBS         7.98      8.85   12.23    27.03     0.43
        TAILS        14.74     11.34   13.30    21.02     0.50
```

TEST # R90

EXPERIMENTAL VALUES
****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	10.10	10.30	13.00	25.70	0.53
TAILS	0.00	1.00	21.30	12.80	14.00	16.40	0.43
Cl-W		410.00					

FLOWRATES AND METAL UNITS
****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	13.55	11.06	13.27	22.63	0.50
CARBS	68.43	7184.99	6.84	7.02	8.86	17.48	0.36
TAILS	31.57	3125.02	6.71	4.03	4.41	5.15	0.14
Cl-W		410.00					

METAL RECOVERIES
****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	50.49	63.51	66.76	77.23	72.71
TAILS	49.51	36.49	33.24	22.77	27.29

METAL GRADES
****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.55	11.06	13.27	22.63	0.50
CARBS	10.00	10.26	12.95	25.54	0.53
TAILS	21.25	12.78	13.98	16.33	0.43

TEST # R90A

EXPERIMENTAL VALUES

****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	10.60	10.50	12.80	25.60	0.52
TAILS	0.00	1.00	19.40	12.10	13.80	17.30	0.43
Cl-W		410.00					

FLOWRATES AND METAL UNITS

****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	13.58	11.04	13.17	22.59	0.49
CARBS	65.06	6850.83	6.82	6.82	8.34	16.57	0.34
TAILS	34.94	3459.17	6.76	4.22	4.83	6.02	0.15
Cl-W		410.00					

METAL RECOVERIES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	50.24	61.74	63.35	73.35	69.26
TAILS	49.76	38.26	36.65	26.65	30.74

METAL GRADES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.58	11.04	13.17	22.59	0.49
CARBS	10.48	10.48	12.82	25.47	0.52
TAILS	19.34	12.09	13.81	17.23	0.43

```
TEST # R90B
                            EXPERIMENTAL VALUES
                            *******************

                flowrates                                   grades
              solids    % sol.    SiO2      Fe2O3    MgO       CaO       Nb2O5

  FEED        100.00     1.00    13.40      11.00    13.20     22.40     0.49
  CARBS         0.00     1.00    10.30      10.30    12.70     25.50     0.50
  TAILS         0.00     1.00    17.00      11.70    13.70     19.30     0.45

      Cl-W              410.00



                         FLOWRATES AND METAL UNITS
                         *************************

                flowrates                                 metal units
              solids     water    SiO2      Fe2O3    MgO       CaO       Nb2O5

    FEED      100.00   9900.00    13.48     10.98    13.19     22.48     0.48
    CARBS      52.05   5562.96     5.34      5.37     6.61     13.25     0.26
    TAILS      47.95   4747.04     8.13      5.61     6.57      9.23     0.22

      Cl-W              410.00



                           METAL RECOVERIES
                           ****************

                       SiO2      Fe2O3     MgO       CaO       Nb2O5


             FEED     100.00    100.00    100.00    100.00    100.00
             CARBS     39.63     48.87     50.16     58.93     54.67
             TAILS     60.37     51.13     49.84     41.07     45.33



                             METAL   GRADES
                             *************

                          SiO2      Fe2O3     MgO       CaO       Nb2O5


             FEED        13.48      10.98     13.19     22.48     0.48
             CARBS       10.26      10.31     12.71     25.46     0.50
             TAILS       16.96      11.71     13.71     19.26     0.45
```

```
TEST # R90C
                              EXPERIMENTAL VALUES
                              *******************

            flowrates                                  grades
         solids     % sol.     SiO2      Fe2O3     MgO       CaO      0
                                                                       Nb2O5

FEED     100.00      1.00      13.40     11.00     13.20     22.40     0.49
CARBS      0.00      1.00      10.60     10.30     13.10     25.60     0.51
TAILS      0.00      1.00      16.60     11.60     13.60     19.50     0.47

    Cl-W            410.00



                           FLOWRATES AND METAL UNITS
                           *************************

            flowrates                                metal units
         solids     water      SiO2      Fe2O3     MgO       CaO       Nb2O5

  FEED   100.00    9900.00     13.52     10.96     13.30     22.52     0.49
  CARBS   50.46    5405.20      5.32      5.21      6.58     12.89     0.26
  TAILS   49.54    4904.80      8.20      5.76      6.71      9.63     0.23

    Cl-W            410.00



                              METAL RECOVERIES
                              ****************

                    SiO2      Fe2O3      MgO       CaO       Nb2O5


         FEED      100.00    100.00    100.00    100.00    100.00
         CARBS      39.36     47.49     49.51     57.23     52.50
         TAILS      60.64     52.51     50.49     42.77     47.50



                                METAL  GRADES
                                *************

                       SiO2      Fe2O3     MgO       CaO       Nb2O5


         FEED          13.52     10.96     13.30     22.52     0.49
         CARBS         10.54     10.32     13.05     25.54     0.51
         TAILS         16.54     11.62     13.55     19.44     0.47
```

```
TEST # R100
                          EXPERIMENTAL VALUES
                          *******************

               flowrates                            grades
            solids    % sol.    SiO2      Fe2O3    MgO       CaO       Nb2O5

FEED        100.00     1.00    13.40     11.00    13.20     22.40      0.49
CARBS         0.00     1.00    11.20      9.93    12.40     25.10      0.47
TAILS         0.00     1.00    13.20     10.50    12.60     22.80      0.45

    Cl-W             410.00



                       FLOWRATES AND METAL UNITS
                       *************************

               flowrates                         metal units
            solids    water     SiO2      Fe2O3    MgO       CaO       Nb2O5

 FEED       100.00  9900.00    13.29     10.74    12.90     22.62      0.47
 CARBS        1.19   527.62     0.13      0.12     0.15      0.30      0.01
 TAILS       98.81  9782.38    13.15     10.63    12.75     22.32      0.46

