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Detection and removal of iron from water

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Abstract

Detection of iron in water involve use of different reagents but αα' dipyridyl along with citric acid was used as a field test in a modified way. Its extraction was studied by means of ion selective chelating resin synthesized from chitosan. The separation is carried out by column chromatography. IR spectra, nitrogen content [calculated by Kjeldahl method] and pH titration [using batch method] were made for resin characterization. Resin characteristic such as moisture content, bulk density, specific bulk volume and ion exchange capacity were also determined by standard methods. Metal uptake analysis was done by batch method using atomic absorption spectrophotometer. The distribution coefficient values at different pH levels were determined from batch method. Fe[ll] showed maximum Dg value at pH 7.0 .

Key words : Chelating resin, chitosan, water, iron

Introduction

Iron is very common constituent of fresh waters. Iron in water is not toxic, but it does have a number of harmful effects[1]. It can cause rust stains on clothing, porcelain materials etc., as well as giving the water itself an undesirable colour and taste, thus making it aesthetically unacceptable.

However, acute exposure[2] to iron is characterised by vomiting, gastrointestinal bleeding, pneumonitis, convulsions, coma and jaundice, but its deficiency may cause anaemia. ICMR has laid down 0.3 mg/l iron in drinking water as maximum permissible limit. Higher iron content may produce undesirable effects such as astringent taste, turbidity and growth of iron bacteria in pipes affecting the acceptability of water for domestic use.

The methods for estimation of iron involve use of different reagents like Potassium ferrocyanide, 8-Hydroxy quinoline, Potassium thiocyanate, Thioglycollic acid, Dimethyl glyoxime, αα' Dipyridyl etc.

Laboratory studies on these reagents were conducted in view of their adaptability for field determinations[3], stability, ease and sensitivity of test at different dilutions specially near permissible limit.

It is found that αα' dipyridyl is most suitable reagent under field conditions because other reagents used in the laboratory are not suitable for field application due to their complexity, unstability and other limitations. It has been found that Fe[ll] is toxicant and must be removed[4] from the water before it is used for drinking purposes. Ion exchange chelating[5,6] resin are used in removal of Fe[ll] in aqueous solution. Determination of Fe[ll] was carried out using cross linked chitosan.

Materials and Method

Synthesis of cross linked chitosan :

1.79 g (0.01 mole) of chitosan[7] was taken in a conical flask and soaked in dioxane, for one hour. 1.8 ml (0.023 mole) of epichlorohydrin and 0.92 g (0.023 mole) of sodium hydroxide was added into the conical flask with continous shaking. The conical flask was then sealed and kept in an oven for 6 hours at 50°C.

The product formed was filtered and washed with dioxane and 80% aqueous methanol containing nitric acid (to remove the inorganic impurities and excess alkali in the content). The washing was continued till the filterate was free from chloride ions and it was no more alkaline. Finally it was washed with solvent ether. The washed product was dried in an oven at 50°C for 2 hours. The crosslinked chitosan was thus formed.

It was further used for derivatization.

Characterisation of resin :

Fromation of crosslinked chitosan was characterized by Kjeldahl method which is used for nitrogen estimation, back titration method for determination of ion exchange capacity and other characteristics like bulk density, moisture content, specific bulk volume, degree of crosslinking were determined using standard methods and data are summarized in table 1.

Table 1

Nitrogen content %	Bulk density g/cm^3	Moisture content %	Specific bulk volume cm^3/g	Degree of crosslinking
5.95	o.363	3	2.75	47

The ion exchange capacity of crosslinked chitosan using back titration was also used and the value came to be 0.618meq/g of H^+ form of dry resin. pH titration curve using batch method is shown in fig. 1.

Determination of Dg value :

A batch equilibrium method was used for the metal uptake investigation by the resin. In glass stoppered conical flasks different amounts of 0.2M CH_3 COOH and 0.2M CH_3 COONa were added to get the desired pH i.e. 4 to 7. In the same way appropriate amounts of 0.2M NH_4OH and 0.2M NH_4Cl were added to get the pH of 8 to 10. 0.070 g of the dry resin and 1 ml of 1000 ppm of ferrous ammonium sulphate solution corresponding to 1mg of Fe(II) was added to each flask. The total volume in each case was kept 40 ml. The contents were stirred magnetically for 1 hour and then filtered. The filterates were analysed for Fe(II) by A.A.S. The results are given in table 2.

