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EXTRACTION OF SULPHUR FROM THE BATAL DEPOSIT OF KOH-I-SULTAN (BALOCHISTAN)

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Flotation concentrates of Batal sulphur assaying over 75% sulphur was purified by metling in a stirred autoclave. Variables controlling the extraction showed that autoclaving minus 85 mesh flotation concentrate in a pulp (15 % solids) at 125° for 60 mins yielded a product assaying 99.8% sulphur at about 92.5% recovery. The residue contained about 19% sulphur which possibly could be recovered by recycling it in a flotation circuit.

Key words: Sulphur extraction, Batal deposit.

Introduction

The processing of volcanic sulphur ores to recover high grade elemental sulphur suitable for industrial use usually involes a combination of physical separation of sulphur from gangue minerals, by employing mineral dressing operations and melt-purification.

Various methods of liquefection were tried previously, on Koh-i-Sultan sulphur ore, by the application of external heat. It was found that on raising the temperature beyond the melting points of sulphur a sticky paste resulted. Only a portion of sulphur could be recovered at higher temperatures. Another experiment by Sulphur and Chemical Ltd. used autoclave heated with steam to melt out sulphur. After repeating the process, commercial grade of sulphur was obtained. The process, however, met with only limited success [1].

In recent years, efforts have been made to extract sulphur from Koh-i-sultan ore at the Balochistan Sulphur Refinery. The run of mine sulphur ore was contacted with steam in batch type autoclaves. The molten sulphur was recovered from the residue, which still contained over 17% sulphur. The refinery was shut down in 1987 becuase of its inefficient operation and uneconomically small size of the plant [2].

The processing of Koh-i-Sultan sulphur ore has been modified such that an average grade of the ore was crushed, ground and concentrated by flotation [3]. In the present work the cleaner concentrate was further processed by autoclaving in order to extract high grade sulphur form the flotation concentrate.

Alternative procedures to melt-purification of sulphur in an autoclave include sublimation, melt-filteration, and solvent extraction.

Sulphur is purified on small scale by sublimation [4] for use in agriculture and pharmaceutical products. The sublimed sulphur, also known as 'flower of sulphur', is a low density material. This process is not used for large scale continuous operations becuase of cumbersome handling problems.

Melt filteration process is particularly suited to high grade ore concentrate containing over 85% sulphur [5]. The molten sulphur is pumped through filters. The filter cakes are collected in a batch type process. This process may be useful on small scale operations, such as extraction of sulphur from the flotation concentrate from Batal ore.

Sulphur may also be extracted from its ores by solvent extraction process, Sulphur is dissovled in a solvent such as carbon disulphide or carbon tetrachloride [6]. The solvent is recovered by distillation and sulphur is retained as residue. Commercial use of solvent extraction processes for sulphur recovery from ores have not proved successful.

Materials and Methods

The melt purification study feature melting the sulphur out of flotation concentrate assaying about 65 - 80% sulphur in an autoclave.

The autoclave was a 2 litre capacity, electrically heated and mechanically stirred unit fitted with variable speed motor and panel to control temperature, time and stirrer speed (Labsco:Model 10285).

The flotation concentrate alongwith sufficient volume of water was added to make pulp containing 5 - 25% solids. The slurry was heated to above the melting point of sulphur (115 - 130°) with continuous stirring and maintained at temperature for 20-30 mins to ensure complete melting. The molten sulphur was formed into globules by stirring with an improvised impeller. At the end of each test, steam in the autoclave was released, the product was allowed to cool and was dumped on a 20 mesh (B.S.S) screen. Sulphur globules retained on the screen were washed, dried and weighed.

Based on preliminary bench autoclaving trials, it was noted that the variables which affected the grade and recovery

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of sulphur in the melt purification operation were particle size of the flotation concentrate, grade of the concentrate, temperature, percent solids in the pulp, and autoclaving time.

For the effect of particle size determination, flotation concentrate of 44,60,80 and 100 mesh (80% passing) was made. Other experimental conditions fixed were pulp density 20% solids, 120° temperature in an autoclave for 2 hrs with continuous stirring.

Effect of temperature variation on the grade and recovery was studied between 115° and 135°. The flotation concentrate grade used assayed 75.5% sulphur.

The residence time in the autoclave was varied from 30 to 180 mins. A pulp density of 15% solids was used while other conditions were maintained as in the previous series of tests.

Analysis of sulphur in the product and the residue was done using carbon disulphide as solvent, filtering through a medium porosity sintered glass crucible, washing and drying until constant weight [6].

Results and Discussion

Recovery of sulphur from the flotation concentrate in an autoclave, was found to be influenced by the operating variable investigated.

It was noted that sulphur recovery increased with increasing mesh number (reduction of particle size). The grade of the product sulphur, however, was found to be maximum for 65- 85 mesh size feed (Table 1). There was apparently no advantage in over-grinding the concentrate beyond 85 mesh.

The feed grade also had positive effect on the grade and recovery of product sulphur (Table 2). A feed above about 75% sulphur yielded a commercial grade, 99.8% sulphur, at 95% recovery. Beyond 75% sulphur in the feed, however, the improvement in the grade and recovery was not found to be substantial.

Autoclaving temperature had a significant effect on the extraction of sulphur (Table 3). At 115° which is close to the melting point of sulphur, the grade of recovered sulphur was 99.7%, at a recovery of 91%. The increased with increase of temperatue upto 130°. Beyond this temperature, however, it was noted that the grade decreased slightly, but there was a substantial decrease in the recovery. This low recovery at higher temperature may be due to the fine distribution of sulphur spherules which passed through 20 mesh screen and reported in the residue. A microscopic examination of the residue showed the presence of fine sulphur globules. It was concluded that a temperature of 125° yielded reasonably good grade and recovery of sulphur.