    Cl-W             410.00



                         METAL RECOVERIES
                         ****************

                  SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED     100.00    100.00    100.00    100.00    100.00
        CARBS      1.00      1.10      1.14      1.32      1.19
        TAILS     99.00     98.90     98.86     98.68     98.81



                          METAL   GRADES
                          *************

                    SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED        13.29     10.74    12.90     22.62      0.47
        CARBS       11.20      9.93    12.40     25.10      0.47
        TAILS       13.31     10.75    12.90     22.59      0.47
```

TEST # R100A

EXPERIMENTAL VALUES

****

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	1.00	13.40	11.00	13.20	22.40	0.49
CARBS	0.00	1.00	12.80	10.40	12.70	24.30	0.49
TAILS	0.00	1.00	16.60	12.20	13.40	19.90	0.43
Cl-W		410.00					

FLOWRATES AND METAL UNITS

****

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	9900.00	13.78	10.98	13.02	22.73	0.48
CARBS	68.43	7184.99	8.58	7.13	8.77	16.48	0.34
TAILS	31.57	3125.02	5.20	3.85	4.25	6.25	0.14
Cl-W		410.00					

METAL RECOVERIES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	62.26	64.91	67.38	72.50	71.35
TAILS	37.74	35.09	32.62	27.50	28.65

METAL GRADES

****

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.78	10.98	13.02	22.73	0.48
CARBS	12.54	10.41	12.82	24.08	0.50
TAILS	16.48	12.21	13.46	19.80	0.43

```
TEST # R100B
                              EXPERIMENTAL VALUES
                              *******************

              flowrates                                grades
            solids     % sol.     SiO2      Fe2O3      MgO       CaO       Nb2O5

 FEED       100.00      1.00     13.40      11.00     13.20     22.40      0.49
 CARBS        0.00      1.00     10.10      10.10     11.00     24.10      0.41
 TAILS        0.00      1.00     13.20      10.50     12.80     13.80      0.43

     Cl-W              410.00



                           FLOWRATES AND METAL UNITS
                           *************************

              flowrates                              metal units
            solids     water      SiO2      Fe2O3      MgO       CaO       Nb2O5

  FEED      100.00   9900.00     11.84      10.51     12.12     21.94      0.44
  CARBS      76.17   7950.80      8.60       7.98      9.01     18.62      0.34
  TAILS      23.83   2359.20      3.23       2.53      3.11      3.31      0.11

     Cl-W              410.00



                               METAL RECOVERIES
                               ****************

                      SiO2      Fe2O3      MgO        CaO       Nb2O5


       FEED         100.00     100.00    100.00     100.00     100.00
       CARBS         72.67      75.92     74.32      84.89      76.34
       TAILS         27.33      24.08     25.68      15.11      23.66



                                 METAL GRADES
                                 ************

                         SiO2       Fe2O3      MgO       CaO       Nb2O5


       FEED              11.84      10.51     12.12     21.94       0.44
       CARBS             11.29      10.47     11.82     24.45       0.45
       TAILS             13.57      10.62     13.06     13.91       0.44
```

```
TEST # R100C
                          EXPERIMENTAL VALUES
                          *******************

                  flowrates                         grades
                solids    % sol.    SiO2      Fe2O3     MgO      CaO      Nb2O5

 FEED           10.00     1.00     13.40     11.00     13.20    22.40     0.49
 CARBS           0.00     1.00     12.30     10.20     12.50    24.40     0.46
 TAILS           0.00     1.00     15.60     11.70     13.80    20.50     0.42

     Cl-W               410.00



                       FLOWRATES AND METAL UNITS
                       *************************

                  flowrates                       metal units
                solids    water     SiO2      Fe2O3     MgO      CaO      Nb2O5

   FEED         10.00    990.00     1.37      1.09      1.31     2.26     0.05
   CARBS         5.51    955.70     0.67      0.56      0.69     1.34     0.03
   TAILS         4.49    444.30     0.70      0.53      0.62     0.92     0.02

     Cl-W               410.00



                          METAL RECOVERIES
                          ****************

                        SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED          100.00    100.00    100.00    100.00    100.00
        CARBS          49.09     51.74     52.69     59.38     57.46
        TAILS          50.91     48.26     47.31     40.62     42.54



                            METAL   GRADES
                            *************

                          SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED              13.65     10.92     13.12     22.57      0.46
        CARBS             12.16     10.25     12.54     24.31      0.48
        TAILS             15.49     11.74     13.83     20.43      0.43
```

```
TEST # R100D
                         EXPERIMENTAL VALUES
                         *******************

              flowrates                         grades
           solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED       100.00     1.00     13.40    11.00    13.20    22.40     0.49
CARBS        0.00     1.00     12.70    10.30    12.60    23.90     0.45
TAILS        0.00     1.00     18.90    12.30    13.70    18.20     0.42

   Cl-W             410.00



                     FLOWRATES AND METAL UNITS
                     *************************

              flowrates                        metal units
           solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED      100.00   9900.00    13.70    10.81    12.97    22.64     0.46
 CARBS      80.65   8393.97    10.05     8.43    10.31    19.12     0.38
 TAILS      19.35   1916.03     3.65     2.39     2.66     3.51     0.08

   Cl-W             410.00



                        METAL RECOVERIES
                        ****************

                 SiO2      Fe2O3      MgO       CaO       Nb2O5


      FEED      100.00    100.00    100.00    100.00    100.00
      CARBS      73.37     77.92     79.49     84.48     82.22
      TAILS      26.63     22.08     20.51     15.52     17.78


                          METAL  GRADES
                          *************

                   SiO2     Fe2O3     MgO      CaO       Nb2O5


      FEED         13.70    10.81    12.97    22.64      0.46
      CARBS        12.46    10.45    12.78    23.71      0.47
      TAILS        18.84    12.34    13.74    18.15      0.43
```

