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RESEARCH ARTICLE

SELECTIVE SEPARATION OF IRON (III) FROM IMMOBILIZATION OF CATION EXCHANGER RESIN WITH TRIOCTYLPHOSPHINE OXIDE SORBENT.

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Abstract

Reversed – phase extraction chromatography (RPEC) is an extremely versatile technique and has been applied successfully to the separation of various metals from organic compounds. In RPEC trioctylphosphine oxide (TOPO) has been extensively used for the extraction chromatographic separation of many metal ions. This paper describes systematic investigation of the extraction chromatographic separation of Iron on a Cation exchanger resin column with triethylphosphine oxide as the stationary phase. Methods are presented for the separation of iron from a large number of elements in multi- component mixtures. The method has been extended to the determination of Iron in real samples.

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Introduction:-

The interest in the separation and determination of trace metals in natural waters has increased in the last decades because of the environmental problems and public health studies. Iron in small amounts is essential for humans and animals. Iron deficiency caused anemia and much may cause several health problems (cancer, heart disease, arthritis, diabetes and liver diseases) [1]. Iron was restricted to 2 mg L⁻¹ by World Health Organization [2] and 200 µg L⁻¹ by European Legislation [3]. Due to the very low concentration of iron and the interfering effect of the matrix, its determination demand very sensitive analytical techniques [4-6]. Separation and pre concentration were applied to overcome these difficulties. Many procedures are well characterized for such a purpose [7-8]. Of all, solid phase extraction (SPE) has attracted a great attention owing to its simple operation, rapid phase separation, no emulsification, high enrichment factor and for easily automation. Organic chelating resins [9], polymer inclusion sorbents [10], modified nanometer-sized alumina [11], C18-bonded silica gel [12], controlled-pore glass [13], activated carbon [14], ion exchange resins [15], thermal modified kaolinite [16], polymeric resin [17-26].

Experimental:-

Apparatus:-

Graduated apparatus of standard calibration were used for measurements. All glassware's were washed with nitric acid (5% v/v) new glassware's were allowed to stand for several hours in nitric acid. After three rinses with distilled water, and the glassware's were stored under dust free conditions.

Equipments:-

Spectrophotometer: Systronic India Ltd. Model No,166, pH meter, Digital Balance

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Reagents:-

All reagents used were of analytical grade. Double distilled water was used for solution preparation. The reagents used in this study were immobilization of baker yeast with trioctyl phosphine oxide polymeric resin, Ferric Chloride (99.5%, hydrochloric acid (37%.

Stock solution:-

Ferric nitrate solution. Dissolve 36.2 grams of reagent grade $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in 500 ml of one per cent HNO_3 to prepare a solution that contains approximately 10 mg of iron(III) per ml. Standardize the solution versus potassium dichromate after reducing the iron(III) to iron(II) with stannouschloride and mercuric chloride. Working solutions for trace analysis were prepared daily by appropriate dilutions.

Preparation of column extractant:-

The chromatographic column was made of a borosilicate glass tube, bore 8mm, length 25 cm, fitted with glass – wool plug at the bottom. 0.1 M TOPO solution was prepared by dissolving 1.94 g of reagent in 25 ml of water. Dowex-50 resin (BDH) (60 – 120 mesh) was dried at 120°C for 2-3 hours and stored in a desiccators. Some of the Dowex -50 resin and methylene blue indicator were mixed with each other and was packed in a U-tube through which was passed a stream of dry nitrogen that had been bubbled through a 20 ml of dimethyldichloro silane (DMCS) vapour was continued for 3-4 h. the Dowex -50 resin containing TOPO was then washed with water and dried. A slurry of the coated Dowex -50 resin in distilled water was prepared by centrifugation at 2000 rpm and be the coated resin was packed into the chromatographic column to given a bed height of 6 cm. The bed was then covered with a glass wool plug and the column extractant

General procedure:-

100 μg of Fe (III) was mixed with Hydrochloric acid in the concentration range of 0.05M to 2.4M in a total volume of 10 mL. The solution was then passed through cation exchange resin with TOPO resin column pre-conditioning with same concentration of hydrochloric acid as that of the sample solution at flow rate of 0.5 mL/min. The column was then washed with the same concentration of hydrochloric acid. The sorbed Fe (III) was then eluted with different eluting agents at the flow rate of 0.5 mL/min. Total 25 mL fractions were collected and the Fe (III) content was determined spectrophotometrically with potassium thiocyanate [17, 18] at 490 nm.

