

DETERMINATION OF FLOTATION CONDITIONS OF
CELESTITE-CALCITE ORE FROM SIVAS-NASIR

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TAKI GÜLER

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Approval of the Graduate School of Natural and Applied Sciences



Prof. Dr. Tayfur Öztürk
Director

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

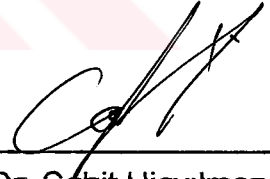


Prof. Dr. Günhan Paşamehmetoğlu
Head of Department

This is to 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.



Assist. Prof. Dr. Ünal Akdemir
Co-Supervisor



Prof. Dr. Cahit Hiçyılmaz
Supervisor

Examining Committee Members

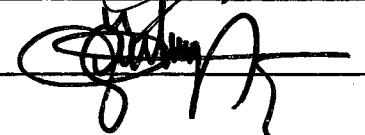
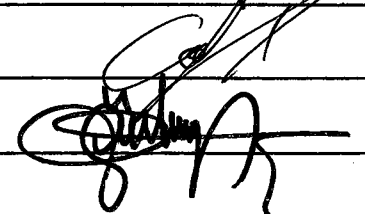
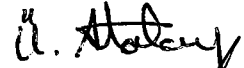
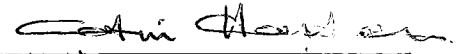
Prof. Dr. Gülhan Özbayoğlu

Prof. Dr. Çetin Hoşten

Prof. Dr. Ümit Atalay

Prof. Dr. Cahit Hiçyılmaz

Assist. Prof. Dr. Özcan Yıldırım Gülsoy



ABSTRACT

DETERMINATION OF FLOTATION CONDITIONS OF CELESTITE-CALCITE ORE FROM SIVAS-NASIR

Güler, Taki

M.Sc., Department of Mining Engineering

Supervisor: Prof. Dr. Cahit Hiçyılmaz

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In this study, optimum flotation parameters of celestite-calcite ore were investigated to obtain a concentrate assaying above 92 % SrSO_4 grade which is saleable in market. Mineralogical analysis of the representative ore sample showed that the major constituents were celestite, calcite and quartz.

Effects of pH, grinding time, type and amount of collectors, depressants and frothers, and flotation time on flotation were examined. Aerofloat-845 (187.5 g/t) and Na-oleate (187.5 g/t) combination as collectors, Na_2SiO_3 as depressant and Aerofroth-65 (60 g/t) as frother were found as the optimum flotation reagents at pH 10 by NaOH. By applying 3 minutes flotation time, a concentrate assaying 93.14 % SrSO_4 was

obtained with 91.32 % recovery from 79.01 % minus 125 μm size ore sample assaying 85.51 % SrSO_4 . At the same conditions, by applying 5 minutes flotation time and 3-stage cleaning, a concentrate assaying 95.34 % SrSO_4 was obtained with 84.93 % recovery.

Keywords: Celestite, Calcite, Flotation, Collector, Depressant, Frother



ÖZ

SİVAS-NASIR SELESTİT-KALSİT CEVHERİNİN
FLOTASYON KOŞULLARININ BELİRLENMESİ

Güler, Taki

Yüksek Lisans, Maden Mühendisliği Bölümü

Tez Yöneticisi: Prof. Dr. Cahit Hiçyılmaz

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Bu çalışmada, satılabilir konsantre (en az % 92 SrSO₄ tenörlü) elde etmek için, selestit-kalsit cevheri için optimum flotasyon parametreleri araştırılmıştır. Temsili cevher örneğinin mineralojik analizi, cevherin başlıca selestit, kalsit ve kuvars içerdiğini göstermiştir.

Toplayıcı, bastırıcı ve köpürtücü türü ve miktarı, pH, öğütme süresi ve flotasyon süresinin flotasyon üzerindeki etkileri incelenmiştir. Toplayıcı olarak Aerofloat-845 (187.5 g/t) ve Na-oleat (187.5 g/t) bileşimi, bastırıcı olarak Na₂SiO₃ (1500 g/t) ve köpürtücü olarak Aerofroth-65 (60 g/t) NaOH ile pH 10'da optimum flotasyon reaktifleri olduğu belirlenmiştir. 3 dakika flotasyon süresi uygulanarak % 85.51 SrSO₄ tenörlü, % 79.01'i 125 µm'nin altında olan cevher örneğinden % 93.14 SrSO₄ tenörlü konsantre % 91.32

verim ile elde edilmiştir. Aynı kořullarda 5 dakika flotasyon süresi ve 3 aşamalı temizleme uygulayarak % 95.34 SrSO₄ tenörlü konsantre % 84.93 verim ile elde edilmiştir.

Anahtar Kelimeler: Selestit, Kalsit, Flotasyon, Toplayıcı, Bastırıcı,
Köpürtücü



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CHAPTER 1

INTRODUCTION

Strontium, found by Aldair Crawford in 1790, is an alkaline earth metal. It is a much less common element in comparison with other alkaline earth metals, such as calcium and barium. It occurs in nature averaging 0.034 % of all igneous rocks (Ober, 1995).

Strontium does not occur in nature in the free state. There are 62 strontium minerals in nature. However, only two of them occurs commonly: Celestite (SrSO_4) and Strontianite (SrCO_3). Although strontianite occurs commonly, it is not an item of commerce due to higher contents of calcium minerals as gangue. Therefore, commercially celestite is the only significant Sr-mineral to produce Sr-compounds, especially SrCO_3 .

Turkey has important celestite reserves, mainly in Sivas province, and since 1972 celestite has been produced. These reserves have gypsum and/or calcite as gangue minerals. Barit Maden Türk A.Ş., which is celestite concentrate producer, processes celestite ore with gypsum as gangue. This deposit is about 1,500,000 tons and liberates in coarser sizes. The company has also 1,000,000 tons celestite reserves with calcite as gangue liberating in finer sizes. This reserve can be concentrated by

using gravity concentration methods. However in finer sizes gravity methods are not recommended due to decreasing efficiency. Therefore in this research it was tried to float celestite selectively from celestite-calcite ore and to increase concentrate grade to 92 % SrSO₄ which is saleable product in market. For this purpose the parameters, such as collector type, frother type, pH, depressant type, grinding time and flotation time were examined.

1.1 PHYSICAL, CHEMICAL AND MINERALOGICAL PROPERTIES

Strontium is a soft, silvery white, alkaline-earth metal which readily tarnishes on exposure to air and ignites spontaneously when powdered. It crystallizes in the cubic system, melts at about 757 °C., has a density of 2.63 g/cm³ and a hardness roughly equal to that of lead (Beard, 1937).

One of the most common Strontium minerals is celestite (SrSO₄). Theoretically it is composed of 56.4 % SrO and 43.6 % SO₃. Celestite (SrSO₄) owes its name to the faint shades of blue which sometimes displays (Beard, 1937). Celestite is a salt type mineral in regard to its physico-chemical properties (Hanna and Somasundaran, 1976). It may be colorless, bluish, gray, white or rarely reddish or greenish. It has orthorhombic crystal structure and is isostructural with barite. The hardness is 3-3.5 on mohs' scale and the specific gravity of the purest forms is 3.95 to 3.97 g/cm³ (Brobst, 1990). But Bery and Mason gave

specific gravity of celestite as changing between 3.96 and 3.98 g/cm³. It melts at 1600 °C. It occurs chiefly in sedimentary rocks, in stratiform deposits of gypsum, anhydrite, or halite, often associated with sulfur; and in cavities, veins, and disseminated limestone or dolomite with fluorite or gypsum (Berry and Mason, 1959).

The other is strontianite (SrCO₃). Theoretically strontianite is composed of 70.19 % SrO and 29.81 % CO₂. It is orthorhombic mineral named after Strantion, in Argyllshire, Scotland, where it was first discovered (Beard, 1937). It occurs as white, pale green or pale yellow. It forms columnar, fibrous and granular masses, spear-shaped crystals and pseudo-hexagonal twin crystals like aragonite. The hardness is 3.5 on mohs' scale and the specific gravity of the purest forms is 3.75 to 3.77 g/cm³ (Karayazıcı and Oral, 1980). Strontianite occurs usually as veins in marls and limestones. Less frequently it forms small crystals in metaliferous veins (Beard, 1937).

1.2.RESERVES

A definite information is not found about world strontium reserves. The USA potential celestite reserve is 135,000 tons and Mexican proved and possible reserves are 450,000 tons and 2,800,000 tons, respectively (Can, 1993)

According to Karayazıcı and Oral, in the USA total estimated celestite and strontianite reserve is 3,200,000 tons with 85 % SrCO_3 and SrSO_4 grade. Spain has 2,000,000 tons proved and 6,000,000 tons possible celestite reserves. Canada has 1,000,000 to 3,000,000 tons possible celestite reserve with 55 % SrSO_4 grade. Iran has 1,000,000 tons proved and 3,000,000 tons possible reserves with approximately 90 % SrSO_4 grade (Karayazıcı and Oral, 1980). Cyprus has 200,000 tons celestite-gypsum reserve with 56 % SrSO_4 grade (Griffiths, 1984).

In 1970s, Lishui deposit in Jiangshu province and Hechuan deposit in Sichuan province with a total reserve of over 1,000,000 tons were found. China's celestite resources are characterized by low grade and concentrated geographical distribution. Over half of the reserves occur in Sichuan province, and the grade of crude ores in most of the mines is between 65-90 % SrSO_4 , typically 80 % SrSO_4 (Hong, 1993)

In Turkey, total amount of Sr-reserve is not definitely known. In Burç district of Gaziantep strontianite was found but this reserve has not been mined because of low content of SrCO_3 (1 % SrCO_3). Another reserve with 46 % SrCO_3 grade was found in Yenipazar district of Bozdoğan, Aydın. This reserve is also not an item of commerce due to low SrCO_3 content and high CaCO_3 content (24 % CaCO_3) (Karayazıcı and Oral, 1980). Commercially important celestite reserves was found in Hafik and Zara district of Sivas. In 1972 Barit Maden Türk A.Ş. found 2,000,000 tons celestite-gypsum reserve in Akkaya village, and since then celestite has

been mined from there. Barit Maden Türk A.Ş. estimates that in Sivas province this company has approximately 1,500,000 tons celestite-gypsum reserves and 1,000,000 tons celestite-calcite reserves.

MTA (Mineral Research and Exploration) has studied four different areas in Sivas. First one of them is in Kabalı village in Hafik. 12,551 tons proved and 26,660 tons possible celestite-calcite reserves were found there (Kayan, Aktürk and Aslan, 1988). The second one is in Battalhöyüğü hill in Ulaş. 91,113 tons proved reserve with 72.38 % SrSO_4 and 11.71 % CaCO_3 grades, and 74,758 tons reserve with 78.65 % SrSO_4 grade was found. SrSO_4 grade still changes: In upper parts of reserve grade is over 90 % SrSO_4 and in lower parts it decreases to 22.54 % SrSO_4 (Çubuk, et.al., 1993). The third one is in Alişanağılı in Zara. Totally 42,515 tons reserve was found. SrSO_4 grade of reserve is not constant: In upper parts, grade is 79.43 % SrSO_4 , and average grade of reserve is 30.14 % SrSO_4 (Kayan and Çubuk, 1995a). The last one is in Atkıran in Zara. 1,449 tons proved and 3,325 tons possible reserve was found. SrSO_4 grade of reserve is 41 % SrSO_4 (Kayan and Çubuk, 1995b).

1.3 PRODUCTION

First celestite production was started in England in 1875. Before World-War-II, world celestite production was 5,000 tpa and as pointed above, over 90 % of this production was from England. World production

increased to 7,375 tons in 1955, to 14,011 tons in 1965, to 16,839 tons in 1968. It sharply increased to 41,108 tons in 1969 and to 111,426 tons in 1971, because it was started to use in TV glass faceplate tube in 1969. World production between 1960-1991 is given in Table-1.1 (Can, 1993; Cebeci, et.al., 1993; Griffiths, 1985; Karayazıcı and Oral, 1980; Kendall, 1995).

Table-1.1: World celestite production

	England	Italy	Mexico	Pakistan	Iran
1960	6,708	830	2,612	1,353	-
1970	9,501	844	26,500	299	300
1980	6,700	1,053	40,761	270	18,000
1985	23,039	4,611	32,320	688	20,000
1986	14,739	4,667	24,289	997	22,000
1987	22,655	177	62,910	1,194	23,000
1988	25,553	200	38,794	941	13,000
1989	20,885	-	37,841	956	25,000
1990	24,734	-	40,130	1,799	30,000
1991	2,000	-	50,000	1,000	25,000

Table-1.1 (continued): World celestite production

	Turkey	Algeria	Argentina	Spain	Cyprus
1960	-	-	-	-	-
1970	-	-	426	7,000	-
1980	20,000	3,000	268	36,000	-
1985	35,000	5,400	1,248	39,100	1,400
1986	35,000	5,400	983	31,740	7,365
1987	35,000	5,400	1,133	26,558	6,300
1988	55,800	5,400	2,241	45,631	7,250
1989	86,375	5,400	1,193	35,134	-
1990	73,790	5,400	1,200	80,352	-
1991	70,000	5,400	1,200	80,000	-

Recently, celestite has been produced at Ksar El Kebir in Morocco. But enough information did not found. In England reserves became exhausted in Bristol area around Yate, tailing has been reprocessed (Griffiths, 1992). In Italy, production was stopped in 1986 (Can, 1993). In Canada, celestite production was started in 1969 and stopped in 1976 (Fulton, 1983). In Turkmenia, in the former USSR, work has apparently been completed on assessing the underground reserves of the Arik celestite deposit. The USA has celestite deposits in Arizona, California, Texas and Washington. However due to low grade, processing of these deposits is not economically feasible at present (Griffiths, 1985). Therefore, the USA, which is the most important celestite consumer, import celestite ore mainly (95 % of total celestite import) from Mexico (Ober, 1991). Spain has mined celestite ore since 1968 and is one of the leading celestite producers. Iran started celestite production in 1970 (Karayazıcı and Oral, 1980). About China's celestite production there is not enough information and Hong gives China's production as 30,000-40,000 tpa (Hong, 1993).

1.4 CONSUMPTION

Little celestite is consumed directly. It is only used in paint industry as a filling material. The majority is converted to SrCO_3 and some part of it is converted into $\text{Sr}(\text{NO}_3)_2$. Other strontium compounds are derived from SrCO_3 . Typical celestite specifications for the carbonate and nitrate markets are shown on Table-1.2 (Fulton, 1983).

SrCO₃ is produced by two different methods: Black ash method to produce chemical grade SrCO₃ (> 98 % SrCO₃) and direct conversion method to produce technical grade SrCO₃ (> 95 % SrCO₃).