```
TEST # R100E
                              EXPERIMENTAL VALUES
                              *******************

              flowrates                         grades
            solids   % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED      100.00    1.00    13.40    11.00    13.20    22.40     0.49
  CARBS       0.00    1.00    12.70    10.30    12.60    23.60     0.44
  TAILS       0.00    1.00    17.60    11.80    12.90    20.00     0.44

      Cl-W           410.00



                           FLOWRATES AND METAL UNITS
                           *************************

              flowrates                       metal units
            solids    water    SiO2     Fe2O3    MgO      CaO      Nb2O5

    FEED    100.00  9900.00   13.64    10.78    12.88    22.64     0.46
    CARBS    77.76  8108.57    9.73     8.14     9.99    18.21     0.36
    TAILS    22.24  2201.43    3.90     2.63     2.88     4.44     0.10

      Cl-W           410.00



                              METAL RECOVERIES
                              ****************

                     SiO2     Fe2O3     MgO       CaO      Nb2O5


            FEED    100.00   100.00   100.00    100.00    100.00
            CARBS    71.38    75.55    77.60     80.41     78.39
            TAILS    28.62    24.45    22.40     19.59     21.61



                                METAL  GRADES
                                *************

                        SiO2     Fe2O3    MgO      CaO      Nb2O5


            FEED        13.64    10.78    12.88    22.64     0.46
            CARBS       12.52    10.47    12.85    23.41     0.46
            TAILS       17.55    11.85    12.97    19.95     0.45
```

```
TEST # R100F
                         EXPERIMENTAL VALUES
                         *******************

            flowrates                          grades
          solids    % sol.   SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED     100.00     1.00    13.40    11.00    13.20    22.40     0.49
 CARBS      0.00     1.00     8.72     8.53    11.40    28.70     0.43
 TAILS      0.00     1.00    14.60    11.20    13.10    21.50     0.49

     C1-W          410.00



                       FLOWRATES AND METAL UNITS
                       *************************

            flowrates                        metal units
          solids    water    SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED    100.00  9900.00    13.61    10.90    13.01    22.47     0.49
  CARBS    14.20  1815.49     1.23     1.21     1.62     4.07     0.06
  TAILS    85.80  8494.51    12.37     9.69    11.38    18.40     0.42

     C1-W          410.00



                         METAL RECOVERIES
                         ****************

                    SiO2     Fe2O3    MgO      CaO      Nb2O5

       FEED       100.00   100.00   100.00   100.00   100.00
       CARBS        9.07    11.13    12.47    18.13    12.60
       TAILS       90.93    88.87    87.53    81.87    87.40


                           METAL  GRADES
                           *************

                      SiO2     Fe2O3    MgO      CaO      Nb2O5

       FEED          13.61    10.90    13.01    22.47     0.49
       CARBS          8.69     8.54    11.43    28.69     0.43
       TAILS         14.42    11.29    13.27    21.44     0.49
```

```
TEST # R100H
                          EXPERIMENTAL VALUES
                          *******************

               flowrates                           grades
             solids    % sol.   SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED        100.00     1.00   13.40    11.00    13.20    22.40     0.49
 CARBS         0.00     1.00   11.20     9.87    12.70    25.70     0.46
 TAILS         0.00     1.00   18.70    12.40    13.20    18.30     0.46

     Cl-W             410.00



                        FLOWRATES AND METAL UNITS
                        *************************

               flowrates                         metal units
             solids     water   SiO2     Fe2O3    MgO      CaO      Nb2O5

   FEED      100.00   9900.00  13.80    10.88    13.00    22.75     0.47
   CARBS      62.55   6602.67   6.85     6.22     8.02    15.94     0.30
   TAILS      37.45   3707.33   6.95     4.66     4.97     6.80     0.18

     Cl-W             410.00



                          METAL RECOVERIES
                          ****************

                     SiO2     Fe2O3     MgO      CaO      Nb2O5


          FEED      100.00   100.00   100.00   100.00   100.00
          CARBS      49.65    57.17    61.74    70.08    62.80
          TAILS      50.35    42.83    38.26    29.92    37.20


                            METAL  GRADES
                            *************

                       SiO2     Fe2O3     MgO      CaO      Nb2O5


          FEED         13.80    10.88    13.00    22.75     0.47
          CARBS        10.95     9.94    12.83    25.48     0.47
          TAILS        18.55    12.44    13.28    18.17     0.47
```

```
TEST # R105
                         EXPERIMENTAL VALUES
                         *******************

                flowrates                            grades
             solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED        100.00     1.00     13.40    11.00    13.20    22.40     0.49
 CARBS         0.00     1.00     10.90     9.71    13.00    24.60     0.46
 TAILS         0.00     1.00     21.50    14.00    13.20    15.00     0.53

     Cl-W              410.00



                     FLOWRATES AND METAL UNITS
                     *************************

               flowrates                          metal units
             solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

  FEED       100.00  9900.00     13.42    10.84    13.11    22.34     0.48
  CARBS       76.05  7939.29      8.28     7.48     9.94    18.74     0.35
  TAILS       23.95  2370.71      5.15     3.36     3.17     3.60     0.13

    Cl-W               410.00



                        METAL RECOVERIES
                        ****************

                  SiO2     Fe2O3     MgO      CaO      Nb2O5


       FEED      100.00   100.00   100.00   100.00   100.00
       CARBS      61.66    68.99    75.84    83.91    73.57
       TAILS      38.34    31.01    24.16    16.09    26.43



