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Determination and Preconcentration of Iron with 4-(2pyridylazo) resorcinol (PAR)by Flotation-FAAS

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Abstract: Sensitive and environmentally friendly method for simple separation and preconcentration of Fe(III) trace in aqueous samples is presented prior to their flame atomic absorption spectrometric determinations. At pH 3;Iron (III) were separated simultaneously with 4-(2-pyridylazo)resorcinol (PAR) as a complexing agent and floated after adding sodium dodecyl sulfate(SDS) as a foaming reagent. The effects of pH, concentration of PAR, type and amount of surfactant as the floating agent, type and amount of eluting agent and influence of foreign ions on the recoveries of the analyteions were investigated. The detection limit of Fe (III) obtained 0.1 μ g L⁻¹. The proposed procedure was then successfully applied for determination of those Irons in water samples.

Introduction

The importance of heavy metals and their complex roles in biological systems come from their diverse functions and low level of existence. For instance, some of the trace heavy metals are essential to life while others are toxic even at very low concentrations. However, some of these heavy metals are of particular concern because of their toxicities to humans [1-8]. In recent years the attention in toxicology has shifted away from readily recognized, acute and usually severe diseases as a result of brief and intense exposure to toxicants, toward delayed, chronic and often less severe illnesses caused by long-term exposure to low levels of toxicants [9]. Therefore, it is worth monitoring traces and ultra traces of the toxic elements in the environmental and biological samples as well as the common ways of their entering human life [9].

Among the various kinds of analytical techniques, the flame atomic absorption spectrometry (FAAS) is one of the most widespread and classical techniques for determination of the trace elements [10]. The major advantage of this technique is its fast function for each determination but it usually suffers from the low sensitivity. Different preconcentration methods can be coupled with FAAS to improve the detection limit and the precision of the results. However, some of these methods suffer from various inconveniences. Coprecipitation method, [11,12] for instance, endure lengthy separation of the colloidal collector precipitate from the mother liquor by filtration or centrifugation. Also, the consumption of organic harmful solvents, in the course of the solvent-solvent extraction method [13,14] is not an environmental-friendly approach. These kinds of problems can be lessened by employing the ion flotation as an almost new preconcentration method. For more than a century, flotation has been used in the mining industry for the recovery of valuable substances from ores and minerals[15]. The technique has found applications in other fields of chemical engineering for removal of suspended solids, surfactants, microorganisms, and other organic contaminants from waste, industrial, sea, and drinking waters. Finally, flotation has employed in analytical chemistry for preconcentration of trace elements, since a half-century ago [16]. Nowadays, flotation is recognized as a simple and reliable preconcentration method in which the trace amount of elements invarious complex materials could be enriched without any significant environmental contamination [17, 18]. The study entails the investigation of different experimental variables that may affect the separation and the determination of this heavy metal. The proposed methodology was successfully applied to the determination of Fe(III) ion in environmental samples.

2 Experimental

2.1 Instrumentation

All determinations were carried out using a Sens AA GBC double beam atomic absorption spectrometer (AAS) equipped with deuterium background correction. Iron hollow cathode lamp was used as radiation source at wavelength max. An adjustable capillary nebulizer and supplier of acetylene and air were used for the generation of aerosols and atomization. A Genway model 3510 pH/Ionmeter with a combined glass electrode was used for pH measurements. The flotation experiments were performed in a cylindrically graduated glass tube (3.0cm id40.0 cm) with a sintered glasssparger (20–30mm nominal porosity) at its bottom to generate airbubbles.

2.2. Reagents

2.2. Reagents and Solutions

All chemicals used were of analytical-reagent gradeand all solutions were prepared with double distilled water. All the plastic and glassware were cleaned by soaking in dilute HNO₃ (1+10) and were rinsed with distilled water prior to use. The surfactant, 0.3 % (w/v) SDS(Merck, Chemise AG, Switzerland) was prepared by dissolving 0.3 g of SDS in 100 mL of distilled water. Stock standard solution of Iron at a concentration of 1000 μ g mL⁻¹ was prepared by dissolving an appropriate amount of the reagent in distilled water and diluting to 100 ml in a volumetric flask. Working standard solution was obtained by appropriate dilution of the stock standard solution. The pH was adjusted by addition of 0.1mol L⁻¹HCl or 0.1 mol L⁻¹NaOH.

