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Removal of Fe (II) using synthesised 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust

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ABSTRACT

Fe and Zn are essential trace elements for living organisms but the high level of accumulation of these metals can produce various problems in human being. Fe (II) is essential part of hemoglobin. Excess amount of Fe (II) may cause conjunctivitis, choroiditis and retinitis, so it is necessary to remove the excessive amount of Fe (II) present in water more than permissible limit[1-3]. Many workers have used different methods for removal of toxic metal from polluted water.[4-11] In present work newly synthesised chelating resin, 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust (AHBSASD) has been used for removal of Fe (II) ions from polluted water samples prepared in laboratory[12-15]. By chelation Fe (II) ions are chelated on the newly synthesised chelating resin and get removed from water sample. The chelation process was studied as a function of pH (3.5 to 6.5), contact time (≃60 min.), initial concentration (10 ppm) and temperature (30°±1°C) keeping constant amount of saw dust (0.1 g). The concentration of Fe (II) ions in the filtrate was determined using corresponding calibration curve. It was observed that the pH has marked effect on removal of Fe (II). Result shows that about 75 % removal of Fe (II) takes place at pH at 5.04. At this pH chelation of Fe (II) ions was studied with varying amounts of resin having same initial concentration, temperature and contact time. It was observed that with increasing amount of AHBSASD resin, the distribution coefficient (K_d) and percentages removal values increase and at 0.5 g dose these reach to maximum 1773 and 78 % respectively and remains constant at higher doses of resin.

Key Words: Heavy metals, Saw dust, Calibration curve, Fe (II), Chelation, Absorbance, Polluted water, 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust (AHBSASD).

INTRODUCTION

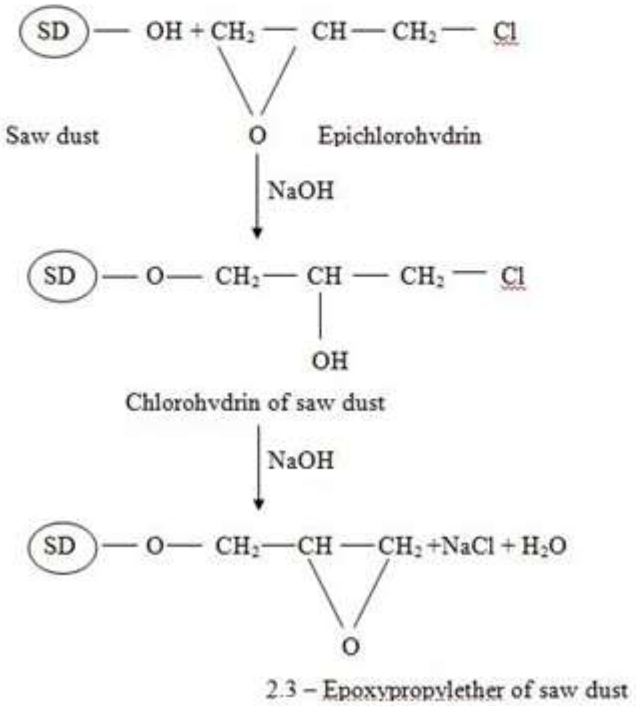
Water is most common liquid on earth. It covers about 71.4% of the earth. It is essential natural resource for sustaining life and environment but over the last few decades the water quality has been deteriorated due to mixing of chemicals, washed down drains and discharged from factories. Factories are point sources of water pollution but quite a lot of water is polluted by ordinary people from non-point sources, virtually every one pours chemicals of one sort or another and down their drains or toilets. Water Pollution is a major environmental issue in our Country. The Largest source of water Pollution in India is untreated sewage and toxic substances released through industrial effluents. Industrial effluents contain many types of toxic trace metal ions like Fe (II), Ni (II), Cr (VI), Zn (II), Cu (II), Hg (II) and Cd (II). Excess dose of heavy metals in to natural environment results various problems in both animals and plants. Out of these heavy metals Fe (II) is essential to almost all living beings, from microorganisms to humans. The human body absorbs iron in animal products faster than iron in plants. It is essential part of hemoglobin. Iron deficiency leads to anemia but chronic inhalation of excessive concentration of Fe (II) results in development of pneumoconiosis and hemochromatosis.

MATERIAL AND METHODS

(A) Synthesis of Cross Linked saw dust

486 g saw dust (corresponding to three anhydroglucose unit) was taken in a round bottom flask and it was slurred with dioxane. 15 ml of 40% (w/v) sodium hydroxide was added to it, to make it alkaline, till pH reached 8.5. The contents of the flask were slurred magnetically at 45°C. Then 92.53 g (1mole) epichlorohydrin was added with constant stirring. The stirring was further continued for four hours at 45°C.

