

8214.

FLOTATION OF KUTLULAR
PYRITIC COPPER ORE

A MASTER'S THESIS
in
Mining Engineering
Middle East Technical University

By

Unal Akdemir
February, 1990

T. C.
YÜKSEKÖĞRETİM KURULU
Dokümantasyon Merkezi

Approval of the Graduate School of Natural and Applied Sciences.

R. Seve
for Prof. Dr. Alpay ANKARA
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science in Mining Engineering.

Gülhan Özbayoğlu
Prof. Dr. Gülhan ÖZBAYOĞLU
Chairman of the Department

We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science in Mining Engineering.

Çetin Hoşten
Assoc. Prof. Dr. Çetin HOŞTEN
Co - Supervisor

Ü. Atalay
Assoc. Prof. Dr. Ümit ATALAY
Supervisor

Examining Committee in Charge:

Prof. Dr. Gülhan Özbayoğlu (Chairman)

Assoc. Prof. Dr. Ümit Atalay

Assoc. Prof. Dr. Çetin Hoşten

Asst. Prof. Dr. Cahit Hiçyılmaz

Dr. Muammer Hacıoğlu (KBİ)

Flotation of pyritic copper ore with 75 g/t collector
LSB yielded a concentrate containing 19.14 % Cu with 69.11 %
recovery after two cleaning steps.



Key Words : Flotation, chalcopyrite, pyrite.

Scientific Code : 607 - 02 - 04

ÖZET

KUTLULAR PİRİTİK BAKIR CEVHERİNİN FLOTASYONU

AKDEMİR, Ünal

Yüksek Lisans Tezi, Maden Müh. Bölümü

Tez Yöneticisi:Doç.Dr. Ümit ATALAY

Yardımcı Tez Yöneticisi:Doç.Dr. Çetin HOŞTEN

Şubat 1990, 45 sayfa

Bu çalışmanın amacı Kutlular piritik bakır cevherinin flotasyon parametrelerinin optimizasyonudur. Yapılan mineralojik analizler, cevherin başlıca pirit ve kalkopirit içerdiğini göstermiştir.

Tane boyutunun, pH'ın, kollektör çeşidi ve miktarının, flotasyon süresinin, köpürtücü türünün ve NaCN ilavesinin konsantre tenör ve verimi üzerindeki etkisi yapılan flotasyon testleriyle incelenmiştir. Ayrıca, laboratuvar sonuçlarından yararlanılarak , "locked - cycle" test sonuçlarının tahmini üzerine de teorik bir çalışma yapılmıştır.

75 g/ton kollektör LSB kullanarak elde edilen kaba konsantrenin iki kez temizlenmesi ile % 19.14'lük bir konsantre % 69.11'lik bir verimle elde edilmiştir.

Anahtar Kelimeler : Flotasyon, Kalkopirit, Pirit

Bilim Dalı Kodu : 607 - 02 - 04

ACKNOWLEDGEMENTS

I wish to express my appreciation to Assoc. Prof. Dr. Ümit Atalay for his kind supervision, valuable suggestions and discussions throughout this research.

I also wish to express my thanks to Assoc. Prof. Dr. Çetin Hoşten for his suggestions and discussions throughout this study.

I express my thanks to Assist. Prof. Dr. Cahit Hiçyılmaz for his kind helps during laboratory studies.

I also wish to thank Savaş Şener for his helps and interest during this study.

Finally, I wish to express my gratitude to Tuncer Gençtan, Hülya Sakınmaz and Yunus Koçak who helped in various ways.

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	iii
ÖZET	v
ACKNOWLEDGEMENTS	vii
LIST OF TABLES	x
LIST OF FIGURES	xi
1. INTRODUCTION	1
2. LITERATURE SURVEY	2
2.1. OCCURRENCE OF COPPER ORES	2
2.2. FLOTATION OF COPPER SULPHIDE ORES	3
2.2.1. Effect of pH	5
2.2.2. Effect of Collector	8
2.2.3. Effect of Frother	10
3. EXPERIMENTAL STUDIES	12
3.1. PREPARATION OF TEST SAMPLES	12
3.2. MINERALOGICAL AND CHEMICAL ANALYSIS	
OF SAMPLE	12
3.3. GRINDING TESTS	14
3.4. FLOTATION TESTS	14
4. EXPERIMENTAL RESULTS AND DISCUSSIONS	17
4.1. EFFECT OF PARTICLE SIZE ON FLOTATION	17
4.2. EFFECT OF COLLECTOR TYPE ON FLOTATION	19
4.3. EFFECT OF pH ON FLOTATION	23
4.4. EFFECT OF AMOUNT OF COLLECTOR	
ON FLOTATION	25

	<u>Page</u>
4.5. EFFECT OF FLOTATION TIME ON FLOTATION . . .	27
4.6. EFFECT OF THE TYPE OF FROTHER ON FLOTATION	29
4.7. EFFECT OF NaCN ON FLOTATION	30
4.8. CLEANING OF THE ROUGHER CONCENTRATE	31
4.9. PREDICTION OF LOCKED - CYCLE FLOTATION TEST RESULTS FROM BATCH DATA	36
5. CONCLUSIONS	42
LIST OF REFERENCES	44

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1. Screen Analysis of Crushed Sample	13
2. Chemical Analysis of the Sample	13
3. Screen Analysis of Various Ground Products	14
4. Effect of Particle Size on Flotation	17
5. Effect of Type of Collector on Flotation	20
6. Screen Analysis of 13 Minutes Ground New Sample	21
7. The Results of Flotation Experiments Carried Out to Compare Collectors LSB and F2791	22
8. Effect of pH on Flotation	23
9. Effect of Collector (LSB) Concentration on Flotation	25
10. Effect of Flotation Time	27
11. Effect of the Type of Frother on Flotation	29
12. The Results of Flotation Experiment Made by Using NaCN	30
13. The Results of 2-Stage Cleaning Experiment at Cleaning pH= 11.5	31
14. The Results of 2-Stage Cleaning Experiment at Cleaning pH= 12.0	33
15. The Results of 2-Stage Cleaning Experiment With Addition of 20 g/t NaCN	34

2. LITERATURE SURVEY

2.1 OCCURRENCE OF COPPER ORES

Oxidized and especially sulfide ores are the primary source of copper. There are more than 170 copper-bearing minerals, out of which only 10 to 15 are of industrial importance. The most common copper sulfide minerals are chalcopyrite (CuFeS_2), chalcocite (Cu_2S), bornite (Cu_5FeS_4), covellite (CuS), tetrahedrite ($\text{Cu}_{12}\text{Sb}_4\text{S}_{13}$), tennantite ($\text{Cu}_{12}\text{As}_4\text{S}_{13}$), digenite (Cu_9S_5) and enargite (Cu_3AsS_4) (Glembotskii et. al, 1972). Pyrite and more rarely pyrrhotite are the regular associates of copper sulfides and may be present in very large amounts. Sulfides of other metals are also encountered, but they are of subsidiary importance. (Glembotskii et. al., 1972).

Copper sulfide ores can be divided into 2 broad basic types; solid pyritic and porphyritic ores. A characteristic of solid pyritic ores is the prevalence of sulfide minerals principally pyrite. On the other hand, impregnated or porphyritic ores contain a minor amount of sulfide minerals and prevalence of gangue minerals (Glembotskii et. al., 1972).

The disseminated or "porphyry" copper deposits are considered mesothermal. Characteristic ore minerals are chalcopyrite, enargite, bornite, tetrahedrite, chalcocite, sphalerite and galena. Gangue minerals include quartz, pyrite, and the carbonates (Park and Mac Diarmid, 1975).

1. INTRODUCTION

The Kutlular copper ore deposit is located near Sürmene at the Black Sea coast in the North-East of Turkey. There are two types of mineralization in the deposit namely pyritic and siliceous. The friable pyritic type ore placed at the inner part of the deposit and it represents the majority of the ore body. The outer crust of the deposit consist the hard siliceous ore. The ore deposit has a reserve of 1.220.000 tons with 2.49 % Cu and 32.5 % S. The plant which belongs to The Black Sea Copper works has been operating with a 400 tons daily capacity since 1985. The tailing of plant is considered as pyrite concentrate.

The flowsheet of operating plant has been changed several times due to poor copper recovery and grade of the concentrate. To improve the grade and recovery of the concentrate, studies were carried out and different results were obtained (Uçurum, 1980; Aytakin, 1983; Bayraktar, 1986).

The results of all these studies were different. This was due to different mineralogical characteristics of studied ore samples. The high pyrite to chalcopyrite ratio and close association of these two minerals are the main causes of poor grade and recovery of chalcopyrite concentrate.

The aim of this study is the optimization of flotation parameters of representatively taken Kutlular pyritic copper ore sample.

LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
1. Relationship Between Concentration of Sodium Diethyl Dithiophosphate and Critical pH Value	7
2. Recovery of Pyrite as a Function of Flotation pH With Various Additions of Potassium Ethyl Xanthate	7
3. Experimental Flowsheet	16
4. Effect of Particle Size on Flotation	18
5. Effect of pH on Flotation	24
6. Effect of Collector Concentration on Flotation	26
7. Effect of Flotation Time	28
8. Flotation Flowsheet With 2 Cleaning Stages	32
9. The Split Factors for 3-Stage Flotation Separation	37

Supergene enrichment has been especially important in the history of many disseminated copper deposits. This enrichment increased the grade of ore from one percent or less to as much as five percent in localized zones. (Park and Mac Diarmid, 1975).

Chalcopyrite is the most widely occurring copper mineral and one of the most important sources of that metal. Most sulfide ores contain some chalcopyrite but the most important economically are the hydrothermal vein and replacement deposits. Chalcopyrite is the principal primary copper mineral in the "porphyry-copper" deposits. Also occurs as an original constituent of igneous rocks; in pegmatite dikes; in contact metamorphic deposits; and disseminated in schistose rocks.

Chalcopyrite is the principal source of copper for the the secondary copper minerals malachite, azurite, covellite, chalcocite and cuprite. Concentrations of copper in the zone of supergene enrichment are often the result of such alteration (Cornelius, 1971).

2.2. FLOTATION OF COPPER SULPHIDE ORES

The production of copper concentrates by flotation exceeds that of all other metallic minerals. Copper sulphide minerals are readily floatable and respond readily to sulphydryl collectors, xanthates and dithiophosphates. Alkaline circuits of pH 8 to 10 are generally used, with lime controlling the pH and depressing pyrite.

In copper ores with a high proportion of pyrite, selectivity between pyrite and chalcopyrite can be an important problem; low copper grades and recoveries are usually recorded. In such cases, it is recommended to increase the pH value to 11 to 12. With higher proportions of pyrite in the ore, a general guideline is to extend the conditioning with air and to raise the pH to 11-12 (LeJa, 1982).

Broadly, the treatment techniques for copper ores can be classified as differential and bulk flotation.

1. Differential Flotation: By using lime to depress iron sulphides and gangue, copper minerals are selectively floated with sulphydryl type collectors in an alkaline medium. The copper concentrate is then cleaned in a number of steps. The iron sulphides can then be separated from the gangue at an acidic or neutral pH. Solid pyritic ores, which contain high proportion of pyrite are usually concentrated by this technique.

2. Bulk Flotation: In this method, the sulphide minerals (copper and iron sulphides) are floated from the gangue in a neutral or slightly alkaline medium. The bulk concentrate consisting of copper and iron sulphides are then separated from each other at an highly alkaline medium. This method is usually used for impregnated copper ores in which a high amount of gangue minerals rather than pyrite is present.

2.2.1. Effect of pH

Pulp alkalinity plays an important role in copper flotation and selectivity in complex separation is dependent on a delicate balance between reagent concentration and pH. Xanthates are usually used in alkaline medium, since they are stable under these conditions. Lime is almost universal pH modifier for copper sulphide ores. It is added to slurry before flotation, usually in grinding mills, to precipitate heavy metal ions from solution (Wills, 1985; Glembotskii et. al., 1972). In this sense, the alkali is acting as a "deactivator" as these ions can activate pyrite and prevent their selective separation from chalcopyrite.

Different approaches are proposed for the mechanism of depression of pyrite by lime. OH^- and H^+ ions modify the electrical double layer and zeta potential surrounding the mineral particles and hence the hydration of surfaces. Glembotskii et. al. (1972), Wills (1985), Charles (1953) states that both OH^- and Ca^{++} ions participate in the depression of pyrite. Charles and Plaksin (1953), have shown that pyrite adsorbs calcium. $\text{Fe}(\text{OH})$, $\text{FeO}(\text{OH})$, CaSO_4 and CaCO_3 formed on the surface of pyrite reduces the adsorption of xanthate. Lime has no such effects with copper minerals.

Competition for adsorption sites on the pyrite surface between the collector and hydroxyl ions could be responsible for pyrite depression by lime. Glembotskii et. al (1972)

states that, the amount of xanthate attached to the pyrite surface is greatly reduced in the presence of lime. For the collector dithiophosphate, hydroxyl depression of pyrite may be due to the creation of a negative surface charge preventing the adsorption of dithiophosphate anion (Ball and Rickard, 1976).

According to Pryor (1978), the depressing action of lime on pyrite flotation is due to desorption by hydroxyls of xanthates from its surface.

With xanthates as collectors, sufficient lime will depress almost any sulphide mineral, and for any concentration of xanthate, there is a pH value below which any given mineral will float and above which it will not float. Figure 1 shows how the critical pH value for pyrite and chalcopyrite depends on the concentration of sodium aerofloat collector. The flotation is possible to the left of the curves. (Sutherland and Wark, 1955).

A study by Gaudin and Mellgren (1956) did indicate the presence of adsorbed dixanthogen on pyrite surfaces. This was also confirmed by Mamiya (1960), Fuerstenau et. al. (1968) and Majimo and Takeda (1968). It is established that xanthate is oxidized to dixanthogen in the presence of pyrite and oxygen. Figure 2 is a typical flotation recovery curve for pyrite using ethyl xanthate collector (Fuerstenau et. al., 1968).

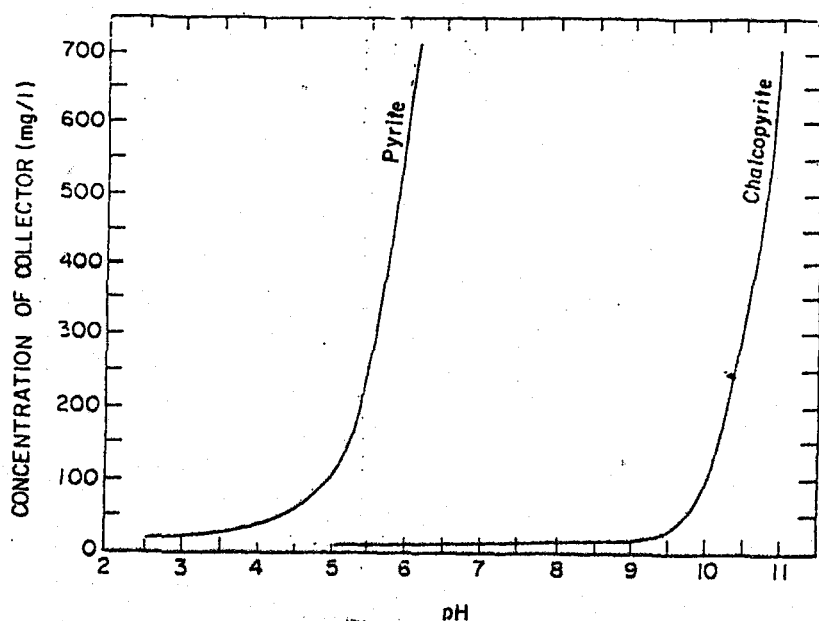


Figure 1. Relationship Between Concentration of Sodium Diethyl Dithiophosphate and Critical pH Value. (Sutherland and Wark, 1955)

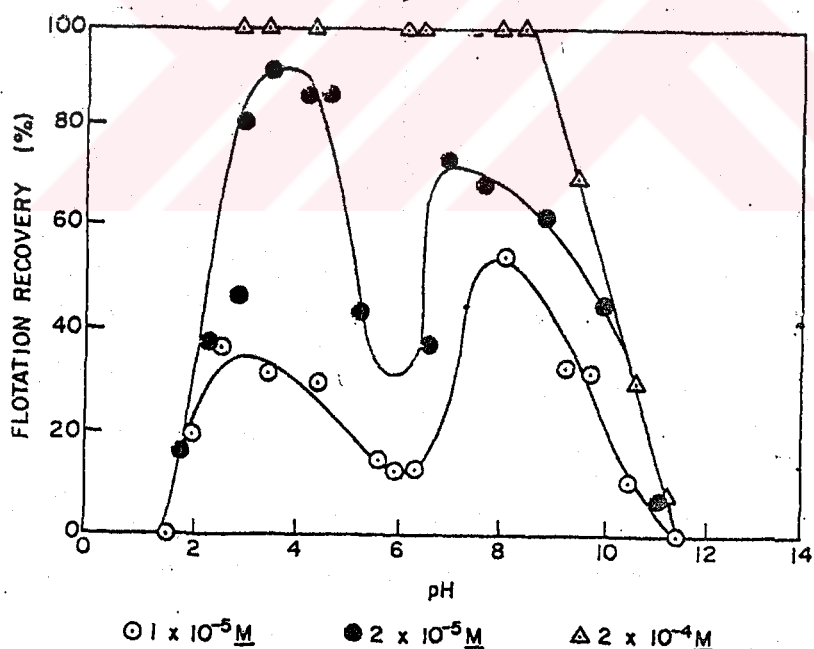


Figure 2. Recovery of Pyrite as a Function of Flotation pH With Various Additions of Potassium Ethyl Xanthate (Fuerstenau, Kuhn and Elgillani, 1968)

Fuerstenau et. al. (1968) suggest that dixanthogen is the specie. primarily responsible for flotation. They found that, almost all xanthate added would exist as dixanthogen below pH 9 and above pH 11, the amount of dixanthogen would be exceedingly small. Thus, the complete depression in flotation above pH 11 was attributed to the lack of dixanthogen.

