Studies on the Speciation of Certain Trace Elements in Aquatic Environment

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دراسات علي تصنيف بعض العناصر الشحيحة في البيئة المائية

إعداد

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رسالة مقدمة
إلي

كلية العلوم

بخصوص الحصول علي درجة
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ABSTRACT

Two analytical techniques namely, atomic absorption and ion chromatography are used for the total content analyses of heavy elements in water and sediments from Ismaelia canal. The water samples are collected from a depth of 1.5 meter from the surface water and adjusting the pH of the samples by nitric acid to prevent the adsorption of the elements on the walls of the sample container. The elements under investigation are copper, lead, iron, nickel, cobalt, cadmium and manganese. For the analysis, using ion chromatographic technique, two different eluents: Pyridine Dicaroboxylic Acid (PDCA) and oxalic acid are used. An agreement in the detected concentrations of the elements is always obtained either on using ion chromatography technique or atomic absorption spectrometry measurements. Element speciation in natural water is important for its toxicity, bioavailability, environmental mobility and biogeochemical behavior.

In general the potential risk depends on the chemical species of the element. Ionic species of [Cr (III)] and the toxic [Cr (VI)] were separated by anion exchange ion chromatography with dionex Ion Pac. CS5A analytical column using Pyridine 2,6-Dicaroxylic acid eluent and post column reagent 1,5-Diphenylcarbazide. The pH of water samples and eluent is selected to be 6.8 to allow optimum separation and detection of both species. Total chromium in water samples is analyzed by flame atomic absorption spectrometer (FAAS). In the present work effect of pH and ionic strength of water samples on the chromatographic separation of [Cr (VI)] is studied.

Hydride generation atomic absorption spectrometry (HGAAS) is highly sensitive and accurate procedure for determination of [As (III)] and [As (V)] in environmental samples. The effect of the concentration of reducing agent sodium borohydride ($NaBH_4$), the effect of flow rate of sodium borohydride, the effect of the concentration of hydrochloric acid (HCl), the effect of the flow rate of argon

gas and the effect of the sample volume on the intensity of the signal of arsenic are studied.

Iron speciation studies of water were analyzed by ion chromatograph and atomic absorption. Iron species [Fe (II)] and [Fe (III)] were separated and detected by anion exchange ion chromatograph, whereas total iron concentration is measured by atomic absorption spectrometry.

CONTENTS

List of Abbreviations

Symbol	Meaning
IC	Ion Chromatograph
HPIC	High Pressure Ion Chromatograph
AAS	Atomic Absorption Spectrometry
HG-AAS	Hydride Generation Atomic Absorption Spectrometry
FAAS	Flame Atomic Absorption Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
PCR	Post column reaction
ppm	part per million
ppb	part per billion
ppt	part per trillion
RSD	Relative Standard Deviation
MAU	Milli Absorbance Unit
UV	Ultra violet
HCL	Hollow Cathode Lamp
PMT	Photo multiplier Tube
PAR	4-(2-Pyidylazo)-resorcinol mono sodium salt
PDCA	Pyridine 2,6 dicarboxylic acid
ATSDR	The Agency for Toxic Substances and Disease Registry

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CHAPTER (1)

INTRODUCTION AND AIM OF THE WORK

1.1. Speciation

Speciation [1, 2] analysis is defined as the separation and quantification of different oxidation states or chemical forms of a particular element. The determination of total element concentrations is not sufficient for clinical and environmental considerations. Although the total concentration of an element is still useful and essential in many areas, the determination of each species is an important task. The concentration of a toxic species is more relevant in the setting of environmental studies than is the total elemental concentration. The collection, treatment and preservation of samples for quantitative analysis of species require careful consideration and planning. Speciation analysis[3-5] is essential for predicting and modeling fate, risk, and effects while it is a must have for designing custom - tailored treatment strategies.

The speciation of an element can directly affect the efficiency of the treatment process. Speciation analysis is not as common as total elemental analysis because while speciation data is accepted by some regulators, there are no set laws or regulations on this matter. The lack of species-specific regulations is due to the absence of methods that can reliably measure the analytes of interest at the regulatory levels. For instance, the analytical methods currently available for elements such as arsenic and chromium are either not selective enough or do not provide sufficiently low detection limits. Another important issue for speciation analysis is its cost. Even though it has been shown time after time that speciation analysis can save time and money with respect to remediation and risk assessment. However, it is usually more expensive than

routine elemental analyses. One of the most important aspects of speciation analysis is the issue of preservation. This is still not possible for most analytes of interest so even the most sophisticated analytical methods for the determination of an element's speciation are "useless" if it cannot be assured that the species distribution in the sample remains unchanged between sample collection and analysis. Therefore, choosing the right preservation techniques for the right matrix is obligatory to ensure that the speciation information in the sample remains intact during shipping and storage until the analysis is performed. Analytical speciation procedures are affected by the sampling and analytical methods depending on the target species and the sample environment.

Application of speciation analysis

- **Environmental risk analysis** with its main tasks hazard identification, dose-response assessment, and exposure assessment heavily depend on detailed speciation data, since the main characteristics of hazardous components, such asecotoxicity, mobility and bioavailability are species dependent.

- **Waste management** is an area, where speciation will provide the information to facilitate remediation and to reduce costs for clean-up procedures.

- **Occupational health and hygiene** will benefit from speciation analysis by identification of the toxic species of exposure, differentiation of the different routes of exposure, biological monitoring of trace element species by measuring biomarkers, and studying the kinetics of trace element species in the body following occupational exposure.

- **Toxicology, pharmacy, medicine**, **clinical chemistry and biology** all benefit from speciation data, since biological activity, toxicity and