International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.6, No.7, pp 3927-3930, Sept-Oct 2014

Removal of Iron Contents in the Crude Sodium Sulphide by using Fuller's Earth as a Cheap Adsorbent

Jyotsna Agarwal*

Vel Tech University, Chennai – 62, India.

***Corres.author: drjyotsnaagarwal@ymail.com**

Abstract: The present work is aimed at studying the adsorption capacity of powdered Fuller's earth for the removal of ferri/ferrous impurities found in commercial grade sodium sulphide. The latter is not suitable for the manufacture of pharmaceutical grade chemicals and dye chemicals. The author has carried out experiments taking different dosage of powdered Fuller's earth as an adsorbent in the aqueous solution of commercial grade sodium sulphide. The findings are quite encouraging and can be very useful in industrial practice.
Keywords: Sodium sulphide, adsorption, barite, carbothermal reduction, Fuller's earth.

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, and 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 adsorbability 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 traces of iron devalues the cost of this valuable compound to the extent of 50 %.However despite such an acute importance of this compound no sustainable technique has been developed for the same. Most of the technique that have been employed in the past are rather expensive, laborious, and not wholly satisfactory. Hence there is an increasing need for effective but cheaper methods.

In the present investigation the author had used powdered Fuller's earth (source- local market of Barmer in Rajasthan) as an adsorbent and it was found that it acted as a fairly strong adsorbent due to its large specific surface area in finely powdered state.Commonly known as Multani mitti the Fuller's earth is chemically reckoned as Montmorillonite which resembles to Bentonite (calcium bentonite) very much.InIndia, Rajasthan possesses good deposits of Fuller's earth mainly in Barmer district, (77%). Fuller's earth is usually used in bleaching, decolourising vegetable oils, petroleum, lubricants, greases, fertilizer industry etc. as well as a carrier for insecticides, fungicides and as a mineral filler and extender. Fuller's earth containscomplex multicentre crystalline structure of oxidesand hydroxide of silicon, magnesium, aluminium, calcium etc. Table 1 shows the weight percent of different oxides present in the Fuller's earth used for the investigation. The author had chosen Fuller's earth purposefully because of its easy availability, cheap cost and large specific surface area[8-12.] These properties play an important role in the adsorption process[1, 2, 5, 13-14.]

Table 1-Weight % of different oxides in Fuller's earth used as an adsorbent

Type of Oxide	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O
Weight %	50.44	22.78	5.90	4.53	1.88	1.11

Materials and Methods

- **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.

- **Chemical Reagents (Potassium thiocyanate, Potassium permanganate, HCl etc.):**Most of the chemical reagents are of AR grade i.e. BDH products.

- **Potassium Thiocyanate Solution:**20 g of AR Potassium thiocyanate was dissolved in 100 ml of distilled water.

- **Potassium Permanganate Solution:**2 g of Potassium permanganate AR was dissolved in 50 ml of distilled water and volume was made up to one litre.

- **HCl (4N):** 36 ml of pure conc. HCl was added into 50 ml of distilled water and volume was made up to 100 ml.

- **Preparation of Sample Solutions:** 5 g of sodium sulphide (iron contaminated) was dissolved in 100 ml of distilled water. In each solution different amounts of finely powdered Fuller's earth (1, 5, 10, 15 and 20 percent by weight of sodium sulphide) was added. The solutions were warmed slightly (up to 30 to 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 permanganate was added in the above solution till it became slightly pink in appearance. 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[13-19].

To find out the percentage of iron contents in treated sodium sulphide, known amount of 50 ml sample solution was placed in a Nesseler cylinder. To the above solution 5 ml of potassium thiocyanate solution and 2-4 ml of 4N-HCl were added. In another Nesseler tube containing 50 ml of distilled water, all the above reagents were added and the standard iron solution was run from burette till the colours matched.

Calculations were made according to the available Indian Standards[13-19].Observed results are summarized in Table 2 (1 ml standard iron solution of the sample contains 0.10 mg of iron) [17].