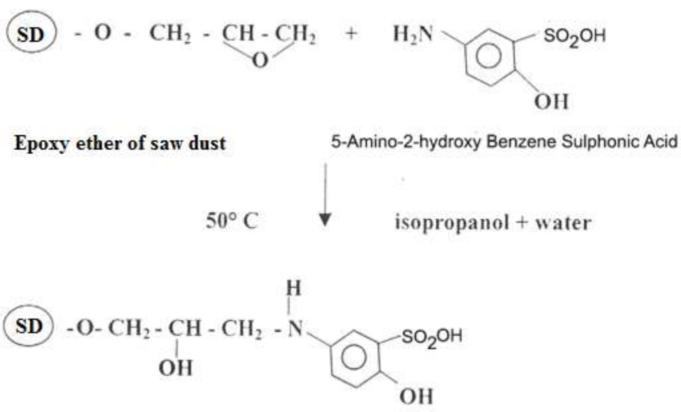
The reaction mixture was then allowed to settle down. The supernatant liquid was decanted off and the product was filtered under vacuum and washed with 80% aqueous methanol containing few drops of nitric acid, to remove inorganic impurities and excess alkali in the contents. Washing was done till the filtrate was free from chloride ions and was no more alkaline. The washed product was dried in an oven at 40°C. Obtained cross linked saw dust was further used for derivatization.



Scheme 1 : synthesis of cross linked saw dust

(B) Synthesis of 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust (AHBSASD)

In a round bottom flask 0.01 mole of cross linked saw dust was taken and it was slurred with 70% aqueous isopropanol. 10 ml of 50% (w/v) aqueous sodium hydroxide was added to it gradually with continuous stirring at 45°C. Then 0.02 mole of 5-Amino-2-Hydroxy Benzene Sulphonic Acid was added slowly with stirring to the contents of flask. The stirring and heating was continued for 6 hours. The product thus formed was filtered on a Buchner funnel. Then washing of product was done with 50% aqueous methanol containing few drops of nitric acid to remove excess alkali and inorganic impurities. The washed product was dried in air and then was suspended in 0.1 N HCl and filtered immediately. The filtered product was successively washed with 0.1 N NaOH and 0.1 N HCl. The supernatant liquid was decanted and resin was washed several times to remove suspended particles and other impurities. The final washing was carried out with absolute alcohol and the product was dried under vacuum. The product was brownish powder.



Scheme 2 : Synthesis of 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust

REAGENTS

All the chemicals used were of analytical grade obtained from E. Merck. Stock solutions of 2000 mg/L each of the Fe (II) were prepared separately by dissolving required amounts in distilled water. Sample solutions of required concentrations were prepared by diluting the stock solutions. The pH of solutions was adjusted using 0.2 M sodium acetate and 0.2 M acetic acid.

INSTRUMENTATION

AGRONIC-511 digital pH meter was used to determine pH of the solutions. Spectrophotometric observations were obtained on an AIMIL-MAKE 'spectrochem' spectrophotometer. Magnetic stirrers manufactured by metrex scientific Pvt. Ltd. were used for stirring.

EXPERIMENTAL METHODS

Measurement of absorbance for standard Fe (II) solutions and Calibration Curve

50 ml sample solution, 5 ml of concentrate HCl and 1 ml of 10% $NH_2OH.HCl$ were taken in a beaker and brought these to room temperature and the contents of beaker were transferred to a volumetric flask. 10ml ammonium acetate buffer was added to a volumetric flask and shaken well, diluted up to mark with distilled water. Allowed, it to stand for 10 to 15 minutes for maximum colour development. Absorbance of extract was measured at 510 nm comparing a reagent blank.

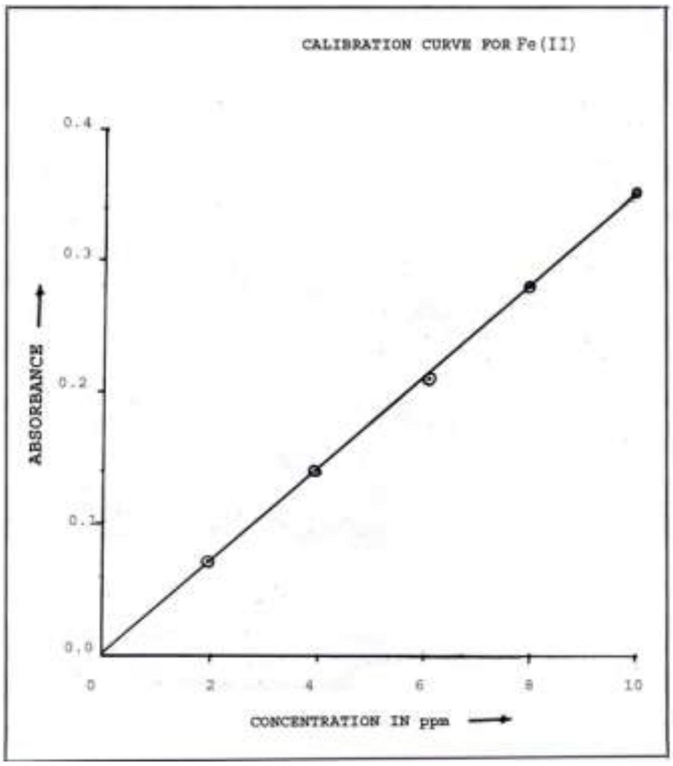
In the same way using standard Fe (II) solutions of different concentrations, calibration curve was plotted. Again the concentration of unknown sample of Fe (II) can be determined by using the calibration curve.

