



Determination of the total and extractable fraction of Ni in lake sediments and natural waters of San Luis (Argentina) by FAAS using a simple solid phase extraction system



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ABSTRACT

An on-line nickel preconcentration and determination system implemented with flame atomic absorption spectrometry (FAAS) was developed. The nickel was retained on the inner walls of a column filled with ethyl vinyl acetate (EVA). The EVA was used as sorbent material for on-line solid-phase extraction (SPE) in the absence of complexing agent. The effects of sampling volume, elution conditions, sample pH and flow rate on the Ni extraction were studied. The analytical efficiency parameters obtained with this methodology were: enrichment factor (EF) of 46 times for a preconcentration time of 120 s, the limit of detection (LOD) was $3.78 \mu\text{g L}^{-1}$, and the precision for six replicate determinations (repeatability conditions) at $20 \mu\text{g L}^{-1}$ Ni concentration was 4.38% relative standard deviation. The sampling frequency for the proposed system was about 20 samples/h. The optimized procedure was applied to the determination of Ni in natural water and lake sediment samples.

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1. Introduction

Nickel is an element that is often found in water as it is used in a number of industrial processes. The exposition to this element may take place by air, soil, drinking water, food samples and mainly by skin contact. In small quantities, nickel is considered an essential element but in excess is reported as toxic. The intake in higher concentrations than permitted, can cause damage to lungs, kidneys, gastrointestinal problems, pulmonary fibrosis, and skin dermatitis, etc. [1].

The Ni determination at trace level is an important topic for the environmental defense, human health, etc. [2]. Ni is normally present at trace level concentration, as a consequence its determination i.e. in water could be carried out directly by electrothermal atomic absorption spectrometry (ETAAS) [3–5] or by inductively coupled plasma mass spectrometry (ICP-MS) [6–9], which has usually enough sensitivity. However, inductively coupled plasma optical emission spectrometry (ICP-OES) [10–20] and flame atomic absorption spectrometry (FAAS) [21–33], in order to obtain an appropriate level of sensitivity, preconcentration/separation procedures are required. In this sense solid phase extraction (SPE) takes an important role in the preconcentration step, due to its simplicity, ease of use, economical nature, environmental friendliness, determination of volatile nickel species by CVG-atomic fluorescence spectrometry (AFS) [34], etc.

The control of the sediments is very important due to its role as final receptors of contaminants in aquatic ecosystems [35]. Furthermore, the use of different digestion methods in the determination of low concentration of Ni in sediments may lead to different results and consequently to different environmental considerations [36], even if these methods are not related to the anthropic fraction of elements.

The dissolution of sample is one of the most crucial steps prior to trace element determination in solid materials. Sample preparation techniques such as hot plate and microwave digestion have widely been used for this purpose and over the years have become established standard methods (i.e.: EPA 3050 standard 3050 B, EPA 3051 standard 3051 A & B [37], ISO standard 11466 [38], The Community Bureau of Reference (BCR) of the Commission of the European Communities [39]) for trace element dissolution from a large number of matrices. The digestion techniques, however, require the use of concentrated mineral acids, high temperatures and, in the case of microwave applications, high pressures. Moreover, the overall sample preparation is basically the most time-consuming part of elemental analysis. Therefore, considerable interest has been expressed for simple and fast sample preparation procedures. The digestion methods generally provide information on the total content of trace elements in sediment without discriminating for the contribution and the level of anthropogenic pollution.

In the recent years sequential extraction procedures have been increased in attention [40]. Some authors defend simple extractions with dilute hydrochloric acid [41–43] for sediment and soil, because

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they focused on the mobile or labile phase which is related with the bioaccumulation and bioavailability. In addition dilute hydrochloric acid leach can be used as a first step to recognize and identify trace metal pollution. Thus, the use of dilute HCl acid is low cost, fast, does not require acids as HClO_4 and HF, considerable reduction of time of analysis in comparison with other extractions and more meaningful than a total digestion. In fact the use of 0.5 M HCl has been recommended and the result obtained with this is comparable with other sequential extractions. A weak acid attack removes loosely bonded metal, absorbed on clay particles (minerals) or other minerals with large surface area (i.e. Fe and Mn oxides) and metal precipitated as its salt.

The aim of the current work was to develop a simple analytical methodology for the preconcentration using solid phase extraction for on-line determination of Ni by FAAS. The effect of the chemical and physical variables on the preconcentration, including pH, sample flow rate, buffer concentration and flow rate, and eluent concentration and flow rate were evaluated and optimized. For the preconcentration of the analyte a column filled with ethyl vinyl acetate (EVA) was used.

The optimized methodology was applied to natural sample waters, lake sediments and certified reference material, (CRM) VKI QC METAL LL1 and lake sediment TRAP-LRM, from IJS. The digestion of the sediment was carried out by partial extraction using 1 M HCl and total digestion using microwave assisted digestion with nitric acid and hydrogen peroxide to compare (EPA standard 3051 A).