Table 2. Effect of powdered Fuller's earth on iron contents of crude sodium sulphide

Sample No.	Na_2S used (gm)	Fuller earth used (gm)	Volume consumed (ml)	Fe present in Na_2S after adsorption (mg)*	% of iron removal after adsorption
1.	5.0	0.00	15.0	1.50	0.0
2.	5.0	0.05	5.0	0.50	67.0
3.	5.0	0.25	4.6	0.46	69.4
4.	5.0	0.50	4.3	0.43	71.4
5.	5.0	0.75	4.1	0.41	72.7
6.	5.0	1.00	4.0	0.40	73.4

***1 ml = 0.10mgFe**

Results and discussion

Adsorbability of iron contents by Fuller's earth powder as an adsorbent in varying amounts is revealed in Table 2. It is noted from Table 2 that incorporation of Fuller's earth 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 0.50 mg as shown in Table 2. Hence this method proved to be very smooth, cost effective and convenient for the reduction of iron contents in the contaminated crude sodium sulphide.

Role of powdered Fuller's Earth as an Adsorbent

The amount of powdered Fuller's earth was varied from 0.00 g to 1.00 g in six samples of sodium sulphide solutions containing 5.00 g of sodium sulphide each. Figure 1 and Figure 2 show that the amount of iron adsorbed per unit mass of the adsorbent is increased by increasing the amount of adsorbent.

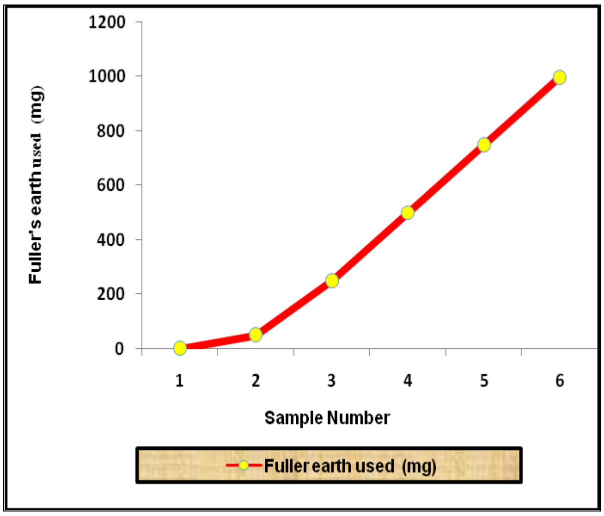

Fig.1 Fuller's earth (mg) used in samples

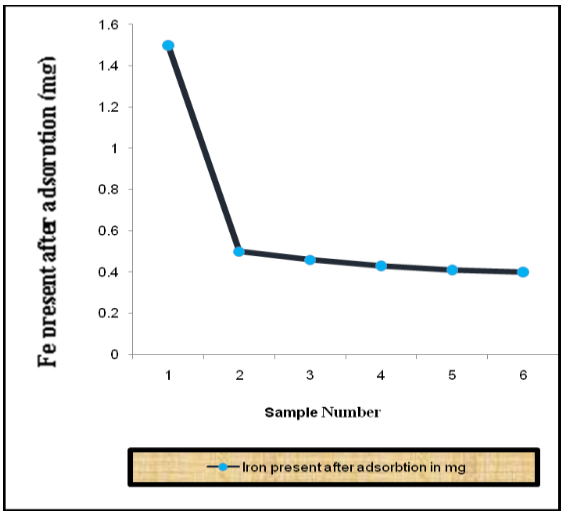

Fig.2 Fe (mg) present after adsorption

This can be explained on the basis of chemisorption. In fine powder state Fuller's earth is comprised of finely divided silica with large surface area and hence a large number of free residual valencies. These valencies interact with iron contents of the sodium sulphide solution resulting in formation of the monomolecular layer of iron contents. The monomolecular layer of iron contents over the particles of Fuller's earth is adsorbed strongly due to the developing chemosorptive forces. At the same time heteroporous nature of Fuller's earth helps to form strong bond between adsorbate and adsorbent. It is interesting to note that additional amount of Fuller's earth (beyond 10% by weight) does not bring any considerable change in the iron contents of the solution as shown in Figure 3.