2.2.2. Effect of Collector

Xanthates and dithiophosphates are almost the universal collectors for the flotation of copper ores. Ethyl xanthate gives completely acceptable results in copper mineral flotation, but increased copper recovery is possible if higher xanthates are used (butyl or amyl xanthate) especially where the copper sulphide surfaces have been subjected to oxidation. Dithiophosphates are more sensitive to depressing action than xanthates, and therefore has special value in differential flotation, particularly when pyrite must be kept down (Glembotskii et. al., 1972; Pryor, 1978).

The amount of collector is important when floating chalcopyrite from pyrite, since an excess of the collector may float pyrite. The amount of xanthate required to float the sulphide minerals is known to be less than a complete monolayer. Plaksin et. al. (1957), Gaudin et. al. (1946) found that, practical collector coatings usually amounted to much less (0.1 to 0.6) than the equivalent of a complete monolayer coverege.

Many studies have been interested in the mechanism of adsorption of xanthates or dithiophosphates on the sulphide mineral surfaces. Xanthates are assumed to adsorb on sulphide mineral surfaces due to chemical forces between the polar group and the surface, resulting in insoluble metal xanthates, which are strongly hydrophobic. The reaction of xanthate with oxidation products of the sulphide surface through an ion-exchange process is considered to be the major adsorption mechanism for the flotation of sulphides (Plaksin, 1959; Finkelstein, 1975). Glembotskii et. al. (1972) states that, xanthate anions render the mineral surfaces water-repellent by their strong chemical attachment to these surfaces.

It appears that the water repulsion imparted to the mineral surface is due to the formation of an oxidation product of the dithiophosphate collector which adsorbs on to the mineral surface.

The presence of oxygen is essential for flotation of sulphide minerals. Poling (1963) showed that in the absence of the oxygen in the system, xanthate ions can not chemisorb on a sulphide surface. Oxidation reactions occurring during the grinding of sulphides have often been found to deplete flotation feed slurries of dissolved oxygen. A study by Plaksin et. al. (1959) gives the following results:

1. In the absence of oxygen, the sulphide minerals are wettable to some extent.

2. Oxygen promotes the dehydration of the mineral surface, thus facilitating the penetration of xanthate molecules.

2.2.3. Effect of Frother

Frother usage in copper flotation has changed significantly in recent years, away from the natural reagents such as pine oil and cresylic acids, to the synthetic frothers such as the higher alcohols and polyglycol esters (Wills, 1985). The important frothers are MIBC, Dowfroth 250, pine oil and cresylic acid. The frother consumption is between 25 and 125 g/t.

Booth and Freyberger (1962) states that, the stability of froths or foams is in large part a function of the rapidity with which the surface tension can vary when a bubble is subjected to mechanical stresses. As a bubble is stretched or compressed, the surface tension must be capable of a corresponding rapid increase or decrease to offset the shock. This quality is referred to as the "Gibbs elasticity". Because of this requirement, it is found that the maximum froth stability arises, not in the region of minimum surface tension, but in a region where the surface tension is capable of rapid change with concentration.

High molecular weight alcohols have the important advantage over industrial products such as pine oil and cresol

in that their composition are much more stable, which makes it easier to control the flotation process and improves performance (Wills, 1985).

Ideally, the frother acts entirely in the liquid phase and doesn't influence the state of the mineral surface. In practise, however, interaction does occur between the frother, mineral and other reagents. Crozier (1980) states that, an optimum reagent system for the flotation of even a simple copper ore will frequently require at least 2 frothers and 2 collectors to obtain an economically attractive combination of maximum recovery at the highest concentrate grade. A typical mill will employ MIBC plus pine oil or a soluble frother such as a polyglycol ether as the frother system. In this mixture, MIBC generates the basic froth characteristics while the pine oil or one of the soluble synthetic frothers affect flotation rate and froth persistence.

3. EXPERIMENTAL STUDIES

3.1. PREPARATION OF TEST SAMPLES

Representative pyritic ore sample taken from Kutlular was crushed to -14 mesh by using a Jaw crusher of 15 cm x 10 cm opening and a 20 cm x 12.5 cm roll crusher successively. The crushed material was riffled in order to obtain representative samples in 500 - gram lots and the samples were packed separately for subsequent operations. The screen analysis of the crushed sample is given in Table 1.

3.2. MINERALOGICAL AND CHEMICAL ANALYSES OF SAMPLE

The major ore minerals of pyritic ore are pyrite and chalcopyrite with minor amounts of sphalerite, galena, quartz, hydrobiotite and tetrahedrite. Secondary copper minerals chalcocite and covellite also exist in minor amounts. The ore does not contain any significant amount of gangue minerals. Grain sizes of minerals range from 5 to 100 microns.

The chemical analysis of pyritic ore is given in Table 2.

Table 1. Screen Analysis of Crushed Sample

Size (micron)	Wt %	Cum wt. % Ret.	Cum. wt. % Pass
+ 833	30.87	30.87	69.13
- 833 + 589	12.32	43.19	56.81
- 589 + 420	12.04	55.23	44.77
- 420 + 300	5.65	60.88	39.12
- 300 + 210	7.29	68.17	31.83
- 210 + 150	4.76	72.93	27.07
- 150 + 105	4.65	77.58	22.42
- 105 + 74	3.24	80.82	19.18
- 74 + 53	3.26	84.08	15.92
- 53 + 44	1.55	85.63	14.37
- 44 + 37	1.14	86.77	13.23
- 37	13.23	-	-

Table 2. Chemical Analysis of the Sample

Element	%
Cu	2.05
Pb	0.40
Zn	0.52
Fe	43.04
S	47.96
SiO ₂	1.01

3.3. GRINDING TESTS

The grinding operations were carried out by using Denver batch laboratory rod mill, having a length of 30 cm and an internal diameter of 18 cm with a rod charge of 14.3 kg. The grinding operations were performed at 56 % solids by weight. (500 gr. solids and 400 cc. water). The desired fineness was obtained by grinding for a predetermined grinding time. Lime was added to the mill to keep the pulp pH high. The screen analyses of the ground products of pyritic ore are given in Table 3.

Table 3. Screen Analyses of various Ground Products

Size (Micron)	GRINDING TIME			
	8 minutes	10 minutes	11 minutes	12 minutes
	cum. %	cum. %	cum. %	cum. %
	Passing	Passing	Passing	Passing
105	87.10	97.80	98.76	99.63
74	62.41	75.40	86.08	89.40
53	47.23	53.12	62.62	61.22

3.4. FLOTATION TESTS

The flotation experiments were carried out to determine the effects of grinding, pH, types and consumption of collector, type of frother flotation time, depressant (NaCN) amount and effect of number of cleaning steps on concentrate grade and copper recovery. These experiments were conducted in

Denver Sub A laboratory type flotation machine with 2- liter glass cell for rougher flotation experiments and 1- liter cell for cleaning purposes. Flotation pulp density was approximately 25 % solids by weight. The impeller speed was adjusted to 1200 rpm. The pH of flotation pulp was measured by a Fischer Accumet, Model 230 pH meter.

Majority of lime was added in the mill to ensure pyrite depression; however small additions were made in the flotation cell for final adjustment. The collectors were diluted with distilled water to obtain a 1 % collector solution for more accurate dosing. The conditioning time for collectors was held constant as 3 minutes.

Figure 3 shows the flowsheet followed during flotation tests.

The collectors tested were Aero 211 (sodium diisopropyl dithiophosphate), Aero 238 (sodium di-secondary butyl dithiophosphate), Aero 249 (sodium diamyl dithiophosphate), KEX (potassium ethyl xanthate), KAX (potassium amyl xanthate), Hostafloat LSB (sodium di-secondary butyl dithiophosphate), Hostafloat F2791 (a combination of di-isobutyl dithiophosphate and mercaptobenzothiazole), Hostafloat LET (sodium di-ethyl dithiophosphate), Hostafloat LIP (sodium di-isobutyl - dithiophosphate).

The frothers used were MIBC (methyl iso butyl carbinol, Dowfroth 250 (polypropylene glycol methyl ether), and Flotanol C-7 (polyalkylene glycol).

