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Aluminium traces determination in biological and water samples using a novel extraction scheme combined with molecular fluorescence

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ABSTRACT

A ternary surfactant system is for the first time proposed as an extraction strategy of aluminium traces using 8-hydroxyquinoline as complexing agent and applied for selective preconcentration of this metal. The analyte was quantified in the enriched solution by molecular fluorescence. After optimization of the complexation and extraction conditions, an enrichment factor superior to 30-fold was obtained with improved sensitivity of 2.5 times compared to the conventional extraction system using only a nonionic surfactant. The calibration curve in the range of 0.853–79.87 μg L^{-1} was linear and the limit of detection was 0.281 μg L^{-1}. The proposed method was successfully applied to the determination of aluminium in biological and water samples.

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1. Introduction

Aluminium (Al) is a non-essential, toxic metal to which humans are frequently exposed and compounds containing Al have been used in manufacturing (e.g., clays, glasses, and alum) for centuries. This metal is also used to manufacture kitchen tools and pharmacological agents including antacids and antiperspirants exposing human body to this element. It has also been considered as a possible cause of renal osteodystrophy, Parkinson and Alzheimer's diseases [1–3]. The determination of very low levels of aluminium has become very important in environmental and clinical chemistry since its negative role in the human life.

Normally Al is found at low levels (μg L^{-1}) in most drinking water because it is still used as a flocculating agent in potable water treatment units. The maximum permissible content of Al in drinking water is 0.2 mg L^{-1} [4]. Therefore, it is important monitoring Al in water and other samples. Moreover, the evaluation of Al levels in biological fluids for prevention of associated diseases has attracted considerable attention in the field of clinical chemistry.

Nowadays, there are many analytical techniques for the direct detection of the Al in real samples like spectrophotometry [5–7], spectrofluorometry [8,9], flame atomic absorption spectrometry (FAAS) [10] and the graphite furnace atomic absorption spectrometry (GFAAS) [11–13]. Before analytical determination of low Al concentration levels in complex samples analysis, it is necessary separation and preconcentration steps.

The most used techniques for the separation and preconcentration of this element include solid-phase extraction [5], conventional liquid–liquid extraction [14,15], and cloud point extraction (CPE) [8,10,11], among others. CPE is becoming an important and practical application of surfactants in analytical chemistry because of the versatility in recuperation of both organic and metallic analytes. CPE has been recognized as green procedure owing to the use of inexpensive surfactant extractants, the generation of less laboratory wastes and the fact that surfactants are non-volatiles, non-toxics and non-inflammable in contrast to organic solvents [16].

To date, nonionic surfactants have been the most widely employed for CPE, although zwitterionic surfactants and mixtures of nonionic and ionic surfactants have been also used [16–17]. Clouding is ascribed to the efficient dehydration of hydrophilic portion of micelles at higher temperature condition. Additionally, it has been reported the ability of different substances to induce phase separation in aqueous solutions of bile salts as sodium cholate (NaC) at room temperature [18]. On the other hand, among cationic surfactants, cetyltrimethylammonium bromide (CTAB) constitutes undoubtedly an example of self-assembled ordered medium as micelles, and other structures and phases, having been widely employed in analytical chemistry with different purposes [19–25].

In the present work, a new extraction scheme that uses polyethyleneglycolmono-p-nonylphenylether (PONPE 5.0) as nonionic surfactant and (CTAB) as cationic and (NaC) as anionic surfactants is

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proposed for separation and preconcentration of Al(III) complexed with 8-hydroxyquinoline (8-HQ), before its determination by molecular fluorescence. Experimental variables affecting sensitivity and precision of the proposed method were in detail investigated and optimized in order its application to determinate of metal traces in biological fluids and water samples.

2. Experimental

2.1. Instrumentals

Shimadzu RF-5301PC spectrofluorometer (Shimadzu Corporation Analytical Instrument Division, Kyoto, Japan) equipped with a discharged Xenon lamp was used for recording fluorimetric measurements using semi-micro quartz cells (300 μL).

Measurements of aluminium were performed with a Shimadzu Model AA-6800 Atomic Absorption Spectrometer (Tokyo, Japan) equipped with a deuterium background corrector, EX7-GFA electrothermal atomizer and ASC-6100 autosampler. L'vov graphite tubes (Shimadzu, Tokyo, Japan) was used in all experiments. Aluminium hollow-cathode lamps (Hamamatsu, Photonics K., Japan) were employed as radiation sources. Wave length used was 309.4 nm (Slit Width: 0.5 nm) using a pyrolysis times of 10 s at 250 °C and atomization time of 3 s at 2500 °C.

Adjustments of pH were carried out using Orion Expandable Ion Analyzer pH-meter (Orion Research, MA, USA) Model EA 940 with a combined glass electrode.

Termostatized bath Arcano 78 HW-1with magnetic stirrer (Arcano, Buenos Aires, Argentine) was used for extraction in this experiment.

A centrifuge equipment (ROLCO SRL, Buenos Aires, Argentine) with an angle rotor (6-place, 3500 rpm) was used to accelerate the phase's separation process.