2.3. Flotation procedure

A combined glass electrode was immersed to a 100mL beaker of sample solution containing $10\mu g L^{-1}$ of Fe (III) ion. After 1 mL of 0.3% (m/v) solution of SDS was added, the pH of the medium was carefully adjusted to 3 with NaOH solution. Then 0.3 mL of 3.0×10^{-3} molL⁻¹PAR in methanol was introduced into the mixture and, after 20 minutes of stirring, the mixture was diluted to 75 mL with DDW. Afterward, the content of the beaker was transferred to a 100 mL separating funnel. The funnel was stoppered and vigorously shaken for 10min, and then left to rest for a few minutes to give a complete scum layer at the surface of the solution. As soon as the floated layer was perfected, the stopcock of the funnel was slowly opened to release the lower aqueous layers. The floated layer adsorbed on the inner walls of the funnel was then eluted by 2mLof 3.0 molL⁻¹ nitric acid in methanol and subjected to the spectrometric determination.

3. Results and Discussion

3.1 Influence of pH



Figure.1. Influences of pH on recovery of metal ion.

pH is an important parameter for the quantitative extraction of analytes during flotation procedure. Therefore, the effect of pH variations (in the range of 1.0–11.0) on the recovery of Fe(III) ion in the foamy layer of the sample solutions was investigated, and theobtained chart of results is presented (Fig. 1). It can be seen that the maximum recovery (88%) is obtained in a pH range of 3.It seems that in this area of pH, Fe–PAR exists as much more probable stable complex.

3.2. Influences of amount of surfactant



Figure.2. Amount of SDS on recovery of metal ion.

In order to optimize the amount of the surfactant, different concentrations of SDS ranging from 0.3-1.5mL of 0.3% were used for the flotation-separation procedure. From the results shown in (Fig. 2), it is obvious that the maximum recovery (93%) was attained with 1mL SDS for the metal ion. In fact, by decreasing the surfactant to 1 (mL), the flotation efficiency was reduced due largely to the inadequate scum layer formed on the surface. On the other hand, by increasing the SDS to 1 mL, the recoveries were moderately declined most probably as a result of forming a viscous scum layer.

3.3. Effect of PAR Concentration

Due to the amount of ligand, which is also an important factor in the preconcentration studies for the quantitative recoveries of analyte ion from the working media, the effect of PAR concentration on the reaction was determined. The concentration of PAR was varied from 0.01 to 0.3 mL⁻¹ with keeping other analytical parameters constant. The results were given in Fig. 3that show the increase absorbance with increasing 0.3 mL PAR with concentration 3×10^{-3} moll⁻¹ which gave >99.0% recovery was selected. A higher concentration of ligand cannot be used due to its solubility.



Figure.3. Effect of PAR amount on recovery of metal ion.

3.4. Influence of eluting solutions

The foamy SDS-rich phase achieved right after the flotation cannot be directly introduced to FAAS without anelution process. In fact, to obtain an exact determination, the retained ions should first be stripped by a proper eluentf rom the viscous layer. Various eluting solutions with different volumes and concentrations

were examined for this purpose. More quantitative recoveries of Fe (III) ions were attained with 1.5-2.0 mL of 3 molL⁻¹ HNO₃ in methanol, in comparison to the other eluents. Therefore, 2.0 mL of the solution was used in the remaining experiments.

3.5. Analytical figures of merit



Figure.4. Iron Calibration curve using flotation method.

The calibration curves for Fe (III) ion was obtained by floating and measuring a serie of standard solution of each of the cation under optimum conditions. The plots exhibited linear ranges of 0.2 to 2 μ gL⁻¹ ion (Fig.4). In order to evaluate the precision of the method, 10 replicate determinations of similar solutions containing 10 μ gL⁻¹ of of the metal ion was performed by FAAS after the floation procedure were applied to this. The results indicated a reasonable repeatability for the method by presenting relative standard deviation (R.S.D.) below 2.5% for the analyte. The limit of detection (LOD), defined as the analyte concentration producing a signal as equal as the blank average signal (N = 10) plus three times the standard deviation of the blanks, was found to have the values of 0.1 μ gL⁻¹ ion.

3.6. Application

In order to test the reliability of the proposed methodology suitable for the assay of the iron, it was applied to determine this concentration in water samples. The results reported in Tables 1 show that the proposed method suitable for the determination of iron in water samples.

Recovery%	Found (µg/ml)	Added (µg/ml)	Samples
-	-	-	Sea water ^a
105	42	40	
104	73	70	
-	-	-	Тар
103	31	30	water ^b
96	58	60	

a) Persian Gulf, Iran.

b)From drinking water system of Bushehr, Iran.

4. Conclusions

With the aid of flotation method and in the presence of PAR as aligand, a sensitive and effective separation for the iron ionin different water samples was attained. The major advantages of the method were the rapidity besides the excellent recoveries of the analyte. Moreover, some other individual features such as simplicity, convenience, ecological safety, and low cost were characterized for this method.

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