In evaluating the effect of percent solids it was observed that at low pulp densities, the grade of the product sulphur was high, although the recoveries were low. At higher pulp densities, on the contrary, the grade decreased gradually, but the recoveries were improved (Table 4). In the extraction of sulphur it is the product grade which is more important from

TABLE.1 EFFECT OF PARTICLES SIZE ON SULPHUR RECOVERY AND PRODUCT GRADE.

Size (BSS)	Sulphur product			Residue		
80%	Wt.	Grade	Recovery	Wt.	Grade	Recovery
passing	%	%S	%S	%	%S	%S
44	71.8	98.2	93.8	28.2	16.5	6.2
60	72.0	99.7	95.6	28.0	11.7	4.4
85	72.0	99.7	95.6	28.0	11.1	4.1
100	72.5	99.6	96.2	27.5	10.4	3.8

TABLE 2. EFFECT OF FEED GRADE TO AUTOCLAVE ON SULPHUR RECOVERY AND PRODUCT GRADE.

Food	Sulphur product			Residue		
grade	Wt.	Grade	Recovery	Wt.	Grade	Recovery
%S	%	%S	%S	%	%S	%S
65.2	60.1	99.5	91.7	39.9	13.5	8.3
71.4	66.9	99.6	93.3	33.1	14.4	6.7
75.5	72.2	99.8	95.2	27.8	13.0	4.8
80.1	76.8	99.6	95.8	23.2	14.4	4.2

TABLE 3. EFFECT OF TEMPERATURE ON SULPHUR RECOVERY AND PRODUCT GRADE.

Tempera-	Sulphur product			Residue		
ture	Wt.	Grade	Recovery	Wt.	Grade	Recovery
°C	%	%S	%S	%	%S	%S
115	70.8	97.7	91.5	29.2	21.8	8.5
120	72.3	99.6	95.4	27.7	13.0	4.6
125	72.0	99.8	95.2	28.0	12.9	4.8
130	71.5	99.8	94.3	28.5	15.0	5.7
135	71.0	99.8	93.6	29.0	16.6	6.4

TABLE 4. EFFECT OF PERCENT SOLIDS ON SULPHUR RECOVERY AND PRODUCT GRADE.

Percent	Autoclave	Sulphur	Wt	Grade	Recovery	Wt.	Grade	Recovery
solids	feed wt (g)	in feed (g)	%	%S	%S	.%	%S	%S
5	75	56.6	68.7	99.8	90.7	31.3	22.36	9.3
10	150	113.2	71.1	99.8	93.9	28.9	15.94	6.1
15	225	169.8	72.2	99.7	95.4	27.8	12.5	4.6
20	300	226.4	72.7	99.5	95.8	27.3	11.6	4.2
25	375	283.0	73.0	99.4	96.1	27.0	10.9	3.9

TABLE 5.EFFECT OF RESIDUE TIME IN AUTOCLAVE ON SULPHUR RECOVERY AND GRADE.

	Sulphur product			Residue		
Time	Wt.	Grade	Recovery	Wt.	Grade	Recovery
(mins)	%	%S	%S	%	%S	%S
30	70.2	96.4	89.6	29.8	26.3	10.4
60	70.0	99.8	92.5	30.0	18.9	7.5
120	72.3	99.7	95.4	27.7	12.5	4.6
180	72.9	99.6	96.1	27.1	10.8	3.9

the commercial point of view. The residue may be recirculated in flotation circuit to recover maximum amount of sulphur. It was condsidered worthwhile to maintain pulp density between 10 and 15% solids to obtain a grade of 99.8% and recovery of over 94% sulphur.

The test results of the effect of autoclaving time on grade and recovery showed that at 30 mins 89.6% of the contained sulphur was recovered having a grade 96.4% sulphur.

The recovery showed a steady increase with the increase in time (Table 5), but there was no significant improvement in the grade after 60 mins autoclaving time. In fact the grade was found to drop slightly with the increase in time beyond 60 mins. The reason might be that during longer periods of stirring the molten sulphur was distributed in to very small globules which passed through 20 mesh screen. The optimum time considered for autoclaving was 60 mins.

Conclusion

Sulphur may be efficiently extracted from the flotation concentrate from Koh-i-Sultan sulphur ore, using a melt purification method in an autoclave. A grade of 99.8% sulphur at 96.2% recovery may be obtained using 80% passing 85 mesh concentrate assaying 75.5% sulphur, made into a pulp containing 10–15% solids in an autoclave maintained at a temperature of 125° for a period of 60 mins. The product sulphur in the shape of globules may be recovered from the pulp on screening.

References

1. M.I. Ahmad, *Volumes and Sulphur of Western Balochistan* (Records of Geol. Sur. Pakistan, 1995), Vol. IV, Part 3, pp. 27-28.
2. W.M. Goldberger, Evaluation of the Commercial Recovery of Elemental Sulphur from Koh-i-Sultan Deposits in Balochistan, Pakistan, A UNDP Project Report, Dec. (1988).
3. M.A. Qazi, Saqib Ahmad, Shafiq Anwar, Mehdi Hassan and Izhar H. Khan, Pak. j. sci. ind. res., **36**, 295 (1991).
4. J.E. Shelton, Sulphur, U.S. Bull. 671, Mineral Facts and Problems (1980).
5. G.F. Eckert, Jr., French Sulphur, Eng. Min. J. Dec. (1987), pp. 46.
6. W.N. Tuller, *The Sulphur Data Book* (MacGraw-Hill Book Co. Inc., 1954), pp.105.