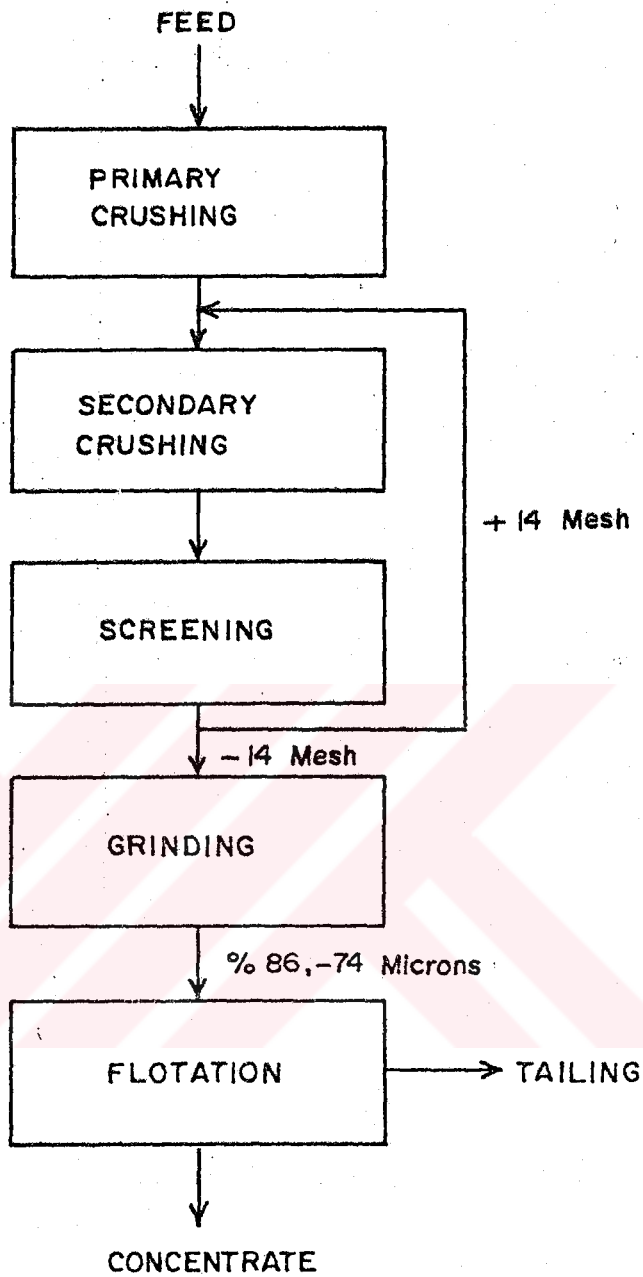


Figure 3. Experimental Flowsheet

4. EXPERIMENTAL RESULTS AND DISCUSSIONS

41. EFFECT OF PARTICLE SIZE ON FLOTATION

4 different size fractions were chosen to investigate the effect of particle size on flotation: 63 % passing 74 microns (8 min. grinding), 75 % passing 74 microns (10 min. grinding), 86 % passing 74 microns (11 min. grinding), 89 % passing 74 microns (12 min. grinding). These experiments indicate that, a grinding of about 86 % -74 microns gives the optimum flotation results; both in grade and recovery. This result was coinciding with the result of microscopical examinations carried for determination of liberation degree of chalcopyrite. Further grinding didn't improve the grade and recovery values. For coarser sizes, the poor metallurgical results may be due to insufficient degree of liberation.

The results of flotation experiments to determine optimum particle size with respect to concentrate grade and recovery are given in Table 4 and plotted in Figure 4.

Table 4. Effect of Particle Size on Flotation.

Exp.Num.	Grind.Time (min.)	% passing 74 microns	Grade (%Cu)	Recovery, %
1	8	62.41	7.61	51.72
2	10	75.40	10.38	67.44
3	11	86.08	12.42	75.20
4	12	89.40	12.06	73.53

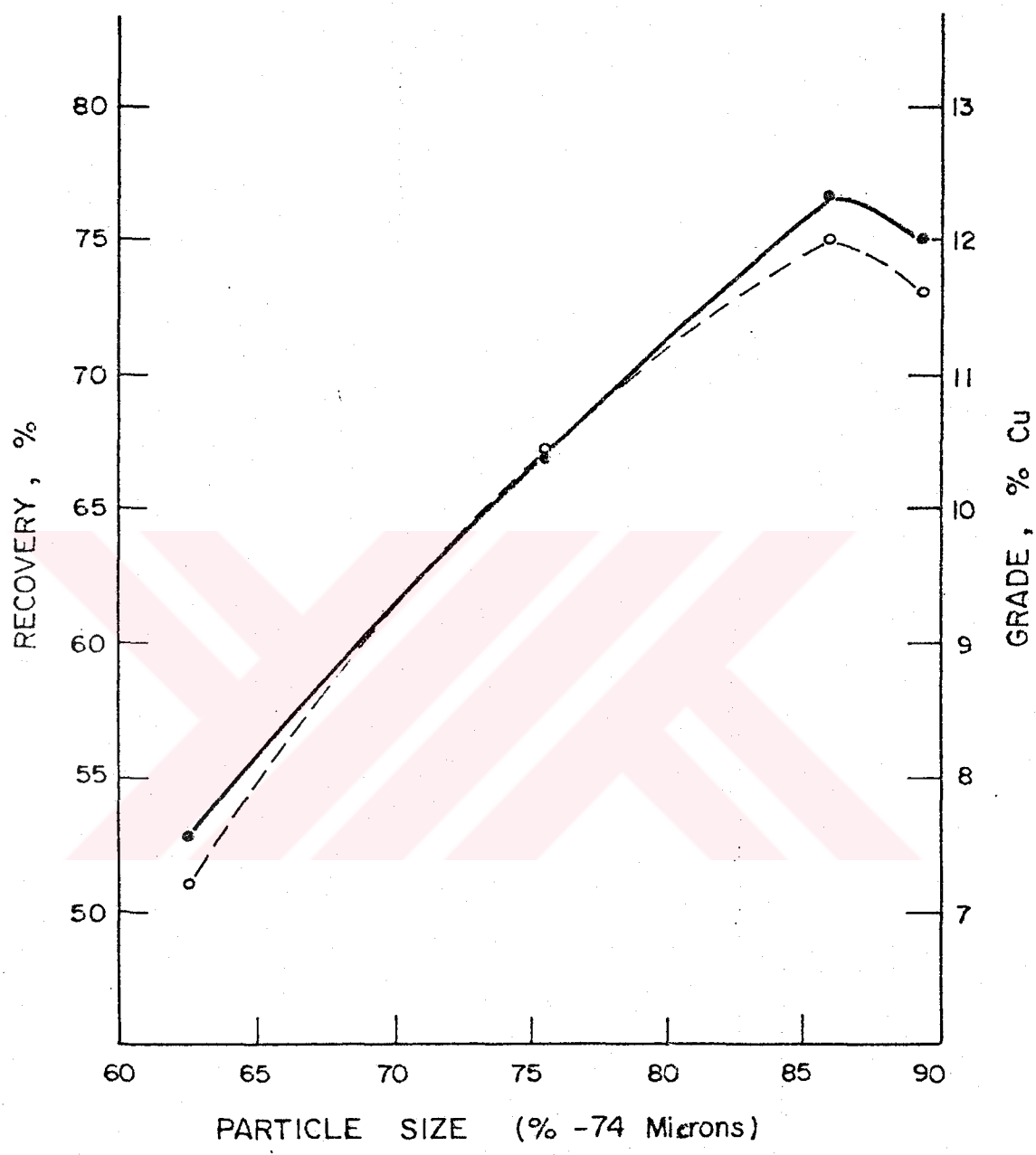


Figure 4. Effect of Particle Size on Flotation

Operational Data:

Collector	: LSB (40 g/t)
Frother	: Dowfroth 250 (20 g/t)
pH	: 11.5
Conditioning Time	: 3 min.
Flotation Time	: 5 min.

4.2. EFFECT OF COLLECTOR TYPE ON FLOTATION

A series of flotation experiments were carried out to determine the effect of 10 different collectors on the flotation recovery and concentrate grade. These collectors were generally chosen such that they were stated as selective collectors in copper-pyrite separations. When conducting these experiments, collector type was changed only; other flotation parameters were kept constant. The results of these experiments are tabulated in Table 5.

Table 5. Effect of Type of Collector on Flotation (Feed = 2.05 % Cu)

Exp. No	Collector Type	Concentrate wt. %	Concentrate Grade, Cu%	Recovery
5	Aero 211	41.32	3.70	80.52
6	Aero 238	30.51	4.80	58.91
7	Aero 249	15.76	8.60	67.35
8	KEX	4.58	3.90	10.13
9	3418 A	7.79	2.00	9.34
10	LET	11.49	11.50	69.90
11	LSB	14.03	11.28	78.64
12	LIP	12.60	11.89	71.89
13	KAX	44.49	3.20	69.19
14	F2791	15.57	10.39	79.30

Operational Data:

Grinding : 86 % -74 microns
Collector Amount : 75 g/ton
pH : 11.5
Frother : Dowfroth 250 (25 g/ton)
Conditioning Time : 3 min.
Flotation Time : 15 min.

The results show that collectors LSB and F2791 give higher recovery with acceptable copper grade than others. To compare LSB and F2791, a number of tests were done. However, it was observed that, there were some deviations between the experiments made at the same conditions. It was thought that, the samples may have been subjected to oxidation. So, new pyritic ore samples were brought and the following experiments were carried out by using this new

sample. The new sample was crushed to -14 mesh by laboratory Jaw and roll crushers sucessively . There were no differences between mineralogical composition of new and old samples. The copper content of new sample was 2.25 % Cu.

The grinding conditions for the new sample were not changed. After some grinding tests, 13 minutes grinding in the rod mill gave a size of 80.86 % passing 71 microns, which was very close to the optimum mesh of grind determined before. The screen analysis for 13 minutes rod mill grinding for the new sample is given in Table 6.