Results and Discussion:-

Adsorption of Fe (III) on cation exchange resin with TOPO resin as a function of Hydrochloric acid concentration. Sorption studies of Fe (III) were carried out from hydrochloric acid medium. The concentration of Hydrochloric acid varied from 0.05 M to 2.4 M. After sorption, Fe (III) was eluted with 2.0 M hydrochloric acid. It was found that there is quantitative (100%) sorption of Fe (III) from 0.5 M to 2M hydrochloric acid concentration. Further decrease in hydrochloric acid concentration there is decrease in sorption of Fe (III) shown in Table 1, Figure 1. The subsequent sorption studies of Fe (III) were carried out with 1.4M hydrochloric acid.

Elution study of Fe (III) with various eluting agents:-

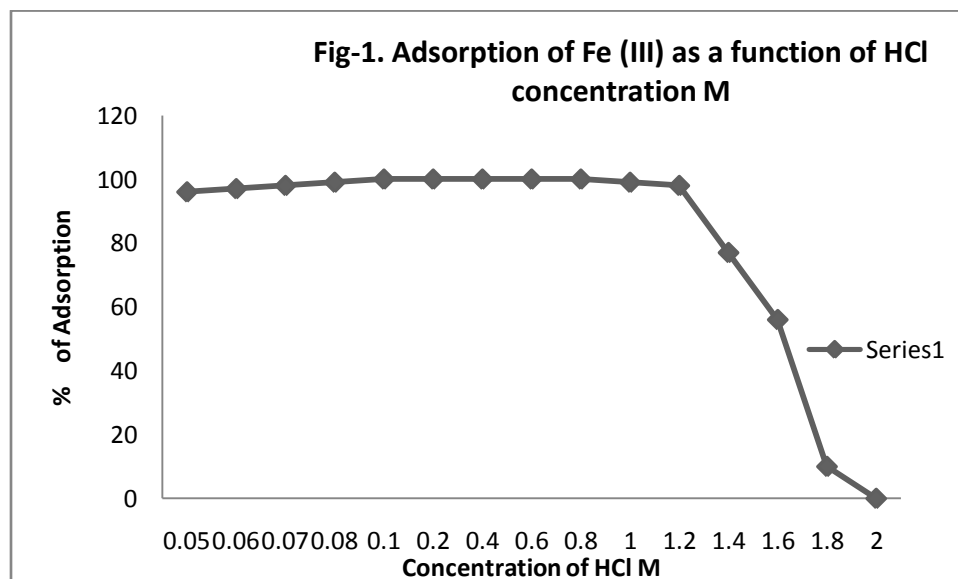
100 μg of Fe (III) in 0.05M to 2.4 M hydrochloric acid was sorbed on cation exchange resin with TOPO resin. After sorption, Fe(III) was eluted from column with various eluents such as hydrochloric acid, hydrobromic acid, sulfuric acid, perchloric acid and acetic acid. The concentration of eluting agent varied from 0.05 M to 4.0 M. Various elution studies revealed that, there is quantitative elution of Fe(III) with 25 mL of 4.0 to 8.0 M hydrobromic acid and 1.0 to 4.0 M sulfuric acid whereas perchloric acid and acetic acid were found to be inefficient eluents. Further elution studies of Fe(III) in this work were carried out with 4.0 M hydrochloric acid.

