

Selective and sensitive spectrophotometric method for determination of sub-micro-molar amounts of aluminium ion

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Abstract

A simple and accurate spectrophotometric method for determination of trace and ultra-trace amounts of Al^{3+} ion in tap and wastewater sample has been described. Using the eriochrome cyanine R (ECR) in the presence of *N,N*-dodecyltrimethylammonium bromide (DTAB) as cationic surfactant spectrophotometric determination of Al^{3+} ion has been carried out. The Beer's law is obeyed over the concentration range of 4–400 ng mL^{-1} of Al^{3+} ion with the detection limits of 0.14 ng mL^{-1} , while the molar absorptivity of complexes is $1.19 \times 10^5 \text{ L mol}^{-1} \text{ cm}^{-1}$. The influence of type and amount of surfactant, pH, and amount of ligand on sensitivity of spectrophotometric method were optimized. The method has been successfully applied for Al^{3+} ion determination in real sample.

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1. Introduction

Aluminium compounds have anti-inflammatory and antibacterial activity and regulate lipid metabolism in the organism. It is recognized as an important toxic substance causing considerable morbidity and mortality, particularly in patients with chronic renal failure. Diseases that have been associated with aluminium include dialysis dementia, renal osteodystrophy and Alzheimer's disease, which have an effect on red blood cells, parathyroid glands and chromosomes.

The European Committee established that diluted dialysis fluids should not contain aluminium concentrations higher than $10 \mu\text{g L}^{-1}$ [1,2]. Aluminium was the most frequently investigated element due to its particular toxicity to patients with chronic renal failure, related to clinical disorders such as dematerializing osteodystrophy [3], dialysis encephalopathy [4] and aluminium-induced anemia [5].

GF-AAS and ICP-AES are accepted reference methods for determining total aluminium in natural waters. However, both methods suffer from serious matrix interferences. Moreover, the

equipment for GF-AAS or ICP-AES is much more expensive and the accurate determination of aluminium requires considerable expertise. Compared to ICP-MS, ICP-AES and Al-NMR methods, electrogravimetry and coulometric methods generally have moderate selectivity, sensitivity and speed, but strong buffering of some fixed pH is necessary to obtain reproducible data in polarography and voltammetric methods. Since the reduction of aluminium at the electrode in aqueous solutions is difficult, it cannot be easily determined by conventional voltammetry [6,7].

Nowadays, spectrophotometric methods due to advantages such as accuracy and good precision, low cost and simple operation have been applied for trace metal determination. Several reagents have been reported for the spectrophotometric determination of aluminium [8,9]. For this purpose, the widely used reagents are aluminon ($\epsilon = 1.4 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$), xylenol orange ($\epsilon = 2.1 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$), stilbazo ($\epsilon = 1.8 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$), chlorophosphonazo I ($\epsilon = 1.96 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$), and methylthymol blue ($\epsilon = 1.9 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$) [8]. These reagents do not have a high sensitivity. Although eriochrome cyanine R has high sensitivity ($\epsilon = 6.5 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$), it requires rigid control of pH and temperature [9]. In addition, for the reagents, such as chromeazurol S and pyrocatechol violet it is needed to use some surfactants for enhancement of selectivity and sensitivity

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Table 1
Characteristic performance of some reported spectrophotometric methods for aluminium determination

Ligand	I ^a	DL ^b	pH ^c	ϵ (L mol ⁻¹ cm ⁻¹)	λ_{\max} (nm)	LR ^d	Reference
HNB	Cu ²⁺ , Hg ²⁺ , UO ₂ ²⁺ , MO ⁶⁺ , Co ²⁺ , Ti ⁴⁺	–	5.5	1.66×10^4	569	0.03–1.6	[13]
CAS	–	5	5.7	1.34×10^5	625	0–0.4	[14]
PCV	–	3	–	–	–	3–10	[15]
PCV	–	–	–	6.4×10^4	710	–	[16]
PVP	NH ₄ ⁺ , SO ₄ ²⁻ , Bi ²⁺ , Cu ²⁺ , Fe ³⁺	–	4.75	1.70×10^4	510	–	[17]
BPR	–	1	–	–	623	0–0.3	[18]
PCV	–	3	5.5–10	8.9×10^4	587	–	[19]
CV	Be ²⁺ , Cr ³⁺ , V ⁵⁺ , Zr ⁴⁺	–	–	1.50×10^3	615	0.27–54	[20]
				5.30×10^3	670		
VIAZ	–	0.5–3.5	5	1.27×10^4	375	–	[21]
AS	Be ²⁺ , F ⁻ , Zr ⁴⁺	–	–	2.15×10^4	567.5	0–1200	[22]
CPI	Zr ⁴⁺ , Ti ⁵⁺	–	4–6	1.96×10^4	610	0.8	[8]
Morin	–	5.0	6.6–6.5	5.3×10^{-3}	421	0.01–5.0	[23]
TSN	Ni ²⁺ , Zn ²⁺ , Co ²⁺ , Ga ³⁺ , In ³⁺	4.0	54,600	479	0.005–1.079	–	[24]
ECR	No interferences	0.14	5	1.19×10^5	584	0.004–0.4	PW

