Pakistan J. Sci. Ind. Res., Vol. 29, No. 1, February 1986

# UTILIZATION OF DEGARI COAL FOR THE PRODUCTION OF BARIUM CARBONATE FROM BARYTE

### Shamshad Ahmed Khan and M. Najmul Hasan

## PCSIR Laboratories, Karachi-39 (Pakistan)

## (Received September 15, 1985; revised November 3, 1985)

The optimum conditions for the conversion of baryte to barium sulphide by reduction with Degari Coal have been studied and 92.8% conversion was achieved at 800°. Barium sulphide was then converted to barium carbonate by the addition of a sodium carbonate solution. During investigations it was found that Degari Coal is as good as imported coal.

#### **INTRODUCTION**

The methods used for the production of barium carbonate are the fusion of baryte with sodium carbonate [1-4], the reaction of barium sulphate with sodium carbonate in fused sodium chloride [5], passing of carbon dioxide into an aqueous solution of barium sulphide [6], fludization in presence of coal [7], and the reduction of baryte by methane and by carbon [9-12].

Usually imported metallurgical coal is used for the reduction of baryte. The reduction takes place at a high temperature (between  $1000-1200^{\circ}$ ) due to the hardness of coal. The analyses of baryte and imported coal are given in Tables 1 and 2 respectively.

Table 1. Analysis of baryte from Bolan Mining, Khizdar, Quetta, Bauchistam

Baryte, Wt. p.c.					
Loss on ignition	1.6				
Silica and aluminia	3.1				
Fe <sub>2</sub> O <sub>3</sub>	0.8				
BaO	61.8				
SO <sub>3</sub>	32.7				
	100.0				
BaSO <sub>4</sub>	94.0				

In our method Degari coal of which an analysis is given in Table 3, has been used for the reduction of baryte which is soft and contains a high content of volatile matter which enhances [13] the conversion of barium sulphate to barium sulphide and 92.8% conversion has been achieved. As the Degari coal is soft, it uses less fuel and the reduction takes place at low temperature of 800°, making the method cheaper.

#### Table 2. Analysis of imported coal obtained from Steel Mill

Imported coal, wt. p.c.				
Moisture	1. 54			
Volatile matter	35.50			
Carbon	53.50			
Sulphur	0.46			
Ash	9.00			
	100.00			

#### Table 3. (Analysis of Degari Coal from Quetta Baluchistan)

	and the second se
Degari Coal, wt. p.c.	
Moisture	11.4
Volatile matter	37.0
Carbon	42.7
Ash	5.5
Sulphur	3.4
	100.00

#### **EXPERIMENTAL**

Materials and Method: The baryte ore was ground to 20-70 mesh. 2.0 g of baryte and 0.4 g of powdered Degari coal were mixed and taken in a porcelein boat which was heated in a silica tube by means of a temperature controlled tube furnace while passing a slow stream of natural gas at the rate of about 50 ml per min. through the silica tube in order to drive out the moisture and maintain reducing conditions during heating. The reduction was carried out at various temperatures ranging from 700 to 900°. After the reduction, the silica tube was removed and allowed to cool at room temperature. The boat along with the material was taken out, extracted with hot distilled water and filtered by suction. The filtrate containing barium sulphide was made upto 250 ml in a volumetric flask and 20 ml aliquot was run slowly into a mixture of 25 ml 0.1 N HCl and 25 ml 0.1 N iodine solution in an iodine flask, and the excess iodine was titrated with 0.1 N sodium thiosulphate solution according to the standard method [14].

#### **RESULTS AND DISCUSSIONS**

The reduction of baryte was carried out with Degari coal as well as with imported coal at different temperatures for different interval of times. The details are given below:

Baryte was reduced with Degari coal at different temperatures ranging from 700 to  $900^{\circ}$  for 30 to 150 min. The results are given in Table 4, 5 and 6 respectively, which show that conversion of barium sulphate to barium sulphide increases with the rise of temperature and is miximum at  $800^{\circ}$ .

Baryte was reduced with imported coal at 800 and 900° for 90 and 150 min. The results are given in Table 7, which show that after heating for 150 min. the maximum conversion of 80.5% barium sulphate to barium sulphide was achieved at  $900^{\circ}$ .

The comparative results for the reduction of baryte with Degari coal and imported coal are given in Table 8 which show that with Degari coal, after heating for 150 min. 92.8% conversion of barium sulphate to barium sulphide was obtained at  $800^{\circ}$  due to the softness of the coal, while with the imported coal it is only 67.8% at  $800^{\circ}$  and 80.5% at  $900^{\circ}$  because metallurgical coal is hard and complete reduction takes place at higher temperature between  $1000-1200^{\circ}$ , which makes the method expensive.

#### PILOT PLANT PRODUCTION

After fixing the parameters for the reduction of baryte

Table 4. Reduction of baryte with Degari coal at 700°

Baryte (g)	Degari coal (g)	Reductin time (min.)	Yield of BaS (g)	f % conversion BaSO <sub>4</sub>
2.0	0.4	34	0.311	22.8
2.0	0.4	60	0.467	34.3
2.0	0.4	90	0.628	46.1
2.0	0.4	120	0.788	57.8
2.0	0.4	150	0.877	64.4

Table 5. Reducation of baryte with Degari coal at 800°

Baryte (g)	Degari coal (g)	at 800 <sup>o</sup> Reducation time (min.)	Yield of BaS co (g)	% onversion BaSO <sub>4</sub>
Ta	ble 6. Red	luctio		
2.0	0.4	30	0.729	53.5
2.0	0.4	60	0.917	67.3
2.0	0.4	90	1.033	75.8
2.0	0.4	129	1.147	84.2
2.0	0.4	150	1.265	92.8

