



# Effective Removal of Iron Contents by Chalk Powder in the Commercial Crude Sodium Sulphide

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## Author's contribution

*This work was carried out by author herself under the guidance of Dr Chandrawat. Author JA did the literature survey, study design, review, performed different experiments, analysis and interpretation of the experimental data, wrote the manuscript and then finalised the paper.*

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## ABSTRACT

**Aims:** This paper reports the investigations pertaining to the adsorbability of ferri/ferrous impurities found in commercial grade sodium sulphide (which is not suitable for manufacture of pharmaceutical grade chemicals and dyes chemicals) by using chalk powder as an adsorbent in different proportions in the aqueous solution of commercial grade sodium sulphide.

**Study Design:** 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.

**Place and Duration of Study:** This study was carried out in Alwar (Raj.), India and the duration of study was two years.

**Methodology:** All required methods, materials, preparations of reagents and experiments were carried out using standard procedures.

**Results:** It is noted that incorporation of chalk powder reduces the iron contents considerably. Even slight addition (as small as 1%) is sufficient to reduce the contamination of iron contents from 1.50 mg to 0.10 mg. 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.

**Conclusion:** Based on the above results, it can therefore be concluded that chalk powder

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is a very good adsorbent. Even using 0.05g of chalk powder reduces the amount of iron from 1.50mg to 0.12mg. The obtained results can be quite useful in industrial practice.

*Keywords: Sodium sulphide; adsorption; barite; chalk powder; surface area.*

## 1. INTRODUCTION

Sodium sulphide is an important industrial chemical and it is largely obtained as a by-product of barium ore processing. Sodium sulphide is widely used in tanneries, dyestuff, textile industries, pharmaceutical grade chemicals, etc [1-7]. The most extensive use is in the depilation of hides for leather before tanning, in wood pulp processing, desulfuration of rayon and cellophane, elastomers, lubrication, in-water treatment as an oxygen scavenger agent, and organic compounds. It is used as a reducing agent, in ore flotation, in the recovery of metals as the insoluble sulfides.

This study investigated the potential use of low-cost class room chalk powder as an adsorbent for removal of ferri/ferrous impurities found in commercial grade sodium sulphide. Sodium sulphide is not suitable for manufacture of pharmaceutical grade chemicals if iron impurities are present. Even traces of iron devalues the cost of this valuable compound to the extent of 50 %. Its utility in the manufacture of dyes also gets affected adversely in presence of iron impurities. Thus to improve the quality of sodium sulphide author has tried chalk powder as an adsorbent.

In the present investigation chalk powder was used as an adsorbent and it was found that it acted as a fairly strong adsorbent due to its large specific surface area in the finely powdered state. This property plays an important part in the adsorption process [1,2,5,8-12].

## 2. MATERIALS

### 2.1 Experimental Materials

#### 2.1.1 Crude sodium sulphide

Supernatant solution obtained after soda ash treatment of barium sulphide extract contained sodium sulphide as the main by-product. This supernatant solution on evaporation gave flakes of crude sodium sulphide, which is usually contaminated with iron contents. The crude sodium sulphide was prepared by the author in the lab by carbothermal reduction of barite.

#### 2.2.2 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.

## 3. EXPERIMENTAL PROCEDURES

Experiments conducted to investigate the adsorbability of chalk powder for iron contents present in commercial crude sodium sulphide are as follows:

### **3.1 Preparation of Reagents**

#### **3.1.1 Potassium thiocyanate solution (2M)**

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

#### **3.1.2 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.

#### **3.1.3 HCl (4N)**

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

#### **3.1.4 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 chalk 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 [8,9,13-17].

### **4. METHOD**

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 Indian Standards [8,9,13-17]. Observed results are summarized in Table 1 (1 ml standard iron solution of the sample contains 0.10 mg of iron) [15].

### **5. RESULT AND DISCUSSION**

The adsorbability of iron content in crude sodium sulphide by using chalk powder as an adsorbent in varying amounts is revealed in Table 1. It is noted from the Table that incorporation of chalk powder reduces the iron contents considerably. Even slight addition (as small as 1%) is sufficient to reduce the contamination of iron contents from 1.50 mg to 0.10 mg as shown in Fig. 1. 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.