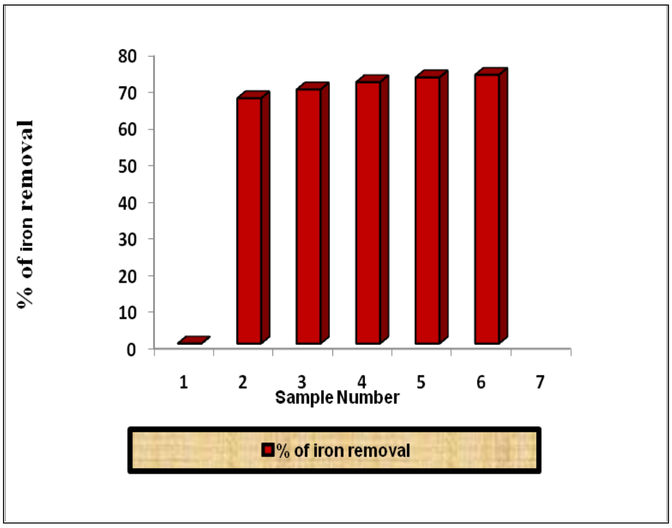

Fig.3 Removal of iron (%) after using fuller's earth as an adsorbent

Plausible reason may be that the amount of powdered Fuller's earth 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.

It is apparent from the results that powdered Fuller's earth is a very good adsorbent and further increase (beyond 10% by weight) brings a very small change in the composition of sodium sulphide solution with respect to its iron contents. Amount left in the solution owe to the thermodynamic equilibrium of iron contents between two phases, i.e. monomolecular heterogeneous phase and the solution phase.

Conclusions

Powdered Fuller's earth is a very good adsorbent for removal of iron from crude sodium sulphide. Even using 0.05 g of powdered Fuller's earth reduces the amount of iron from 1.50 mg to 0.50 mg. From Table.2 it is quite significant to note that by using only 1% (by weight) of powdered Fuller's earth eliminates approximately 67.0% of iron impurities from crude sodium sulphide. After adding an optimum amount (approximately 10% by weight) of powdered Fuller's earth, further additions do not bring any remarkable change in iron contents.

References

1. Kirk & Othmer, Encyclopaedia of Chemical Technology, vol 2, John Wiley & Sons, New York 1978, 41,441.
2. Kirk & Othmer, Encyclopaedia of Chemical Technology, vol 8, John Wiley & Sons, New York 1978, 540,612.
3. Kirk & Othmer, Encyclopaedia of Chemical Technology, vol 18, John Wiley & Sons, New York 1978,438,510.
4. Kirk & Othmer, Encyclopaedia of Chemical Technology, vol 19, John Wiley & Sons, New York1978, 424, 431.
5. Shereve Norris R, Chemical ProcessIndustries, 3rd ednMcGraw hill, New York 1976.
6. Aimalohi Esechie1& Perenlei Enkhbaatar Br. J. Pharmaco., 2009, 158,1442-1453.
7. Mitchell J Best N Sundstrom LE& Wheal HV, PMID: 8468197 Histrochemistry; 1993, 99(1),91-4.
8. Gupta V. K & Suhas J. Envroni. Manag., 2009, 90,2313-2342.
9. A.K. Bajpai & N. Vishwakarma Physicochem. Eng. Aspects,2003, 220,117-130.
10. Elzea J, Nikhil C, James M,&Stanley T Industrialminerals and rocks: Commodities, markets and uses,7th ed. Society for Mining-Metallurgy and Exploration Inc 2005.
11. McKay G Otterburn M S & Sweeney AG Water Air Soil Pollut.,1985, 24147-61.
12. Indian Minerals Year book,50thedn, Indian Bureau of Mines, Nagpur 2012.
13. Kirk & Othmer, Encyclopaedia of Chemical Technology, vol 1, John Wiley & Sons, New York 1978, 25, 421,460.
14. Perry Roberts H & Chilton Cecil Chemical Engineers Handbook,5th edn. McGraw Hill, New York 1973.
15. IS: 297, 2001 (New Delhi: BIS)
16. IS: 915, 1975 (New Delhi: BIS)
17. IS: 4161, 1975(New Delhi: BIS)
18. IS: 1997, 1982(New Delhi: BIS)
19. A. I. Vogel,Quantitative Inorganic Analysis,3rd edn.Longman 1966.

*****