                         METAL  GRADES
                         *************

                    SiO2     Fe2O3    MgO      CaO      Nb2O5


       FEED         13.42    10.84    13.11    22.34     0.48
       CARBS        10.88     9.83    13.07    24.65     0.47
       TAILS        21.49    14.04    13.22    15.01     0.53
```

APPENDIX 8. Summary of Laboratory Column Flotation Test Conditions

TABLE A.8.1 Summary of Flotation Conditions

TEST	DISPERSANT (CONC. %)	FA	NaSiO3	STARCH	FROTHER (ppm)	pH	Jg (cm/s)	OBSERVATIONS
		kg/tonne						
R1	-	4.5	1	-	DF250 (20)	8.1	0.6	stable
R2	-	4.5	2	-	DF250 (20)	8.1	0.6	stable
R3	-	4.5	4	-	DF250 (20)	8.1	0.6	stable
R4	-	4.5	3	-	DF250 (20)	8.1	0.6	stable
R5	-	2.25	3	-	DF250 (20)	8.1	0.6	stable
R6	-	6.5	3	-	DF250 (20)	8.1	0.6	stable
R10	-	4.5	5.5	-	DF250 (20)	8.1	0.6	stable
R11	-	4.5	7	-	DF250 (20)	8.1	0.6	stable
R18	calgon (0.21)	4.5	-	-	DF250 (20)	11.5	0.6	carb. depress.
R19	calgon (0.21)	4.5	-	-	DF250 (20)	7.4	0.6	carb. depress.
R20	EDTA (0.098) calgon (0.053)	4.5	-	-	DF250 (20)	11	0.6	carb. depress.
R21	calgon (0.053)	4.5	-	-	DF250 (20)	11.6	0.6	poor dispersion
R22	calgon (0.105) ultrasonic treat	4.5	-	-	DF250 (20)	11.6	0.6	poor dispersion
R23	lignosulph. SFX (0.1)	4.5	-	-	DF250 (20)	11.6	0.6	poor silicate - depression froth unstable
R24	lignosulph. SFX (0.1)	4.5	2	-	DF250 (20)	11.5	0.6	froth unstable better results
R25	lignosulph. SFX (0.1)	4.5	4	-	DF250 (20)	11	0.6	froth unstable
R26	calgon (0.6)	4.5	-	-	DF250 (20)	11.5	0.6	carb. depress.

TABLE **A.8.1 Summary of Flotation Conditions**
(Continuation)

TEST	DISPERSANT (CONC. %)	FA	NaSiO3 kg/tonne	STARCH	FROTHER (ppm)	pH	Jg (cm/s)	OBSERVATIONS
T27	DA 811 (0.05)	4.5	-	0.2	DF250 (20)	8.1	0.6	carb. depress.
T27A	DA 811 (0.05)	4.5	-	0.2	DF250 (20)	8.1	0.6	froth collapse
T27B no solids	DA 811 (0.05)	4.5	-	0.2	DF250 (20)	8.1	0.6	stable
T27C	DA 811 (0.05)	4.5	-	0.2	DF250 (20)	8.1	0.6	froth collapse
T27D	DA 811 (0.05)	4.5	-	0.2	DF400 (20)	8.1	0.6	froth collapse
T27E	DA 811 (0.0250)	4.5	-	0.2	DF250 (20)	8.1	0.6	froth collapse
T29	DA 811 (0.05)	4.5	-	0.2	DF250 (20)	11	1.0	froth collapse
T29	DA 811 (0.05)	4.5	-	0.2	DF250 (20)	11	1.0	froth collapse
T29A	DA 811 (0.025)	4.5	-	0.2	DF250 (20)	11	1.0	froth collapse
T29B	DA 811 (0.0125)	4.5	-	0.2	DF250 (20)	11	1.0	froth collapse
T29C	DA 811 (0.0625)	4.5	-	0.2	DF250 (20)	11	1.0	poor dispersion
R32	-	4.5	3	0.5	DF250 (20)	8.2	1.0	froth unstable
R33	-	4.5	3	0.5	DF250 (20)	8.2	1.0	froth unstable
R30	DA 811 (0.00625)	4.5	3	0.4	DF250 (20)	8.1	1.0	carb. depress. and froth collapse