Sorption of Fe (III) on cation exchange resin with TOPO resin as a Function of hydrochloric acid concentration:-

Sorption studies of Fe (III) were carried out from hydrochloric acid medium. The concentration of hydrochloric acid varied from 0.05M to 2.4M. After sorption, Fe(III) was eluted with 4.0 M hydrochloric acid. It was found that there is quantitative (100%) sorption of Fe(III) from 0.5M to 2 M. hydrochloric acid concentration. Further decrease in hydrochloric acid concentration with decrease in sorption of Fe (III). The subsequent sorption studies of Fe(III) were carried out with 1.4 M. hydrochloric acid

Table 1:- Sorption of Fe (III) as a function of hydrochloric acid concentration.

Hydrochloric acid Concentration M	Adsorption of Fe (III) %
0.05	94
0.06	98
0.07	99
0.08	99
0.1	100
0.2	100
0.4	100
0.6	100
0.8	100
1.0	100
1.2	98
1.4	77
1.6	56
1.8	10
2.00	00

**Fig 1:-** Adsorption of Fe (III) as a function of Hydrochloric acid concentration M.**Effect of varying concentration of Fe(III):-**

The capacity of the cation exchange resin with TOPO resin for Fe (III). was evaluated using 4.0 g of the cation exchange resin with TOPO resin and sorption studies were carried out from 1.4 M hydrochloric acid and 4.0 M hydrochloric acid as an eluent. The volume of Fe (III) sample solution was 10mL. The concentration of Fe(III) was varied from 100-2000 μg of Fe(III). Per 10 mL of solution. The result shows that sorption of Fe (III) is quantitative up to 1500 $\mu\text{g}/10\text{ mL}$. Further increase in concentration of Fe(III) there is decrease in percentage sorption as shown in Table 2, The capacity of the cation exchange resin with TOPO resin for Fe (III) was found to be 0.87 ± 0.01 mmol/g cation exchange resin with TOPO resin.

Table 2:- Effect of varying concentration of Fe(III).

Concentration of Iron μg	Adsorption of Fe (III).
100	100
200	100
400	100
600	100
800	100
1000	100
1200	80
1400	65
1600	45
1800	30
2000	20

Column reuse:-

To test the long-term stability of the column containing the modified resin, successive sorption and elution cycles were carried out by passing Fe (III) solution through the column at the optimum flow rate, then eluted. The procedure was carried out several times and the stability of the column was assessed by monitoring the change in the recoveries of the sorbed Fe (III). The results of 100 sorption/desorption cycles indicate that, the recovery decreases by $\leq 3\%$ reflecting good stability of the modified resin

Determination of Iron in real sample:-

Geological sample: 0.550 g of Hematite ore was heated up to 700 °C for 2 hrs to remove organic matter and was treated with concentrated HNO_3 , the mixture was heated to moist dryness. The procedure was repeated for thrice and extracted with distilled deionized water. The hot solution was filtered, diluted and Fe (III) Content was determined by proposed method. The obtained results are shown in Table 3.

Table 3:- Determination of Fe (III). From geological samples.

Sample	Amount of Fe(III). found By		
	AAS	Proposed Method	Standard deviation ($\pm 2\%$)
Hematite ore	43%	42.8%	0.2%

Determination of Fe (III) in pharmaceutical samples:-

A tablet of drug or vitamin was digested using 5 mL of concentrated HNO_3 and dryness. After cooling, the residue was dissolved with another 5 mL of the acid. The solution was gently evaporated on a water bath till a residue was again left. It was heated with 50 mL of doubly distilled water, filtered off and completed to 100 mL in a calibrated flask. After adjusting the pH, the procedure for the determination of Fe(III) was applied (column mode). The recovered amount of Fe (III) was determined and the results are listed in Table 5.

Pharmaceutical tablets	Mineral Composition mg/tablet	Recovery % Fe(III)
Gerimax	Mg(II) 150 mg ,Fe(II) 14mg Mn(II)2.5 mg ,Zn (II)15 mg Cr(III) 0.05 mg	14 ± 0.02 100%
Centrum	Ca(II) 162 mg, Mg(II) 100 mg ,Fe(II) 27mg, Mn(II)7.5mg ,Zn (II)22.5 mg K 7.5mg, Cu 3.00 mg.	26.75 ± 0.02 99.15 %
Totavit	Cr 25 mg, Mn 2.5 mg, Fe 18 mg, Cu 2.00 mg Zn 15 mg	17.88 ± 0.02 99.65 %

Conclusion:-

A simple, rapid and selective column chromatographic method for the separation and determination of Fe (III) from other toxic metal ions using cation exchange resin with TOPO resin in hydrochloric acid medium has been developed. In addition to adopting a simple method, the method extends for the determination of Fe (III) from real samples. The results obtained by this method were in good agreement with AAS results.

