

Evaluation of Antimony Ores Flotation Data by Topographical Isohips Method

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In this study, batch flotation experiments were performed on representative antimony ore samples from Emirli mines of Odemis region in Turkey. Stibnite mineral was shown to be flatable using different type and amount of reagents, such as collectors and activators. The tests were conducted by use of Denver laboratory type flotation machine and cells whose operating parameters were also controlled. Flotability results of stibnite ores were evaluated by taking some crucial parameters into account, *i.e.* dosages of collector and activator. Finally, topographical isohips depending on flotation data were constructed to optimize the parameters by using a special software package.

Key Words: Antimony, Ores, Flotation, Topographical isohips method.

INTRODUCTION

Antimony is found in the earthcrust at an average amount of 65 ppm. Its specific gravity is 6.69 g/cm^3 and melting point is 631°C . Antimony having silverish colour and little conductivity for heat and electricity is a brittle material and easily powdered. Cold hydrochloric acid and weak sulphuric acid do not necessarily dissolve antimony. It is easily oxidised and forms antimony trioxide (Sb_2O_3) that vaporises at temperature of $350\text{-}450^\circ\text{C}$.

By considering the use of stibnite (Sb_2S_3) for production of matches and special bullets, antimony can be classified into three main categories on the basis of its uses *e.g.*, antimony sulphide, antimony oxide and metallic antimony.

Consumption rates of antimony are 55 % as fire retardants for paint, plastics, paper and textile industries, 15 % as additive for paint pigments, glass and ceramics industries, 15 % for production of car batteries and 15% for production of special alloys¹.

Although the production of antimony is mainly realised from scraps containing antimony alloys but good quality of antimony metal is only obtained from processing and treatment of antimony ores. There are at

least 100 different minerals containing antimony in their crystal structure, but few of them can be economically exploited. Table-1 shows commercially important antimony minerals in detail².

TABLE-1
COMMERCIALY IMPORTANT ANTIMONY MINERALS AND
THEIR PROPERTIES

Mineral name	Formulas	Specific gravity (g/cm ³)	Mohs hardness	Theoretical Sb (%)
Stibnite	Sb ₂ S ₃	4.5-4.6	2.0	71.83
Valentinite	Sb ₂ O ₃	5.6-5.8	2.5	83.30
Senarmontite	Sb ₂ O ₃	5.2-5.3	2.0	83.30
Servantite	Sb ₂ O ₄	-	-	78.90
Stibiconite	Sb ₃ O ₆ (OH)	-	-	-
Kermesite	2Sb ₂ S ₃ Sb ₂ O ₃	4.6	1.0	75.10

The antimony sulphide ores should contain at least 15-20 % Sb for metallurgical treatment. If these ores contain less than 8 % Sb, they are generally floated by using anionic type collectors with addition of specific frothers, depressants and activators according to the structure at fine particle sizes followed by optical sorting and jigging at coarse particle sizes. Flotation concentrates are saleable at grades of 55-65 % Sb. Metallic antimony is produced by well-known metallurgical methods of reduction by iron, leaching followed by electrolysis and reduction with oxidation roasting¹.

Floatability of stibnite from Odemis region of Turkey is investigated with use of a number of batch flotation tests. Stibnite flotation is shown to be achieved for representative ore samples from Emirli mines in Odemis region using different type and amount of reagents, such as collectors and activators. The tests were performed in a Denver laboratory type flotation machine of which operating parameters were also controlled. The results were illustrated by drawing three dimensional graphs and topographical isohips. The graphs were constructed to evaluate the flotation data for optimization taking crucial parameters into account, *i.e.* dosages of collector, frother, activator and depressant by using a special software package.

EXPERIMENTAL

Source of the material and sample preparation: The test samples used in this study were supplied from Emirli mines in Odemis region of Turkey. About 60 kg of stibnite ore sample, sized at 300 mm, was first crushed to 60 mm with a primary jaw crusher and then quartered five times. Half of the sample was saved for records and the rest was crushed to 8 mm through a secondary jaw crusher. Crushed samples were passed through a

tertiary roll crusher in order to obtain convenient particle sizes for grinding tests, for which a laboratory ball mill was used to grind samples down to 0.106 mm No desliming procedure was used.