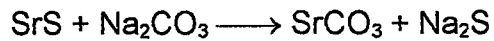
Table-1.2: Celestite specifications for SrCO₃ and Sr(NO₃)₂ production

	For SrCO ₃	For Sr(NO ₃) ₂
SrSO ₄	90 % Min.	95 % Min.
CaSO ₄	-	1.5 % Max
BaSO ₄	2 % Max.	2 % Max.
F	0.1 % Max.	-
Size	6 in. Max.	-6 in.+0.25 in.

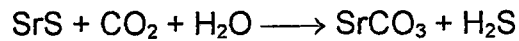
In black ash method, SrCO₃ is produced by using Na₂CO₃ (soda ash method) or CO₂ (carbon dioxide method). Celestite concentrate is mixed with coke and then fed into a kiln where the ore is reduced at 1300°C to SrS. Coke is added to the ore as a source of energy and carbon. The carbon combines with oxygen of sulfate group and escapes as CO₂. SrS is removed from the kiln, purified, and dissolved in water. Sulfide solution is then reacted with either Na₂CO₃ (soda ash method) or CO₂ (carbon dioxide method). Chemical reactions for soda ash method and carbon dioxide method are given bellow (Fulton, 1983).



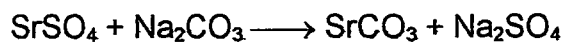
The soda ash method:



Carbon dioxide method:



In direct conversion method, celestite is ground and washed. It is then thickened and most of water is removed. Thickened ore is mixed with soda ash and treated with steam for 1-3 hours, causing celestite and soda ash to react and form solid SrCO_3 and water soluble Na_2SO_4 . Finally, insoluble SrCO_3 is separated by centrifuging. The reaction is as follows (Ober, 1995; Griffiths, 1992):



Produced SrCO_3 is directly used or converted into other Sr-compounds. The largest end use of SrCO_3 is in the production of color TV tube faceplate glass to block X-ray emission. Almost 70 % of the USA's SrCO_3 consumption, about 80% of Japan's consumption and 20 % of European countries' consumption are for color TV tube faceplate glass. The USA's and Japan's color TV manufacturers use SrCO_3 to shield X-ray produced by high operating voltage. On the other hand, color televisions manufactured in Europe operate with low voltage, so the amount of

produced harmful X-ray is low and this amount is shielded by using barium in color TV tube faceplate glass. SrCO_3 is also used in electrolytic zinc production. In Turkey, ÇİN-KUR buy 20 tpa celestite concentrate from Barit Maden Türk A.Ş. and convert into SrCO_3 by soda ash method (Kayan and Çubuk, 1995a). World SrCO_3 consumption in zinc electrolysis is approximately is 3,000–4,000 tpa. Other SrCO_3 consumption areas are in production of permanent ceramic ferrite magnets in ceramic and glass application, in fireworks, in signal systems and in the production of Sr-compounds. Specifications of manufactured carbonate made from celestite for glass grade to glass industry and ceramic grade for ferrites to electronic industry are given on Table-1.3 (Fulton, 1983).

Table-1.3: Specifications of manufactured SrCO_3 made from celestite for glass and ceramic industries

	Glass Grade	Ceramic Grade
SrCO_3	96 % min.	96 % min.
BaCO_3	3 % max.	1.5 % max.
CaCO_3	0.5 % max.	-
Total S	0.4 % max.	0.4 % max.
Fe_2O_3	0.01 % max.	-
Na_2CO_3	1.0 % max.	-
Size (Tyler mesh)		
on 10 mesh	0.0 %	no size specification
on 14 mesh	2.0 % max.	
on 100 mesh	75-85 %	
on 150 mesh	95 % min.	
thru 150 mesh	5.0 % max.	

$\text{Sr}(\text{NO}_3)_2$ is produced by reacting SrCO_3 and HNO_3 . It is used in pyrotechnic devices such as fireworks, distress signals and tracer bullets,

since it produces bright red flame when it burns. Other Sr-compounds are used in many areas. End use of some of other Sr-compounds are as follows: Strontium chromate, strontium chloride and strontium hydrate are used in corrosion resistant paints, in toothpastes for temperature sensitive teeth, and in Sr-oils and soaps, respectively.

Celestite is generally sold on the basis of a minimum SrSO_4 content with premiums payable for each additional 1 % per ton above the basis. Of course, price is negotiable depending on tonnages involved. Turkish celestite is priced at \$80 per ton FOB Iskenderun for a minimum 92 % SrSO_4 with premiums of \$6 for every 1 % over the base. Mexican celestite is priced in the low \$90s basis FOB vessel and Spanish celestite is currently priced at \$85 per ton FOB Motril basis minimum 94 % SrSO_4 (Griffiths, 1992).

CHAPTER 2

PREVIOUS STUDIES ABOUT CELESTITE CONCENTRATION

Most of the currently operating celestite facilities produce sufficient supplies with minimal processing necessary to achieve acceptable specifications. Hand sorting and some washing are all that are necessary at many strontium mines to produce celestite of acceptable quality. A few operations use froth flotation or gravity separation to beneficiate ore (Ober, 1995). Up to now, hand picking, gravity separation and flotation have been successfully used in the mineral processing of celestite in order to upgrade the ore to 92 % SrSO_4 , suitable for making SrCO_3 . Usually high grade ore with 90 % or higher SrSO_4 could meet the requirement simply by hand picking or crushing and screening, whereas for lower grade ore some other beneficiation must be used (Hong, 1993).

2.1 STUDIES ON CELESTITE CONCENTRATION BY GRAVITY

METHODS

Bayraktar, et.al. studied on hand picking tailings of Sivas-Budaklı celestite ore of Barit Maden Türk A.Ş.. Sample contains celestite, gypsum, calcite, quartz, anhydrite, limestone and clay. Since, anhydrite, limonite

and clay minerals can be ground easily, at first step a rotating drum was used to grind these minerals to slime sizes and then sample was classified into three fraction: Coarse, fine and slime. Coarse and fine fractions were concentrated on jigs and shaking tables, respectively. Slime was discarded as tailing. A concentrate assaying 97.18 % SrSO_4 was obtained with 91.07 % recovery. Figure-2.1 shows the proposed flowsheet to Barit Maden Türk A.Ş. (İ.T.Ü., 1981).

Aslan and Canbazoğlu studied the concentration of $-100 \mu\text{m}$ size celestite-gypsum ore by using Multi Gravity Separator (MGS). 94.40 % SrSO_4 grade concentrate was obtained from a feed assaying 76.78 % SrSO_4 with 87.35 % recovery. Optimum operating parameters of MGS were determined as follows (Aslan and Canbazoğlu, 1996):

Drum speed	: 150 rpm
Tilt angle	: 6°
Shake amplitude	: 20 mm
Shake frequency	: 4 stroke/sec.
Washwater	: 4 l/min.
Feed pulp density	: 27.5 % solid by weight

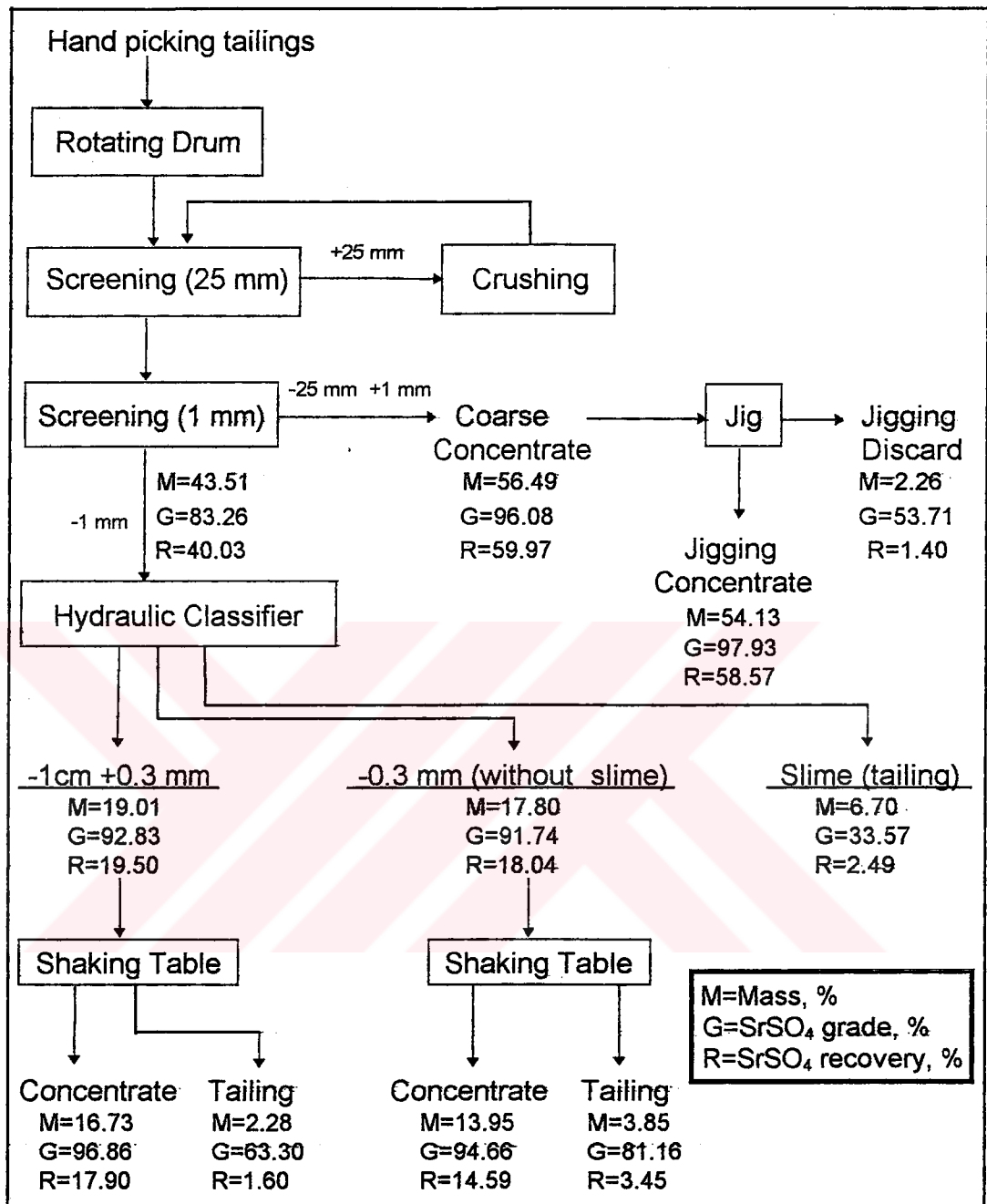


Figure-2.1: Concentration flowsheet for hand picking tailings of Barit Maden

Türk A.Ş.

Canbazoğlu, et.al., studied the concentration of four different samples with 83.60 %, 32.20 %, 84,10 % and 40.10 % SrSO₄ grades from four different mine areas of Barit Maden Türk A.Ş.. In experimental studies, MGS was used. Samples were ground to -104 µm. Concentrates assaying 85.27 % SrSO₄, 61.65 % SrSO₄, 87.30 % SrSO₄ and 52.50 % SrSO₄ were obtained with 58.04 %, 60.69 %, 56.05 % and 82.87 % recoveries, respectively, by applying the following operating parameters (C.Ü., 1996):

Drum speed	: 150 rpm
Tilt angle	: 4°
Shake amplitude	: 15 mm
Shake frequency	: 4 /sec.
Washwater	: 6 l/min.
Feed pulp density	: 30% solid by weight

MTA studied to concentrate -210 µm size and 80 % SrSO₄ grade celestite ore on shaking table from Alişanağılı at Zara, Sivas. As a result, a concentrate assaying 92.20 % SrSO₄ was obtained with 70.80 % recovery (Kayan and Çubuk, 1995a). MTA also studied to concentrate Kabalı Köyü celestite ore on shaking table from Hafik, Sivas. A concentrate containing 80.22 % SrSO₄ was recovered with 63.22 % from -1.41 mm size and 54.27 % SrSO₄ grade ore (Kayan, Aktürk and Aslan, 1988).

Özkan studied to concentrate celestite-gypsum ore assaying 86.49 % SrSO₄ by shaking table and jigging. 96.87 % SrSO₄ grade of concentrate

had been obtained with 91.48 % recovery from minus 8 mm size sample (Özkan, 1996).

2.2 STUDIES ON CELESTITE CONCENTRATION BY FLOTATION

Alkaline salts of oleic acid, particularly sodium oleate, are among the collectors traditionally employed for the flotation recovery of celestite, barite and fluorite, as well as of the other minerals which can also be included in the group of slightly soluble solids (Bahr, 1975; Clement, et al., 1973; Glembotskii, et al., 1961; Glembotskii and Uvarov, 1962; Taha, et al., 1984; Taha, et al., 1985; Uvarov and Glembotskii, 1964). But high grade concentrates are sometimes difficult to obtain because of contamination with calcite and barite, which float well with the same reagents. Quebracho, sodium silicate and copper sulfate have all been used with varying degrees of success in the system for improvement of selectivity (Zaman, 1985).

Sodium silicate is currently one of the most widely used non-sulphide mineral depressant (Hanna and Somasundaran, 1976; Mishra, 1982; Yongxin and Changgen, 1983). It had shown strong depressing effects on celestite from the Jake Enon deposit of Canada. The crude ore, grading 55 % SrSO_4 , was ground to 100 % $-75 \mu\text{m}$, and a final concentrate containing 84.5 % SrSO_4 with a recovery of 78 % was obtained with Igepon (oleate) T33 as collector and citric acid as depressant for fluorite (Hong, 1993).

Tests undertaken in Spain had shown that with sodium oleate, at pH 5-12, sodium metasilicate gave a much stronger depressant effect on calcite than celestite. However, at higher concentration of sodium oleate, the depressing action of sodium metasilicate decreased significantly (Hong, 1993).

Falconer and Crawford were able to separate SrSO_4 from an ore containing CaO, MgO and feldspar by using sodium silicate and pine oil together with oleic acid as reagents for flotation (Falconer and Crawford, 1944).

Hernández and Calero studied influence of quebracho and Na_2SiO_3 on flotation of celestite and calcite with sodium oleate. Experiments were carried out with pure minerals and mixed samples ground below 75 μm size from Montevives Mount in Las Gabias, Granada, Spain. They stated that quebracho depressed calcite and celestite effectively although the depressor action of the agent depended largely on the concentration of collector. They also found that Na_2SiO_3 had a depressing action more intense than quebracho on celestite and calcite, but its effect was more accused on calcite mainly when low concentrations of sodium oleate are used (Hernández and Calero, 1993).

