



Separation and preconcentration of trace amounts of aluminum ions in surface water samples using different analytical techniques

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ARTICLE INFO

Article history:

Received 24 April 2009

Received in revised form 16 June 2009

Accepted 17 June 2009

Available online 3 July 2009

Keywords:

Aluminum

Water sample

8-Hydroxyquinoline

Triton X-114

Cloud point extraction

ABSTRACT

A separation/preconcentration of aluminum (III) (Al^{3+}) has been developed to overcome the problem of high matrix species, which may interfere with the determination of trace quantity of Al^{3+} in natural water samples. The separation of Al^{3+} in water samples was carried out from interfering cations by complexing them with 2-methyl-8-hydroxyquinoline (quinaldine) on activated silica. Whereas the separated trace amounts of Al^{3+} was preconcentrated by cloud point extraction (CPE), as prior step to its determination by spectrofluorimetry (SPF) and flame atomic absorption spectrometry (FAAS). The Al^{3+} react with 8-hydroxyquinoline (oxine) and then entrapped in non-ionic surfactant Triton X-114. The main factors affecting CPE efficiency, such as pH of sample solution, concentration of oxine and Triton X-114, equilibration temperature and time period for shaking were investigated in detail. The validity of separation/preconcentration of Al^{3+} was checked by certified reference material of water (SRM-1643e). After optimization of the complexation and extraction conditions, a preconcentration factor of 20 was obtained for Al^{3+} in 10 mL of natural water samples. The relative standard deviation for 6 replicates containing $100 \mu\text{g L}^{-1}$ of Al^{3+} was 5.41 and 4.53% for SPF and FAAS, respectively. The proposed method has been applied for determination of trace amount of Al^{3+} in natural water samples with satisfactory results.

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1. Introduction

Aluminum (Al^{3+}) is a non-essential, toxic metal to which humans are frequently exposed. Normally it is very insoluble and in most neutral natural waters its concentration is very low. In recent years, however, a large amount of Al^{3+} has been released into the environment through water acidification, waste discharge, and soils extract from acidic rain. The maximum permissible content of Al^{3+} in drinking water is only $200 \mu\text{g L}^{-1}$ [1]. Nowadays, much interest has been raised by the toxicity and biological effect of Al^{3+} [2]. Some studies suggested that Al^{3+} may be accumulated in the brain via different routes (drinking waters, food, and medicines) and interfere with the normal activities of nervous system [3,4]. This metal ion has been considered as a possible cause of renal osteodystrophy, Parkinson and Alzheimer disease [4]. The determination of very low levels of Al^{3+} has become increasingly very important in environmental

and clinical chemistry since its negative role in the human life [5]. Therefore, there is a strong need for Al^{3+} monitoring in natural water resources [6].

There are a variety of analytical techniques such as, atomic absorption spectrometry [7], inductively coupled plasma-atomic emission spectrometry (ICP-AES) and spectrofluorimetry are used for the determination of Al^{3+} in environmental samples [8,9]. However, all of the techniques are required enrichment methods for the determination of trace amounts of Al^{3+} [10].

Due to presence of interfering cations such as iron (Fe^{3+}), chromium (Cr^{3+}), copper (Cu^{2+}), lead (Pb^{2+}) and zinc (Zn^{2+}) in environmental samples or the presence of Al^{3+} below the detection limit, becomes difficult for accurate determination of Al^{3+} by spectroscopic techniques [11]. So, the separation and preconcentration steps for Al^{3+} contents are still necessary to applying simple and less expensive techniques such as spectrofluorimetry (SPF) and flame atomic absorption spectrometry (FAAS) [12,13]. The widely used techniques for the separation and preconcentration of Al include liquid–liquid extraction [14], ion exchange [15], solid–phase extraction (SPE) [16] and cloud point extraction (CPE) [17]. Separation and preconcentration based on SPE and CPE are important alternatives and have a lot of practical applications in the field of surface chemistry [16,18]. The SPE and CPE are offered the most frequently used extraction methodologies, which are simple, cheap, most

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efficient and less toxic than other extraction methods [19]. Recently, the SPE technique has become increasingly popular, by using a suitable complexing reagent which initiates the separation or elimination of interference [20]. The CPE is based on the preconcentration of metal ions after the formation of sparingly water-soluble complexes in the surfactant-rich phase prior to the determination by different spectrophotometric techniques [21,22]. The CPE coupled with spectrophotometry for the determination of Al^{3+} has also been reported [23]. In addition, the cloud point strategies can enlarge the FAAS applications, because depending on preconcentration factors obtained, its sensitivity can significantly be increases, thus making the method more advantageous when compared with those bases on direct determination using electrothermal atomic absorption spectrometry and ICP-AES techniques [24].

The complexing reagent 8-hydroxyquinoline (oxine) has been acknowledged as one of the most sensitive organic ligands used for the determination of Al^{3+} [25,26]. It forms a highly fluorescent complex, without showing any intrinsic fluorescence itself. In the extraction of Al^{3+} by complexing with oxine, the interference of Fe^{3+} , Cu^{2+} , Cr^{3+} , Zn^{2+} and other metals ions, which are made complex with oxine, can be eliminated partly by using a masking agent, but it is inconvenient in operation and not very sensitive because of the use of organic reagent [16].