The results of the detailed mineralogical and chemical analyses showed that the ore samples used in this study mainly consist of stibnite as a major component and some arseno-pyrites, pyrites, realgar, orpiment and quartz as gangue minerals. The main chemical components² are as follows; 8-9% Sb, 8-9% S, 45-50% SiO₂, 5-7% Fe, 4-5% Al₂O₃, 0.8-1.0% As.

Laboratory apparatus and reagent preparation: Batch flotation tests were conducted in a Denver Sub2A type flotation machine with an impeller speed of 1200 rpm and a cell capacity of 1 L. Izmir tap water was used at ambient temperature, *i.e.* 25 ± 5°C throughout the tests. Unless otherwise required, 30 % solid/liquid ratio, the reagents at 5 % concentration and conditioning and flotation times of 15 min per stage were used.

Batch flotation tests: The main aim of this study is to investigate the practical floatability of stibnite ores from the Emirli mines of Odemis in Turkey using conventional reagents, particularly collectors and activators, to determine the optimal dosages for those reagents.

A number of stibnite flotation reagents were obtained from several mining chemical companies, such as Hoechst, Dow Chemicals and Cytec (formerly known as Cyanamid). Conventional stibnite flotation reagents are mainly anionic type dithiophosphates. Hostaflo-LIP, which has a dithio phosphate (DTP) structure, is known to be an efficient collector for stibnite in the literature³⁻⁵.

Collector and activator dosages were taken as main variables for batch flotation tests, since stibnite is known to be floatable under certain conditions, such as pH value of 6.5 with some alkali addition, lead nitrate as an activator and anionic type collector^{6,7}.

Batch flotation tests were carried out according to the following flotation flowsheet (Fig. 1). Since presently applied processing method is mainly dependent upon the selective floatability of stibnite from gangue minerals, a rougher flotation stage was followed by a single stage scavenging flotation for rougher tailings and two separate stages of cleaning flotation for rougher concentrates in this study.

The reagents used in this study can be classified according to their order of addition. First a certain amount of depressant for quartz, *i.e.* 100 g/t Na₂SiO₃ for mixing with the pulp during first 3 min, then varied amount of activator and collector for mixing with the pulp for another 3 min per reagent were added into flotation cell. Finally, a frother, namely Dowfroth-250 at an amount of 70 g/t and *ca.* 4000 g/t of pH regulator, *i.e.* Na₂CO₃ for 3 min per reagent was mixed with the pulp. The pH value of the pulp was continuously controlled during the tests.

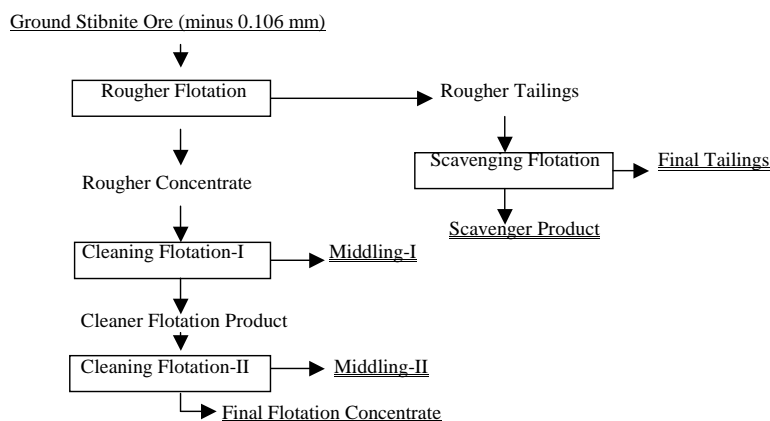


Fig. 1. Laboratory flotation flow-sheet of Emirli stibnite ore samples

Determination of optimal dosages for collectors: Hostaflo-LIP collector has an anionic type structure, specifically a sodium diisopropyl DTP and may be classified on the basis of dialkyl dithiophosphates and its salts.