Hernández and Calero also studied the influence of conditioning and flotation times for celestite and calcite separately. 10 min. conditioning time

and 5 min. flotation time for celestite and 3 min. flotation time for calcite had been found as optimum values at pH 9 and with 600 g/t sodium oleate. It is also indicated in this study that calcite is rapidly coated by sodium oleate. (Hernández and Calero, 1995).

Houot, Joussemet and Fabre studied on an ore from Escuzar mine on laboratory, pilot plant and mill scale. Mineralogy of the crude ore was given on Table-2.1.

Table-2.1: Mineralogy of crude ore from
Escuzar mine

Mineral	Distribution, %
BaSO ₄	0.5
CaCO ₃	29.3
SrSO ₄	57.5
CaSO ₄ .H ₂ O	5.0
Clays et.al.	7.7

In laboratory scale, 300 g/t Na₂SiO₃ as depressant, 13-19 kg/t Na₂CO₃ as pH regulator, 1000 g/t of alkylsulphate as collector containing 50 % of active substance were used. As a result, 95.1 % SrSO₄ grade of concentrate with 68.5 % recovery and 42.3 % SrSO₄ grade of middling with 18.2 % recovery had been obtained. A final tailing and slimes assaying 13.9 % SrSO₄ and 36.8 % SrSO₄ had been discarded with 7.5 % and 5.8 % recoveries, respectively.

In pilot plant scale, pulp density and pH was adjusted to 40 % and 9.5, respectively. In the circuit, 1 kg/t Na_2SiO_3 , 14-15 kg/t Na_2CO_3 and 360 g/t collector were consumed and a concentrate assaying 95,8 % SrSO_4 was produced with 65.2 % flotation recovery. Tailings and slimes were discarded with 27.4 % SrSO_4 and 57.0 % SrSO_4 grades and 14.6 % and 20.2 % recoveries, respectively.

In flotation plant scale, 800-1,000 g/t Na_2SiO_3 , 5-7.7 kg/t Na_2CO_3 (pH=10) and 200-295 g/t collector were consumed. In conclusion, a concentrate assaying 94.8 % SrSO_4 was produced with 63,1 % flotation recovery. Tailings and slimes were discarded with 29.8 % SrSO_4 and 49.1 % SrSO_4 grades and 21.5 % and 15.4 % recoveries, respectively (Houot, Joussemet and Fabre, 1993).

2.3 CELESTITE CONCENTRATION APPLICATIONS OVER THE WORLD

2.3.1 TURKEY

Barit Maden Türk A.Ş. has a concentrator at Sivas where ore containing 65-70 % celestite, 25-30 % gypsum and 5-10 % clay minerals. The ore is concentrated by using jig, stripa heavy medium separator and

shaking table. A concentrate assaying minimum 94 % SrSO_4 is obtained. Flowsheet of this plant is given in Figure-2.2 (Cebeci, et.al.,1993).

2.3.2 MEXICO

Cia Minera La Valenciana SA has a processing plant, consisting of bendelari jig and wilfley tables. Minimum 92 % SrSO_4 of concentrate is produced (Griffiths, 1985). In Sales y Oxidos SA de CV mine at San Pedro in Coahuila, only hand picking method is applied to concentrate the ore containing 93-94 % SrSO_4 . Concentrate grade was not given. Cia Minera La Roja SA upgrade ore assaying 94 % SrSO_4 in a washing and flotation plant at Madero to minimum 95 % SrSO_4 grade (Kendall,1995).

2.3.3 ALGERIA

Enterprise Nationale des Produits Miniers Non-Ferreux et des Substances Utiles (ENOF) upgrade celestite ore by hand picking method to 95-98 % SrSO_4 from a deposit at Beni Mansour (Griffiths, 1992).

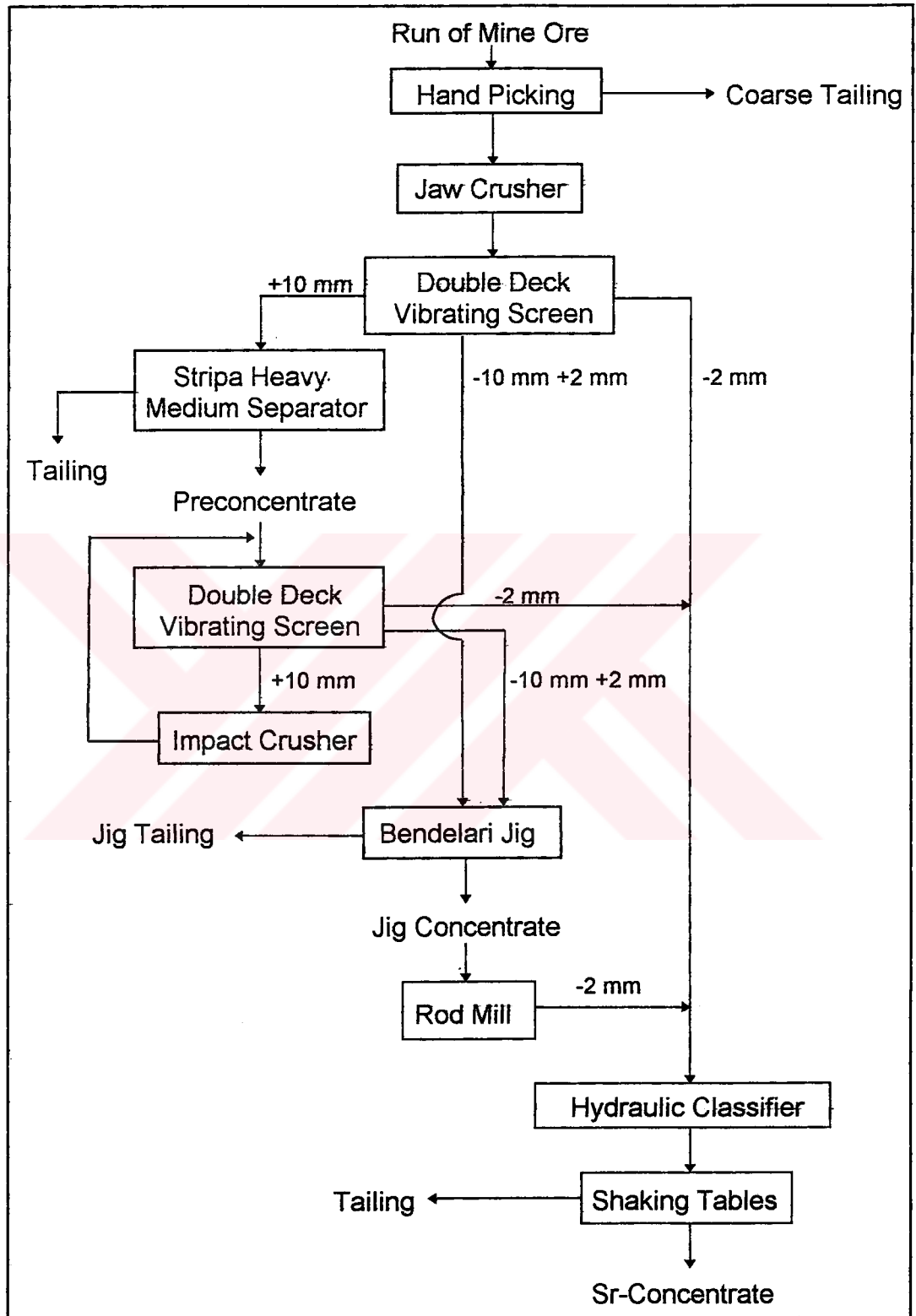


Figure-2.2: Celestite ore concentration plant flowsheet of Barit Maden Türk

A.Ş.

2.3.4 SPAIN

Bruno SA upgrade celestite ore by hand sorting method from 80 % SrSO_4 grade to 94 % SrSO_4 grade. Concentrate is sold in minus 4 inch and minus 2 inch sizes (Griffiths, 1992).

In Escuzar flotation plant of Kali Chemie Co., ore assaying 50 % SrSO_4 , 45 % CaCO_3 , and 5 % gypsum and clay minerals, is processed. Liberation size of ore is below 40-50 μm . A concentrate grading 93-94 % SrSO_4 is obtained by using 700-1200 g/t alkylsulphate as collector, 4 kg/t Na_2CO_3 as pH regulator (pH=10, 12) and 100-500 g/t Na_2SiO_3 as depressant. Sometimes, prophanosphate is used as depressant. Flow sheet of this plant is as shown in Figure-2.3 (Teke, 1990).

2.3.5 IRAN

Iran Strontium Co. upgrade celestite ore by hand sorting method from 88-89 % SrSO_4 to 90-92 % SrSO_4 (Griffiths, 1985).

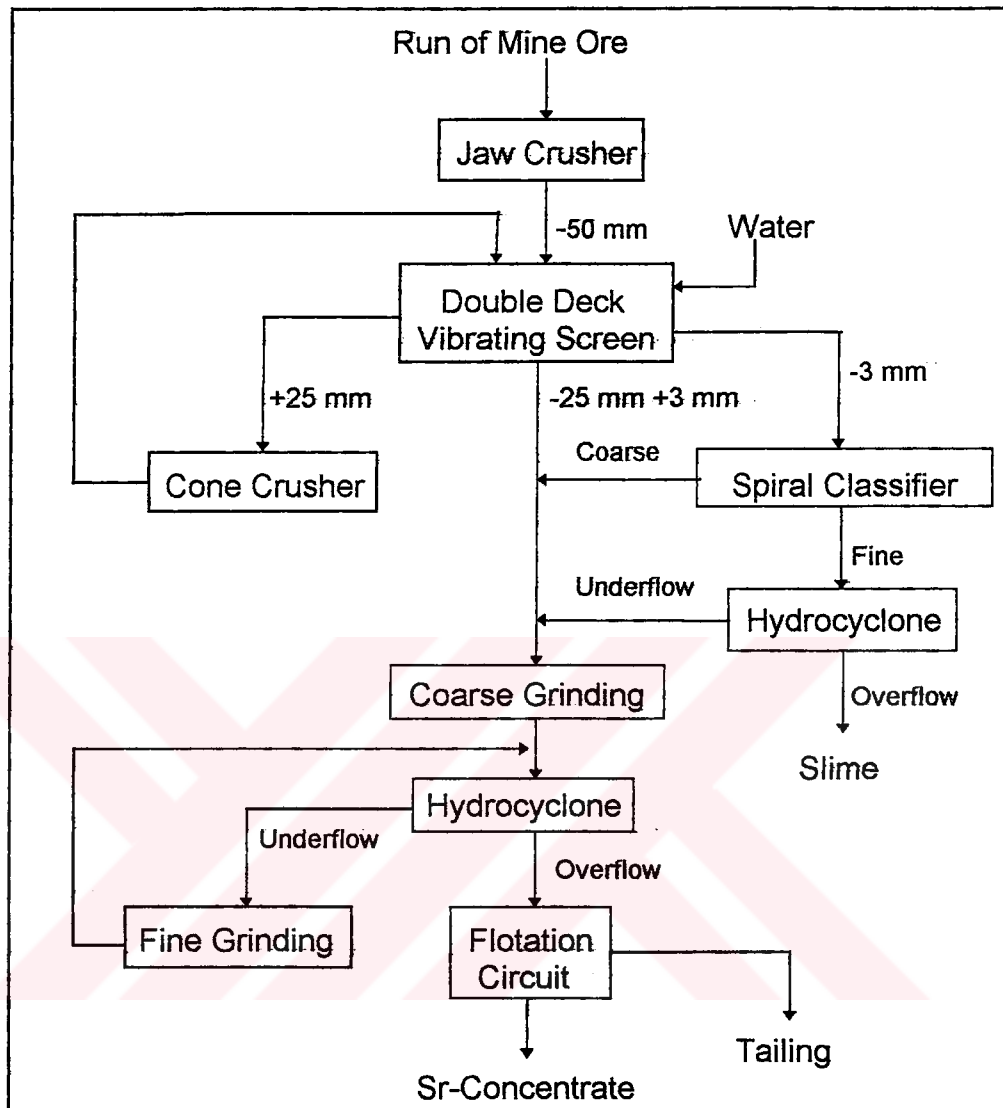


Figure-2.3: Escuzar flotation plant flowsheet of Kali Chemie Co.

2.3.6 ENGLAND

In England there are two celestite processing plant. A simplified flowsheet of process at Gloucestershire is as shown in Figure-2.4 (Zaman,1985). However no other information was found about this plant.

Bristol Mineral Co. concentrate celestite ore containing 70-75 % SrSO_4 , 20-25 % CaCO_3 and 5-10 % clay minerals to a concentrate assaying 95 % SrSO_4 . Flow sheet of this plant is given in Figure-2.5 (Teke, 1990).

