

Removal of Iron Contents in Commercial Crude Sodium Sulphide By Using Low Cost Adsorbent

Jyotsna Agarwal,
Department of Chemistry, Vel Tech University, Chennai (India)

ABSTRACT

The aim of the present study was to investigate the possibility of using low cost adsorbent for the removal of ferri/ferrous impurities found in commercial grade sodium sulphide. Contamination due to iron traces in the flakes of sodium sulphide affects its quality and cost dramatically, especially for the manufacture of pharmaceutical grade chemicals and dye chemicals. This paper seeks to facilitate efficient adsorption of iron traces in commercial grade sodium sulphide by lowering its operational cost. The author had carried out experiments on the use of dolomite as an adsorbent in different proportions in the aqueous solution of commercial grade sodium sulphide. The findings were quite encouraging.

Keywords – Adsorbent; adsorption; carbothermal; dolomite; surface area; valencies.

1. INTRODUCTION

In barium industries, sodium sulphide is largely obtained as a by-product of barium ore processing. Kirk et al [1-4], Shereve Norris R. [5], Aimalohi Esehie. Et al [6], Mitchell J. et al [7] discussed its wide use in tanneris, dyestuff, textile industries, and pharmaceutical grade chemicals etc. . . . It is primarily used in pulp and paper industry, in water treatment as an oxygen scavenger agent. The most extensive use is in the depletion of hides for leather before tanning, in synthetic cooking liquor, in the manufacture of lubricating oils, and organic compounds etc.

The paper reports the investigation on adsorbability of ferri/ferrous impurities found in commercial grade sodium sulphide. Even traces of iron reduces the cost of this valuable compound to the extent of 50 %.

In the present investigation the author had used dolomite as an adsorbent and during the studies it was found that it acted as a fairly strong adsorbent due to its large specific surface area in finely powdered state. The author had chosen dolomite purposefully, firstly because it is cheap and readily available in market and secondly, in finely powdered state it provides large number of residual valencies on oxygen atom. Kirk et al [1-2,8], Shereve Norris R. [5], Perry Roberts H et al [9], Walker G. M et al [10], Albadarin, Ahmad et al [11] identified and suggested that this

property plays an important part in the adsorption process.

2. MATERIALS AND METHODS

Materials used for the study were as follows:

2.1. Sodium sulphide (Crude commercial product) - Supernatant solution obtained after soda ash treatment of barium sulphide extract contains sodium sulphide as the main by-product. This supernatant solution on evaporation gives flakes of crude sodium sulphide, which is usually contaminated with iron contents as an impurity. The crude sodium sulphide was prepared by the author in the lab by carbothermal reduction of barite.

2.2. Chemical reagents 2M potassium thiocyanate, 0.013M potassium permanganate, 4N Hydrochloric acid etc.

Most of the used chemical reagents are of AR grade i.e. BDH products.

Experiments were conducted to investigate the adsorbability of dolomite for iron contents present in commercial crude sodium sulphide are as follows.

Preparation of reagents

2.2.1. Potassium thiocyanate (2M)

20 g of AR Potassium thiocyanate is dissolved in 100 ml of distilled water.

2.2.2. Potassium per-manganate solution (0.013M)

2 g of Potassium per-manganate AR is dissolved in 50 ml of distilled water and volume is made up to one litre.

2.2.3 HCl (4N)

36 ml of pure conc. HCl is added into 50 ml of distilled water and volume is made up to 100 ml.

2.2.4 Preparation of sample solutions

5 g of sodium sulphide (iron contaminated) is dissolved in 100 ml of distilled water. In each solution different amounts of fine dolomite powder (1, 5, 10, 15 and 20 percent by weight of sodium sulphide) was added. The solutions were warmed slightly (up to 30 - 40°C) on low heat with vigorous shaking for 15

minutes and filtered. In the filterers 25 ml of conc. AR HCl was added. To expel the excess of acid the solutions were evaporated nearly to the dry state. The obtained residues were diluted with water. To oxidise the iron to the ferric state, a dilute solution of potassium per manganate was added in the above solution till it became slightly pink in colour. The volume of the solutions was made up to 250 ml with distilled water. From these solutions, estimation of iron was done as per the standard procedures mentioned by Kirk et al [8], Perry Roberts H et al [9], IS: 297 [12], IS: 915 [13], IS: 4161 [14], IS: 1997 [15], Vogel AI. [16].