Dg values for Fe(II) on CL- CH resin

Table 2

pH	Concentration of Fe (II) in filterate (ppm)	Amount of Fe(II) in solution (mg)	Amount of Fe (II) in resin (mg)	Dg ml g^{-1}
4	16.7	0.6847	0.3153	269
5	12.3	0.5043	0.4957	575
6	7.0	0.2870	0.7130	1455
7	2.2	0.0902	0.9098	5907
8	3.0	0.1230	0.8770	4176
9	5.1	0.2091	0.7909	2215
10	11.5	0.4715	0.5285	656

Maximum distribution co-efficient value for Fe(II) on CL – CH resin is attained at pH 7.0 and the Dg value at pH 7.0 is 5907. The Dg values were calculated using standard formula

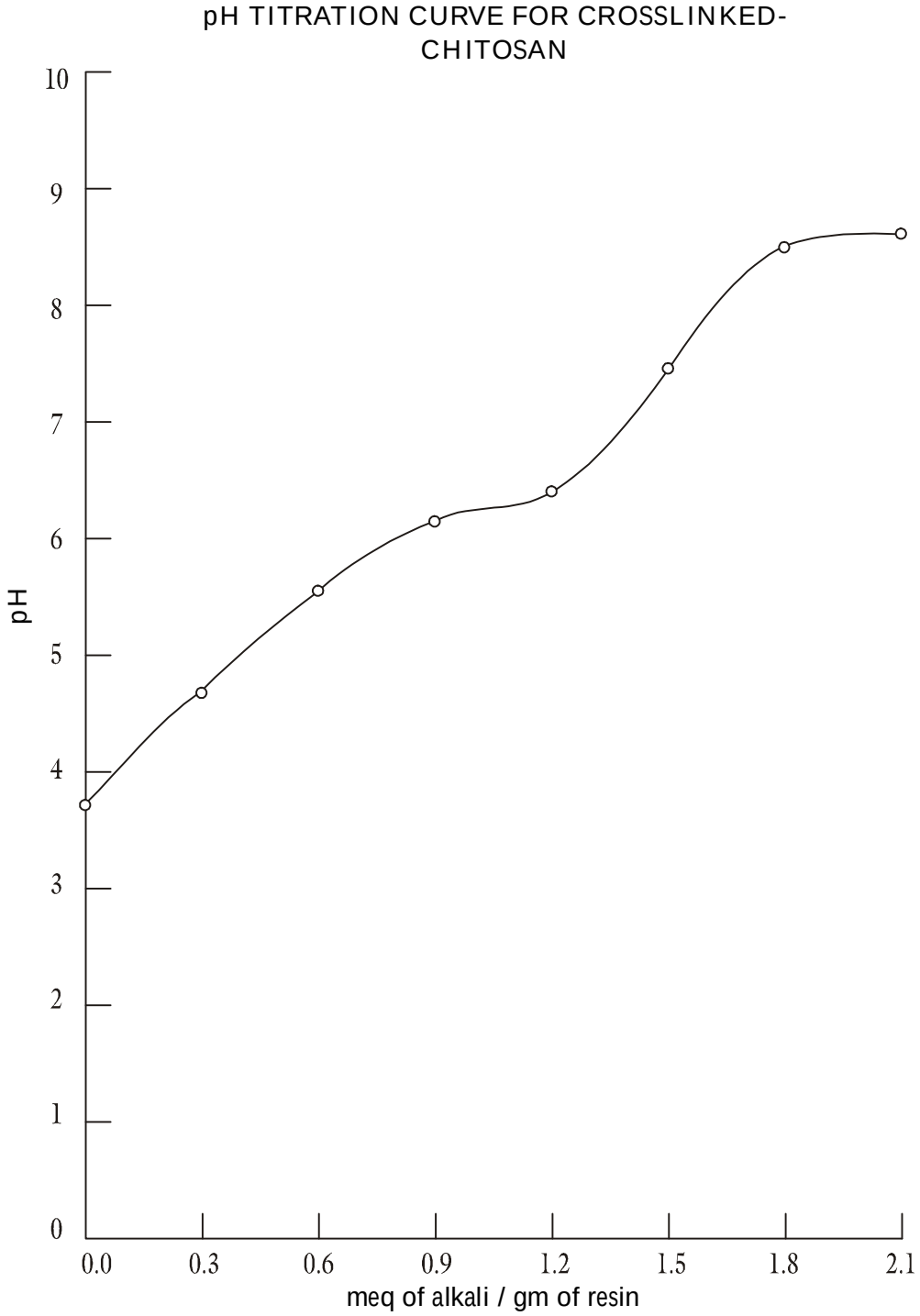
$$Dg = \frac{\text{Amount of metal ion in resin phase / gm of dry resin}}{\text{Amount of metal ion in solution / ml of solution}}$$