Table 6. Reduction of baryte with Degari Coal at 900°

Baryte (g)	Degari coal (g)	At 900 <sup>0</sup> Reducation time (min.)	Yield of BaS (g)	% Conversion BaSO <sub>4</sub>
2.0	0.4	30	0.868	63.7
2.0	0.4	60	1.066	78.2
2.0	0.4	90	1.161	85.2
2.0	0.4	120	1.206	88.5
2.0	0.4	150	1.233	90.5

to barium sulphide, the reduction was carried out in a rotary furnace at a temperature of 800° in the following manner:

50 kgs. ground baryte ore (containing 94%  $BaSO_4$ ) of mesh size 20-70 was mixed with 10 kg powdered Degari coal. The mixture was heated in a firebrick linked rotary furnace (2 rpm). The furance was fired with natural gas. The charge was heated with the evoluation of volatile gases and the temperature approached 800°. Reducing atmos-

Baryte ore (g)	Imported Coal (g)	Temperature °C	Reduction time (min.)	Yield of BaS BaS (g)	% conversion BaSO <sub>4</sub>
2.0	0.4	800	90	0.725	53.2
2.0	0.4	800	150	0.924	67.8
2.0	0.4	900	90	0.985	72.3
2.0	0.4	900	150	1.124	80.5

Table 7. Reduction of baryte with imported coal at 800° and 900°

Table 8. Comparative results of reduction of baryte with Degari coal and imported coal at 800 and 900°

Degari coal					Imported coal						
Baryte ore (g)	Degari coal (g)	Reduction time (min.)	Temperature <sup>O</sup> C BaS (g)	Yield of BaS (g)	% Conversion BaSO <sub>4</sub>	Baryte ore (g)	Imported coal (g)	Reduction time (min.)	Temperature °C	Yield BaS (g)	% conversion BaSO <sub>4</sub>
2.0	0.4	90	800	1.033	75.8	2.0	0.4	90	800	0.725	53.2
2.0	0.4	150	800	1.265	92.8	2.0	0.4	150	800	0.924	67.8
2.0	0.4	90	900	1.161	85.2	2.0	0.4	90	900	0.985	72.3
2.0	0.4	150	900	1.233	90.8	2.0	0.4	150	900	1.124	80.5

Table 9. Analysis of barium carbonate BaCO<sub>3</sub>, wt. p.c.

Barium carbonate	99.00
Loss on ignition	0.80
Iron	0.05
Sulphur as BaS	0.005
Sulphur as SO <sub>3</sub>	0.03
Acid (insoluble)	0.115
	100.00

phere was maintained during heating. After an hr the charge becomes slightly sticky which indicates that the reduction is practically complete. The fire was turned off and the charge was allowed to cool in the absence of air.

The black ash was taken out from the furnace and ground in a ball mill. The yield of black ash was 35.0 kg containing 75.6% soluble barium sulphide.

The black ash was dissolved in hot water with stirring and the barium sulphide solution was filtered to separate it from insoluble materials. The clear solution was then treated with 5% sodium carbonate solution and the precipitated barium carbonate was filtered leaving behind sodium sulphide and washed with water till it was free from sodium sulphide.

Barium carbonate was dried at 120° to remove the moisture and some precipitated sulphur and then pulverized to fine powder and 30 kg. barium carbonate of 99% purity was obtained. The analysis of barium carbonate is given in Table 9.

The sodium sulphide solution (by product) was then concentrated by distillation and crystallization. 30 kg. sodium sulphide  $(30\% \text{ Na}_2\text{S})$  was obtained.

#### REFERENCES

- 1. H.S. Booth and Pollard, Ind. Eng. Chem., 40, 1983 (1984).
- 2. A.C. Herbert, U.S. Pat. 2, 637, 700, May 5 (1983).
- 3. J. Marwedel and J. Boser, Ger. Pat., 493267, April 6, 1922.

- 4. U.N. Ipalieff and C. Freitag. Ind. Eng., Chem., 27, 342 (1935).
- 5. Wells, Ind. Eng., Chem., 8, 770 (1916).
- 6. J.B. Pierce, J.U.S. Pat. 1, 774, 523, Sept. 2 (1930).
- 7. Fr. Patent, 1, 467, 067 1967.
- 8. Osaunn Kamiik, et al. J. Soc. Chem. Ind. Japan, 46, 879 (1943).
- 9.Kirk Othmer, Encyclopedia of Chemical Technology,

2nd ed., Vol. 3, p. 95 (1964).

- 10. E. Reitler, Metallborse, 20, 1966 1936.
- 11. Friedrich Rusberg and Paul Schmid (Kali-Chemic) U.S. Patent, 1, 829, 108, Oct. 27 (1932).
- 12. Brit. Patent, 499, 925 (1939).
- 13. W. Hirschel Chem. Zntq. 50, 692 (1926).
- F.P. Treadwell, Analytical Chemistry, 9th ed., Vol. 11, p. 17 (1958).

7

-

2

1.00

tran meanite material. The cost ministrate descents of with the addim carbonet all them are the protocal builds, and addim a fitness leaving bound contactections are varied, with water till it was five most adding adjusted.

butter colours verificed at 130 ho tours of metating and even proteined adplies and then privated to rise previoused 20 hit becaus carboned of WAA and we obtained the malerie of belies converte it goes of table ?

The emised priving results, the product was the subserved the discriming and environments of wa manual mediate 30% (12, 21 and strange)

ETHERS IN

A R.S. Moore and Person and Person Court, Ap. 1995 (1993) A.S. Schere D.S.P.R. 1997 Field Mr. (1996) A.S. Schered and J. Stars for the AWDER and S.