Table 1

Absorbance for standard Fe (II) Solution

S.No.	Concentration (ppm)	Absorbance
1	2	0.08
2	4	0.14
3	6	0.21
4	8	0.28
5	10	0.35

Figure 1 : Calibration Curve for Fe (II) Solutions



RESULT AND DISCUSSION

A. Chelation of Fe (II) ion on constant amount of AHBSASD resin with varying pH.

0.1 g of dry resin and 25 ml of 20 ppm solution of Fe (II) were taken in different sets. Appropriate amounts of 0.2 M acetic acid and 0.2 M sodium acetate were added to each set to obtain desired pH. The total volume of sodium acetate-acetic acid buffer was kept 25 ml in each set. The contents were stirred magnetically. The filtrates were analysed for Fe (II) concentration spectrophotometrically. The results are given in Table 2.

The distribution coefficient (Kd) and percentage removal of Fe (II) are calculated by applying following Formula -

$$K_d = \frac{\text{Amount of Fe (II) in saw dust derivate (AHBSASD) Phase/g of dry saw dust derivate}}{\text{Amount of Fe (II) in solution/ml of solution}}$$

$$\%\text{Removal of Fe (II)} = \frac{\text{(Initial concentration of Fe (II) sol. — concentration of Fe (II) solution after treatment with saw dust derivate)}}{\text{Initial concentration Fe (II) solution}} \text{X100}$$

Table 2
Chelation of Fe (II) on constant amount of AHBSASD resin with varying pH.

Amount of AHBSASD added = 0.1 g Initial concentration = 10 ppm

Volume of Fe (II) of 20 ppm =25 ml

Total volume = 50 ml. Temperature = 30°± 1° C

S.No	Vol. of 0.2 M acetic acid (ml)	Vol. of 0.2 M sodium acetate (ml)	pH	O.D. of filtrate	Conc. Of Fe (II) in filtrate (ppm)	Amount of Fe (II) in sol. (mg)	Amount of Fe (II) in AHBSASD (mg)	K_d	% Removal
1	23	2	3.51	0.27	7.8	0.385	0.115	149	23
2	19	6	4.02	0.21	6.0	0.300	0.200	333	40
3	15	10	4.51	0.14	4.1	0.195	0.305	782	61
4	7	18	5.04	0.09	2.5	0.125	0.375	1500	75
5	3	22	5.53	0.12	3.3	0.165	0.335	1015	67
6	1	24	6.03	0.16	4.4	0.225	0.275	611	55

Inference

It is observed that with the increase of pH the K_d values for Fe (II) on AHBSASD increases. At pH 5.04 the distribution coefficient value is maximum (1500) and removal percentage is 75 %. On pH more than 5.04 the K_d value and removal percentage decreases.

B.Chelation of Fe (II) on varying amount of AHBSASD resin at constant pH.

Different amounts of AHBSASD resin were taken in each flask and 7 ml of 0.2 M acetic acid 18 ml of & 0.2 M sodium acetate were added to get the pH 5.04. Now 25ml (20 ppm) solution of Fe (II) was then added to each set. The contents were stirred magnetically and equilibrated over night. The filtrates were analysed for Fe (II). The results are given in Table 3.

Table 3
Chelation of Fe (II) ion on varying amounts of AHBSASD resin at constant pH.

Volume of Buffer = 25 ml
(7 ml Acetic acid + 18 ml Na-Ac)
Volume of Fe (II) of 20 ppm =25 ml
Total volume = 50 ml.

Initial concentration = 10 ppm
Temperature = 30°± 1° C
pH = 5.04

S. No.	Amount of AHBSASD added (mg)	O.D. of Filtrate	Conc. Of Fe (II) in Filtrate (ppm)	Amount of Fe (II) in solution (mg)	Amount of Fe (II) in AHBSASD (mg)	K_d	% Removal
1	100	0.13	3.8	0.165	0.335	1015	67
2	200	0.12	3.5	0.150	0.350	1167	70
3	300	0.11	3.2	0.135	0.365	1352	73
4	400	0.10	2.9	0.125	0.375	1500	75
5	500	0.09	2.7	0.110	0.390	1773	78
6	600	0.09	2.7	0.110	0.390	1773	78

Inference

It is observed that at constant pH 5.04, the K_d value and percentage removal of Fe (II) increases with amount of AHBSASD. It reaches maximum at 500 mg amount of AHBSASD. At this amount, K_d is 1773 and percentage removal is 78%. It remains constant on further increase of amount of resin.