R31	DA 811 (0.00625)	4.5	3	0.4	DF250 (20)	8.1	1.0	carb. depress. and froth collapse
T28	AQ 55D (0.025)	4.5	-	0.2	DF250 (20)	11.2	1.0	no selectiv.
T28A	AQ 55D (0.0125)	4.5	4	-	DF250 (20)	11.2	1.0	froth collapse
T28B	AQ 55D (0.0125)	4.5	4	-	DF250 (20)	11.2	1.0	froth collapse
T28C	AQ 55D (0.0125)	4.5	4	-	DF250 (20)	11.2	1.0	froth collapse
T28D	AQ 55D (0.00625)	4.5	3	0.5	DF250 (20)	11.2	1.0	froth collapse

TABLE A.8.1 Summary of Flotation Conditions
(Continuation)

TEST	DISPERSANT (CONC. %)	FA	NaSiO3	STARCH	FROTHER (ppm)	pH	Jg (cm/s)	OBSERVATIONS
			kg/tonne					
R28	AQ 55D (0.05)	4.5	3	0.5	DF250 (20)	11.2	1.0	stable
R29	AQ 55D (0.02)	4.5	3	0.5	DF250 (20)	11.2	1.2	froth collapse
R29A	AQ 55D (0.02)	4.5	3	0.5	DF250 (20)	11.2	1.0	stable
R29B	AQ 55D (0.00625)	4.5	3	0.5	DF250 (20)	11.2	1.4	stable
R35	AQ 55D (0.05)	-	3	0.5	DF250 (20)	11.2	1.45	stable
R36	AQ 55D (0.03)	-	3	0.5	DF250 (20)	11.2	1.45	stable
T40	ALCOMER							see details in Table 2
T41	ALCOMER							see details in Table 2.
T42	ALCOMER							see details in Table 2.
R50 no solids	-	4.5	3	1	DF250 (20)	8.1	1.0	froth collapse
R50A	-	4.5	3	2	DF250 (20)	8.1	1.0	froth collapse
R50B	-	4.5	3	4	DF250 (20)	8.1	1.0	froth collapse
T50	-	4.5	3	0.5	DF250 (20)	8.1	1.0	strong coales. froth unstable
T51	-	4.5	3	-	DF250 (60)	8.1	1.0	strong coales. froth unstable
T52	-	4.5	3	4	DF250(120)	8.1	1.0	strong coales. froth unstable
T53 no solids	-	4.5	3	4	DF250 (60)	8.1	1.0	strong coales. froth unstable

TABLE A.8.1 Summary of Flotation Conditions
(Continuation)

TEST	DISPERSANT (CONC. %)	FA	NaSiO3 kg/tonne	STARCH	FROTHER (ppm)	pH	Jg (cm/s)	OBSERVATIONS
T54 no solids	-	4.5	3	4	DF250(120)	8.1	1.0	strong coales. froth unstable
T55 no solids	-	4.5	3	4	PINE OIL (40)	8.1	1.0	strong coales. froth unstable
T56 no solids	-	4.5	3	4	PINE OIL (80)	8.1	1.0	bubbly flow regime
T57 no solids	-	4.5	3	4	PINE OIL (60)	8.1	1.0	strong coales. froth collapse
T58 no solids	-	4.5	3	4	MIBC (120)	8.1	1.0	strong coales froth collapse
T59 no solids	-	4.5	3	4	TEB (20)	8.1	1.0	less coales.
T59A no solids	-	4.5	3	4	TEB (40)	8.1	1.0	stable
TESTS CONDUCTED WITH RECYCLE WATER 2								
R80A	-	3	3	2	TEB (20)	8.1	1.0	stable
R80B	-	3	3	4	TEB (20)	8.1	1.0	stable
R80C	-	3	3	6	TEB (20)	8.1	1.0	stable
R81	-	1.5	3	4	TEB (20)	8.1	1.0	stable
R82	-	0.75	3	4	TEB (20)	8.1	1.0	stable
R83	-	0.25	3	4	TEB (20)	8.1	1.0	stable
R90	-	1.5	3	1	TEB (20)	8.1	1.0	stable
R90A	-	1	3	1	TEB (20)	8.1	1.0	stable
R90B	-	1	3	2	TEB (20)	8.1	1.0	stable
R90C	-	1	3	4	TEB (20)	8.1	1.0	stable