2. Experimental

2.1. Standards and reagents

All solutions were prepared with ultrapure water (18 M Ω cm) obtained from an EASY pure RF (Barnstead, Iowa, USA). All the glasses and plastic materials were cleaned by soaking in a 10% HNO_3 and were rinsed with distilled water prior to use.

Hydrochloric acid, puriss. p.a. ACS (Sigma-Aldrich), was used as eluent in the flow injection system and prepared by appropriate dilution with distilled water from the concentrated acid. To alkalize solutions, NaOH (Merck, Germany) was used.

Working standard solutions of the metal were prepared by a dilution of a 1000 mg L⁻¹ Ni(II) atomic spectroscopy standard solution Trace CERT® (Fluka, Switzerland.)

Buffer solution was prepared from sodium tetraborate puriss p.a. standard substance (Riedel-de Haën, Seelze, Germany) adjusted to the appropriate pH (9.00) by adding a diluted hydrochloric acid or sodium hydroxide solution. Fresh buffer solution was prepared daily.

2.2. Instrumentation

Experiments were performed using a Shimadzu Model AA-6800 atomic absorption spectrophotometer (Tokyo, Japan) equipped with a deuterium background corrector. Nickel hollow-cathode lamps (Hamamatsu, Photonics K., Japan) were employed as the radiation sources. The wavelength used was 221.47 nm for Ni. The FAAS instrumental and operating conditions are listed in Table 1. A Minipulse 3 peristaltic pump Gilson (Villiers-Le-Bell, France) was also used. Sample injection was

achieved using a Rheodyne (Cotati, CA, USA) Model 50, four-way and of six ports, two positions, and rotary valves.

Microwave-assisted acid digestion was carried out in a microwave oven Milestone START D (Milestone, Bergamo, Italy), equipped with high pressure digestion rotor in segmented format (maximum pressure 100 bar or 1440 psi) and direct temperature monitoring and control up to 300 °C in a reference vessel. Installed power 1.200 W and the operating frequency is 2.5 GHz. Tetrafluorometoxil (TFM) reaction vessels were used in all digestion procedures. The reaction vessels were cleaned by using 5 mL of concentrated nitric acid before each digestion.

2.2.1. Microwave digestion

For microwave digestion 0.25 g of each sample was transferred to 100 mL Teflon vessels. Samples were digested by making use of HNO_3 , $\text{HCl}/\text{H}_2\text{O}_2$ (3:1) acid mixtures in a microwave digestion system. Blank digest was carried out in the same way.

2.3. Sampling

The samples were collected in two stations (A, B) affected to different degrees by the rivers, located along the mouths of the La Florida lake, San Luis, Argentina.

La Florida lake, situated in the central part of Argentina, is a semi-enclosed lake that stores 105 million cubic meters of water, irrigates an area of 10,000 ha and creates a significant hydraulic power take for the city of San Luis and Villa Mercedes, while attenuates the big floods of the rivers.

It is exposed mainly to the presence of two affluent rivers, Trapiche and Grande rivers respectively.

In this work two sampling points of different degrees of potential pollution were considered, they are in the mouths of the lake. Surface sediments, the first 10 cm layer, free of animal, were taken from these points and then put into polyethylene containers, after that each sample was hand ground to a powder and dried at room temperature.

With respect to the natural water samples, immediately after collection, the samples were filtered using 0.45 μm pore diameter membrane filters. After that, the water samples were acidified to pH 2 with nitric acid and stored in glass bottles at 4 °C.

2.4. Microwave digestion

In order to determine the total metal concentration in sediments with conventional acidic digestion, we applied the EPA standard 3050 (A). An acid mixture of 69% HNO_3 (6 mL), and 30% H_2O_2 (2 mL) was added to 0.25 g of sample put into PTFE vessels and submitted to the temperature program, with the following parameters: 10 min up 200 °C ramp–10 min at 200 °C step. Finally the samples or CRMs were diluted to 100 mL and transferred to polyethylene tubes. The procedure was applied in triplicate. For each digestion, reagent blanks were obtained. The blanks were prepared in the same conditions as the samples without the sediments and CRMs.

2.5. Column preparation

Ethyl vinyl acetate is a flexible (rubbery), transparent polymeric material with good low temperature flexibility, chemical resistance (to acids, alkalis, and alcohols) and high friction coefficient properties. EVA-polymer pellets (containing 28 wt.% vinyl acetate) (LAQUIMAR, Buenos Aires, Argentina) were used to create the preconcentration minicolumn as follows: the minicolumn was prepared by placing 100 mg approximately of EVA turnings (small shaving with a media length of 1.5 mm and a media wide of 0.45 mm) into an empty cylindrical EVA tubing using the dry packing method. Small amount of quartz wool was placed on both ends of the minicolumn. Finally, the column was connected with PTFE tubing to the peristaltic pump to form the preconcentration system.

Table 1
Operating FAAS conditions.

FAAS instrumental operating conditions	
Spectral bandpass	0.2 nm
Lamp current	8.0 mA
Slit width	7.0 mm
Flow rate gas	1.8 L min ⁻¹
Wavelength of Ni	231.91 nm
Background correction	Deuterium lamp
Sample flow rate	4.5 mL min ⁻¹

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