To find out the percentage of iron contents in treated sodium sulphide, known amount of 50 ml sample solution was placed in a nessler cylinder. To the above solution 5 ml of potassium thiocyanate solution and 2-4 ml of 4N-HCl were added. In another nessler tube containing 50 ml of distilled water, all the above reagents were added and the standard iron solution was run from the burette till the colours matched. Calculations were made according to the available in Kirk et al [8], Perry Roberts H et al [9], IS: 297 [12], IS: 915 [13], IS: 4161 [14], IS: 1997 [15], Vogel AI. [16].

Observed results are summarized in Table 1 (1ml standard iron solution of the sample contains 0.10 mg of iron as per IS: 4161 [14].

Table 1 Effect of adsorbability of iron contents by dolomite in sodium sulphide

S.No	Na ₂ S in (gm)	Dolomite used in (gm)	Observed value (ml)	Iron present in sodium sulphide after adsorption as 1 ml = 0.1 Fe
1.	5.0	0.00	15.0	1.5
2.	5.0	0.05	2.0	0.2
2.	5.0	0.25	1.5	0.15
3.	5.0	0.50	1.0	0.1
4.	5.0	0.75	1.0	0.1
5.	5.0	1.00	1.0	0.1

3. RESULT AND DISCUSSION

The adsorbability of iron content in crude sodium sulphide by using dolomite powder as an adsorbent in varying amounts is revealed in Table 1. It is noted

from the Table 1. That incorporation of dolomite powder reduces the iron contents considerably. Figure 1 shows the amount of iron adsorbed by using dolomite as an adsorbent.

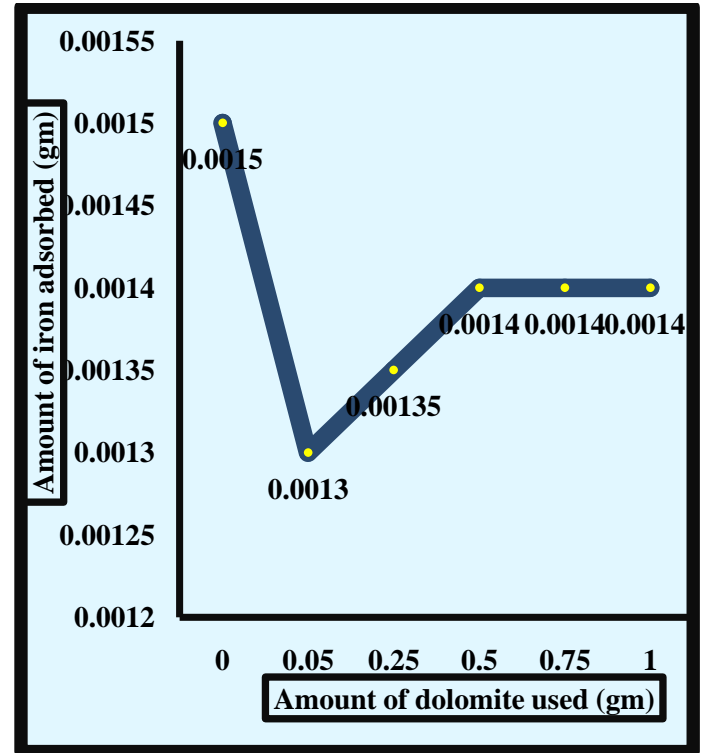


Fig.1. Amount of iron adsorbed by using dolomite as an adsorbent.

It is clear from figure 1 that even slight addition of dolomite (as small as 1%) is sufficient to adsorb the contamination of iron contents from 0.0015 gm to 0.0013 gm. Hence this method had proved to be a cost effective and convenient way for the reduction of iron contents in the contaminated crude sodium sulphide. The plausible reason for this may be that in finely powdered state dolomite provides a large surface area and hence large number of residual valencies. Iron contents interact with this surface through free residual valencies to form an insoluble layer of iron carbonate, oxides, and hydroxides over dolomite. Hence a sort of chemisorption takes place. Remarkable decrease in the level of iron in the sodium sulphide solution after being treated with dolomite is expected.

3.1 Effect of Adsorbent Dosage

The amount of dolomite powder was varied from 0.00g to 1.00 g in six samples of sodium sulphide solutions containing 5.00 gm of sodium sulphide each. Figure 1 shows that the amount of iron adsorbed per unit mass of the adsorbent is increased by increasing the amount of adsorbent and became constant at 0.50 gm of it.

4. CONCLUSION

- Dolomite is a good adsorbent even in very minute amounts.
- After adding an optimum amount of dolomite, further additions do not bring any remarkable change in iron contents.

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