TABLE A.8.1 Summary of Flotation Conditions
(Continuation)

TEST	DISPERSANT (CONC. %)	FA	SALT + Na_2SiO_3 kg/tonne	STARCH	FROTHER (ppm)	pH	Jg (cm/s)	OBSERVATIONS
R100	$FeCl_3.6H_2O$+ Na_2SiO_3	1	2.86+ 2.95	-	TEB (20)	9.3	1.0	stable
R100A	$FeCl_3.6H_2O$+ Na_2SiO_3	1	0.95+ 0.98	-	TEB (20)	9.3	1.0	stable
R100B	$(NH_4)_2SO_4$+ Na_2SiO_3	1	14.7+ 2.94	-	TEB (20)	9.3	1.0	stable
R100C	$(NH_4)_2SO_4$+ Na_2SiO_3	1	4.9+ 0.98	-	TEB (20)	9.3	1.0	stable
R100D	$ZnSO_4.7H_2O$+ Na_2SiO_3	1	3.05+ 2.77	-	TEB (20)	9.3	1.0	stable
R100E	$ZnSO_4.7H_2O$+ Na_2SiO_3	1	1.02+ 0.92	-	TEB (20)	9.3	1.0	stable
R100F	Na_2SO_3+ $FeSO_4.7H_2O$+ Na_2SiO_3	1	0.1044+ 0.1044+ 1.88	-	TEB (20)	9.3	1.0	stable
R100H	Na_2SO_3+ $FeSO_4.7H_2O$+ Na_2SiO_3	1	0.052+ 0.052+ 0.940	-	TEB (20)	9.3	1.0	stable
R105	-	4.5	3	0.5	TEB (20)	8.1	1.0	stable

TABLE A.8.2 EFFECTS OF ALCOMER AND Na2SiO3 IN COALESCENCE

TEST No.	ALCOMER 75-L	Na2SiO3	STARCH	FATTY ACID	SOLIDS	COALESCENCE
	kg/tonne					
40	12.5	-	0.5	4.5	YES	none
40A	6	-	-	-	YES	none
40B	4	3	0.5	4.5	YES	strong
40C	4	3	0.5	4.5	NO	strong
40D	4	3	-	-	NO	none
40E	4	3	-	4.5	NO	strong
40F	4	3	-	4.5	NO	some
40E'	4	3	-	4.5	NO	strong
40H	4	-	0.5	4.5	YES	strong
40J	4	-	0.5	4.5	NO	some
41	4	-	-	4.5	NO	some
41A	4	-	-	4.5	NO	some
41B	4	3	-	4.5	NO	strong
41C	4	3	-	4.5	NO	some
41D	-	3	-	4.5	NO	strong
42	-	3	-	4.5	NO	some
42A	-	3	-	-	NO	none
42B	-	-	-	4.5	NO	some
42C	4	3	-	4.5		some

APPENDIX 9 . Observed (using water manometers) vs Actual Gas Holdup, for Pulp Densities from 1.0 to 1.4.

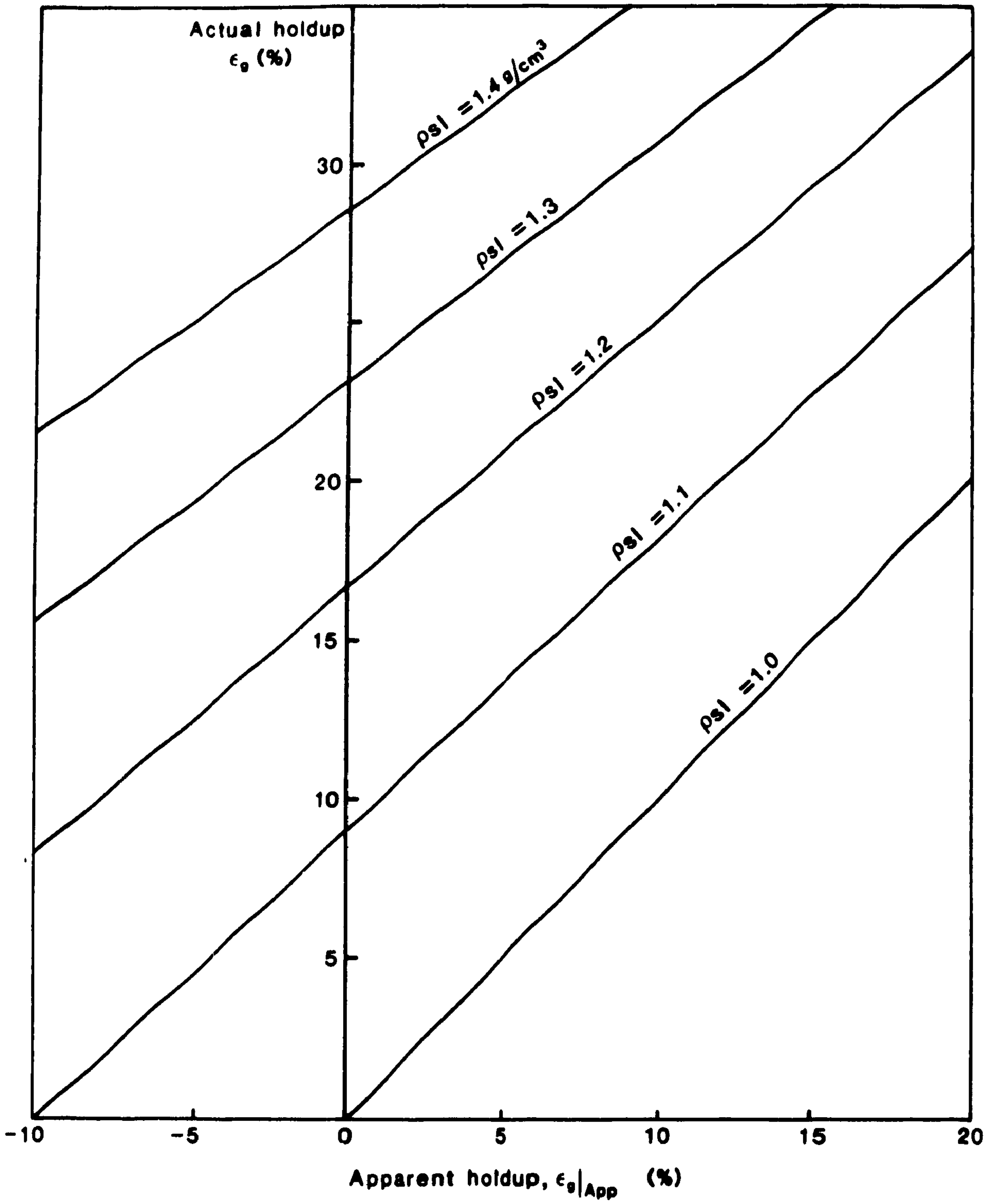
Using Eqs. 9 and 11 a relationship can be calculated to correct the observed (using water manometers) vs the actual gas holdup at any pulp density. Figure A.8.1 shows these relationships for slurry densities from 1.1 to 1.4 g/cm..

$$Eg = 1 - \frac{\rho_w}{\rho_{sl}} \left(1 - \frac{\Delta h}{\Delta L}\right) \qquad (9)$$

$$Eg = \frac{\Delta h}{\Delta L} \qquad (11)$$

For example, if for a slurry $\rho_{sl} = 1.1$ the observed gas holdup is 5.% the actual is about 12.%. Note that negative values of observed holdup are possible.

Figure A.8.1


Actual holdup
ϵ_g (%)
30
20
15
10
5
-10
-5
0
5
10
15
20
Apparent holdup, $\epsilon_g|_{App}$ (%)
ρsl =1.4 g/cm³
ρsl =1.3
ρsl =1.2
ρsl =1.1
ρsl =1.0

APPENDIX 10. Gas Flowmeter Calibration for the Pilot Size Column used at Niobec.

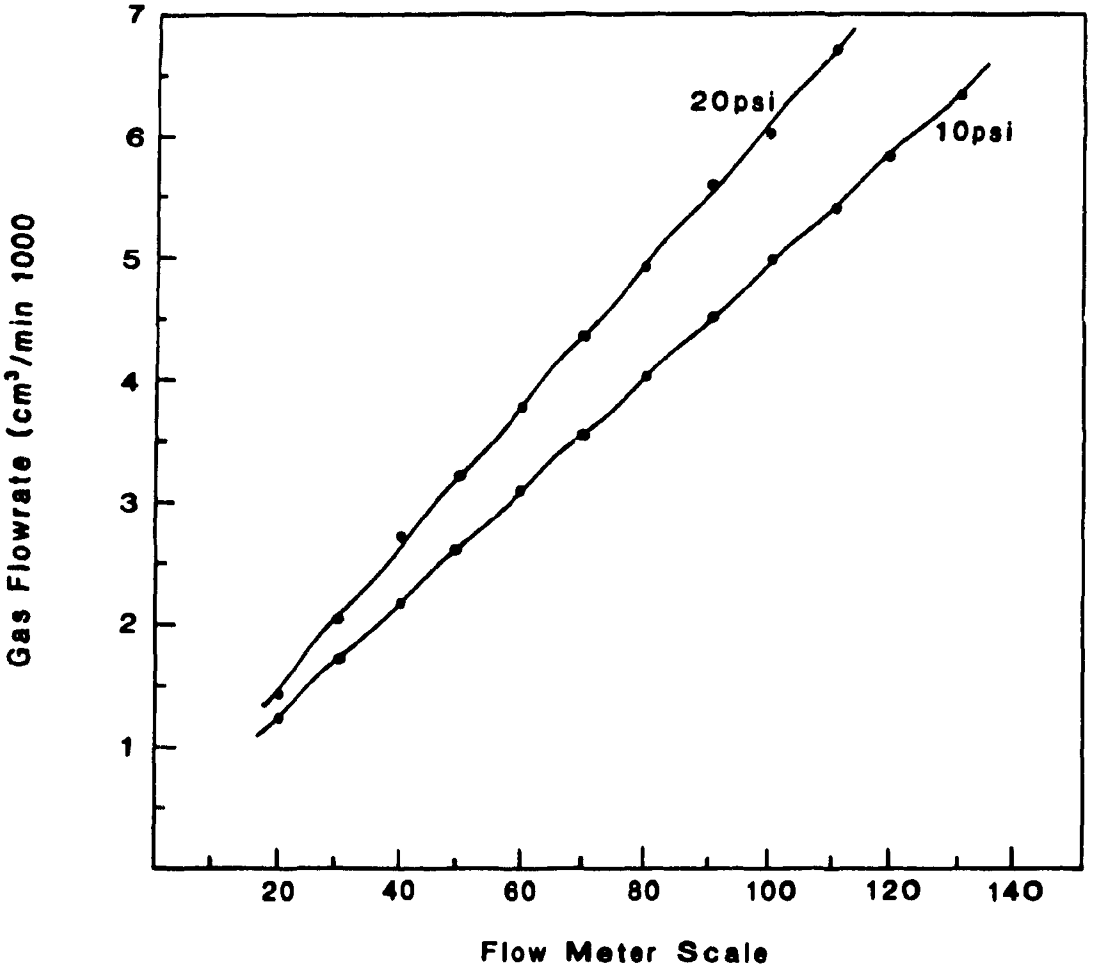


Figure A.10.1 Gas flowmeter calibration

APPENDIX 11. Flotation Results Obtained at Niobec Experimental and Adjusted Assys: Metal Distribution.