Table 6. Screen Analysis of 13 Minutes Ground New Sample

Size (microns)	Wt %	Cum. wt % retained	Cum. wt. % passing
+ 104	4.20	4.20	95.80
- 104 + 71	14.94	19.14	80.86
- 71 + 53	17.56	36.70	63.30
- 53 + 44	10.26	46.96	53.04
- 44	53.04	-	-

A set of experiments were carried out to compare LSB and F2791 collectors by using new sample. The results of these experiments are given in Table 7.

Table 7. The Results of Flotation Experiments Carried out to Compare Collectors LSB and F2791 by using New Sample (Feed: 2.25 % Cu)

Exp. No	Collector Type and Amount	Flotation Time(min)	Concentrate Grade,Cu%	Recovery %
15	F2791,100 g/t	15	10.07	84.85
16	LSB, 100 g/t	15	7.88	88.73
17	LSB, 75 g/t	10	9.96	86.36

Operational Data:

Grinding : 81 % -71 microns

pH : 11.5

Frother : MIBC (25 g/t)

Conditioning Time : 3 minutes

The results show that, LSB gives higher recovery values than F2791 even at lower dosages. The grade values for experiment number 15 and 17 are almost equal.

As mentioned before, there were two entirely different mineralization at Kutlular copper ore deposit. The results of flotation tests carried on siliceous ore sample with collector F2791 were not promising. Therefore, collector LSB, which also give good results with siliceous copper sample, was chosen as best collector type.

4.3 EFFECT OF pH ON FLOTATION

The effect of pH on the flotation of Kutlular pyritic ore was studied by a series of flotation experiments, carried out at pH values from 10 to 12. The adjustment of pulp pH was done with addition of lime. The results of these experiments are given in Table 8 and represented in Figure 5.

Table 8. Effect of pH on Flotation

Exp. No	pH	Concentrate wt. %	Concentrate grade (% Cu)	Recovery %
18	10.0	31.91	1.42	20.42
19	10.5	55.91	1.69	41.43
20	11.0	60.07	3.02	80.65
21	11.5	15.73	11.67	81.79
22	12.0	12.39	13.60	73.98

Operational Data:

Grinding : 81 % -71 microns
Collector : LSB (75 g/t)
Frother : MIBC (25 g/t)
Conditioning Time : 3 minutes
Flotation Time : 5 minutes

The results show that, the pH is a very important parameter for flotation of Kutlular pyritic ore. The grades and recoveries are all very low at pH's lower than 11.5. It is concluded that, the optimum rougher flotation pH should be 11.5 to obtain a high recovery with a reasonable grade.

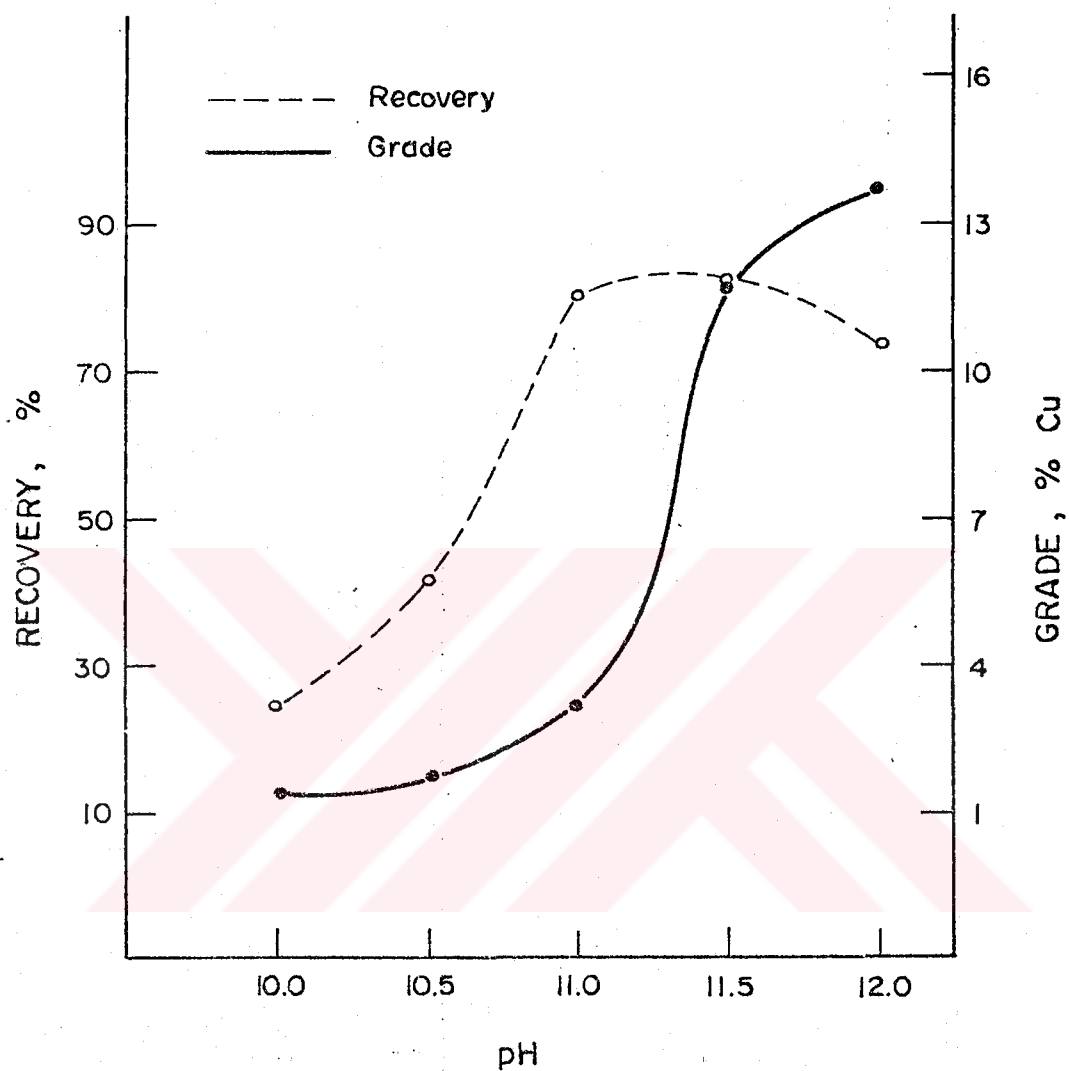


Figure 5. Effect of pH on Flotation

4.4. EFFECT OF AMOUNT OF COLLECTOR ON FLOTATION

A series of flotation experiments were carried out to investigate the effect of concentration of collector LSB on flotation recovery and grade. The LSB consumption was changed from 25 g/ton to 100 g/ton. Table 9 and Figure 6 show the effect of LSB concentration on flotation.

Table 9. Effect of Collector (LSB) Concentration on Flotation

Exp. No	Collector Conc. (g/ton)	Concentrate wt. %	Concentrate Grade(%Cu)	Recovery %
23	25	20.47	8.43	80.97
24	50	20.42	9.02	84.69
25	75	21.51	9.12	87.41
16	100	24.16	7.88	88.73

Operational Data:

Grinding : 81 % -71 microns
pH : 11.5
Frother : MIBC (25 g/t)
Conditioning Time: 3 minutes
Flotation Time : 15 minutes

The results indicate that, 75 g/t LSB consumption gives better grade with a high recovery. The grade decreases considerably at higher concentrations with almost no increase in recovery.