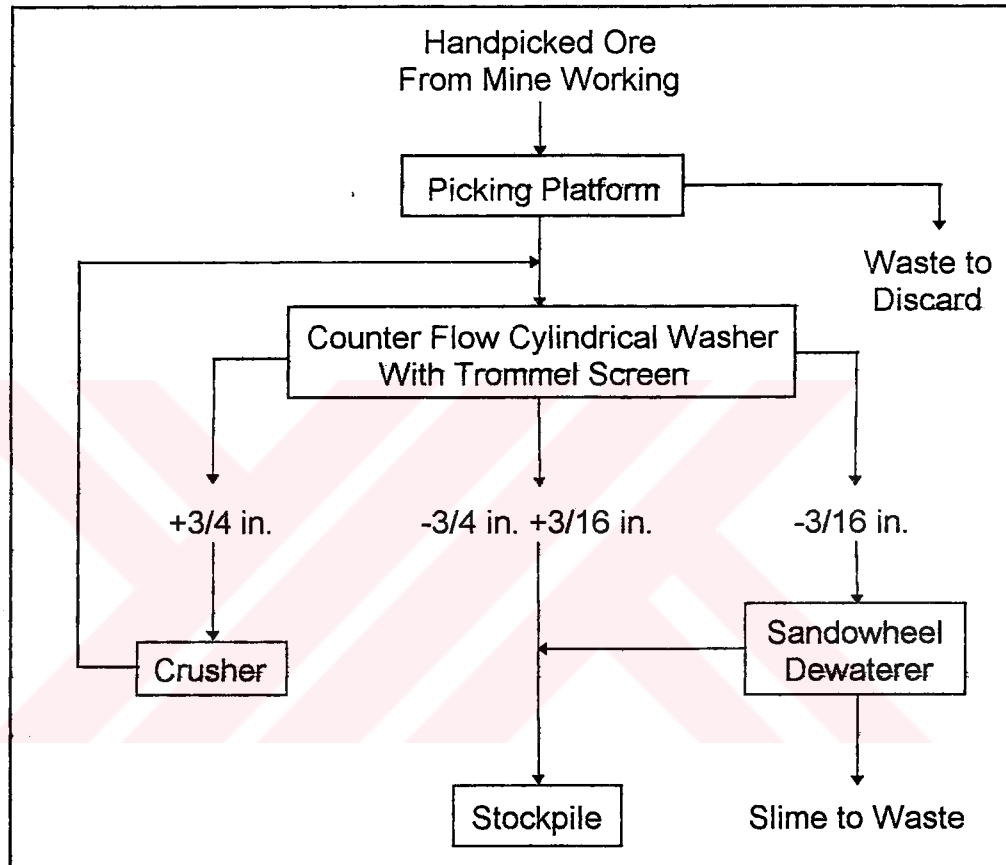


Figure-2..4: Simplified flowsheet of celestite processing plant at Gloucestershire, England

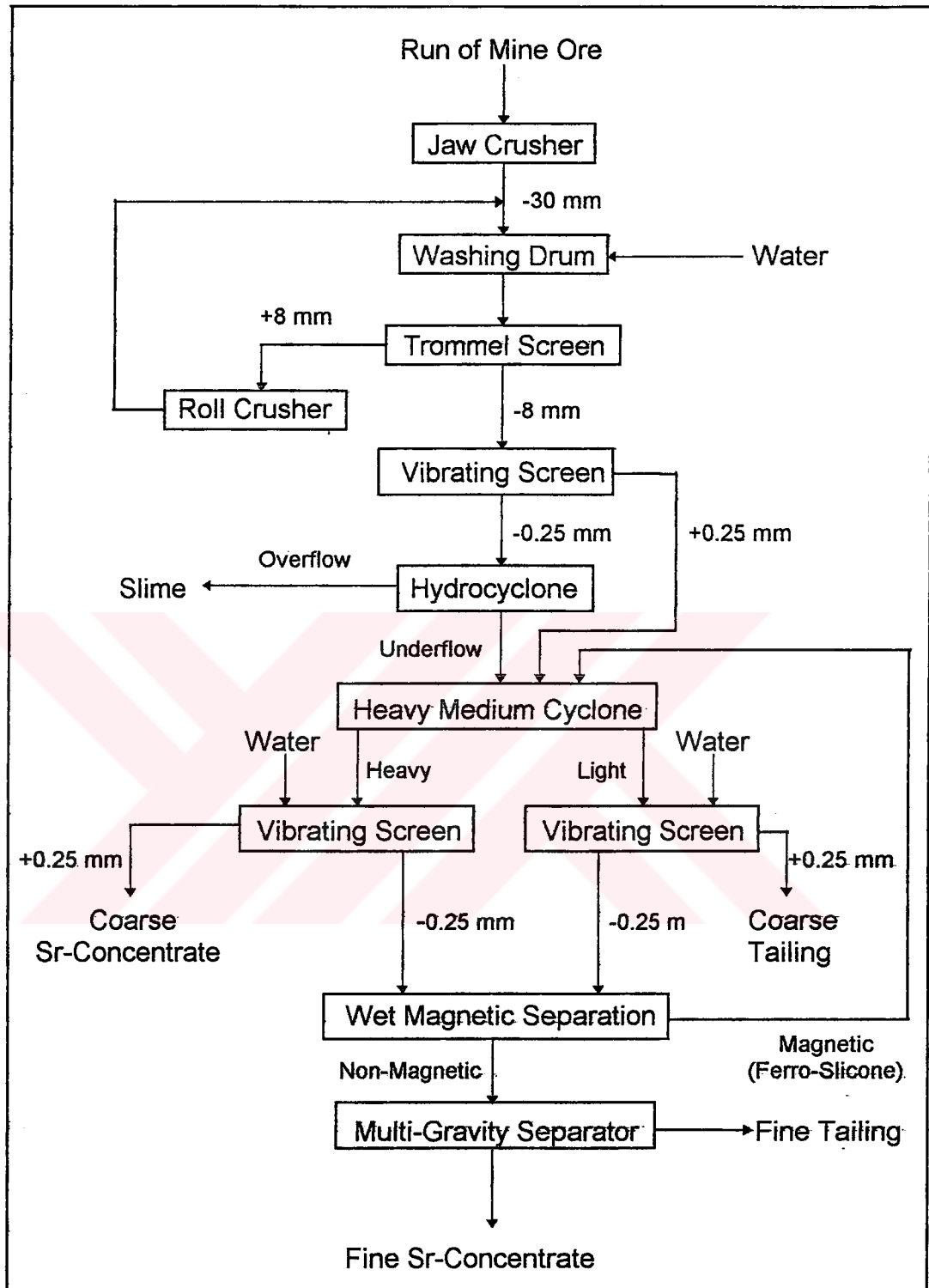


Figure-2.5: Celestite ore concentration plant flowsheet of Bristol Mineral

Co.

CHAPTER 3

EXPERIMENTAL STUDIES

3.1 PREPARATION OF TEST SAMPLES

About 300 kg representative celestite-calcite ore sample was taken from Nasır Village celestite-calcite ore deposit of Barit Maden Türk A.Ş. at Zara, Sivas. Sample was 100 % minus 15 cm in size. It was crushed by using jaw crushers of 16 cm x 12 cm and 11 cm x 9 cm openings. The crushed material was first of all quartered and half of sample was saved. Then material was riffled in order to obtain representative samples in 330 g lots and the samples were packed separately for subsequent operations.

3.2 ANALYSIS

To determine mineralogical composition of ore sample, X-Ray Diffraction (XRD) method was applied. Result given in Figure-3.1 shows that sample contains celestite, calcite and quartz.

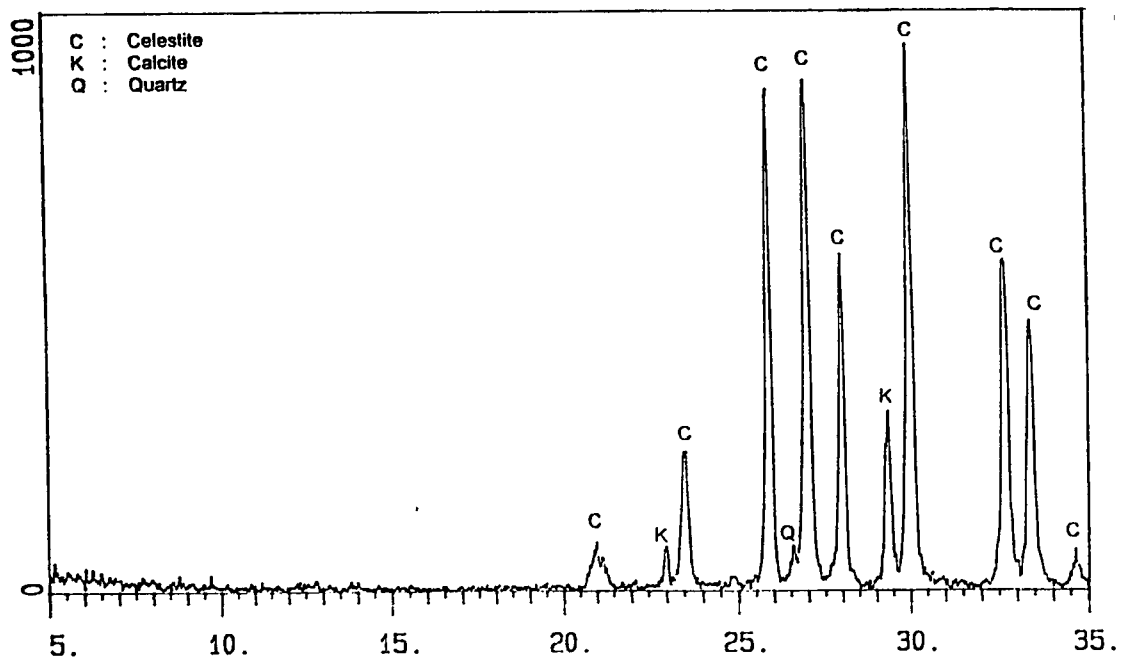


Figure-3.1:Result of X-Ray Diffraction (XRD) method

Chemical analysis of sample was made by three different method: Fluxation method, chemical method and gravity method (See Appendix). Table-3.1 shows the chemical composition of sample. From Table-3.1, SrSO_4 , CaCO_3 and SiO_2 grades of sample were found as 85,51 % SrSO_4 by gravity method, 9.19 % CaCO_3 by fluxation method and 3.31 % SiO_2 by chemical method, respectively. Gravity method was applied in the determination of SrSO_4 grade of products taken from experimental studies.

3.3 GRINDING TESTS

Dry grinding was applied by using a rod mill having 215 mm inner diameter and 300 mm length. In grinding tests, 5 of 30 mm diameter, 8 of 25 mm diameter and 15 of 20 mm diameter rods and 330 g sample were

used. Rods were 280 mm in length. Rotational speed was adjusted to 61.01 % of critical speed (98.35 rpm) of mill.

Table-3.1:Chemical composition of sample

Element	Content
Sr	40.78 %
Ca	3.68 %
Si	1.55 %
Al	0.55 %
Mg	2084 ppm
Fe	2059 ppm
Na	659 ppm
K	543 ppm
Ni	241 ppm
Pb	137 ppm
Co	77 ppm
Cr	69 ppm
Zn	62 ppm
Mn	48 ppm
Cu	7 ppm

3.4 SCREEN ANALYSIS

ASTM standard Retsch screens and Retsch screen vibrating equipment were used in screen analysis. Vibration frequency and screening time were applied as 30 stroke/sec and 20 min, respectively. For crushed ore sample and for 15 sec, 30 sec, 1 min, 3 min, 5 min and 7 min grinding times, wet screening was made. Results were given on Table-3.2 and Table-3.3 and in Figure-3.2.

Table-3.2: Screen analysis for crushed ore sample

Screen Size mm	Cum. Passing Weight %
0.250	21.67
0.425	26.23
0.710	30.88
1.700	44.42
3.350	69.75

Table-3.3: Screen analysis for different grinding times

Screen Size μm	Cumulative Passing, Weight %					
	15 sec	30 sec	1 min	3 min	5 min	7 min
38	14.12	19.62	30.62	56.81	74.92	89.69
75	26.02	36.66	57.73	92.75	99.24	99.68
125	36.31	51.15	79.01	99.61	100.00	100.00
180	44.26	62.38	90.37	100.00	-	-
250	53.03	73.41	98.68	-	-	-

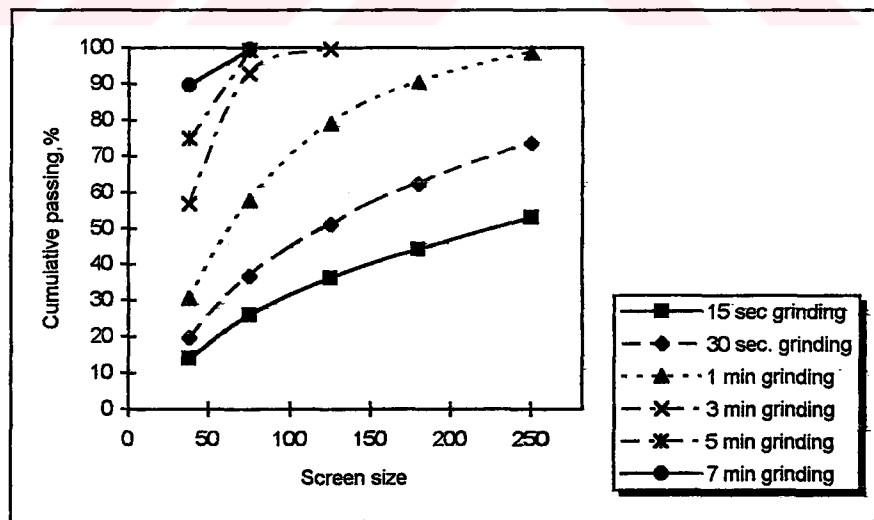


Figure-3.2: Screen analysis for different grinding times

3.5 FLOTATION TESTS

Flotation experiments were carried out to determine the effects of pH, grinding time, types and consumption of collectors, depressants and frothers, flotation time, and effect of number of cleaning steps on concentrate grade and recovery. Experiments were conducted in Denver Sub A laboratory type flotation machine with 1.3 liter cell. Flotation pulp density was approximately 23 % by weight. Impeller speed was adjusted to 1100 rpm.

3.5.1 COLLECTORS

Sodium oleate, oleic acid, linoleic acid, Amine-D, Aerofloat-801, Aerofloat-830 and Aerofloat-845 were examined as collector.

Sodium oleate, oleic acid and linoleic acid are anionic, oxyhydri collectors, and Amine-D is cationic, dodecyl amine type collector. Oleic acid and linoleic acid are carboxylic acid type collectors. They were rarefied by distilled water. Because of formation of emulsion, they were mixed well and then used in experiments.

Sodium oleate is carboxylate type collector. It was produced by reacting oleic acid with NaOH. Oleic acid was solved by ethyl alcohol and

NaOH was dissolved in distilled water. These two solution were mixed and stirred up by a magnetic mixer for 4 hours. Reaction occurs as follows:



Aerofloat-801 (A-801) is anionic, petroleum sulfonate type promoter. It is water soluble at room temperature. It has strong frothing property (Anonymous, 1986).

Aerofloat-830 (A-830) and Aerofloat-845 (A-845), anionic, alkyl succinamate type promoters, had developed to provide more selectivity than can be had with fatty acids. These are excellent promoters for barite, celestite and sheelite in alkaline circuits. They are also used as secondary collectors with fatty acids and petroleum sulfonates to provide improved metallurgy and circuit control. They have application wherever fatty acids or petroleum sulfonates are principal collectors. Generally, the short conditioning times with A-830 and A-845 have favored best metallurgy. When used as principal collectors these promoters tend to produce large volume of froth having little load-carrying capacity. A-845 is a completely water soluble collector, can be fed in any concentration. A-830 is semi-liquid to soft paste, dispersible in water (Anonymous, 1986).

3.5.2 FROTHERS

Flotal-B, Aerofroth-65 (A-65), Aerofroth-73 (A-73), Aerofroth-76 (A-76) and Aerofroth-88 (A-88) were examined as frother. Flotal-B is water soluble, alcohol type collector. A-65 is a polypropylen glycol. It is completely water soluble and a powerful frother. A-73 is a mixture of C₄ to C₇ alcohols and hydrocarbon oil. It does not dissolve in water well. A-76 is a mixture of C₄ to C₇ alcohols. It is useful wherever a light yet lively frothing action is desired. A-88 is composed of higher alcohol. It is used where a strong action is required with less persistence than polypropylene glycol frothers. It forms emulsion in water (Anonymous, 1986).

3.5.3 MODIFIERS

Dilute solutions of NaOH, Na₂CO₃ and H₂SO₄ were used as pH-regulators.