```
TEST # T1
                              EXPERIMENTAL VALUES
                              *******************

              flowrates                              grades
           solids     % sol.    SiO2      Fe2O3     MgO       CaO       Nb2O5

 FEED      140.40     14.80     8.30      7.85     14.30     27.20      0.72
 CARBS       0.00     19.90     1.10      4.35     15.10     33.10      0.18
 TAILS       0.00      5.80    15.10     10.50     12.60     21.10      1.19

     C1-W             600.00



                           FLOWRATES AND METAL UNITS
                           *************************

              flowrates                              metal units
           solids     water     SiO2      Fe2O3     MgO       CaO       Nb2O5

  FEED     140.40    808.25    11.58     10.67     19.64     38.00      0.99
  CARBS     69.03    249.12     0.78      3.09     10.53     22.89      0.13
  TAILS     71.37   1159.13    10.80      7.58      9.11     15.11      0.86

    C1-W             600.00



                              METAL RECOVERIES
                              ****************

                       SiO2      Fe2O3     MgO       CaO       Nb2O5


         FEED        100.00    100.00    100.00    100.00    100.00
         CARBS         6.72     28.93     53.63     60.25     13.21
         TAILS        93.28     71.07     46.37     39.75     86.79



                                 METAL  GRADES
                                 *************

                          SiO2      Fe2O3     MgO       CaO       Nb2O5


         FEED              8.24      7.60     13.99     27.07      0.70
         CARBS             1.13      4.47     15.25     33.17      0.19
         TAILS            15.13     10.63     12.76     21.17      1.20
```

```
TEST # T2
                        EXPERIMENTAL VALUES
                        *******************

           flowrates                            grades
           solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED       138.80    13.90     8.26     7.67     14.10    27.50    0.68
CARBS        0.00    16.30     1.06     4.44     15.30    33.10    0.14
TAILS        0.00     7.10    12.60     9.46     13.00    23.60    1.02

    Cl-W            600.00



                      FLOWRATES AND METAL UNITS
                      *************************

           flowrates                          metal units
           solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED      138.80    859.76    11.31    10.49    19.39    38.00    0.94
 CARBS      54.19    352.67     0.60     2.43     8.32    17.96    0.08
 TAILS      84.61   1107.09    10.72     8.06    11.07    20.03    0.86

    Cl-W            600.00



                        METAL RECOVERIES
                        ****************

                  SiO2      Fe2O3    MgO       CaO       Nb2O5


        FEED      100.00    100.00   100.00    100.00    100.00
        CARBS       5.28     23.16    42.91     47.28      8.12
        TAILS      94.72     76.84    57.09     52.72     91.88


                         METAL  GRADES
                         *************

                     SiO2      Fe2O3    MgO       CaO       Nb2O5


        FEED         8.15      7.56     13.97     27.37     0.68
        CARBS        1.10      4.48     15.35     33.15     0.14
        TAILS       12.67      9.53     13.08     23.68     1.02
```

TEST # T3

EXPERIMENTAL VALUES

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	130.50	14.30	8.07	7.59	14.10	27.00	0.65
CARBS	0.00	23.50	15.80	4.58	15.20	32.80	0.20
TAILS	0.00	5.30	15.80	10.80	13.00	21.30	1.17
Cl-W		616.00					

FLOWRATES AND METAL UNITS

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	130.50	782.09	17.26	9.99	18.40	35.28	0.88
CARBS	65.30	233.04	8.63	2.97	9.92	21.41	0.12
TAILS	65.20	1165.04	8.62	7.02	8.48	13.88	0.76
Cl-W		616.00					

METAL RECOVERIES

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	50.03	29.72	53.94	60.67	14.01
TAILS	49.97	70.28	46.06	39.33	85.99

METAL GRADES

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	13.22	7.66	14.10	27.04	0.67
CARBS	13.22	4.55	15.20	32.78	0.19
TAILS	13.23	10.77	13.00	21.28	1.16