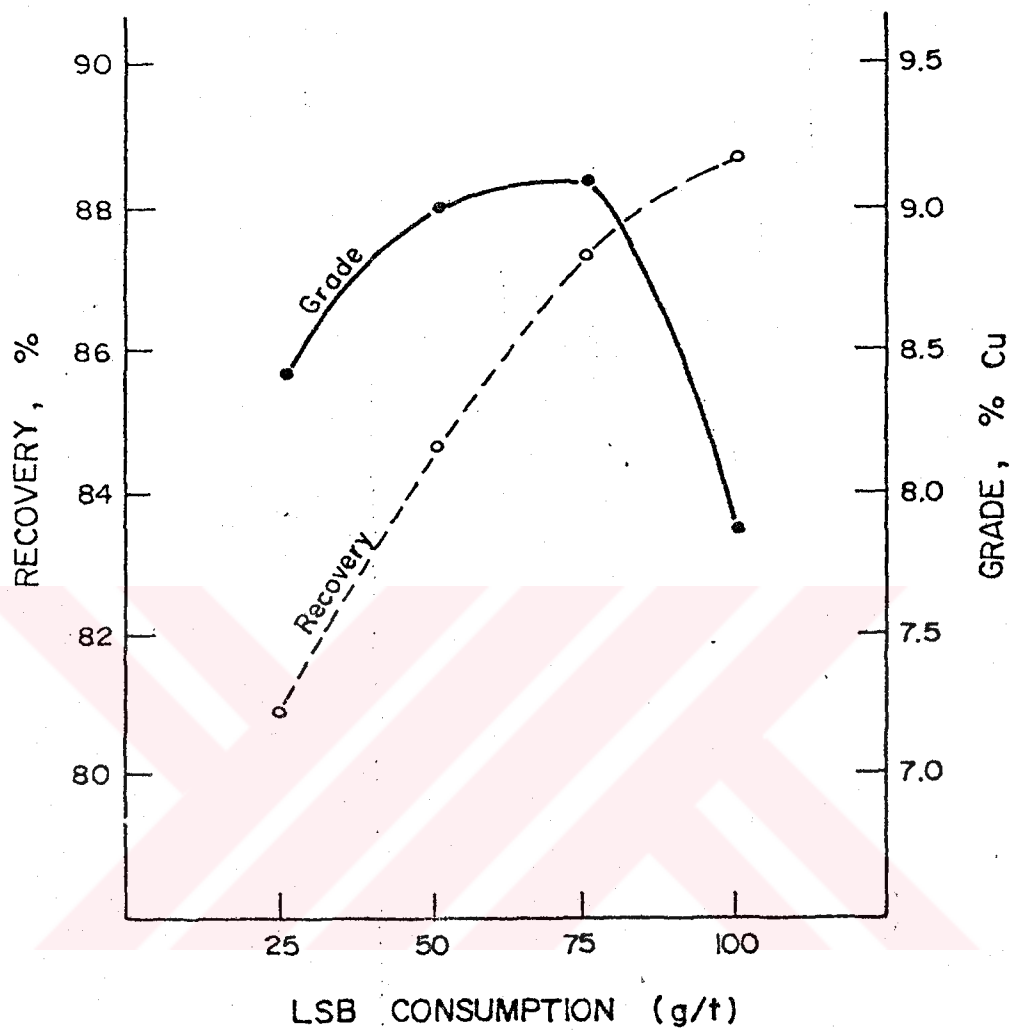


Figure 6. Effect of Collector Concentration on Flotation.

4.5. EFFECT OF FLOTATION TIME ON FLOTATION

In order to determine optimum flotation time, an experiment was carried out. The froth was collected in timed increments. The cumulative results are given in Table 10 and plotted in Figure 7.

Table 10. Effect of Flotation Time for 75 g/t LSB Consumption

Exp. No	Flotation Time (min)	Concentrate wt. %	Concentrate Grade, %Cu	Recovery %
26	1	7.55	14.54	48.90
27	2	11.45	13.62	69.46
28	3	13.67	12.67	76.85
21	5	15.73	11.67	81.79
29	7	17.33	10.93	84.39
17	10	19.46	9.96	86.36
30	12	20.39	9.56	86.87
25	15	21.51	9.12	87.41

Operational Data:

Grinding : 81 % -71 microns
pH : 11.5
Collector : LSB (75 g/t)
Frother : MIBC (25 g/t)
Conditioning Time : 3 min.

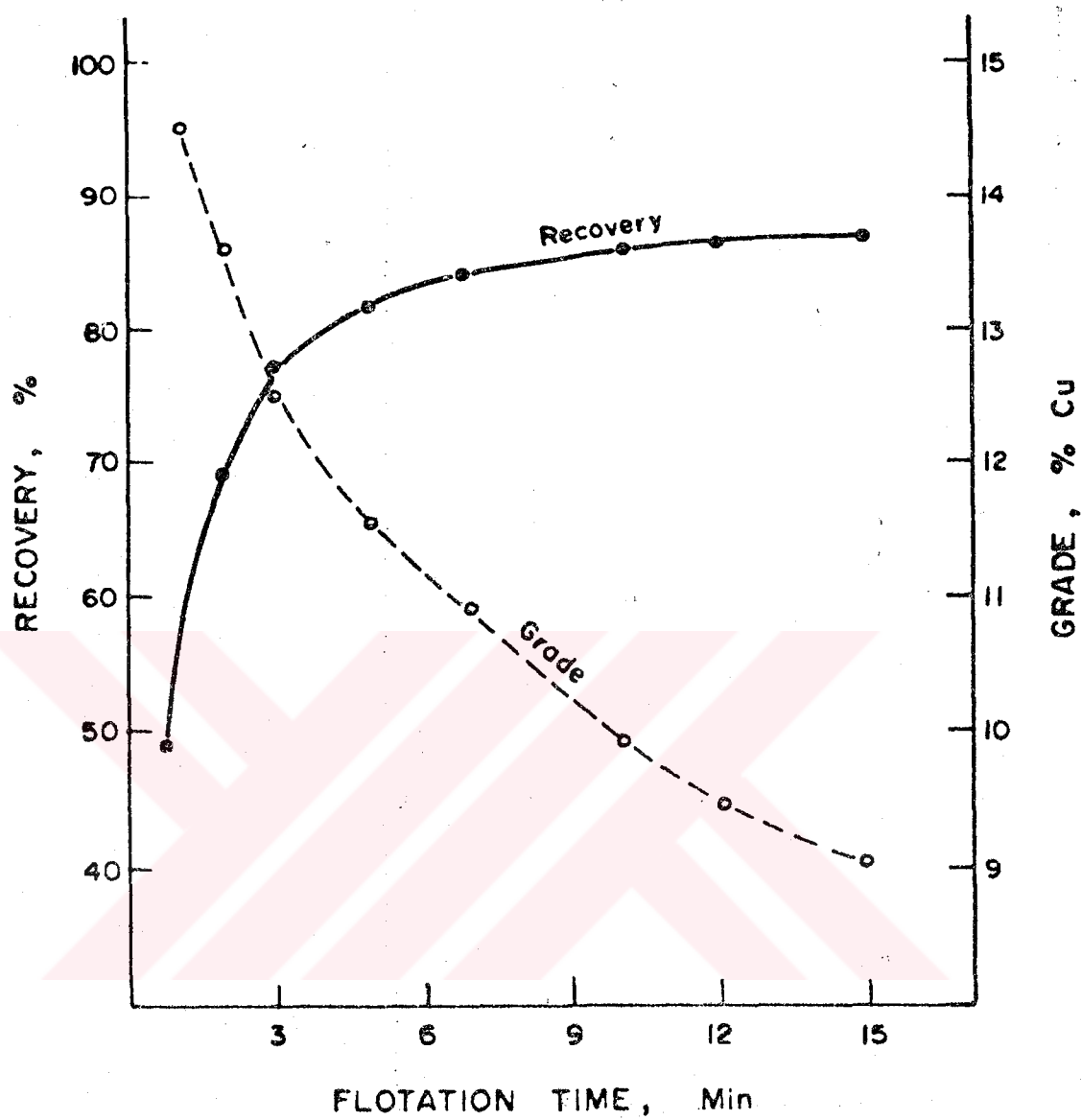


Figure 7. Effect of Flotation Time

If the grades for time intervals are calculated, it is seen that, the grade of material added to the float portion is below feed grade after 10 minutes flotation. The grade decreases although the recovery increases slightly after a flotation of 10 minutes. Consequently, a flotation time of 10 minutes was chosen as optimum rougher flotation time.

4.6. EFFECT OF THE TYPE OF FROTHER ON FLOTATION

A number of flotation experiments were carried out by using MIBC, Dowfroth 250, Flotanol C-7 and a combination of MIBC and Dowfroth 250 to determine the effect of type of frother on the flotation. The frother consumptions were 25 g/t for individual frothers and (12.5 + 12.5) g/t for MIBC - Dowfroth 250 combination. The results of these experiments are given in Table 11.

Table 11. Effect of the Type of Frother on Flotation

Exp.No.	Type of Frother	Concentrate wt. %	Concentrate Grade, %Cu	Recovery %
17	MIBC	19.46	9.96	86.36
31	Dow 250	22.58	8.43	84.24
32	Flotanol C-7	23.00	8.62	84.56
33	MIBC+Dow 250 (1:1)	19.92	9.47	86.11

Operational Data:

Grinding : 81 % -71 microns
 pH : 11.5
 Collector : LSB (75 g/t)
 Conditioning Time : 3 minutes
 Flotation Time : 10 minutes

The results show that MIBC is a suitable frother since it gives higher grade and recovery values than the other frothers. MIBC and Dowfroth 250 combination gives almost equal results.

4.7. EFFECT OF NaCN ON FLOTATION

The depressive effect of sodium cyanide on pyrite was examined by a series of experiments. The NaCN consumptions were 20, 30 and 40 g/tons. Table 12 gives the results of these flotation experiments.

Table 12. The Results of Flotation Experiments Made by Using NaCN

Exp.No.	NaCN Consumption (g/t)	Concentrate wt. %	Concentrate Grade, %Cu	Recovery %
34	20	23.07	7.97	83.59
35	30	21.04	8.66	82.48
36	40	20.98	8.77	82.33

Operational Data:

Grinding : 81 % -71 microns
 pH : 11.5
 Collector : LSB (75 g/t)
 Frother : MIBC (25 g/t)
 Conditioning Time : 3 minutes
 Flotation Time : 10 minutes

The results show that, NaCN has no significant effect on depression of pyrite in rougher flotation stage. It also causes a decrease in recovery values about 3-4 %.