References:-

1. Niederau, C.; Fischer, R.; Purschel, A.; Stremmel, W.; Haussinger, D Gastroenterology. 1996, 110, 1107–1119.
2. WHO, Rolling revision of the WHO guidelines for drinking water quality, Nutrient minerals in drinking-water and the potential health consequences of long-term consumption of demineralized and remineralized and altered mineral content drinking-waters, 2003.
3. European Community, Directive 98/83/EC on the quality of water intended for human consumption, 1998.
4. Ceccarini, A. Cecchini, I.; Fuoco, R.. Microchem. J. 2005, 79, 21–24.
5. Elci, L.; Kartal A. A.; Soylak, M. J. Hazard. Mat. 2008, 153, 454–461.
6. Mortada, W.I.; Hassanien, M. M.; El-Asmy, A. A.. Anal. Methods, 2013, 5, 530-535
7. Xia, L.B.; Wu, Y.L.; Jiang, Z.C.; Li, S.Q.; Hu, B J. Environ. Anal. Chem. 2003, 83, 953-962.
8. Kara, D.; Alkan, M. Talanta, 2001, 55, 415-423
9. Mondal, B.C.; Das, D.; Das, A.K. Talanta 2002, 56, 145-152.
10. Scindia, Y.M.; Pandey, A.K.; Reddy, A.V.R.; Manohar, S.B. Anal. Chem. 2002, 74, 4204-4212.
11. Pu, X.L.; Jang, Z.C.; Hu, B.; Wang, H.BJ. Anal. At. Spectrom. 2004, 19, 984.
12. Mahmoud, M.E.; Gohar, G.A. Talanta 2000, 51, 77-87.
13. Bruhn, C.G.; Pino, F.E.; Campos, V.H.; Nóbrega, J.A Anal. Bioanal. Chem. 2002, 374, 131.
14. Cerutti, S.; Silva, M.F.; Gásquez, J.A.; Olsina, R.A.; Martinez, L.D. Spectrochim. Acta Part B 2003, 58, 43-50.
15. Hassanien, M.M.; Hassan, A.M.; Mortada, W.I., El-Asmy A.A. Am. J. Anal. Chem. 2011, 2, 697-709.
16. Afzali, D.; Taher, M.A.; Mostafavi, A.; Mobarakeh, S.Z.M. Talanta 2005, 65, 476-480.
17. Taher, M. A. Talanta 2000, 52, 301-309.
18. Taher, M.A. Analyst 2000, 125, 1865-1868.
19. Mortada, W.I.; Hassanien, M.M.; El-Asmy A.A. J. Trace Elements in Medicine and Biology, 2013, 27, 267-272.
20. Huang, W.H.; Hu, B.; Xiong, H.C.; Jiang, Z.C.. J. Anal. Chem. 2000, 367, 254.
21. Xiong, H.C.; Hu, B.; Peng, T.Y.; Chen, S.Z.; Jiang, Z.C. Anal. Sci. 1999, 15, 737.
22. B. S. Mohite, B. E. Mahadik & S. H. Burungale, *Res. J. Chem. & Environment*, 1998, 2, 43-45.
23. B. S. Mohite, S. H. Burungale, B. E. Mahadik & D. N. Zambare, *Ind. J. Chem.*, 1998, 37A, 1035-37.
24. B. S. Mohite & S. H. Burungale, *J. Chemical & Environ. Research*, 1998, 7(3-4), 205-212.
25. S. H. Burungale International Journal of Analytical and Bioanalytical Chemistry 2015; 5(2): 48-52