```
TEST # T4
                         EXPERIMENTAL VALUES
                         *******************

               flowrates                          grades
             solids    % sol.   SiO2    Fe2O3   MgO      CaO      Nb2O5

 FEED        149.60    14.20    8.06    7.54   14.20    27.10     0.64
 CARBS         0.00    19.70    1.11    4.46   15.30    32.90     0.13
 TAILS         0.00     6.00   16.50   10.80   12.80    20.40     1.22

     Cl-W              631.00



                       FLOWRATES AND METAL UNITS
                       *************************

               flowrates                        metal units
             solids    water    SiO2    Fe2O3   MgO      CaO      Nb2O5

   FEED      149.60   903.92   12.17   11.11   21.20    40.61     0.95
   CARBS      80.99   460.00    0.87    3.66   12.40    26.63     0.11
   TAILS      68.61  1074.93   11.30    7.45    8.79    13.98     0.84

     Cl-W              631.00



                         METAL RECOVERIES
                         ****************

                    SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED      100.00    100.00    100.00    100.00    100.00
        CARBS       7.13     32.97     58.52     65.57     11.42
        TAILS      92.87     67.03     41.48     34.43     88.58



                          METAL  GRADES
                          *************

                       SiO2      Fe2O3     MgO       CaO       Nb2O5


        FEED           8.13      7.43     14.17     27.14      0.63
        CARBS          1.07      4.52     15.32     32.88      0.13
        TAILS         16.47     10.85     12.81     20.38      1.22
```

TEST # T5

EXPERIMENTAL VALUES

	flowrates		grades				
	solids	% sol.	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	137.10	13.90	7.97	7.46	14.40	27.30	0.60
CARBS	0.00	22.50	1.57	4.72	15.20	31.80	0.29
TAILS	0.00	2.90	28.60	15.50	10.90	11.40	1.70
Cl-W		646.00					

FLOWRATES AND METAL UNITS

	flowrates		metal units				
	solids	water	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	137.10	849.23	10.78	10.01	19.58	37.26	0.83
CARBS	105.52	437.88	1.74	5.11	16.13	33.65	0.30
TAILS	31.58	1057.35	9.04	4.91	3.45	3.61	0.54
Cl-W		646.00					

METAL RECOVERIES

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	100.00	100.00	100.00	100.00	100.00
CARBS	16.16	51.01	82.38	90.32	35.78
TAILS	83.84	48.99	17.62	9.68	64.22

METAL GRADES

	SiO2	Fe2O3	MgO	CaO	Nb2O5
FEED	7.86	7.30	14.28	27.18	0.61
CARBS	1.65	4.84	15.29	31.89	0.28
TAILS	28.62	15.54	10.93	11.43	1.70

```
TEST # T6
                         EXPERIMENTAL VALUES
                         *******************

              flowrates                           grades
            solids    % sol.    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED       158.90    14.70     7.72     7.23    14.10    27.00     0.63
 CARBS        0.00    28.30     1.29     4.70    15.50    32.60     0.20
 TAILS        0.00     5.20    16.30    10.90    12.80    20.80     1.14

     Cl-W            631.00


                      FLOWRATES AND METAL UNITS
                      *************************

              flowrates                        metal units
            solids    water     SiO2     Fe2O3    MgO      CaO      Nb2O5

   FEED     158.90    922.05    12.53    11.73    22.61    43.27     0.99
   CARBS     88.19    263.87     1.06     4.07    13.60    28.64     0.18
   TAILS     70.71   1289.19    11.47     7.66     9.01    14.64     0.81

     Cl-W            631.00


                         METAL RECOVERIES
                         ****************

                   SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED     100.00   100.00   100.00   100.00   100.00
         CARBS      8.43    34.70    60.16    66.18    18.22
         TAILS     91.57    65.30    39.84    33.82    81.78


                            METAL   GRADES
                            **************

                      SiO2     Fe2O3     MgO      CaO      Nb2O5


         FEED          7.89     7.38    14.23    27.23     0.62
         CARBS         1.20     4.62    15.43    32.47     0.20
         TAILS        16.23    10.83    12.74    20.70     1.14
```

```
TEST # PLANT
                          EXPERIMENTAL VALUES
                          *******************

              flowrates                           grades
            solids   % sol.   SiO2     Fe2O3    MgO      CaO      Nb2O5

FEED        100.00    1.00     7.77     7.32    14.30    27.10     0.65
CARBS         0.00    1.00     2.54     5.26    15.30    31.30     0.30
TAILS         0.00    1.00    13.20     9.52    13.50    23.20     0.99

   C1-W              410.00



                       FLOWRATES AND METAL UNITS
                       *************************

              flowrates                        metal units
            solids    water    SiO2     Fe2O3    MgO      CaO      Nb2O5

 FEED       100.00  9900.00    7.84     7.37    14.37    27.20     0.65
 CARBS       49.96  5356.44    1.25     2.62     7.63    15.61     0.15
 TAILS       50.04  4953.56    6.59     4.75     6.74    11.58     0.50

   C1-W              410.00



                         METAL RECOVERIES
                         ****************

                  SiO2      Fe2O3     MgO       CaO       Nb2O5


       FEED      100.00    100.00    100.00    100.00    100.00
       CARBS      15.97     35.51     53.10     57.41     23.30
       TAILS      84.03     64.49     46.90     42.59     76.70


                          METAL  GRADES
                          *************

                    SiO2      Fe2O3     MgO       CaO       Nb2O5


       FEED          7.84      7.37     14.37     27.20      0.65
       CARBS         2.51      5.24     15.27     31.25      0.30
       TAILS        13.17      9.50     13.47     23.15      0.99
```