4.8. CLEANING OF THE ROUGHER CONCENTRATE

It was necessary to clean the rougher concentrate to obtain a saleable copper concentrate. The cleaning experiments were carried out at pH 11.5 and 12. An experiment was also made by addition of 20 g/t NaCN as a depressant. No collector and frother were used at cleaning stages. The flowsheet which is followed during the cleaning is shown in Figure 8. Tables 13, 14, 15 give the results of these cleaning experiments.

Table 13. The Results of the 2-Stage Cleaning Experiment at Cleaning pH= 11.5 (Experiment Number= 37)

Product	Wt. %	Cu %	Recovery %
Final Tailing	74.03	0.38	12.98
1 st Cleaner Tailing	11.50	1.47	7.80
2 nd Cleaner Tailing	6.00	4.12	11.41
Final Concentrate	8.47	17.35	67.81
Calculated Feed	100.00	2.17	100.00

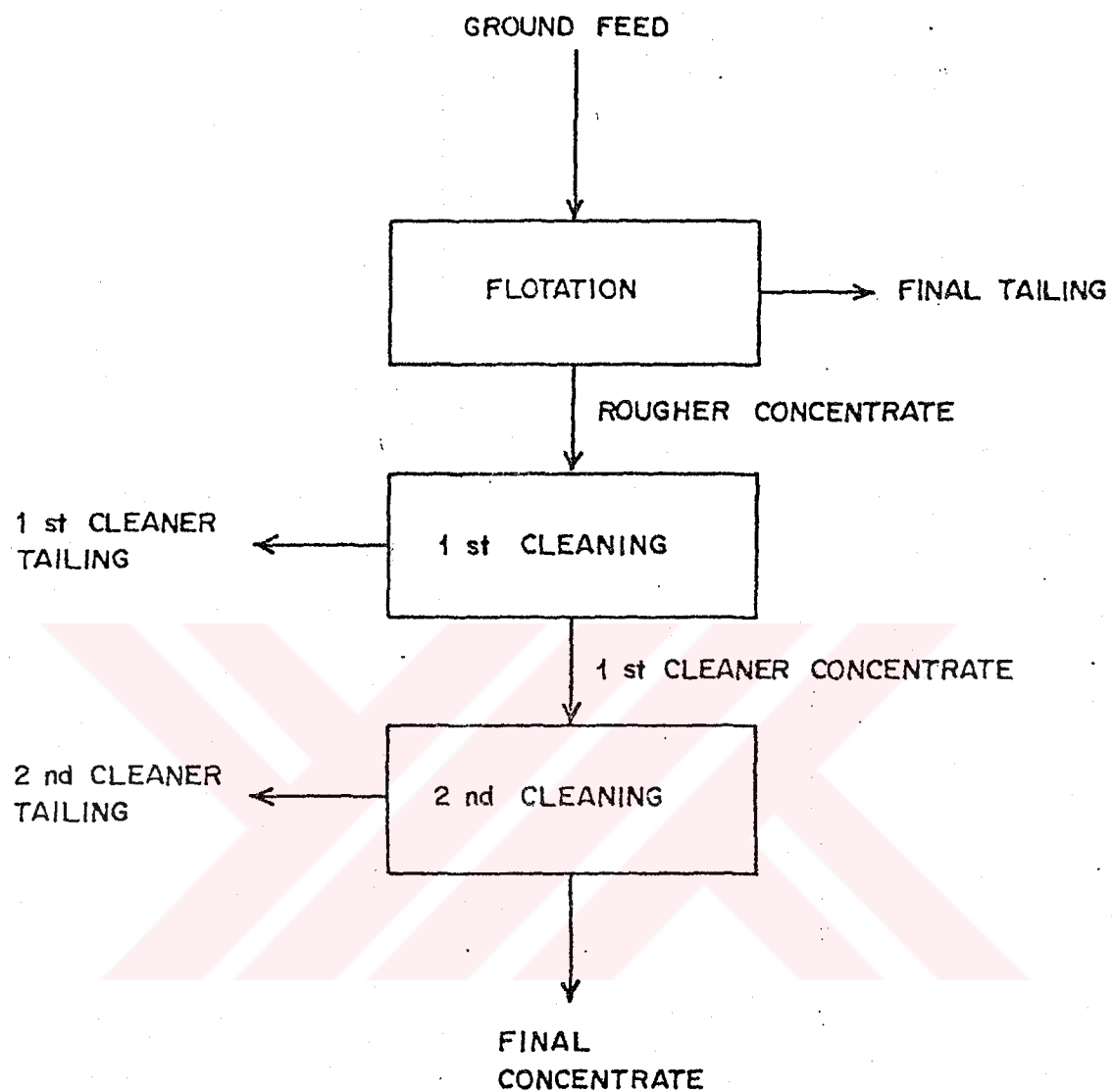


Figure 8. Flotation Flowsheet
with 2 Cleaning Stages

Operational Data:

Grinding : 81 % -71 microns
 pH : 11.5
 Collector : LSB (75 g/t)
 Frother : MIBC (25 g/t)
 Conditioning Time : 3 minutes
 Flotation Time : 10 minutes
 Cleaning pH : 11.5
 Flotation Time : 5 min. (for both cleaning steps).

Table 14 . The Results of 2-Stage Cleaning Experiment at Cleaning pH= 12.0 (Experiment Number 38)

Product	Wt %	Cu %	Recovery %
Final Tailing	75.99	0.39	13.85
1st Cleaner Tailing	10.69	1.32	6.59
2nd Cleaner Tailing	5.52	4.05	10.45
Final Concentrate	7.80	19.14	69.11
Calculated Feed	100.00	2.14	100.00

Operational Data:

Grinding : 81 % -71 microns
 pH(rougher) : 11.5
 Collector : LSB (75 g/t)
 Frother : MIBC (25 g/t)
 Conditioning Time : 3 minutes
 Flotation Time : 10 minutes
 Cleaning pH : 12.0 (for both cleaning steps)
 Flotation Time : 5 minutes (for both cleaning steps).

Table 15. The Results of 2-Stage Cleaning Experiment With Addition of 20 g/t NaCN (Experiment number 39)

Product	Wt. %	Cu %	Recovery %
Final Tailing	76.93	0.47	16.41
1stCleaner Tailing	11.32	1.78	9.15
2ndCleaner Tailing	4.23	4.23	8.13
Final Concentrate	7.52	19.41	66.31
Calculated Feed	100.00	2.20	100.00

Operational Data:

Grinding	: 81 % -71 microns
pH	: 11.5
Collector	: LSB (75 g/t)
Depressant	: NaCN (20 g/t)
Frother	: MIBC (25 g/t)
Flotation Time	: 10 minutes
Conditioning Time	: 3 minutes
Cleaning pH	: 11.5
Flotation time	: 5 minutes (for both cleaning steps)

If we compare the results of experiment 37 and 38, cleaning at pH 12 gives better grade and recovery values than cleaning at pH 11.5.

Comparison of the results of experiment 37 and experiment 39 showed that addition of NaCN have a positive effect on the grade of final concentrate.

4.9. PREDICTION OF LOCKED CYCLE FLOTATION TEST RESULTS FROM BATCH DATA

A method has been developed for the prediction of locked cycle flotation results from data obtained from individual batch tests (Agar and Kipkie, 1978). A postulate is made that the distribution of material in a separation stage is determined by the chemical conditions and the flotation time. This distribution can be characterized by "split factor" which are nothing more than the fraction of the feed that reports to the non-float or tailing stream from each separator. The split factors for three-stage flotation separation are shown in Figure 9. Each stream is numbered so it can be readily identified; the diamond-shaped symbols represent junctions where two streams join to form a third stream. The separation stages, identified as rectangles, are numbered in sequence. The split factor for the first separator is referred as SF1, similarly, the second separator split factor is identified as SF2 and the third separator split factor as SF3. The system which is shown in Figure 9 can be solved algebraically, the equations are as follows.

$$[1] = 100$$

$$[3] = [1] / \frac{1 - (1 - SF1)(SF2)}{(1 - (1 - SF2)(SF3))}$$

$$[4] = [3] (SF1)$$

$$[5] = [3] (1 - SF1)$$

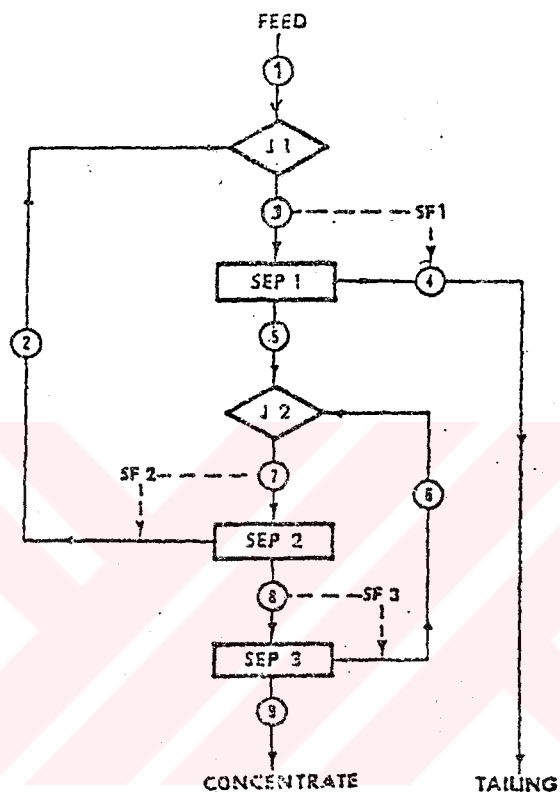


Figure 9 . The Split Factors for 3-Stage Flotation Separation

$$\begin{aligned}
[7] &= [3] (1-SF1) / (1-(1-SF2) (SF3)) \\
[2] &= [3] (1-SF1) (SF2) / (1-(1-SF2) (SF3)) \\
[8] &= [3] (1-SF1) (1-SF2) / (1-(1-SF2) (SF3)) \\
[6] &= [3] (1-SF1) (1-SF2) (SF3) / (1-(1-SF2) (SF3)) \\
[9] &= [3] (1-SF1) (1-SF2) (1-SF3) / (1-(1-SF2) (SF3))
\end{aligned}$$

SF1: Split factor in first separation (rougher).

