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NOTE

Solid Phase Extraction of Iron Using 2′-Pyridyliminosalicyl Cellulose

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2′-Pyridyliminosalicyl cellulose has been used for the adsorption and estimation of iron by both column and batch techniques. The distribution coefficient has been determined for iron. Methods have been developed to estimate iron in medicinal tablets.

Key Words: Extraction, Iron, 2′-Pyridyliminosalicyl cellulose.

Recently cellulose immobilized with phosphoric acid and carboxymethyl group[1, 2], dithiocarbamate and 2,2′-diaminoethylamine[3] have been used for the preconcentration of lead, cadmium and some transition metals. From literature, it is apparent that Schiff bases have been largely used in the past to synthesize various stable transition and non-transition metal complexes[4–6]. 2′-Pyridyliminosalicylaldehyde has also been reported to form stable complexes with various transition metal ions[7]. The present work describes a novel method developed for the preconcentration and estimation of iron ions in various samples. In the present work, both batch and column techniques have been employed.

All chemicals and solvents used were of analytical reagent grade. Distilled deionized water was used throughout the investigation. Acetate buffer solutions were prepared by mixing acetic acid (0.2 M) and sodium acetate (0.2 M) in suitable proportions. A digital pH-meter 5651A and a Shimadzu AA-640-13 atomic absorption spectrophotometer were used. A glass tube of 100 mm length and 7 mm i.d. was used as a chromatographic column.

Preparation of immobilized cellulose: 2-Aminopyridine was immobilized on to cellulose in two separate steps as follows:

Step 1: Cellulose (10 g) was suspended in a 100 mL solution of 2-hydroxy-5-bromobenzaldehyde (HBB) (1 M) in dry ether. The mixture was refluxed on a water bath for 2 h in the presence of triethylamine; the resulting product HBB-cellulose (HBBC) was filtered, washed consecutively with ether, ethanol and acetone and then heated at 60°C for 8 h in vacuum.

Step 2: HBBC (5 g) was reacted with 50 mL of 2-amino pyridine (1 M) in dry diethyl ether with constant stirring in the presence of an acid. The solid product 2′-pyridyliminosalicyl cellulose (PSC) was filtered, washed and dried by heating in vacuum.

Procedure: Immobilized cellulose (0.1 g) was equilibrated with 30 mL (10 ppm) solution of iron in a 100 mL pyrex conical flask. Solution of iron was maintained at pH 6–6.8 by sodium acetate and the total volume of the solution was made to 32 mL. The solution was maintained at 40°C for 20 min in a thermostat. Immobilized cellulose was allowed to settle. Supernatant solution was separated and the amount of iron in supernatant solution were determined by atomic absorption spectrophotometric technique.

Glass columns were packed with 0.1 g of modified cellulose to a height of about 10 mm and then 30 mL (10 ppm) solution containing iron, maintained at the required pH 6–6.8 with sodium acetate-acetic acid buffer, were passed through the column at a flow rate of 2 mL/min adsorbed iron were then eluted from the column using 30 mL of 0.01 N HNO_3. The time required for the solid-liquid system to attain the equilibrium condition was determined at a definite temperature by placing 30 mL (10 ppm) of metal ions in a conical flask and shaking it with 0.1 g of immobilized cellulose. The supernatant solution from the flask was separated off at different time intervals. The time intervals were studied between 2 to 20 min. Unextracted iron contents were determined with atomic absorption spectrophotometry.

The speed with which the solid phase adsorbs iron from the solution and attains the equilibrium condition is of considerable importance. It was found that the time required to reach the equilibrium is 12 min. The percentage extraction of 10 ppm of iron by immobilized cellulose was best found with 0.1 g of PSC and it was maintained throughout the study. Retention of metal ions on a column packed with immobilized cellulose was studied as a function of pH. The solutions containing iron were adjusted to different pH values and passed at a rate of 2 mL/min through the column.

The adsorbed iron were eluted from the column with 0.01 N HNO_3 and determined by AAS. It was found that the maximum adsorption occurred at pH range 6–6.8. For adsorption isotherms, the distribution coefficient D, calculated for iron, is found to be 3.9×10^2 mL/g. The equilibrium constant of the complexation reaction has been found to decrease with the decrease in temperature. It has been observed that adsorption increases with increase in temperature up to 35°C and practically remains constant after that. The dependence of uptake of iron on the flow rate was studied at pH 6.5 where maximum adsorption took place. The flow rate varied from 1 to 5 mL/min. and 2 mL/min was found to be the most suitable. In order to investigate preconcentration and recovery results, column method was employed. When the volume of eluent was kept 30 mL, the percentage recovery was found in the range of 98–99%.

The effect of various electrolytes such as sodium chloride, potassium nitrate and potassium chloride on adsorption of iron on modified cellulose has no significant impact on the adsorption of metal ions.

Determination of iron contents in medicinal tablets: One tablet of the brand vitamin, or accurately weighed capsule powder to be analyzed, was placed in a 100 mL beaker and heated to slow boil with 25 mL HCl (6 N) for 15 min and then boiled gently with 10 mL HNO_3 (1 N) for 30 min. The mixture was then

diluted slightly with distilled water and filtered while hot. The solution was made up to 100 mL.

To a 2.5 mL aliquot of this sample, 2.5 mL of buffer solution of pH 6.5 was added. The volume was made up to 25 mL with distilled water and the solution was passed through a column packed with 0.1 g of PSC and then it was eluted by HNO_3 (0.1 N) and the iron content was determined by means of AAS.

The amount of iron was cross-checked by eluting the column with 20 mL HNO_3 (0.1 N). The results are represented in Table-1.

TABLE-1
DETERMINATION OF IRON USING
2′-PYRIDYLIMINOSALICYL CELLULOSE

Sample	Fe determined (mg/g)	Fe present (mg/g)	Recovery (%)
1	92.5	94.0	98.4
2	60.2	61.36	98.2
3	60.1	61.36	98.0
4	64.8	65.7	98.6

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