Determinatio of efficiency of the resin :

The efficiency of the resin was determined by column chromatography. In this method a column of 20cm height and 1.5cm diameter was taken in which upto 1 to 2cm height crosslinked chitosan was filled. For assessing the efficiency of the resin for Fe [ll], varying amount of ferrous ammonium sulphate solution of 5 to 50ppm concentration was added at pH 7.0 of its maximum adsorption. The elute were then analysed by atomic absorption spectro-photometer. The result are analysed in table 3. The advantage of chelating resin is their regeneratability. The resin is recovered by using a solution of pH more than 7.

Table 3.

Concentration of Fe(II) before adsorption (ppm)	Concentration of Fe(II) in filterate after adsorption (ppm)	Amount of Fe(II) adsorbed (ppm)	% Efficiency
5	0.3	4.7	94
10	0.58	9.42	94.2
20	1.1	18.9	94.5
30	1.8	28.2	94
40	2.2	37.8	94.5
50	2.8	47.2	94.4
60	3.6	56.4	94


pH TITRATION CURVE FOR CROSSLINKED-CHITOSAN
pH
10
9
8
7
6
5
4
3
2
1
0
0.0
0.3
0.6
0.9
1.2
1.5
1.8
2.1
meq of alkali / gm of resin

Result and Discussion

IR spectra of newly synthesised chelating derivatives were recorded using Shimadzu IR – 400 spectrophotometer. Spectrophotographic grade KBr was used for preparation of pallets.

In the IR spectra of crosslinked chitosan, primary NH_2 group gives two peaks at 3500 and 3400 cm^{-1} while O – H group gives a peak at 3600 cm^{-1}. In our case the three peaks merged together to give a broad peak at 3680 – 3000 cm^{-1} assigned to γ(N – H) and γ (O– H) vibrations, and a band at 1600 cm^{-1} is assigned to (N–H) bending.

The metal exchange capacity of Fe[ll] in meq/gm at pH 7.0 is o.2327. Maximum distribution coefficient value for Fe[ll] on cross linked chitosan resin is attained at pH 7.0 and the Dg value at this pH is 5907. At pH 7.0 efficiency of the resin for iron was determined using column chromatography. It was assessed varying the concentration of the metal at pH of the maximum adsorption. It has been found experimentally that 94% efficiency was recorded for the metal concentration in the range of 5 to 60ppm.

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References

1. R.K. Trivedy and P.K. Goel "Water pollution causes, effects and control" A book published in (1996).
2. B.L. Carson, H.V. Ellis and J. L. Mc Cann., "Toxicology and biological monitoring of metals in human," Lewis publishing Chelsea, MI, pp (328), (1987).
3. R. Gopal, T. N. Bhargava, Mankanwar Bhutra *et al*. "A field kit for examination of water quality", J. IWWA, Vol. XV, PP (59-64), Jan-March, (1983).
4. S. John and M. Ahammed, "A simple house hold method for the removal of iron from water", *J. Water SRT* – Aqua, Vol. *47*, No. 2, PP 47–49, (1998).
5. S. K. Sharma, M. R. Greetham and J. C. Schippers "Adsorption of iron(II) onto filter media", *J. AQUA*, Vol. *48*, No. *3*, June (1999).
6. L. R. Brown, Skrobot Brown, C. Teicheri, D. J. Balsingame and C. M. Lander in chitin and chitosan, The Japanese society on chitin and chitosan, Tittori, pp 227–232, (1982).
7. D. R. Nagraj, "Chelating agents in mineral processing paper presented at the special session on chelation chemistry in mineral processing", IIIth AIME Annual meeting Dallas, Feb., pp 14–18 (1982).