



Analytical Methods

Preconcentration of heavy metals on activated carbon and their determination in fruits by inductively coupled plasma optical emission spectrometry



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ABSTRACT

A method of separation and preconcentration of cadmium, cobalt, copper, nickel, lead, and zinc at trace level using activated carbon is proposed. Activated carbon with the adsorbed trace metals was mineralised using a high-pressure microwave mineraliser. The heavy metals were determined after preconcentration by inductively coupled plasma optical emission spectrometry (ICP-OES). The influence of several parameters, such as pH, sorbent mass, shaking time was examined. Moreover, effects of inorganic matrix on recovery of the determined elements were studied. The experiment shows that foreign ions did not influence recovery of the determined elements. The detection limits (DL) of Cd, Co, Cu, Ni, Pb, and Zn were 0.17, 0.19, 1.60, 2.60, 0.92 and 1.50 $\mu\text{g L}^{-1}$, respectively. The recovery of the method for the determined elements was better than 95% with relative standard deviation from 1.3% to 3.7%. The preconcentration factor was 80. The proposed method was applied for determination of Cd, Co, Cu, Ni, Pb, and Zn in fruits materials. Accuracy of the proposed method was verified using certified reference material (NCS ZC85006 Tomato).

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1. Introduction

Contemporary instrumental techniques, such as inductively coupled plasma optical emission spectrometry (ICP-OES) or atomic absorption spectrometry (AAS) allow for simultaneous or sequential determination of a large number of elements. Unfortunately, concentrations of heavy metals in fruits are usually below detection limits. Therefore, the trace metals have to be preconcentrated before the measurement.

Activated carbon is still the most important sorbent due to its large surface area, high adsorption capacity, porous structure and selective adsorption. Activated carbon has been widely used for many purposes both in the laboratory and industrial settings. This is due to its ability to adsorb organic compounds and organic metal complexes. Since its introduction in analytical chemistry, enrichment of trace metals using unmodified and modified activated carbon has been carried out with very high preconcentration factors in various matrices, e.g. of water, vegetable sample, sediment, fish, meat, etc. (Cerutti et al., 2003; Chakrapani, Mahanta, Murty, & Gomathy, 2001; Jankowski, Jackowska, & Łukasiak, 2005; Jankowski, Yao, Kasiura, Jackowska, & Sieradzka, 2005; Su et al., 2003).

A literature survey revealed that enrichment of heavy metals on activated carbon can be carried out after chelation with 1,10-phenanthroline (Mikula & Puzio, 2007), bis(2-hydroxyacetophe-

none)ethylenediimine (Ghaedi et al., 2009), ammonium pyrrolidinedithiocarbamate (Daorattanachai, Unob, & Imyim, 2005), methyl thymol blue (Ensafi & Ghaderi, 2008), bis-(salicylaldehyde)-propane-1,3-diimine (Ghaedi et al., 2008), pyrocatechol violet (Narin, Soylak, Elci, & Dogan, 2000), 8-hydroxyquinoline (Cerutti, Silva, Gasquez, Olsina, & Martinez, 2003), O,O-diethylidithiophosphate (Quinaia, De Silva, Rollemberg, & Curtius, 2001), thiourea and bromide ion (Madrakian, Afkhami, & Esmaeili, 2003), 1-(2-thiazolylazo)-2-naphthol (Starvin & Rao, 2004a), diarylazobisphenol (Starvin & Rao, 2004b), pyrogallol red (Ensafi, Khayamian, & Karbasi, 2003), zinc-piperazinedithiocarbamate (Cesur, Macit, & Bati, 2000), and ethylenediamine (Li, Chang, Zou et al., 2009).

The aim of this paper was to investigate the performance of activated carbon in absence of a complexing reagent in preconcentration and separation of cadmium, copper, nickel, cobalt, lead, and zinc traces. Activated carbon with the adsorbed trace metals was mineralised using a high-pressure microwave mineraliser and metal ions were determined using ICP-OES technique.

2. Experimental

2.1. Apparatus

An optical emission spectrometer, ICP Model M (Spectro Analytical Instruments, Germany) was used for determination of Cd, Co, Cu, Ni, Pb, and Zn. The sequential spectrometer was used with fol-

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lowing parameters: frequency, 27.12 MHz; power, 1.1 kW; demountable quartz torch, Ar/Ar/Ar; coolant gas Ar, 14.0 L min⁻¹; auxiliary gas Ar, 0.5 L min⁻¹; nebulizer gas Ar, 1.0 L min⁻¹; nebulizer pressure, 2.4 bar; glass spray chamber according to Scott, sample flow rate, 1.0 mL min⁻¹; observation height 11 mm; holographic grating, 2400 grooves mm⁻¹; dispersion of grating in the first reciprocal order, 0.55 nm mm⁻¹; wavelength range of the monochromator 165–460 nm. The wavelengths (integration time) were 228.80 nm (3 s), 228.62 nm (4 s), 324.75 nm (3 s), 221.65 nm (5 s), 220.35 nm (5 s), 213.87 (4 s) for Cd, Co, Cu, Ni, Pb, and Zn, respectively.

A UniClever microwave mineraliser (Plazmatronika BM-1z, Poland) was used for dissolution of the fruits and tomato samples. An N-1570 pH-metre (Mera-Elwro, Poland) with a glass ESAGP-309 electrode was employed for measurement of pH values in the aqueous phase. A Pulverisette 14 variable speed rotor mill (Fritsch, Germany) was used to obtain a homogeneous sample.