2.2. Reagents

Working standard Al(III) solutions were obtained by appropriate dilution of standard solution of $Al(NO_3)_3.9\ H_2O$ (E-Merck, Darmstadt, Germany) of 1000 mg L^{-1}, using ultrapure water.

A 1×10^{-3} mol L^{-1} solution of 8-HQ (E-Merck) was prepared by dissolving appropriate amount of this reagent in ethanol (Sigma Chemical Co., St. Louis, MO, United States) and was kept in refrigerator (4 °C) for one week.

Surfactant PONPE 5.0, (Tokyo Kasei Industries, Chuo-Ku, Tokyo, Japan) 50% (v/v) in ethanol (Sigma Chemical Co.), was employed without further purification.

NaC ($C_{24}H_{39}NaO_5$, Sigma Chemical Co.) 1×10^{-2} mol L^{-1}, CTAB ($C_{16}H_{33}N(CH_3)_3Br$, Tokyo Kasei Industries, Chuo-Ku, Tokyo, Japan) 1×10^{-2} mol L^{-1} and sodium dodecylsulphate (SDS, Tokyo Kasei Industries) 1×10^{-2} mol L^{-1} solutions were prepared using an adequate weight of reagents, respectively, and dissolving in ultrapure water.

A stock solution, acetic acid (1.0 mol L^{-1}) (Riedel-de Haen) was prepared by diluting appropriate amounts of this compound in ultrapure water and adjusting to pH 5.8 by adding diluted NaOH (Mallinckrodt Chemical Works, St. Louis, Mo.) solution. A 25% (*w*/*v*) NaCl (*E*-Merck, Darmstadt, Germany) solution was used, in order to adjust ionic strength.

Methanol (Sigma Chemical Co.) was used as diluents of surfactant rich phase.

All used chemicals were of analytical grade and ultrapure water was throughout used.

2.3. Samples treatment

2.3.1. Tap and beverage waters

Bottled mineral water samples were obtained from local sources. Tap water samples were freshly collected after allowing the water flow for 5 min. All samples were filtered through a 0.45 μm pore size membrane filter to remove suspended particulate matter and were stored at 4 °C in the dark. 1 mL was taken of each water sample and was subjected to the General Procedure as described in the previous section.

2.3.2. Collection and treatment of biological samples

According to regulations, all participants of present research signed the written informed consent.

The first morning urine sample was collected from occupationally unexposed subject in polystyrene test tubes, between 8 and 10 h to reduce possible circadian contributions. Sample was centrifuged for 10 min at 1000 × *g* and processed immediately after arriving to the laboratory. It is not recommended to add a none stabilizer because of the risk of incorporating analyte as impurity.

Blood sample, from the same subject (each 10 mL), was obtained by vein puncture of forearm. It was placed in two tubes, one with Li-heparin (anticoagulant) and the other without it. The tubes with anticoagulant were homogenized and centrifuged (1500 g) during 15 min. Then the clear and transparent supernatant corresponding to plasma was extracted and reserved at 4 °C until Al(III) assays. In order to accelerate the coagulation process to make the serum separation, tubes containing blood without heparin were maintained thermostated at 37 °C during 30 min. Then, systems were centrifuged (1500 g) during 15 min and the supernatant was put in polypropylene tubes with hermetic closing.

They were taken 100 μL of each sample, then were diluted to 10 mL with ultrapure water. General Procedure was applied to 100 μL of each diluted sample.

2.4. General Procedure

A volume of 0.5 mL chelating solution 8-HQ 1×10^{-3} mol L^{-1}, sample/standard containing from 0.853 to 79.87 μg L^{-1} of Al(III), 0.25 mL of buffer solution 1 mol L^{-1} (pH 5.8), 0.5 mL CTAB 1×10^{-2} mol L^{-1}, 0.5 mL NaC 1×10^{-2} mol L^{-1}, 0.10 mL PONPE 5.0 50% (v/v) and 0.5 mL NaCl 25% (*w*/*v*) were placed in a centrifuge tube. The mixture was diluted to 10 mL with ultrapure water then it was homogenized. The resultant solution was equilibrated at 70 °C for 15 min. In order to separate the phases, the turbid solution was centrifuged 10 min at 3500 rpm (1852.2 × *g*). The supernatant aqueous phase was separated with an automatic pipette. This phase was later discard. A volume of 100 μL of MeOH was added to the surfactant rich phase (200 μL). The diluted surfactant rich phase was transferred to a 300 μL quartz cell and fluorescent emission was determined at 515 nm using $\lambda_{exc} = 373$ nm (Fig.1).

2.5. Interferences study

Different amounts of ions, which may be present in samples, (1/1, 1/10, 1/50 and 1/100 Al(III)/interferent ratio) were added to the test solution containing 24.95 μg L^{-1} Al(III) and the General Procedure was applied. Interferences studies were realized in samples without addition of masking or anticoagulant agents.

2.6. Accuracy study

Adequate volume of each sample was spiked with increasing amounts of Al(III) (9.98 and 24.95 μg L^{-1}). Analyte concentrations were determined by proposed methodology.

2.7. Validation

Al(III) contents in water samples were determined by ETAAS, using operational conditions previously consigned in apparatus item.

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