$R_2P_2O_5 \cdot M^+$: Dialkyl dithiophosphates and its salts (namely dithiophosphates) where R represents an alkyl group which is C_nH_{2n+1} ($n: 1,2,3,\dots,n$) and contains saturated Cl_2 and M^+ represents a cation, *i.e.* Na^+ , K^+ , *etc.* These are used in 5-20 % solutions for flotation of metal sulphides such as antimony, copper, lead and zinc ores. Because bonding is usually electrostatic, pH of the pulp is critical especially for the lower molecular weight collectors where chain-chain interaction is less important.

Hostaflo-LIP, commercially produced by Hoechst is known to be very efficient as a collector for stibnite, as well as some other metallic sulphides, *i.e.* complex type Cu, Pb, Zn and Ag ores according to the literature⁸⁻¹⁴.

A set of batch flotation tests for determination of optimal dosage for Hostaflo-LIP was performed on the stibnite ore samples at level of 50, 75, 100, 125 and 150 g/t. In addition, 100 g/t of Dowfroth-250 as a frother and 800 g/t lead nitrate as an activator were employed. The other variables were kept constant, particle size of minus 0.106 mm, solid liquid ratio of 30 %, conditioning and flotation times of 15 min per stage. Another set of batch flotation tests for determination of optimal dosage for lead nitrate as an activator was performed on the stibnite ore samples at level of 400, 600, 800, 1.000 and 1.200 g/t. In addition, a 100 g/t of Dowfroth-250 and a 100 g/t Hostaflo-LIP as a collector were employed. The other variables were kept constant again, particle size of minus 0.106 mm, solid liquid ratio of 30%, conditioning and flotation times of 15 min per stage¹⁴. Concentration tables are given in Table-2 for comparison of variable collector and activator dosages against grade and recovery values for stibnite ore sample.

TABLE-2
CONCENTRATION TABLES OF EMIRLI STIBNITE SAMPLES
ACCORDING TO VARIABLE PARAMETERS

Conditions	Products	Weight (%)	Grade (%) Sb	Sb (%) Recovery
Activator* 400 g/t lead nitrate	Final Conc.	6.11	56.80	47.37
	Mid-II	2.50	25.20	8.60
	Mid-I	7.25	16.40	16.23
	Scavenger	10.30	12.60	17.72
	Final Tailing	73.84	1.00	10.08
	Total	100.00	7.32	100.00
Activator* 600 g/t lead nitrate	Final Conc.	8.66	60.80	67.06
	Mid-II	2.91	28.30	10.49
	Mid-I	6.93	12.10	10.68
	Scavenger	5.44	10.00	6.93
	Final Tailing	76.06	0.50	4.84
	Total	100.00	7.85	100.00
Activator* 800 g/t lead nitrate	Final Conc.	8.39	64.70	67.72
	Mid-II	2.11	20.30	5.34
	Mid-I	8.46	14.20	14.99
	Scavenger	5.64	10.30	7.25
	Final Tailing	79.78	0.80	8.48
	Total	100.00	8.02	100.00
Activator* 1000 g/t lead nitrate	Final Conc.	7.71	60.10	64.30
	Mid-II	2.12	30.10	8.86
	Mid-I	6.63	18.80	17.30
	Scavenger	1.90	10.40	2.74
	Final Tailing	81.64	0.60	6.80
	Total	100.00	7.21	100.00
Activator* 1200 g/t lead nitrate	Final Conc.	8.00	58.60	59.46
	Mid-II	3.19	23.80	9.63
	Mid-I	9.21	18.00	21.03
	Scavenger	2.55	9.40	3.04
	Final Tailing	77.05	0.70	6.84
	Total	100.00	7.88	100.00
Collector** 50 g/t Hostafлот LIP	Final Conc.	5.40	60.00	41.58
	Mid-II	8.00	28.80	2.95
	Mid-I	5.21	20.10	13.44
	Scavenger	3.96	18.60	9.45
	Final Tailing	84.63	3.00	32.58
	Total	100.00	7.79	100.00