2.2. Reagents and solutions

All chemicals were of analytical grade. All reagents were dissolved and diluted with high purity water obtained from Milli-Q system. The following reagents were used in the experiment: activated carbon (AC), powder (J.T. Baker, Phillipsburg, USA), nitric acid, hydrochloric acid, hydrogen peroxide, sodium hydroxide, nitrates (V) of sodium, potassium, magnesium, calcium, strontium, barium, aluminium, iron(III), manganese(II) (all from POCh, Gliwice, Poland), stock standard solutions of cadmium, cobalt, copper, nickel, lead, and zinc with concentration of 1000 mg L⁻¹ (Merck, Darmstadt, Germany). Potassium phthalate buffer solution was prepared by adding an appropriate amount of potassium phthalate to sodium hydroxide solution until pH 6 was obtained. Activated carbon was kept in nitric acid (1:1) solution for 24 h so as to remove the metal ions and other impurities sorbed on it. Then it was filtered and rinsed with deionized water until it was free from acid. It was dried in a drying furnace at 110 °C. The accuracy of the method was assessed by analysing the certified reference material (CRM): NCS ZC85006 Tomato (China National Analysis centre for Iron & Steel 2000).

2.3. Procedures

2.3.1. Preconcentration of activated carbon by static method

50 mg of activated carbon were added to 100 mL of solution containing 1 µg of Cd and Co, 5 µg of Pb and Ni, and 10 µg of Cu and Zn. The pH value of the obtained solution was adjusted to 6 using the phthalate buffer. Then, the solution was stirred for 20 min to facilitate adsorption of the metal ions onto the sorbent. Next, the solution was filtered through a paper filter. The paper filter covered with activated carbon was mineralised by adding 3 mL of nitric acid in microwave mineraliser in the conditions which are described in Section 2.3.2. The solution was diluted to 5 mL with water (final volume) and analysed. The concentration of metals was determined by ICP-OES. The calibration samples were prepared using 100 mL of the standard solution containing analytes in the ranges: 0.1–2 µg of Cd and Co, 0.5–10 µg of Ni and Pb, 1–20 µg of Cu and Zn. In the next step, calibration samples were prepared using preconcentration procedure described above.

2.3.2. Preparation of the plant material for the investigations

Apple and pear were selected for the analysis. The analysed material was cleaned, broken up and dried in a laboratory drier at a temperature of 50 °C for 48 h. Dried apple and pear were ground in a high-speed rotor mill (Fritsch, Germany), in order to obtain a homogeneous sample with grain diameter of 0.5 mm.

The fruit material was stored in labelled, tightly sealed polyethylene containers.

The fruit sample was mineralised in the following way: 0.5 g of the sample was digested in 6 mL of concentrated nitric acid using a microwave pressure mineraliser. The following program was applied: step 1 (time: 3 min, power: 60%), step 2 (time: 5 min, power: 80%), and step 3 (time: 7 min, power: 100%). Then, the obtained solution was diluted to a volume of about 50 mL. Next, the sample was prepared using the preconcentration procedure. The same procedure was used for the blank solutions.

In the case of certified reference material (NCS ZC85006 Tomato), 0.5 g of the sample was digested in 6 mL of concentrated nitric acid using a microwave pressure mineraliser and the program described above.

3. Results and discussion

3.1. Effect of pH

The influence of pH on the recovery was studied in the range from 2 to 11. The effect of pH on the recoveries of the studied ions is shown in Fig. 1. It can be seen that all the metal ions were adsorbed poorly at pH < 3. A quantitative recovery (>90%) for the studied ions was obtained in the pH range of 6–11 for Cd, Co and Ni, 5–11 for Cu, 4–6 and 10–11 for Pb and 6–7 and 10–11 for Zn. So, a pH of 6.0 was selected as the compromising condition. The adsorption experiments were performed in triplicates.

3.2. Effect of the mass of sorbent

The effect of mass of the sorbent activated carbon on the sorption of cadmium, cobalt, copper, nickel, lead, and zinc was tested in the range of 25–150 mg. The results showed that a quantitative adsorption for metal ions was obtained in the range of 25–150 mg. In the subsequent experiment, 50 mg of AC were used.

3.3. Effect of stirring time

In this work, different stirring time (range from 10 to 120 min) was studied for the extraction of Cd, Co, Cu, Ni, Pb, and Zn by activated carbon. It was found that the recovery of the metal ions is close to 100% even if adsorption process was performed for 20 min. Therefore, the stirring time of 20 min was chosen as the adsorption equilibrium time. It indicated that kinetics of equilibrium is very fast.

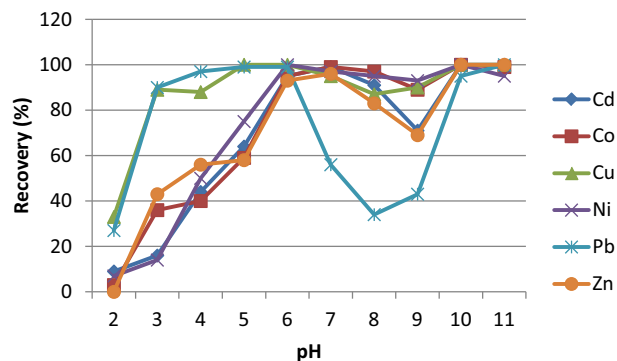


Fig. 1. Effect of pH on adsorption of the elements studied.

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