CONCLUSION

The saw dust gives hydrophilic base for preparation of chelating resins. The polysaccharide based resins are more effective and compatible in metal ion separation from solutions. The Naturally occurring polysaccharides are fibrous in nature, which imparts the case of accessibility of functional groups even to macro molecules in the surrounding solutions. Keeping this aim in view, we have synthesized 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust, resin for removal of Fe (II) from standard solutions prepared in laboratory. It is clearly seen from Table-3 that 78% of Fe (II) is removed at pH 5.04 and its distribution coefficient (1773) is also maximum at this pH. Results in Table-3 show that percentage removal of Fe (II) is increased with increase in amount of AHBSASD resin at constant pH 5.04. It remains constant on further increase of amount of resin. Finally we concluded that 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust is a good and economical resin for removal of toxic metals from solution and waste water. It may be used in water treatment plants because of its effectiveness, low cost and eco-friendly nature.

REFERENCES

1. S.S. Dara;
   A Text Book of Environmental Chemistry and Pollution Control.
   S. Chand & Company Ltd. New Delhi, 1993
2. A.K. De;
   Environmental Chemistry.
   Wiley Eastern Limited, New Delhi, 1989
3. H. Kaur;
   Environmental Chemistry.
   Pragati Prakashan, Meerut, 2005
4. Dingwag Chen, Ajay K Rasy;
   Removal of toxic metal ions from waste water by semiconductor photocatalysis.
   Chemical engineering science, Volume 56, Issue 4, 2001, 1561-1570.
5. Ashok K Pandey, Shri Dhar Pandey and Virendar Mistra;
   Removal of toxic metals from leachates from hazardous solid waste and reduction of toxicity to microtox by the use of calcium alginate beads containing humic acid.
   Ecotoxicology and Environmental Safety, Volume52,Issue 2, 2002, 92-96.
6. A L Zouboulis, M X Loukidou and K A Matis;
   Bio sorption of toxic metals from aqueous solution by Bacteria strain isolated from metals polluted soils.
   Process Biochemistry, Volume 39,Issue 8, 2004, 909-916

7. Sandra Maria Dal Basco, Ricardo Sarti Jimer and Wagner Alves Carvalho;
   Removal of toxic metals from waste water by Brazilian natural Scolecite.
   Journal of colloid and Interface Science, Volume 281, Issue 2, 2005, 424-431.
8. Syed Moosa Hasany and Rashid Ahmad;
   The potential of cost effective coconut husk for the removal of toxic metal ions for environmental protection.
   Journal of environmental management, Volume 81, Issue 3, 2006, 286-295.
9. Amany H Sikaily, Ahmed Nemr, Azza Khaled and Ola Abdel Wehab;
   Removal of toxic chromium from waste water using green alga ulva lactuca and its activated carbon.
   Journal of hazardous materials, Volume 148, Issue 1-2, 2007, 216-288.
10. Sarika Singh , K C Baruck and D Bahadur;
    Surface engineered magnetic nano particles for removal of toxic metal ions and bacterial pathogens.
    Journal of hazardous materials, Volume192, Issue 3, 2011, 1539-1547.
11. Mikko Vepsalainen, Heli Kivisari, Martti Pullianen and Aimo Oikri;
    Removal of toxic pollutants from pulp mill effluents by electroagulation.
    Separetion and purification technology, Volume 81, Issue 2, 2011, 141-150.
12. Harsukh Ram Chharang and Atul Kumar Sharma;
    Romoval of Mercury (II) using Synthesised 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of wood flour.
    Journal of Interdisciplinary Cycle Research, Vol 3, Issue 2, July-December 2011.
13. Harsukh Ram Chharang and Atul Kumar Sharma;
    Romoval of Chromium (VI) using Synthesised 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of saw dust.
    IJREAS, Vol 2, Issue 10, October 2012.
14. Harsukh Ram Chharang and Bhanwar Lal;
    Removal of Copper (II) using Synthesised 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of wood flour.
    Bull. Env. Pharmacol. Life Sci., Vol 2 (7) June 2013 : 38-40.
15. Bhanwar Lal;
    Removal of Nickel (II) using Synthesised 5-Amino-2-Hydroxy Benzene Sulphonic Acid derivative of wood flour.
    Research Journal of Chemical and Environmental Sciences, Vol 1, Issue 3, Augest 2013 : 03-07.