The aim of present work was to develop and establish a separation/preconcentration of trace quantity of Al^{3+} in surface water (river, canal and lake) samples. For separation of Al^{3+} from other interfering cations, 2-methyl 8-hydroxyquinoline (quinaldine) was used which is selective for complexation with different metal ions adsorbed on activated silica, while for enrichment of separated Al^{3+} , a CPE was used to react with oxine and resulted complex entrapped by Triton X-114 prior to its determination by SPF technique, and for comparative purpose FAAS with nitrous oxide-acetylene flame. Several experimental variables affecting the method sensitivity and stability were investigated in detail. The proposed method has been applied for determination of trace amount of Al^{3+} in surface water samples of different origin with acceptable results.

2. Experimental

2.1. Instrumentation

A centrifuge of WIROWKA Laboratoryjna type WE-1, nr-6933 (speed range 0–6000 rpm, timer 0–60 min, 220/50 Hz, Mechanika Phyczyjna, Poland) used for centrifugation. The pH was measured by pH meter (720-pH meter, Metrohm) and global positioning system (iFinder GPS, Lowrance, Mexico) was used for sampling locations [27]. Fluorescence measurement of Al–oxine was made on a Shimadzu RF-510 (Kyoto, Japan) spectrofluorophotometer equipped with a 150 W Xenon lamp and using 1.00 cm quartz cells. Instrument excitation and emission slits were adjusted to 10 nm. The concentration of Al^{3+} in extracts was also determined by a double beam PerkinElmer model A Analyst 700 atomic absorption spectrometer (Norwalk, CT, USA) equipped with deuterium background correction. An aluminum hollow-cathode lamp was used as radiation source at wave length (nm) 309.3, slit width (nm) 1.3, and lamp current (10 mA). The Al^{3+} was measured under optimized operating conditions by FAAS with fuel (acetylene 0.45 kg cm^{-1}) and oxidant (nitrous oxide as oxidant = 1.6 kg cm^{-2}) at burner height (12.5 mm).

2.2. Reagents

Ultrapure water obtained form ELGA labwater system (Bucks, UK), was used throughout the work. The non-ionic surfactant Triton X-114 was obtained from Sigma (St. Louis, MO, USA) and was

used without further purification. Stock standard solution of Al^{3+} at a concentration of $1000 \mu\text{g L}^{-1}$ was obtained from the Fluka Kamica (Bush, Switzerland). Working standard solutions were obtained by appropriate dilution of the stock standard solutions before analysis. Concentrated nitric acid, hydrochloric acid were analytical reagent grade from Merck (Darmstadt, Germany) and were checked for possible trace Al^{3+} contamination by preparing blanks for each procedure. The 8-hydroxy quinoline and 2-methyl 8-hydroxyquinoline were obtained from (Merck), both reagents were prepared by dissolving appropriate amount of these reagents in 10 mL ethanol (Merck) and diluting to 100 mL with 0.01 M acetic acid and were kept in refrigerator (4°C) for one week. The 0.01 mol L^{-1} acetate buffer was used to control the pH of the solutions. The pH of the samples was adjusted to the desired pH (3–8) by the addition of 0.1 mol L^{-1} HCl or NaOH solution in acetate buffer. For the accuracy of methodology, certified reference material of water SRM-1643e (National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA) was used.

For analysis of trace quantity of Al^{3+} , contamination is a serious problem. Hence, the utmost care was taken in preparing standards and samples, and the working area around the instruments was kept as clean as possible, especially for the determination of Al^{3+} by SPF technique. The glass and plastic wares were soaked in 10% nitric acid overnight and rinsed many times with deionized water prior to use to avoid contamination.

2.3. Sampling

The surface water samples of different origin (river, canal and lake) were collected on alternate month in 2008 from 210 sampling sites of Jamshoro, Sindh (southern part of Pakistan) with the help of Global positioning system (GPS). The understudy district positioned between $25^\circ 19' - 26^\circ 42' \text{N}$ and $67^\circ 12' - 68^\circ 02' \text{E}$. The sampling network was designed to cover a wide range of determinates of whole district. From each sampling site, a fresh surface water samples from canal (CS), river (RS), and lake (LS) were collected from main stream of five to six different sampling points at a depth of 20–30 cm. The collection of surface water samples was performed by using Van Dorn plastic bottles (1.5 L capacity) and was kept in well stoppered polyethylene plastic bottles previously soaked in 10% nitric acid for 24 h and rinsed with ultra pure water. All water samples were filtered through a $0.45 \mu\text{m}$ pore size membrane filter to remove suspended particulate matter and were stored at 4°C .

2.4. Solid-phase extraction

For separation of Al^{3+} from different interfering cationic species present in matrices of water samples, a glass column with an inner diameter of 20 mm and a length of 25 cm was filled up to a height of about 20 cm with activated silica gel at 100°C . Prior to use, the column was preconditioned with buffer solution (acetate buffer pH 6.2), and passed 10 mL of 0.1 mol L^{-1} quinaldine solution prepared in ethyl alcohol-acetic acid at pH 6.2. A 25 mL of replicate six sample of SRM and real water samples were passed through this column to separate Al^{3+} from other interfering cations at the flow rate of $0.5 - 5.0 \text{ mL min}^{-1}$. The treated SRM and real water samples were then divided into two sub-samples of 10 mL each. After several separation experiments ($n = 20$), the column was rinsed with 10% ethanol in $0.1 \text{ mol L}^{-1} \text{ HNO}_3$ and deionized water then used for further experiments.

2.5. Cloud point extraction

For Al^{3+} preconcentration, aliquots of 10 mL of standard solutions containing Al^{3+} (in the range of $10 - 200 \mu\text{g L}^{-1}$), replicate six samples of SRM, surface water samples, treated with quinaldine to

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