Gum arabic, quebracho, Na₂SiO₃, starch, gelatine, albumin, dextrin and calgon, all in powder form, were examined as depressants. Gum arabic, quebracho and calgon were dissolved in distilled water. Albumin and dextrin were dissolved in 0.9 % NaCl solution. Gelatine was added to distilled water, and heated and dissolved in water, and then hot solution was used in experiments.

For the preparation of starch to flotation, 4 unit starch and 1 unit NaOH were mixed well and diluted 20 times of total starch and NaOH with distilled water. Then solution was boiled, and then cooled and used in experiments. This is known as caustic starch solution.

3.6 SELECTION CRITERIA

As mentioned in section 1.4, it was given that Turkish celestite was priced at \$80 per ton FOB Iskenderun for a minimum 92 % SrSO₄ grade of concentrate and for every 1 % over the base, \$6 per ton additional payment was made. Therefore, income was used as a selection criteria and following formula was applied.

$$\text{Income} = \% C \times (80 + A \times 6)$$

% C = Weight percentage of concentrate

A = (Concentrate grade for above 92 % SrSO₄) - 92

CHAPTER 4

EXPERIMENTAL RESULTS AND DISCUSSIONS

4.1 EFFECT OF pH

8 different pH values were chosen to investigate the effect of pH on flotation. pH was regulated with NaOH and H₂SO₄. Experimental results indicate that pH is very important parameter for flotation of this celestite ore sample. Grade and recovery of concentrate increase up to pH 10 and then decrease. Optimum flotation results was obtained at pH 10. Results of flotation experiments to determine optimum pH value with respect to concentrate grade and recovery are given on Table-4.1 and plotted in Figure-4.1. In the following experiments pH of flotation pulp was adjusted to 10.

Table-4.1:Effect of pH on flotation

pH	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
5	34.53	73.81	29.81
6	39.49	83.58	38.60
7	51.06	88.38	52.78
8	61.56	89.01	64.08
9	68.90	89.49	72.10
10	84.07	90.98	89.45
11	83.29	89.13	86.82
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH, H ₂ SO ₄ Conditioning time for pH regulator: 5 min Collector: 375 g/t Na-Oleate Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

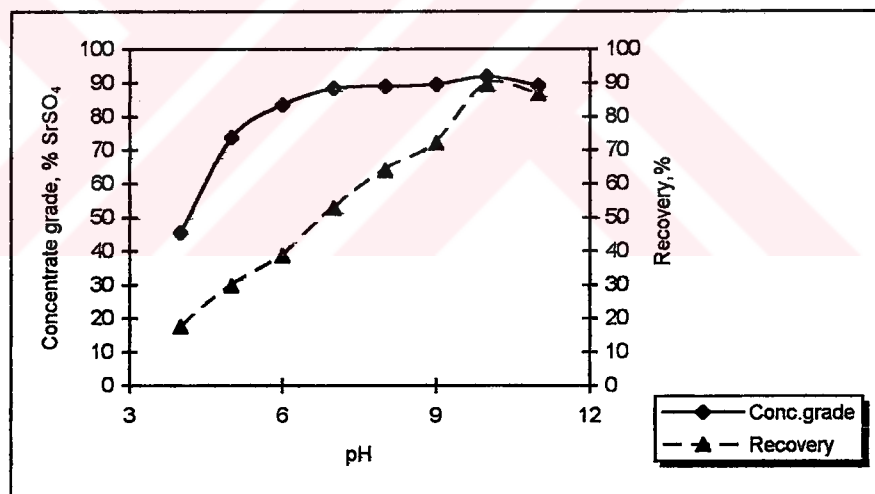


Figure-4.1:Effect of pH on flotation

4.2 EFFECT OF GRINDING TIME

6 different grinding times (15 second, 30 second, 1 minutes, 3 minutes, 5 minutes, 7 minutes) were chosen to investigate the effect of particle size on flotation.

Experiments were carried out by using both NaOH and Na₂CO₃ as pH regulator. These experiments indicate that as the grinding time increases, recovery increases up to 1 minute grinding (80 % passing 129 µm screen size) while SrSO₄ grade of concentrate decreases. Optimum flotation result was obtained with 1 minute grinding time and by using NaOH as pH regulator. In the following experiments, 1 min grinding time was used. For coarse grinding (by 15 sec and 30 sec grinding times), poor metallurgical results may be due to coarse size of product and insufficient degree of liberation. Fine grinding (by 3 min, 5 min and 7 min grinding times) cause intruding of froth by fine gangue and so grade of concentrate slightly decreases while recovery increases.

The results of flotation experiments to determine optimum grinding time with respect to concentrate grade and recovery are given on Table-4.2 and plotted in Figure-4.2 and Figure-4.3.

As seen in Table-4.3, saleable concentrate was obtained from sample ground 15 sec with NaOH as pH regulator. However concentrate weight percentage and therefore flotation recovery are very low.

Table-4.2: Effect of grinding time on flotation

pH Regulator	Grinding Time (80 % Passing)	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
Na ₂ CO ₃	15 sec (540 μm)	29.63	89.66	31.07
Na ₂ CO ₃	30 sec (301 μm)	61.99	88.12	63.88
Na ₂ CO ₃	1 min (129 μm)	80.31	86.54	81.28
Na ₂ CO ₃	3 min (57 μm)	83.45	85.03	82.98
Na ₂ CO ₃	5 min (44 μm)	83.67	83.94	82.13
Na ₂ CO ₃	7 min (28 μm)	84.92	83.37	82.79
NaOH	15 sec (540 μm)	35.30	93.64	38.66
NaOH	30 sec (301 μm)	58.62	90.04	61.33
NaOH	1 min (129 μm)	84.07	90.98	89.45
NaOH	3 min (57 μm)	89.58	87.45	91.61
NaOH	5 min (44 μm)	90.49	86.29	91.32
NaOH	7 min (28 μm)	90.13	87.56	92.29
Experimental Conditions	pH regulator: NaOH, Na ₂ CO ₃ (pH=10) Conditioning time for pH regulator: 5 min (pH=10) Collector: 375 g/t Na-Oleate Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min			

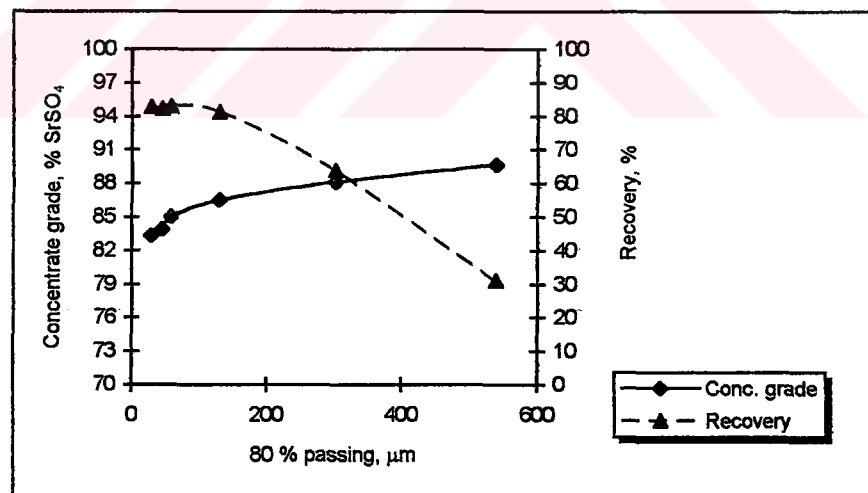


Figure-4.2: Effect of grinding time on flotation when Na₂CO₃ is used as pH regulator

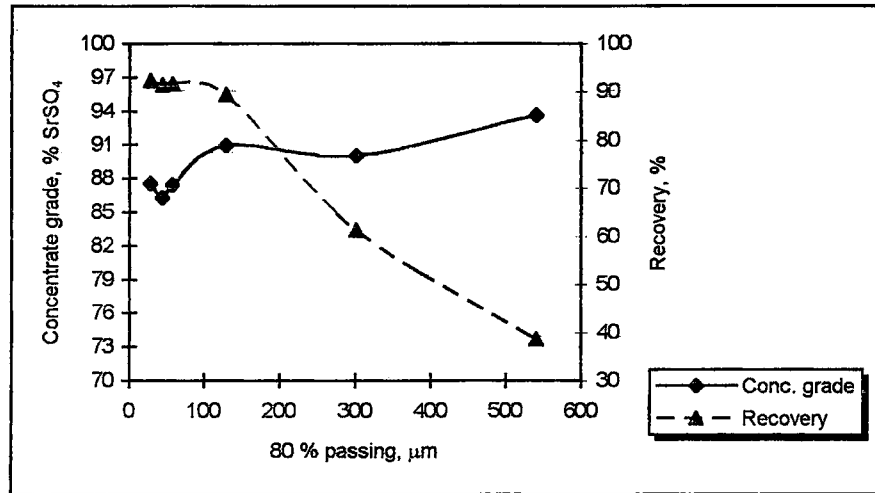


Figure-4.3: Effect of grinding time on flotation when NaOH is used as pH regulator

4.3 EFFECTS OF TYPE AND AMOUNT OF COLLECTORS

A series of flotation experiments were carried out to determine the effect of 7 types of collectors on the flotation recovery and concentrate grade. These collectors were generally chosen such that they were recommended for celestite-calcite separation by flotation. Results of experimental studies are given on Table-4.3.

Table-4.3: Effect of collector type on flotation

Collector	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
Oleic Acid	80.13	88.65	80.26
Linoleic Acid	71.91	87.93	73.95
A-801	64.73	93.76	70.97
A-830	61.53	88.38	63.59
A-845	84.96	90.33	89.75
Na-Oleate	84.07	90.98	89.45
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Collector: 375 g/t Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

As seen from Table-4.3, in the case of Aerofloat-801 (A-801), concentrate grade is high enough but recovery and percentage of concentrate are low. Therefore, amount of A-801 was examined. However desired results were not obtained. The results of flotation experiment to determine the effect of amount of A-801 consumption on flotation recovery and concentrate grade are given on Table-4.4 and potted in Figure-4.4.

Table-4.4: Effect of Aerofloat-801 on flotation

A-801 g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
225	53.58	91.60	57.40
375	64.73	93.76	70.97
600	73.13	89.68	76.70
900	79.37	89.90	83.45
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Collector: Aerofloat-801 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

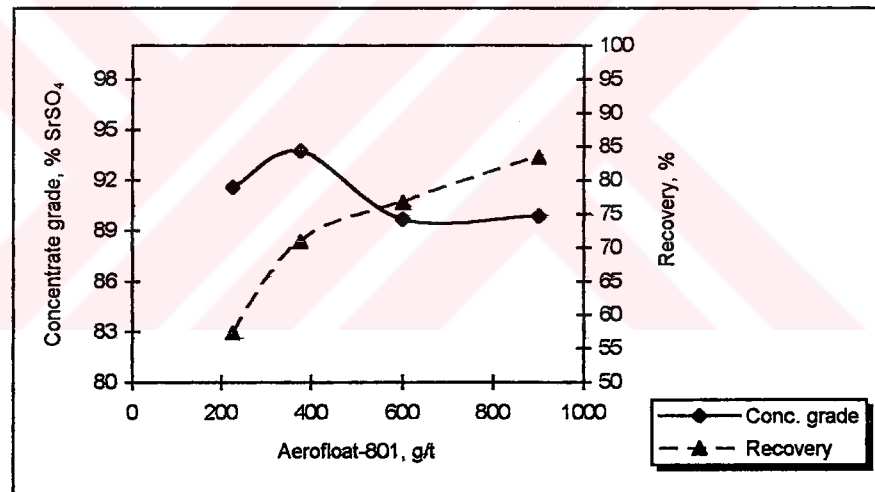


Figure-4.4: Effect of Aerofloat-801 on flotation

Reasonable recovery and grade values were obtained by using A-845 and Na-oleate as seen in Table-4.3. So a series of flotation experiments were carried out to determine the effect of amount of Na-oleate and A-845, and the use of Na-oleate and A-845 combination as

collectors in the same flotation experiments in different ratios on concentrate grade and recovery.

Na-oleate consumption was changed from 125 g/t to 625 g/t. As seen from experimental results on Table-4.5 and in Figure-4.5, when Na-oleate consumption increases, grade decreases while recovery increases. High amount of Na-oleate caused to the flotation of less hydrophobic minerals which increased recovery and decreased grade. The results indicate that 375 g/t collector consumption gives optimum grade and recovery.

Table-4.5: Effect of Na-oleate on flotation

Na-Oleate g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
125	60.75	87.38	62.07
250	65.22	92.39	70.46
375	84.07	90.98	89.45
500	86.96	89.12	90.64
625	89.95	88.62	91.12
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Collector: Na-Oleate Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

A-845 consumption was changed from 250 g/t to 625 g/t. According to experimental results given on Table-4.6 and in Figure-4.6, when A-845 consumption increases, grade decreases while recovery increases. High

amount of A-845 caused to decrease in recovery. This may be due to micelle formation. The results indicate that 375 g/t collector consumption gives optimum grade and recovery.

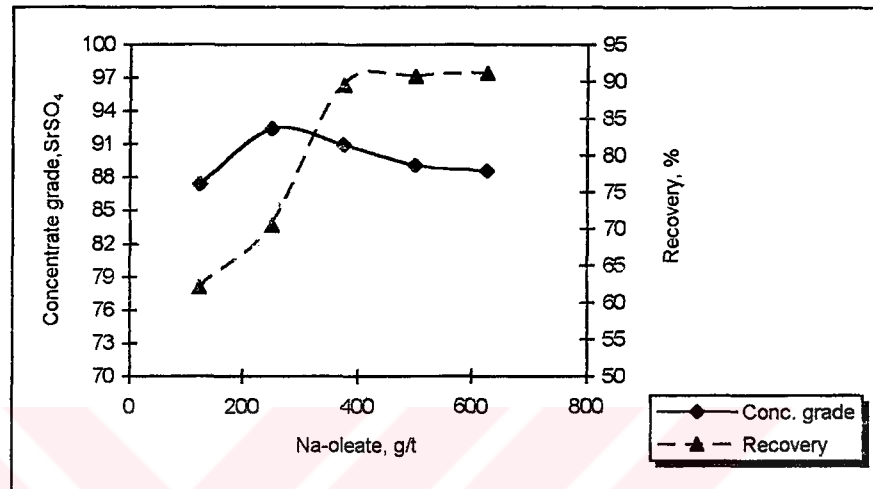


Figure-4.5: Effect of Na-oleate on flotation

Table-4.6: Effect of Aerofloat-845 on flotation

A-845 g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
250	70.88	92.74	76.87
375	84.96	90.33	89.75
500	86.47	88.35	89.34
625	85.70	86.29	86.48
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Collector: Aerofloat-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

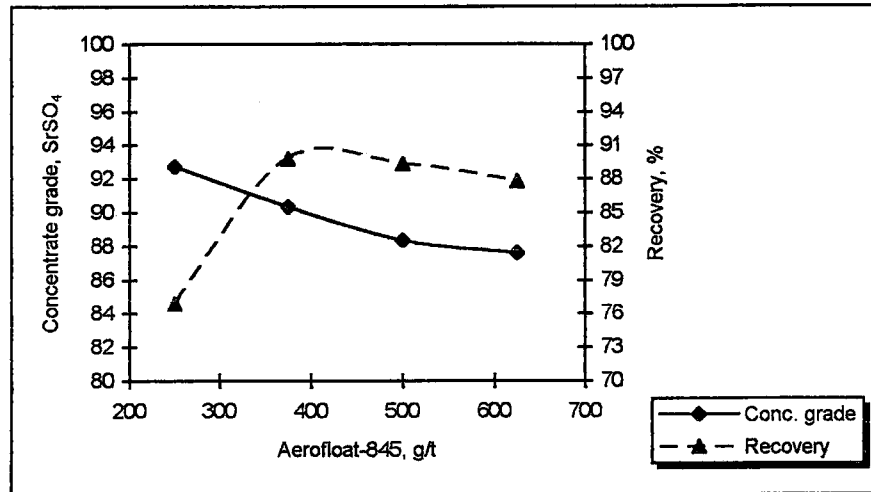


Figure-4.6: Effect of Aerofloat-845 on flotation

In the case of Na-oleate and A-845 combination, as seen from experimental results given on Table-4.7 and plotted in Figure-4.7, optimum result was obtained by using 50 % Na-oleate and 50 % A-845 combination.