Conditions	Products	Weight %	Grade % Sb	Sb % Recovery
Collector** 75 g/t Hostafлот LIP	Final Conc.	7.90	62.00	65.13
	Mid-II	2.21	20.00	5.88
	Mid-I	15.84	17.00	18.20
	Scavenger	4.27	12.95	7.35
	Final Tailing	79.78	0.80	8.48
	Total	100.00	7.52	100.00
Collector** 125 g/t Hostafлот LIP	Final Conc.	13.72	58.00	85.78
	Mid-II	1.17	15.00	1.89
	Mid-I	4.39	9.50	4.5
	Scavenger	4.54	9.30	4.55
	Final Tailing	76.18	0.40	3.28
	Total	100.00	9.28	100.00
Collector** 150 g/t Hostafлот LIP	Final Conc.	13.78	56.00	83.58
	Mid-II	2.00	11.00	2.38
	Mid-I	6.33	9.60	6.58
	Scavenger	5.79	9.40	5.90
	Final Tailing	72.10	0.20	1.56
	Total	100.00	9.23	100.00

*Collector dosage is stable at 100 g/t and **Activator dosage is stable at 800 g/t

RESULTS AND DISCUSSION

Test results (Fig. 2) showing grade and recovery graphs against dosages of collector and activator. A new approach depending on topographical isohips evaluation techniques was used to determine optimal dosages for the reagents.

From Fig. 2, the acceptable dosage for Hostafлот-LIP was found to be around 100 g/t, while the acceptable dosage for lead nitrate was around 800 g/t. Although the concentrate grade is just above 60% Sb grade, yield and recovery data clearly show that almost all of the feed material is floated, when this amount of collector is employed at feed grade of 7-9% Sb. When the concentration results were evaluated cumulatively, the high values for yield and recovery data could be easily seen, although a sharp decrease in cumulative grade values was obtained.

Finally it can be seen that stibnite from the Emirli mines of Odemis in Turkey is floatable under certain conditions which involve acceptable values of collector and activator dosages.

This three dimensional topographical isohips technique for evaluation of flotation data is quite novel for taking the least number of experiments into account and enables researchers to finalize the optimum amount of parameters and their effects on the results.