SF2: Split factor in second separation (1st cleaner).

SF3: Split factor in third separation (2nd cleaner).

The numbers in square brackets represent the mass of a given component in that stream. This set of equations is used to calculate the weight distribution, then the distribution of copper. Finally, the assays of streams are calculated from these distributions and known feed assays.

Application of Method for the Data of Experiment 38.

Data of Experiment 38.

	<u>Wt %</u>	<u>Cu %</u>
Feed	100	2.14 (Calculated)
Final Tailing	75.99	0.39
1st Cleaner Tailing	10.69	1.32
2nd Cleaner Tailing	5.52	4.05
Final Concentrate	7.80	19.14

Calculations for weight distributions:

$$\text{Split Factor} = \frac{\text{weight of Tailing taken from the separator}}{\text{weight of feed in the same separator}}$$

$$\text{SF1} = \frac{75.99}{100} = 0.76$$

$$\text{SF2} = \frac{10.69}{100 - 75.99} = 0.445$$

$$\text{SF3} = \frac{5.52}{100 - (75.99 + 10.69)} = 0.414$$

The mass of a given component in the stream were found as follows:

$$[3] = 116.10$$

$$[5] = 27.86$$

$$[2] = 16.10$$

$$[6] = 8.32$$

$$[4] = 88.24$$

$$[7] = 36.18$$

$$[8] = 20.08$$

$$[9] = 11.76$$

Calculations for copper distributions:

$$\text{Split Factor} = \frac{\text{Copper content in tailing taken from separator}}{\text{Copper content in feed in the same separator}}$$

$$\text{SF1} = \frac{13.85}{100} = 0.139$$

$$\text{SF2} = \frac{6.59}{100 - 13.85} = 0.076$$

$$\text{SF3} = \frac{10.45}{100 - (13.85 + 6.59)} = 0.131$$

The copper content of a given component in the stream were found as follows:

$$[3] = 108.04$$

$$[4] = 15.02$$

$$[5] = 93.02$$

$$[7] = 105.83$$

$$[2] = 8.04$$

$$[8] = 97.79$$

$$[6] = 12.81$$

$$[9] = 84.98$$

From weight and copper distributions, the grades for each stream are calculated as follows:

$$[1] = 2.14 \% \text{ Cu}$$

$$[2] = 1.07 \% \text{ Cu}$$

$$[3] = 1.99 \% \text{ Cu}$$

$$[4] = 0.36 \% \text{ Cu}$$

[5] = 7.15 % Cu

[6] = 3.29 % Cu

[7] = 6.26 % Cu

[8] = 10.42 % Cu

[9] = 15.46 % Cu

From the calculated results, we can predict that in a closed circuit flotation process a production of copper concentrate with a 15.46 % Cu grade and 84.98 % recovery is possible.

5. CONCLUSIONS

In the light of the tests, performed on a pyritic copper ore sample from the Kutlular deposit, the following conclusions can be drawn:

1. The optimum liberation size can be achieved by grinding the ore to 81 % to -71 microns.
2. The highest copper grade and recovery is obtained with the use of the collector Hostafloat LSB (Sodium di-secondary butyl dithiophosphate).
3. The pH of the pulp has a very significant effect on both grade and recovery of concentrate. It must be kept above 11.5.
4. The frother MIBC and the combination of it with Dowfroth 250 in a ratio of 1:1 gave the best flotation results.
5. The optimum dosage of collector LSB is 75 g/ton.
6. The optimum rougher flotation time was found as 10 minutes. After 10 minutes, the grade of material added to the concentrate was lower than the grade of the feed.
7. Two stage cleaning was necessary to obtain saleable copper concentrate. During the cleaning steps the pH of the pulp must be kept above 11.5.
3. Production of final copper concentrate of 19.14 % Cu with a recovery 69.11 % is possible.

9. The higher amount of copper lost in the tailing was due to very fine chalcopyrite grain size down to 5 micron.
10. From the batch test results, it can be predicted that a concentrate assaying 15.46 % Cu with a recovery of 84.98 % can be obtained.
11. Comparison between predicted and observed locked cycle test must be done to conclude the accuracy of predicted locked cycle test method.

LIST OF REFERENCES

- Agar, G.E., Kipkie, W.B., 1978. "Predicting Locked Cycle Flotation Test Results From Batch Data", CIM Bulletin, Vol. 71, pp.119.
- Aytekin, Yavuz, 1983. "Kutlular Piritik Bakır Cevherinin Optimal Flotasyon Şartlarının Tesbiti", 9 Eylül Üniversitesi, Maden Mühendisliği Bölümü, 326 pages.
- Ball, B., Rickard, R.S., 1976. "The Chemistry of Pyrite Flotation and Depression", in Flotation, A.M. Gaudin Memorial Volume, ed. Fuerstenau, M.C., AIME, New York, pp.458-484.
- Bayraktar, I., 1986, "Karadeniz Bakır İşletmeleri Kutlular Flotasyon Tesisi Optimizasyon Çalışmaları Raporu", Demir Export A.Ş., Ankara.
- Booth, R.B., Freyberger, W.L., 1962, "Froths and Frothing Agents", "Froth Flotation-50th Anniversary Volume", ed. Fuerstenau, D.W., AIME, New York, pp.261-262.
- Charles, W.D., 1953, Trans. AIME, Vol.196, pp.195.
- Crozier, R.D., 1980, "Frother Function In Sulphide Flotation", Mining Magazine, Vol.142, pp.26-35.
- Finkelstein, N.P., 1975, "Natural and Induced Hydrophobicity in Sulphide Mineral Systems", A.I.Ch.E. Symposium Series, Vol.71, pp.185.
- Fuerstenau, M.C., Kuhn, M.C., Elgillani, D.A., 1968, Trans. AIME, Vol.241, pp.148.
- Gaudin, A.M., Mellgren, Olav, 1956, Trans. AIME, Vol.207, pp.65.
- Gaudin, A.M., Preller, G.S., 1946, AIME Tech. Pub. 2002.
- Glembotskii, V.A., Klassen, V.I., Plaksin, I.N. 1972, "Flotation", Primary Sources, New York, pp.126-127 and 445-478.

- Hurlbut, C.s., 1977, Manual of Mineralogy, John Wiley and Sons, New York, pp.258-263.
- LeJa, J., 1982, Surface Chemistry of Froth Flotation, Plenum Press, New York, pp.33-35 and 498-514.
- Majima, H., Takeda, M., 1968, Trans AIME, Vol.241, pp.431.
- Mamiya, M, 1960, Nippan Kogyo Kaishi, Vol.76, pp.555.
- ODTÜ, Maden Mühendisliği Bölümü, 1988, "Kutlular Flotasyon Tesisi İyileştirme Projesi Gelişme Raporu".
- Park. Q., MacDiarmid, R., 1975, Ore Deposits, Freeman and Company, San Francisco, pp.311-312 and 419-492.
- Plaskin, I.N., Zaitseva, S.P., Shaefeyev, R.S. 1957, Institution of Mining and Metallurgy Trans., Vol.67, pp.1.
- Plaksin, I.N., 1959, Trans. AIME, Vol.214, pp.319.
- Plaksin, I.N., 1960, International Mineral Processing Congress, London, pp.253.
- Poling, G.W., 1976, "Reactions Between Thiol Reagents and Sulphide Minerals", Flotation A.M. Gaudin Memorial Volume, AIME, New York, pp.334-363.
- Pryor, E.J., 1978, Mineral Processing, Applied Science Publishers Limited, London, pp.472-477.
- Rogers, J., 1962, "Principles of Sulphide Mineral Flotation", Froth Flotation 50th Anniversary Volume, ed. Fuerstenau, D.W., AIME, New York, pp.139-167.
- Sutherland, K.L., Wark, I.W., 1955, Principles of Flotation, Australian Institute of Mining and Metallurgy, Melbourne.
- Uğurum, M., 1980, "Sürmene Kutlular Piritli Bakır Cevherinin Laboratuvar Ölçeğinde Zenginleştirme Çalışmaları", MTA Raporu, pp.14.
- Wills, B.A., 1985, Mineral Processing Technology, Pergamon Press, Oxford, pp.389-398 and 462-463.