Table-4.7: Effect of use of Na-oleate and Aerofloat-845 combination as collectors in different ratios on flotation.

Collector %		Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
Na-Oleate	A-845			
0	100	84.96	90.33	89.75
25	75	83.90	91.16	89.44
50	50	84.33	91.56	90.30
75	25	84.85	90.01	89.32
100	0	84.07	90.98	89.45
Experimental Conditions		Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Collector: 375 g/t (Na-oleate + A-845) Conditioning time for collectors: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min 0		

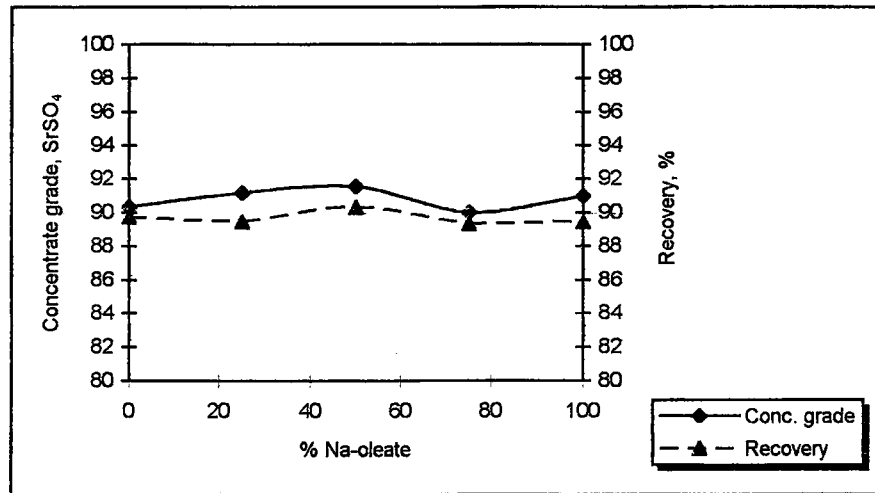


Figure-4.7: Effect of percentage of Na-oleate in Na-oleate and Aerofloat-845 combination on flotation.

Effect of Amine-D as a collector on concentrate grade and recovery was also examined for different pH values. Desired results could not be obtained. The effect of Amine-D on concentrate grade and flotation recovery at different pH values are given on Table-4.8 and plotted in Figure-4.8.

In conclusion, 50 % Na-oleate (187.5 g/t) and 50 % A-845 (187.5 g/t) combination was found as optimum collector types and amount for the flotation of this celestite ore sample. In the following experiments this combination was used as collector.

Table-4.8: Effect of pH on flotation when using Amine-D as a collector

pH	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
5	33.68	80.15	31.57
6	40.14	84.46	39.65
7	47.35	83.90	46.46
8	59.92	85.31	59.78
9	44.68	79.92	41.76
10	46.11	80.44	43.37
11	47.76	78.93	44.08
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH, H ₂ SO ₄ Conditioning time for pH regulator: 5 min Collector: 850 g/t Amine-D Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

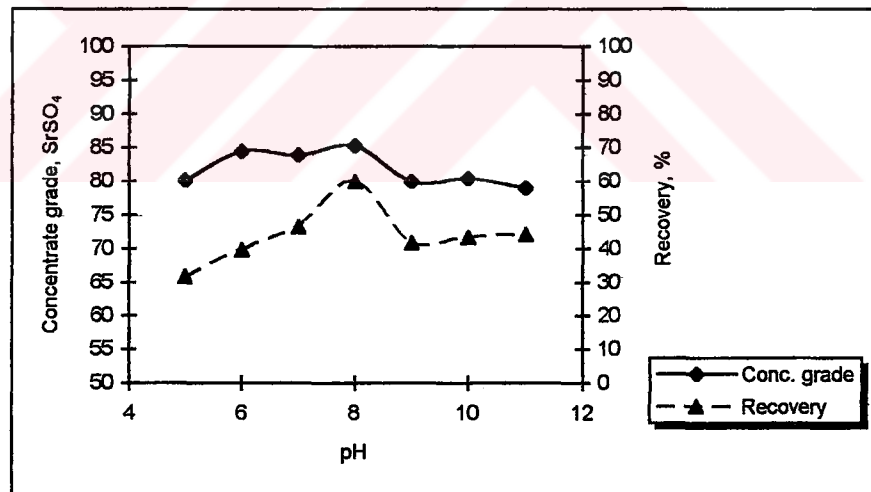


Figure-4.8: Effect of pH on flotation when using Amine-D as a collector

4.4 EFFECTS OF TYPE AND AMOUNT OF DEPRESSANTS

A series of flotation experiments were carried out to determine the effect of 8 (caustic starch, calgon, dextrin, albumin, gelatine, Na_2SiO_3 , gum arabic and quebracho) depressants on the concentrate grade and flotation recovery. These depressants were generally chosen such that they were recommended as effective depressants on gangue minerals of celestite-calcite ore.

When the using of caustic starch, calgon, dextrin and albumin, as the depressant consumption increases, then weight percentage of concentrate, concentrate grade and flotation recovery decreases. These results indicate that these depressants depressed celestite as well as calcite.

Results of flotation experiments to determine the effect of caustic starch, calgon, dextrin and albumin as on concentrate grade and recovery are given on Table-4.9-4.12 and plotted in Figure-4.9-4.12.

Table-4.9:Effect of caustic starch on flotation

Caustic Starch g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	82.97	91.23	88.52
50	80.44	90.12	84.78
100	75.57	89.84	79.40
250	70.74	90.36	74.75
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Caustic starch Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

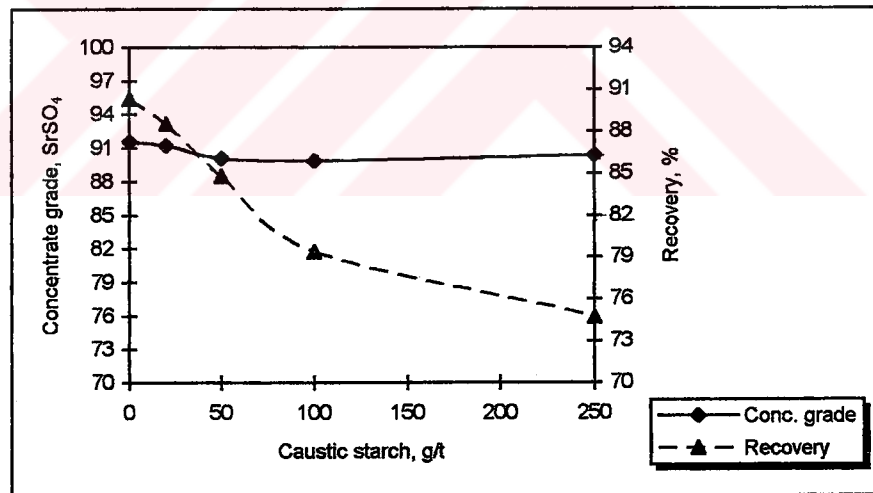


Figure-4.9:Effect of caustic starch on flotation

Table-4.10:Effect of calgon on flotation

Calgon g/t	Concentrate Weight %	Con. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	84.96	91.10	90.51
50	80.68	90.05	84.96
100	80.72	89.01	84.03
250	64.01	86.11	64.46
500	46.56	84.59	46.06
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Calgon Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

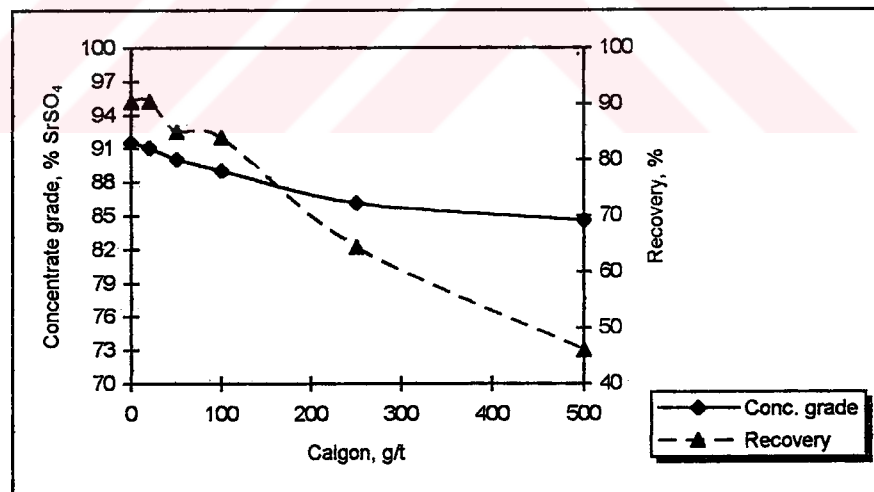


Figure-4.10:Effect of calgon on flotation

Table-4.11:Effect of dextrin on flotation

Dextrin g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	80.71	89.47	84.45
50	78.76	89.23	82.19
100	77.09	88.31	79.61
250	76.60	87.76	78.61
500	63.20	82.26	60.80
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Dextrin Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

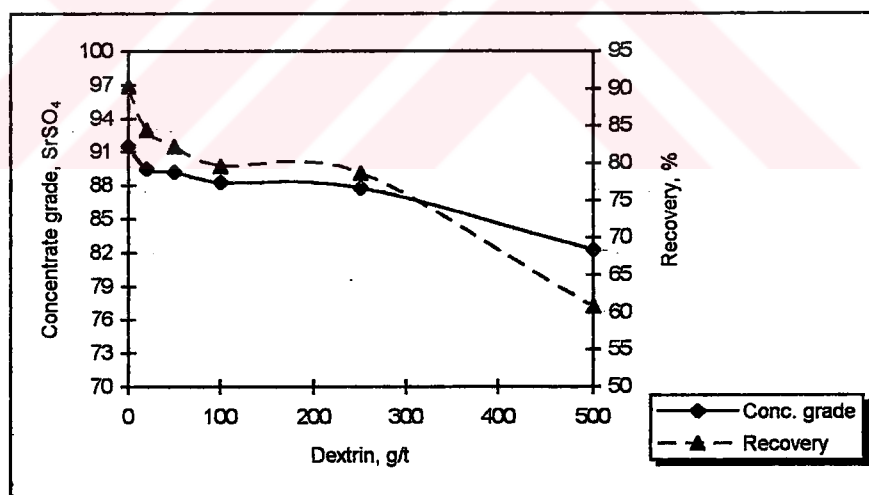


Figure-4.11:Effect of dextrin on flotation

Table-4.12:Effect of albumin on flotation

Albumin g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	81.95	90.11	86.36
50	82.13	89.59	86.05
100	81.18	89.72	85.18
250	75.65	88.35	78.16
500	55.48	85.54	55.50
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Albumin Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

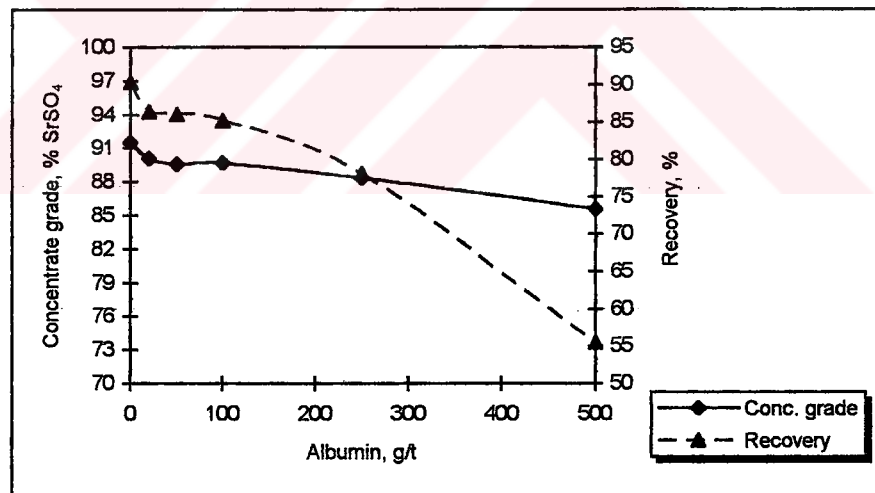


Figure-4.12:Effect of albumin on flotation

When gelatine was used, percent of concentrate almost did not changed but concentrate grade and recovery slightly decreased indicated

that gelatine depressed celestite more than calcite. Experimental results are given on Table-4.13 and plotted in Figure-4.13.