PW: present work; HNB: hydroxynaphthol blue; CAS: chromeazurol S; CPI: chlorophosphonazo I; PVP: polyvinylpyrrolidone; CV: catechol violet; AS: azurol S; PCV: pyrocatechol violet; BPR: bromopyrogallol red; IAZ: 1-isonicotinoyl-2-salicylidenehydrazine; TSN: 2,2',3,4-tetrahydroxy-3'-sulpho-5'-nitroazobenzene.

^a Interference.

^b Detection limit (ng mL⁻¹).

^c Applicable pH range.

^d Linear range ($\mu\text{g mL}^{-1}$).

of the methods [10,11]. Moreover, using reagents such as 8-hydroxyquinoline [10] and alizarin [9] some separation techniques like extraction are required. However, some of the problems of the method based on ECR are the serious interferences by Fe(III), Mn(II) and F⁻, which widely exist in environment samples, and its sensitivity is not high enough to determine low concentrations of Al³⁺ ion. The characteristic performances of some spectrophotometric method for aluminium determination are presented in Table 1 [8,12–24].

Application of micelles in analytical chemistry involves the beneficial alteration of metal ion–ligand complex spectral properties via surfactant association. The surfactant has high capacity to solubilize an insoluble complex and/or ligands [25,26] by micellar solubilization or formation of ternary complexes containing surfactant monomers, which led to an improvement in sensitivity (molar absorptivity) and red shift (approximately 50–150 nm) [27,28] could be achieved.

The purpose of this work is to develop a sensitized and selective spectrophotometric method for trace and ultra-trace Al³⁺ ion determination in the presence of DTAB using ECR. The influence of some analytical parameters including pH, type and amount of surfactant, ligand concentration, and type of buffer were examined.

2. Experimental

2.1. Instrumentation

Absorbance measurements were carried out with a Perkin-Elmer UV–vis spectrophotometer EZ201. All pH measurements were made using pH meter Model 691 Metrohm and Titrionic model T23680 (SCHOTT Company) equipped with a combined glass–calomel.

2.2. Reagent and solution

All chemicals such as ECR, nitrate of Al³⁺, hexamethylenetetramine buffer and other cations were of the analytical grade purchased from Merck Company. A 1% (w/v) solution of all surfactants (all from Merck) including sodium dodecyl sulphate (SDS), Triton X-100, Triton X-114, Brij 35, cetyltrimethylammonium bromide (CTAB), *N,N*-dodecyltrimethylammonium bromide (DTAB) was prepared by dissolving 1 g of surfactant in 100 mL volumetric flask while stirring.

2.3. Spectrophotometric titrations

Standard stock solutions of ligand (1.0×10^{-3} M) and the Al³⁺ ions (1.0×10^{-3} M) were prepared by dissolving appropriate and exactly weighed (with an accuracy of 0.0001 g) pure solid compounds in pre-calibrated 25.0 mL volumetric flasks and diluted to the mark with deionized water. Working solutions were prepared by appropriate dilution of the stock solutions. According to the spectra reported in Fig. 1, titration of the ligand solution (9.1×10^{-5} M, 2.75 mL) at pH 5 (using hexamethylenetetramine buffer) was carried out by the addition of microliter amounts of a concentrated standard solution of the Al³⁺ ion solution (1.0×10^{-3} M) using a pre-calibrated microsyringe, followed by absorbance intensity reading at 25.0 °C at the related λ_{\max} .

2.4. Potentiometric pH titrations

All potentiometric pH measurements were made on solutions in a 75-mL double-walled glass vessel using a Titrionic model T23680 equipped with a combined glass–calomel electrode. The temperature was controlled at 25.0 ± 0.1 °C by circulating water

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