**Table 1. Effect of chalk powder on iron contents of crude sodium sulphide**

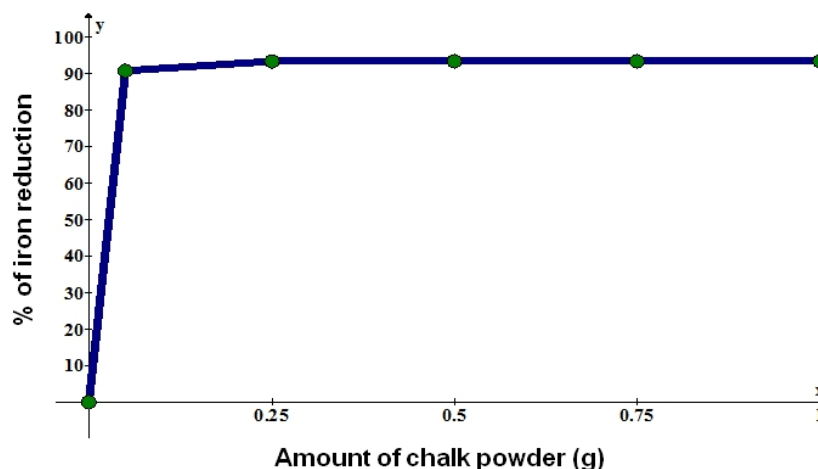
| Sample No. | Na <sub>2</sub> S used (g) | Chalk powder used (g) | Volume consumed (ml) | Fe present in Na <sub>2</sub> S after adsorption (mg)* | Removal of Fe (%) |
|------------|----------------------------|-----------------------|----------------------|--|-------------------|
| 1.         | 5.0                        | 0.00                  | 15.0                 | 1.50   | 0.0               |
| 2.         | 5.0                        | 0.05                  | 1.2                  | 0.12   | 90.7              |
| 3.         | 5.0                        | 0.25                  | 1.0                  | 0.10   | 93.4              |
| 4.         | 5.0                        | 0.50                  | 1.0                  | 0.10   | 93.4              |
| 5.         | 5.0                        | 0.75                  | 1.0                  | 0.10   | 93.4              |
| 6.         | 5.0                        | 1.00                  | 1.0                  | 0.10   | 93.4              |

\*1 ml = 0.10 mg Fe

### 5.1 Effect of Adsorbent Dosage

The amount of chalk powder was varied from 0.00g to 1.00 g in six samples of sodium sulphide solutions containing 5.00 g of sodium sulphide each. Fig.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 0.25 g of it.

This can be explained on the basis of chemisorption. In fine powdered state, chalk powder provides a large surface area and, therefore, a large number of free residual valencies. On this surface the iron contents interact with the free residual valencies of surfacial oxygen atom of chalk powder through weak chemical bonds. As a result of this chemisorption, a monomolecular layer of iron oxide or iron sulphide is formed over the chalk powder. Hence, it is concluded that the amount of chalk powder suspended in the sodium sulphide solution is just sufficient to provide required surface area for the chemisorption and formation of monomolecular layer of iron contents. So, further addition of adsorbent (beyond this optimum amount) should not show any remarkable improvements in the adsorption. Consequently, it is apparent from the results that suspended chalk powder is a very good adsorbent and further increase (beyond 5%) in its amount does not show any improvement (in the results).

**Fig. 1. % of iron reduction versus the amount of chalk powder used**

## **6. CONCLUSION**

The reduction of iron from 1.50 mg to 0.10 mg (Table 1) is quite significant and it indicates that chalk powder is a good adsorbent for removal of iron from crude sodium sulphide. Even using 1% (by weight) of chalk powder, it eliminates approximately 90.7% of iron impurities from crude sodium sulphide. After adding an optimum amount (approximately 5% by weight) of chalk powder, further additions do not bring any remarkable change in iron content.

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## **COMPETING INTERESTS**

Author has declared that no competing interests exist.

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