Table-4.13:Effect of gelatine on flotation

Gelatine g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	84.37	90.12	88.92
50	83.95	89.59	87.96
100	84.42	89.23	88.09
250	85.13	89.07	88.67
500	85.24	88.92	88.64
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Gelatine Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

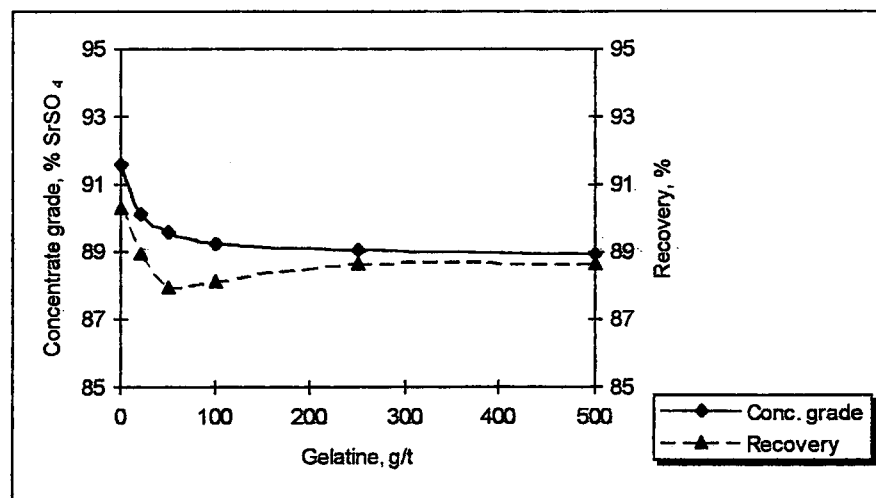


Figure-4.13:Effect of gelatine on flotation

When Na_2SiO_3 , gum arabic and quebracho were used, percent of concentrate and flotation recovery decreased, instead concentrate grade increased. Results are given on Table-4.14-4.16 and in Figure-4.14-4.16.

Table-4.14: Effect of Na_2SiO_3 on flotation

Na_2SiO_3 g/t	Concentrate Weight %	Conc. Grade % SrSO_4	Recovery %
0	84.33	91.56	90.30
500	83.96	92.06	90.39
1000	84.12	91.64	90.15
1500	84.02	92.94	91.32
2000	78.23	93.04	85.12
2500	69.66	95.52	77.81
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Na_2SiO_3 Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min.		

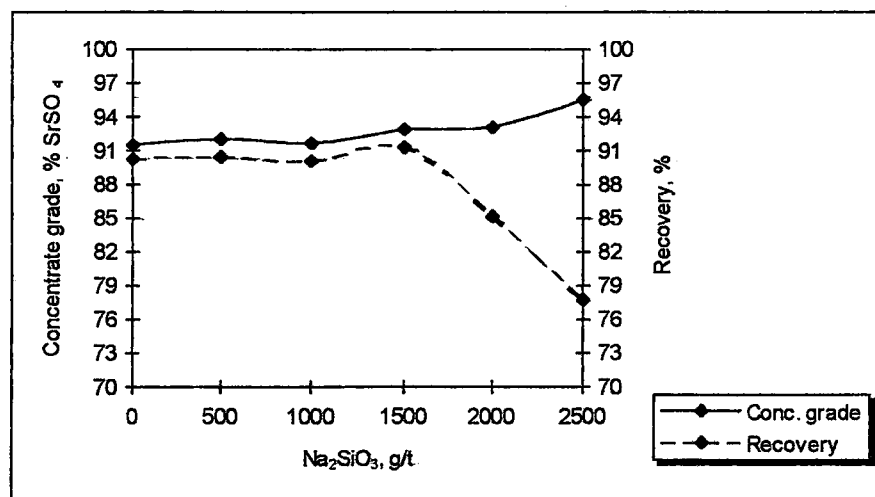


Figure-4.14: Effect of Na_2SiO_3 on flotation

Table-4.15:Effect of gum arabic on flotation

Gum Arabic g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	82.90	91.61	88.11
50	81.36	91.80	87.34
100	81.55	92.07	87.81
250	82.71	92.54	89.51
500	53.84	93.10	58.62
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min. Depressant: Gum arabic Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

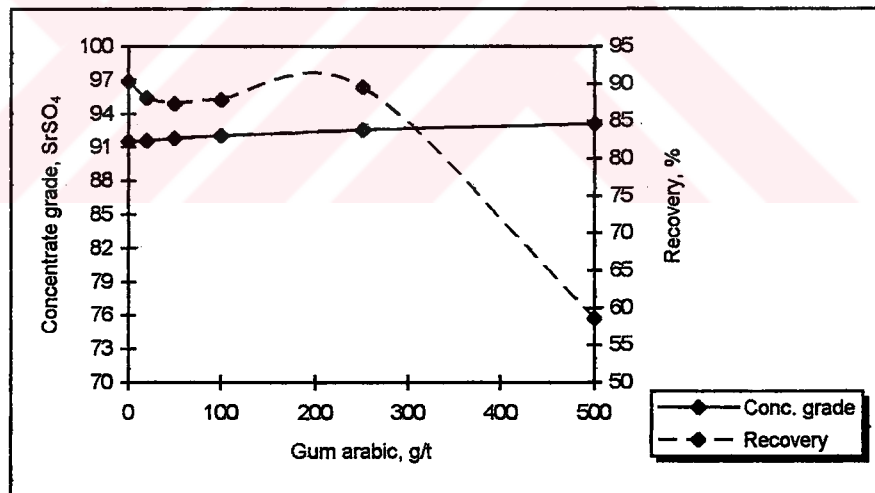


Figure-4.15:Effect of gum arabic on flotation

Table-4.16: Effect of quebracho on flotation

Quebracho g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	84.33	91.56	90.30
20	83.60	91.56	89.51
50	82.22	92.01	88.47
100	83.05	92.44	89.97
250	63.96	94.78	70.90
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: Quebracho Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65 Flotation time: 5 min		

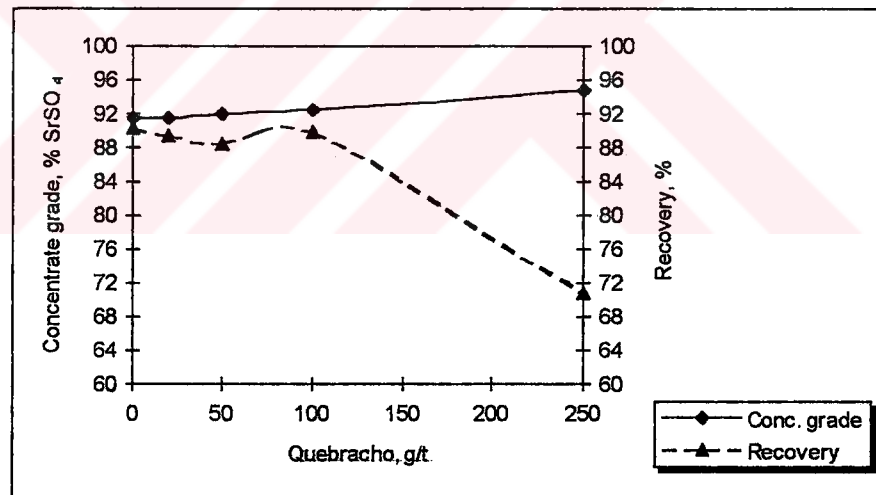


Figure-4.16: Effect of quebracho on flotation

Saleable concentrate was obtained when Na₂SiO₃ (500 g/t, 1,500 g/t, 2,000 g/t, 2500 g/t), gum arabic (100 g/t, 250 g/t, 500 g/t) and quebracho (50 g/t, 100 g/t, 250g/t) were used as depressants. When selection criteria given in section 3.6 is applied, it can be seen that

maximum income can be taken with 1.500 g/t Na_2SiO_3 . Then in the following experiments 1500 g/t Na_2SiO_3 was used as depressants.

4.5 EFFECTS OF TYPE AND AMOUNT OF FROTHERS

A series of experiments were carried out to determine the effect of amount and types (Flotal-B, Aerofroth-65, Aerofroth-73, Aerofroth-76, Aerofroth-88) of frothers on concentrate grade and recovery.

Experimental results given on Table-4.17-4.21 and plotted in Figure-4.17-4.21 show that types and amount of frother have considerable effect on weight percentage of concentrate, concentrate grade and recovery. Increase in the frother consumption results in an increase in weight percentage of concentrate and recovery up to a level of almost 90 g/ton, and then a decrease in concentrate grade and recovery could be seen probably due to micelle formation.

Table-4.17:Effect of Flotal-B on flotation

Flotal-B g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	25.69	91.62	27.53
30	63.37	92.41	68.48
60	79.63	92.03	85.70
90	84.99	91.88	91.32
120	84.78	88.30	87.54
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: 1,500 g/t Na ₂ SiO ₃ Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: Flotal-B Flotation time: 5 min		

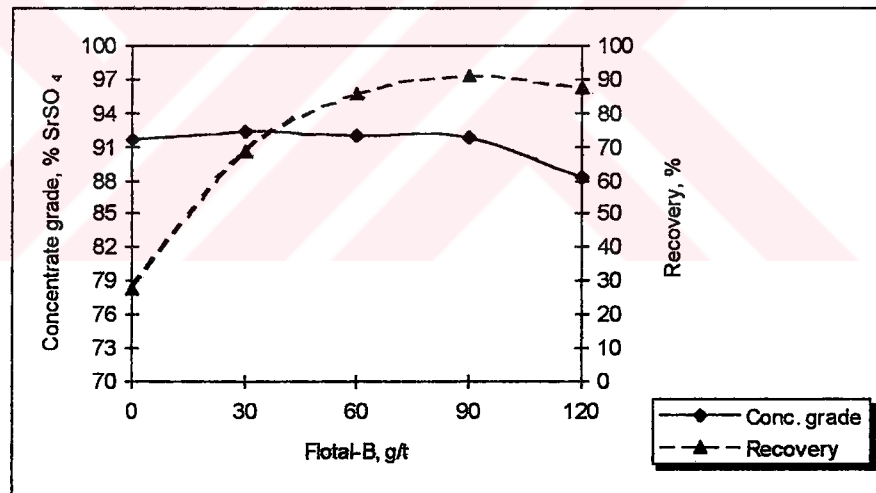


Figure-4.17:Effect of Flotal-B on flotation

Table-4.18:Effect of Aerofroth-65 on flotation

Aerofroth-65 g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	25.69	91.62	27.53
30	80.44	92.56	87.08
60	84.02	92.94	91.32
90	84.80	90.98	90.22
120	84.62	86.05	85.15
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: 1,500 g/t Na ₂ SiO ₃ Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: Aerofroth-65 Flotation time: 5 min		

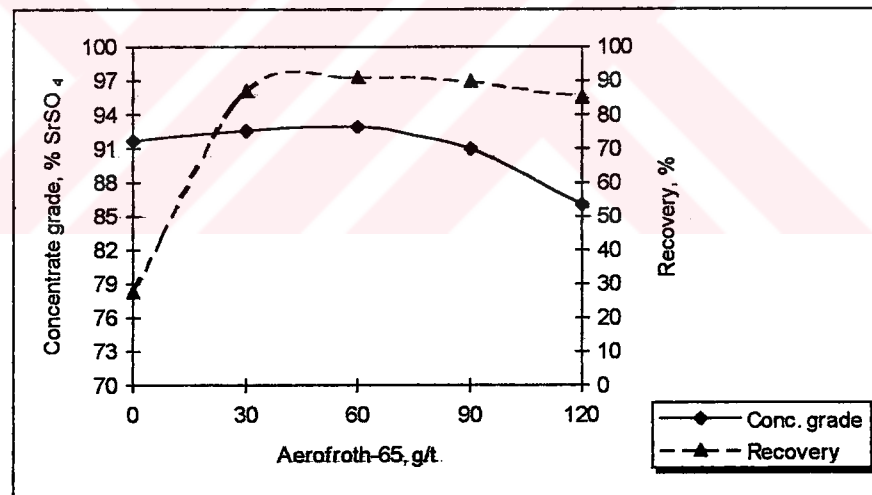


Figure-4.18:Effect of Aerofroth-65 on flotation

Table-4.19: Effect of Aerofroth-73 on flotation

Aerofroth-73 g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	25.69	91.62	27.53
30	76.37	91.28	81.52
60	83.69	91.33	89.39
90	85.54	92.04	92.07
120	86.42	90.26	91.22
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: 1,500 g/t Na ₂ SiO ₃ Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: Aerofroth-73 Flotation time: 5 min		

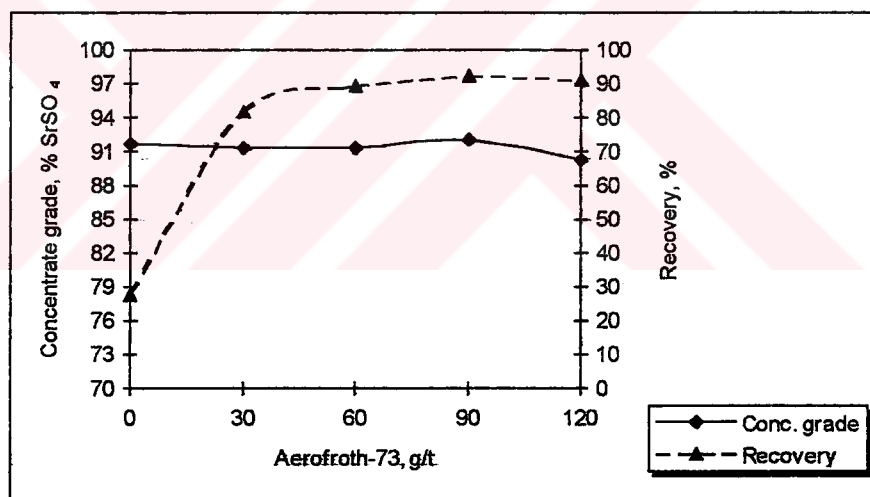


Figure-4.19: Effect of Aerofroth-73 on flotation

Table-4.20: Effect of Aerofroth-76 on flotation

Aerofroth-76 g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	25.69	91.62	27.53
30	80.39	91.70	86.21
60	85.25	91.44	91.16
90	85.51	91.97	91.97
120	87.81	90.55	92.99
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: 1,500 g/t Na ₂ SiO ₃ Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: Aerofroth-76 Flotation time: 5 min		

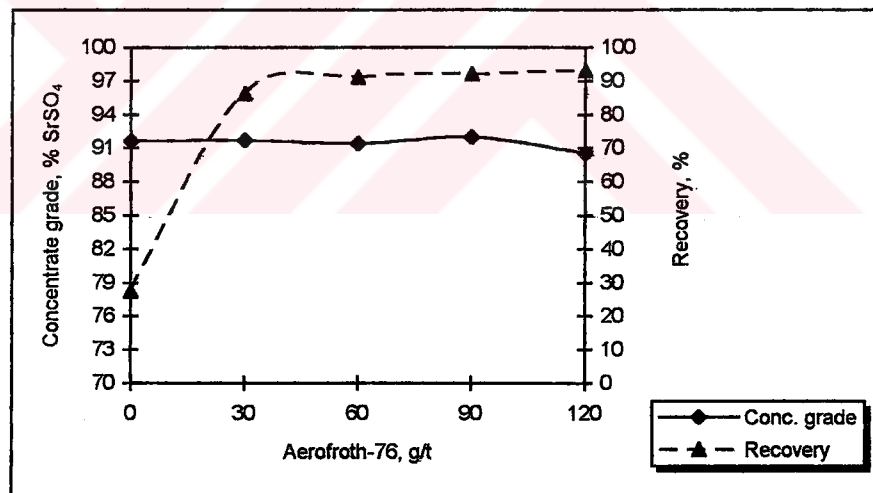


Figure-4.20: Effect of Aerofroth-76 on flotation

Table-4.21:Effect of Aerofroth-88 on flotation

Aerofroth-88 g/t	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
0	25.69	91.62	27.53
30	72.75	90.98	77.40
60	83.24	90.42	88.02
90	83.83	90.84	89.07
120	83.09	90.56	88.00
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: 1,500 g/t Na ₂ SiO ₃ Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: Aerofroth-88 Flotation time: 5 min		

