

Effective removal of iron contents by chalk powder in the commercial crude sodium sulphide

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Authors' contributions

This work was carried out by author herself under the guidance of Dr Chandrawat. She 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.

ABSTRACT

Aims: This paper reports the investigations pertaining to the adsorb ability of ferry/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: Sodium sulphide: 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 the slight addition (as small as 1%) is sufficient to reduce the contamination of iron contents from 1.5 mg to 0.1 mg. This amounts to a very smooth and convenient reduction of iron contents to the extent of 0.005 percent or even less in the contaminated commercial sodium sulphide.

Conclusion: Based on the above results, it can therefore be concluded that chalk powder is a very good adsorbent even in very minute amounts.

Keywords: Sodium sulphide, adsorption, barite, precipitated chalk powder, surface area.

1. INTRODUCTION

Sodium sulphide is largely obtained as a by-product of barium ore processing. It is a commercially important compound. 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, and organic compounds. It is used as a reducing agent, in ore flotation, in the recovery of metals as the insoluble sulfides.

The paper reports the investigations on absorbability of ferri/ferrous impurities found in commercial grade sodium sulphide. Sodium sulphide is not suitable for manufacture of pharmaceutical grade chemicals and dyes if iron impurities are present. Even the traces of iron reduces the cost of this valuable compound to the extent of 50 %. Its utility in the manufacture of dyes also affected adversely in presence of iron impurities. Thus to improve the quality of sodium sulphide author have tried chalk powder as adsorbent.

In the present investigation author had used chalk powder 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

Materials used for 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 (Potassium thiocyanate, KMnO_4 , HCl – 4 N etc.) – Most of the chemical reagents are of AR grade i.e. BDH products.

3. EXPERIMENTAL PROCEDURES

Experiments were conducted to investigate the adsorb ability of aluminium oxide for iron contents present in commercial crude sodium sulphide are as follows.

3.1. Preparation of reagents –

3.1.1 Potassium thiocyanate solution - 20 gm of AR Potassium thiocyanate is dissolved in 100 ml of distilled water.

3.1.2 KMnO_4 solution – 2 gm of KMnO_4 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 gm of sodium sulphide (iron contaminated) is dissolved in 100 ml of distilled water. In each solution the different amounts of finely powdered precipitated barium s aluminium oxide (1,5,10,15 and 20 percent by weight of sodium sulphide) was added. The solutions were warmed slightly on the 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 dryness. The obtained residues were diluted with water. To oxidise the iron to the ferric state, a dilute solution of potassium per magnate was added in the above solution till the slight pink colour appeared. 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 is placed in a nessler cylinder. In this solution 5 ml of potassium thiocyanate solution and 2-4 ml of 4N- HCl is added.

In another nessler tube 50 ml of distilled water, all the above reagents are added and the standard iron solution is run from burette till the colours are matched.

Calculations were made according to the available Indian Standards [8, 9, 13-17]. Observed results are summarized in the Table -1 (1 ml standard iron solution of the sample contains 0.1 mg of iron) [15].

5. RESULT AND DISCUSSION

Adsorbability of iron contents by chalk powder is revealed in the Table 1. It is noted from the Table 1 that incorporation of chalk powder reduces the iron contents considerably. Even the slight addition (as small as 1%) is sufficient to reduce the contamination of iron contents from 1.5 mg to 0.1 mg as shown in fig-1. This amounts to a very smooth and convenient reduction of iron contents to the extent of 0.005 percent or even less in the contaminated commercial sodium sulphide.

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

S.No	Na ₂ S used in grams	chalk powder used in grams	Observed value (ml)	Iron present in sodium sulphide after adsorption as 1 ml = 0.1 Fe	% Removal of iron
1.	5.0	0.00	15.0	1.5	0.0
2.	5.0	0.05	1.2	0.1	90.7
3.	5.0	0.25	1.0	0.1	93.4
4.	5.0	0.50	1.0	0.1	93.4
5.	5.0	0.75	1.0	0.1	93.4
6.	5.0	1.00	1.0	0.1	93.4

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5.1. Effect of Adsorbent Dosage:

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The amount of chalk powder was varied from 0.0 gm to 1.00 gm in six samples of sodium sulphide solution. Each solution is containing 5 gm of sodium sulphide. It is observed from figure.2 that the amount of iron adsorbed per unit mass of the adsorbent is increases with increase in amount of adsorbent and became constant at 0.05 gm of adsorbent.