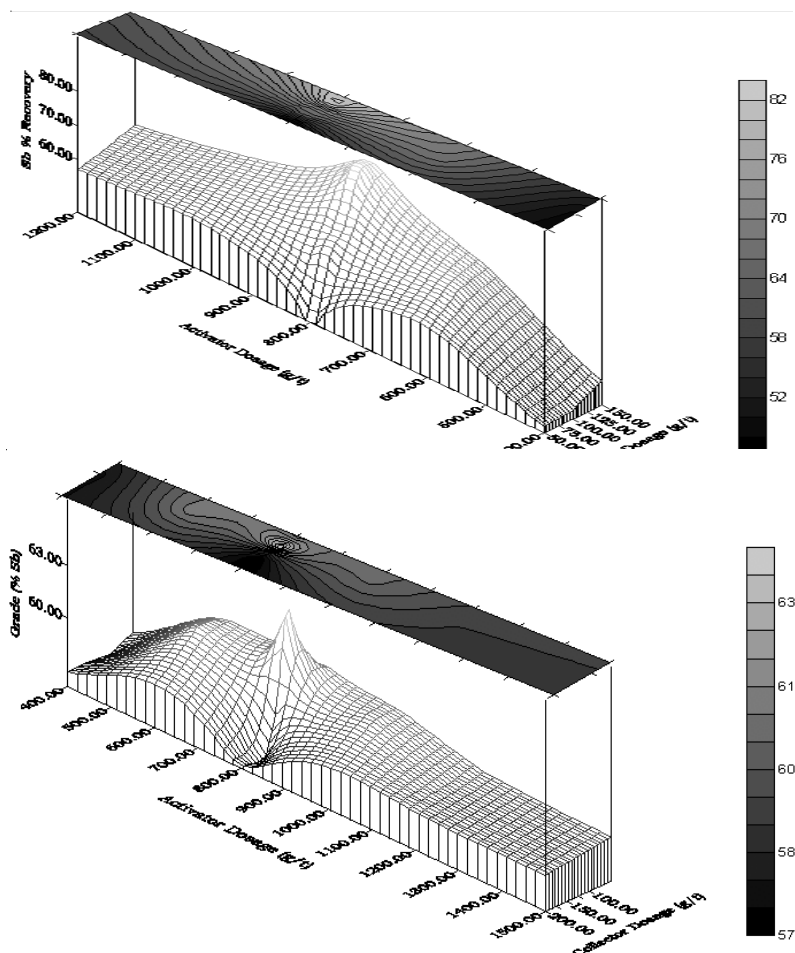


Fig. 2. Topographical isohips of flotation data of Emirli stibnite samples according to grade and recovery values against collector and activator dosages

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REFERENCES

1. Y. Cilingir, *Metallic Ores and Their Concentration Methods*, Vol. 1, Publication of DEUMMF, MM/MAD-90 EY 198, Izmir (1990) (In Turkish).
2. S.G. Ozkan and Z. Ergin, *Determination of Arsenic and Mercury Distribution and Correlation between Antimony Grade and Recovery in the Flotation Products of Etibank's Halikoy Mines*, B.Sc. Final Year Project, Dokuz Eylul University, Eng.-Arch. Fac., Mining Eng. Dept., Bornova, Izmir, Turkey (1990) (In Turkish).
3. Anonymous, *Several Flotation Reagent Leaflets from Hoechst* (1992).

4. Anonymous, Several Flotation Reagent Leaflets from Cyanamid (1992).
5. Anonymous, Cytec's Mining Chemicals Handbook, Revised Edition, USA (2002).
6. A. Akar, Beneficiation of Arseniferous Odemis-Halikoy-Emirli Stibnite Ores and Separation of Arsenic Minerals, The Proceedings of the 7th Turkish Mining Congress, The Chamber of Mining Engineers of Turkey, Ankara, (Turkish Text) pp. 239-274 (1971).
7. S. Atak, Flotation Principles and Applications, Istanbul Technical University Books, Istanbul, Turkey, edn. 1, pp. 206-207 (1982) (In Turkish).
8. R.D. Crozier, Plant Reagents, Part 1: Changing Patterns in the Supply of Flotation Reagents and Part 2: Some of the Manufacturers, Mining Magazine, 151 (Sept.) pp. 202-219 (1984).
9. R.D. Crozier, Flotation Theory, Reagents and Ore Testing, Pergamon Press, London, England, ISBN 0-08-041864-3 (1992).
10. D.W. Fuerstenau, Froth Flotation, Fiftieth Anniversary Volume, SME/AIME, New York, USA (1976).
11. A.M. Gaudin, Flotation, McGraw Hill Book Co. Inc., New York, USA, edn. 2, (1957).
12. V.A. Glembofskii, V.I. Klassen and N.I. Plaksin, Flotation, Primary Sources, New York, USA (1963).
13. J. Leja, Surface Chemistry of Froth Flotation, Plenum Press, New York, USA (1982).
14. R.D. MacDonald, R.J. Brison and W.C. Hellyer, in ed.: N.L. Weiss, Flotation test procedures and sampling and testing, SME Mineral Processing Handbook, New York, USA, ISBN 0-89520-433-6, pp. 30.96-30.104 (1985).
15. S.G. Ozkan, Evaluation of Antimony Ores Flotation Data by Using Three Dimensional Topographical Isohips Method, Proceedings of the International Conference on Mineral Process Modelling, Simulation and Control, Laurentian University, Sudbury, Canada, pp. 249-257 (2006).

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