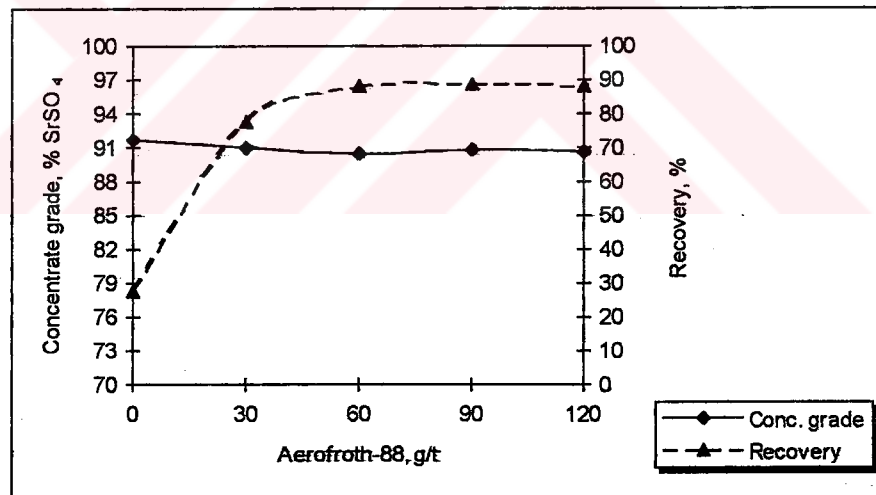


Figure-4.21:Effect of Aerofroth-88 on flotation

Saleable concentrate was obtained when Flotal-B (30g/t, 60g/t), A-65 (30 g/t, 60g/t) and A-73 (90 g/t) were used as frothers. When selection criteria given in section 3.6 is applied, it can be seen that maximum income

can be taken with 60 g/t A-65. Therefore in the following experiments 60 g/t A-65 was used as frother.

4.6 EFFECT OF FLOTATION TIME

In order to determine optimum flotation time, a set of experiments was carried out. The froth was collected in the time increments. The cumulative results are given on Table-4.22 and plotted in Figure-4.22.

Table-4.22: Effect of flotation time on flotation

Flotation Time min.	Concentrate Weight %	Conc. Grade % SrSO ₄	Recovery %
30 sec	50.60	94.98	56.20
1	65.73	94.17	72.38
2	80.35	93.66	88.01
3	83.47	93.14	90.92
5	84.02	92.94	91.32
7	85.46	92.01	91.95
Experimental Conditions	Grinding time: 1 min pH regulator: NaOH (pH=10) Conditioning time for pH regulator: 5 min Depressant: 1,500 g/t Na ₂ SiO ₃ Conditioning time for depressant: 5 min Collector: 187.5 g/t Na-oleate + 187.5 g/t A-845 Conditioning time for collector: 3 min Frother: 60 g/t Aerofroth-65		

As the flotation time increased, concentrate percentage and recovery increased and concentrate grade decreased. When selection criteria given in section 3.6 is applied to these results, it is shown that

maximum income can be taken with 3 min flotation time. Therefore 3 min flotation time was chosen as optimum rougher flotation time.

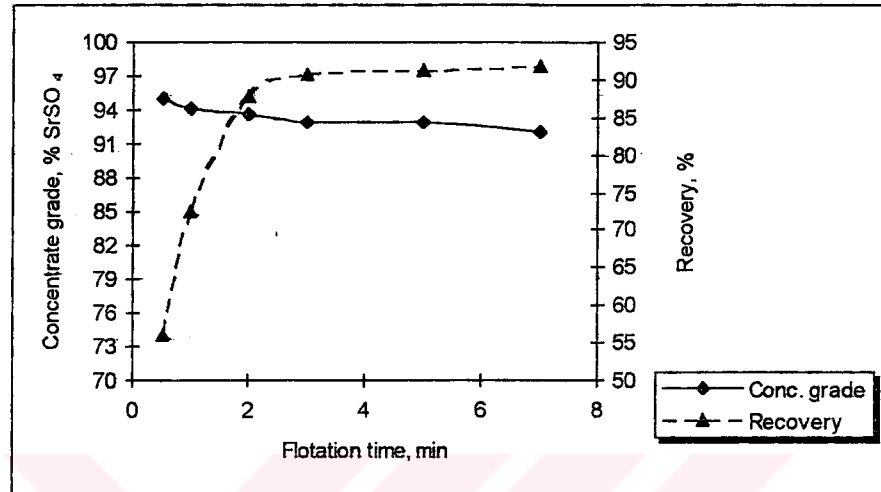


Figure-4.22: Effect of flotation time on flotation

4.7 CLEANING OF THE ROUGHER CONCENTRATE

To increase the economic value of concentrate, 3 stage cleaning was investigated. Optimum conditions found throughout this research were used in rougher stage. Solid weight percentages in first, second and third cleaning stages were 20.15 %, 19.37 % and 18.95 %, respectively. Collector, frother and depressant were not used in cleaning stages. Only pH was adjusted. Flotation time was applied in rougher stage and cleaner stages as 5 minutes to take SrSO₄ in concentrate as high as possible. The flowsheet which was followed during cleaning is shown in Figure-4.23. Results of cleaning experiments were given on Table-4.23.

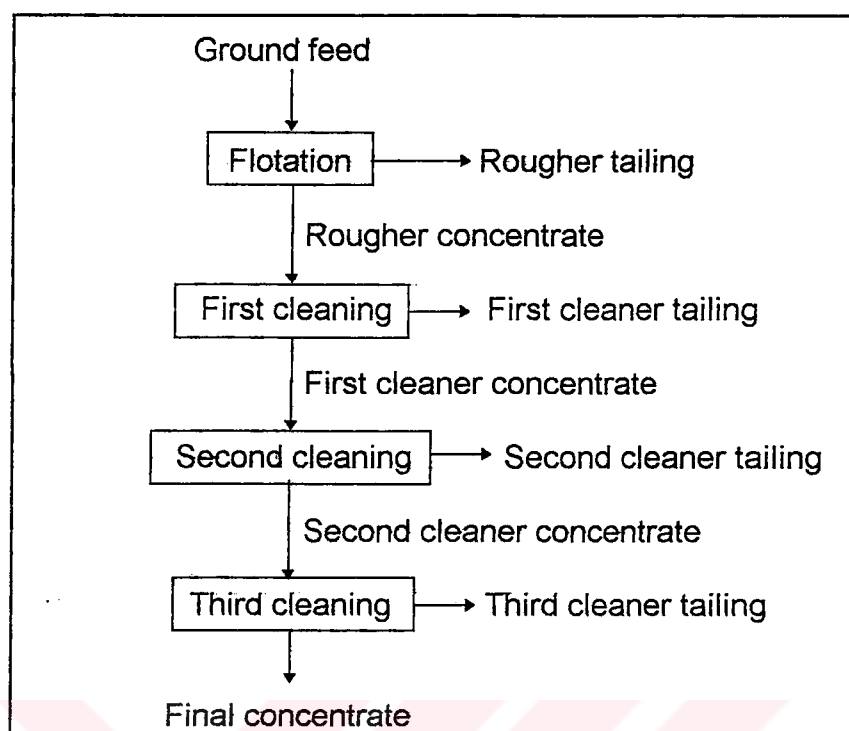


Figure-4.23: Flotation flowsheet with 3 cleaning stages

Table-4.23: Results of 3-stage cleaning experiments.

Product	Weight %	Grade % SrSO ₄	Recovery %
Final Concentrate	76.17	95.34	84.93
Rougher Tailing	15.98	46.44	8.68
First Cleaner Tailing	4.03	63.98	3.01
Second Cleaner Tailing	2.12	72.16	1.79
Third Cleaner Tailing	1.68	80.92	1.59
Feed	100.00	85.51	100.00

CHAPTER 5

CONCLUSIONS

In the light of the flotation tests, performed on a celestite-calcite ore from Nasır Village deposit of Barit Maden Türk A.Ş., the following conclusions can be drawn:

1. The highest SrSO_4 grade and recovery is obtained at pH 10 with NaOH as pH regulator.

2. The optimum flotation size is 79.01 % passing 125 μm size screen which is achieved by grinding the ore for 1 minute.

3. The optimum SrSO_4 grade and recovery is obtained with the combination of Na-oleate and Aerofloat-845 in equal percentage. The optimum dosages of collectors are 187.5 g/t Na-oleate and 187.5 g/t Aerofloat-845.

4. Na_2SiO_3 selectively depresses calcite in the celestite-calcite ore. Optimum dosage of Na_2SiO_3 is 1500 g/t.

5. Frother Aerofroth-65 gives the best results with 60 g/t frother dosage.

6. The ore can be concentrated in rougher flotation as a saleable product by using 187.5 g/t Na-oleate and 187.5 g/t Aerolfoat-845 as collectors, 1,500 g/t Na_2SiO_3 as depressant and 60 g/t Aerofroth-65 as frother at pH 10 by NaOH, where 93.14 % SrSO_4 was obtained with 90.92 % recovery.

7. Optimum rougher flotation time is 3 minutes.

8. Celestite concentrate of 95.34 % SrSO_4 with a recovery of 84.93 % is possible with 3-stage cleaning by using 187.5 g/t Na-oleate and 187.5 g/t Aerolfoat-845 as collectors, 1,500 g/t Na_2SiO_3 as depressant and 60 g/t Aerofroth-65 as frother at pH 10 by NaOH.

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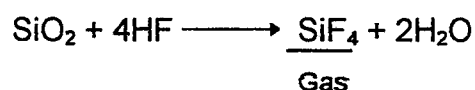
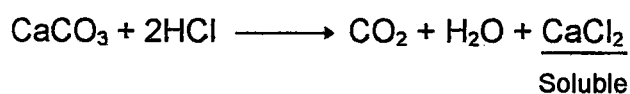


APPENDIX

METHODS FOR GRADE ANALYSIS

To determine grade of sample three types of method was applied. First one of them is fluxation method. In fluxation method, at first 0.5 g sample and 2.5 g di-Lithium Tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) put in a carbon crucible, and left in a furnace at 1100 °C for 30 minutes. Then it was allowed to cool slowly. And then it was dissolved in 20 % HCl solution. Solution was evaluated by atomic absorption spectrophotometer and thus chemical composition of sample was determined.

The second is chemical method. In this method, to find CaCO_3 and SiO_2 contents of sample, it was first reacted by HCl in a ceramic crucible. At the end of the reaction with HCl, sample was washed in a gooch crucible to remove CaCl_2 , and dried in a furnace at 105 °C. Then sample was reacted with HF to remove SiO_2 as SiF_4 . Reaction formulas are as follows.



In gravity method, a 250 ml Chatelier glass, kerosene and a water tank was used. Pipe part of Chatelier glass is measured from 0 ml to 24 ml. In the tank, water is heated to above 10-12 °C of room temperature by a heater, and stirred up by a mixer. Chatelier glass is filled with kerosene to a level of below 0 ml level (Figure-A.1) and fitted by a holder in the tank. It is left in the tank until the expansion of kerosene stop and then level is recorded. Then moistureless sample is filled to Chatelier glass to about 19-20 cc level. It is waited until level of sample-kerosene pulp is not change, and this level is also recorded. Grade of sample is calculated by using following formulas.

$$d_{ave} = \frac{m}{V_2 - V_1}$$

$$t_v = \frac{d_{ave} - d_g}{d_v - d_g} \times 100$$

$$t_m = \frac{t_v \times d_g}{d_{ave}}$$

- d_{ave} : Average density, g/cm³
- d_g : Density of gangue mineral, g/cm³
- d_v : Density of valuable mineral, g/cm³
- m : Mass of sample, g
- V_1 : Kerosene level in Chatelier glass, ml
- V_2 : Pulp level in Chatelier glass, ml
- t_v : Grade of sample in volume, %
- t_m : Grade of sample in mass, %

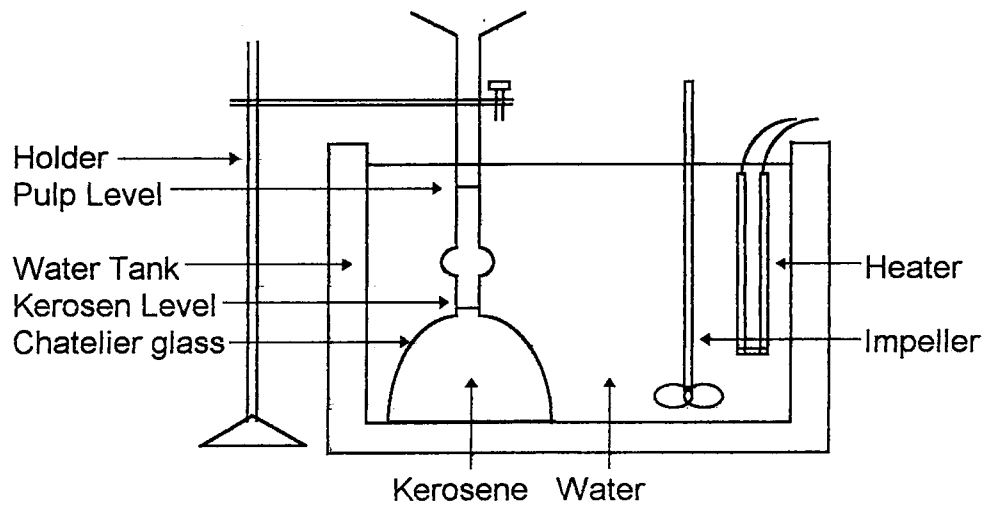


Figure-A.1:Experimental apparatus of gravity method

As shown from these formulas, sample must contain only two material of different specific gravities. In this sample, valuable mineral is celestite with 3.98 g/cm^3 , and gangue minerals are calcite and quartz with 2.70 g/cm^3 and 2.65 g/cm^3 , respectively. Major gangue mineral is calcite, then specific gravity of gangue minerals were taken as 2.70 g/cm^3 .