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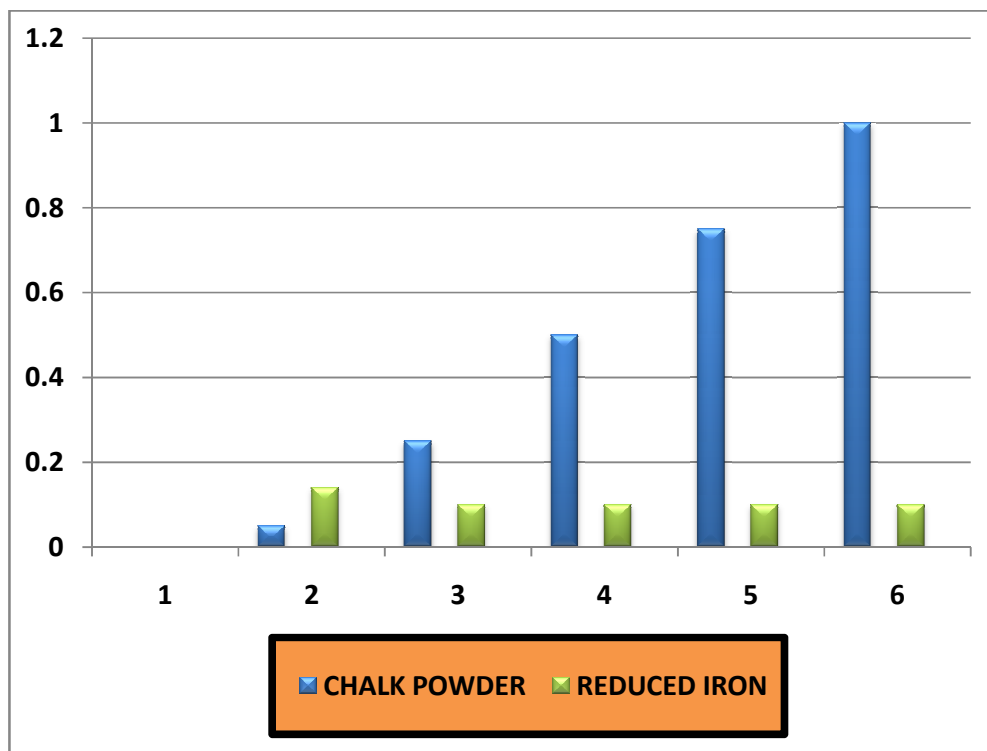
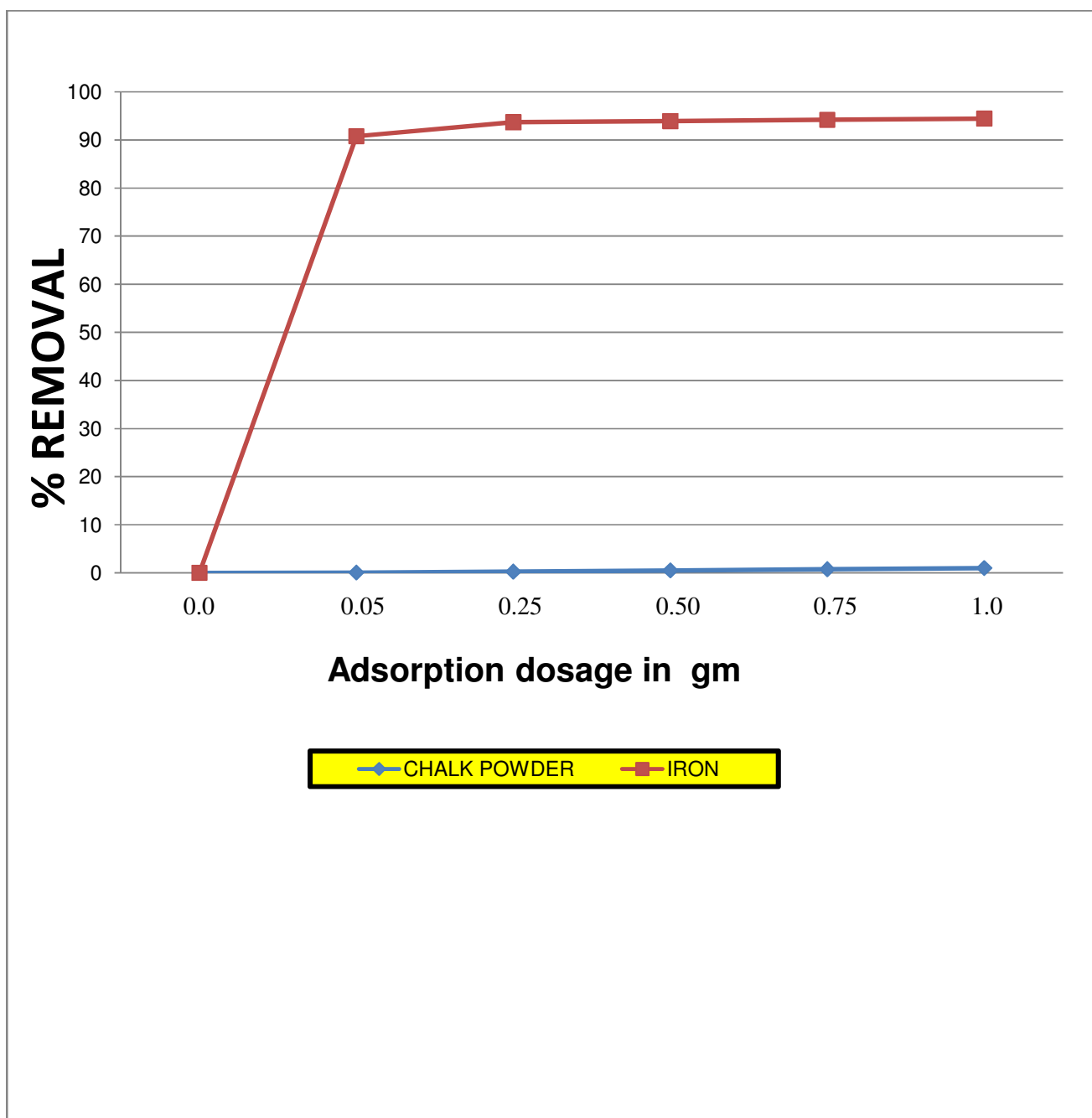


Figure 1. Variation in chalk powder dosage and iron adsorption



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121 **Figure.2. % removal of iron at different amount of chalk powder**

122 This can be explained on the basis of chemisorption. In fine powdered state, chalk powder provides a
 123 larger surface area and hence large number of free residual valencies. On this surface the iron
 124 contents interact with the free residual valencies of surfacial oxygen atom of chalk powder through
 125 weak chemical bonds. Resulting this chemisorption a monomolecular layer of iron oxide or iron
 126 sulphide is formed over the powdered chalk powder. Hence it is expected that the amount of
 127 powdered chalk powder suspended in the sodium sulphide solution is just sufficient to provide
 128 required surface area for the formation of monomolecular layer and chemisorption of iron contents
 129 should be the optimum amount. Therefore further addition of adsorbent (beyond this optimum
 130 amount) should not show any remarkable improvements in the adsorption. Accordingly it is apparent

from the result that powdered suspended chalk powder is a very good adsorbent and beyond 5% further increase in its amount does not show any improvements in the results.

6. CONCLUSION

Chalk powder is a good adsorbent even in very minute amounts. After adding an optimum amount of chalk powder, further additions do not bring any remarkable change in iron content

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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