

Direct determination of toxic trace metals in honey and sugars using inductively coupled plasma atomic emission spectrometry

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Abstract

A rapid method for the determination of Pb, Cd, Cu, Cr, Co, Ni, Mn and Zn in honey and sugars without prior digestion or ashing of the sample was developed, using inductively coupled plasma atomic emission spectrometry (ICP-AES). The critical instrumental parameters such as sample flow rate and radio frequency incident power were thoroughly optimized. The effect of matrix type and its concentration was also examined for glucose/fructose, sucrose and honey matrices. The sensitivity was investigated using calibration curves obtained in presence of the above matrices. The obtained recoveries for Cd, Cu, Cr, Co, Ni and Mn at the $\mu\text{g l}^{-1}$ level were satisfactory and practically independent of the matrix used for the calibration standards. The recoveries of Pb and Zn were less sufficient. Various commercial samples of honey, sugar, glucose and fructose were analyzed with respect to their toxic metal content. The method can be applied for routine analysis, quality and environmental pollution control purposes at the $\mu\text{g l}^{-1}$ level of concentration, after suitable dilution of the samples.

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1. Introduction

Honey is high-viscosity liquid foodstuff containing a range of nutritiously important complementary elements. It contains a mixture of carbohydrates, such as fructose (25–45% m/m), glucose (25–37% m/m), maltose (2–12% m/m), sucrose (0.5–3% m/m) with traces of many other sugars depending on the floral source and water (15–18% m/m) [1]. The mean content of mineral substances in honey has been calculated to be 0.17% m/m, although it varies within a wide range. Honey is the result of a bio-accumulation process useful for the collection of information related to the environment where the bees live. Since the forage area of the hive is very large (more than 7 km^2) and the bees come in contact not only with air but also with soil and water, the concentration of heavy metals in honey reflects their amount in the whole region. Therefore, honey has been recognized as a biological indicator of environmental pollution [2]. But nevertheless, determi-

nation of heavy metals in honey is of high interest mainly for quality control and nutritional aspect. High levels of metals are undesirable because of their known or supposed toxicity, so that, for instance a limit of 1 mg kg^{-1} for lead is set in some countries [3].

Furthermore, sugars (such as glucose, fructose or sugar) are classified as generally recognized safe food ingredients. Because of their extended usage, high concern exists about the contribution of these ingredients to the total dietary intake of trace metals that may be present as contaminants. Specific attention has focused on lead because of increasing knowledge of adverse health effects from lead at various levels of exposure [4]. However, these ingredients also serve as potential sources of exposure to other metals, such as cadmium, copper and tin, which occur at various levels in the environment [5,6].

Metals determination in sugar-rich foodstuffs has been a challenging analytical task due to the interference arising from the matrix. Sample pre-treatment is usually required to destroy the organic matrix and to extract the metal ions bound in organic complexes. Obviously, the selection of the digestion procedure must take into account the analytes, the

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sample matrix and the time requirements of the analytical technique considered. Several techniques have been proposed for the determination of heavy metals in honey and other sweeteners, but in most cases the matrix mineralization is required [3,4,7]. However, in common mineralization procedures there are risks of contamination or analyte loss as a result of prolonged sample manipulation and heating.

Flame atomic absorption spectrometry (FAAS), due to its relatively low cost and quite good analytical performance has been widely used for determination a various metals in honey [8–11]. The conventional way to carry such determinations involves a mineralization stage to obtain a final solution suitable for introduction into the flame nebulizer [9]. The destruction of the organic matter eliminates both spectral interferences and the accumulation of residues in the burner head and spray chamber. However, a drawback is posed since the linear response range of the AAS is narrow. Difficulties associated to the high organic matter content are overcome by dissolving the samples in acidified water and then directly introducing the resulting solution in the nebulizer [10]. Electrothermal atomic absorption spectrometry (ETAAS) has been proposed as a more sensitive analytical technique compared to FAAS for the determination of toxic metals in honey and sugars [1,12].

Inductively coupled plasma-based techniques (ICP-AES and ICP-MS) have been applied as multi-elemental techniques for the determination of heavy metals in honey and other sweeteners [7,13–16]. ICP-AES is attractive for trace analysis, owing to the satisfactory sensitivity coupled with the advantage of simultaneous determinations of several metals at several spectral lines. However, the great disadvantage of sample dry or wet digestion still remains. Hence, the sample is usually digested by wet-acid or dry ashing by heating in a microwave oven or a furnace.

The aim of this study was to investigate the possibility of the simultaneous and direct measurement of heavy metals in honey and sugars using ICP-AES without any dry or wet sample dissolution. The effect of the matrix type and its concentration was examined using aqueous (AQ) matrix and three different carbohydrate matrices: a 1 + 1 mixture of glucose and fructose mixture (GF), sucrose (SU) and honey (HO). The sensitivity of the method with respect to each metal was evaluated using the resulted slope of the calibration curves. As there are not readily available standard reference materials [17,18], the recoveries of the analytes were measured at different concentrations using spiked solutions and used as estimation of the accuracy of the method. The precision was evaluated by measuring the repeatability of the method for all analytes and at different sugar matrixes. The capability of the method as a routine analysis method was estimated through the determination of the detection limits of every heavy metal studied. The proposed method was applied in a number of commercial samples of honey, sugar, glucose and fructose.

Table 1
Operating conditions and description of the ICP-AES instrument

rf generator	40 MHz, free-running
rf incident power	Optimized
Torch, injector, i.d.	Fassel type, Alumina, 2.0 mm
Argon flow rates	Auxiliary 0.5 l min ⁻¹ ; nebulizer 0.85 l min ⁻¹ ; plasma 15 l min ⁻¹
Air flow rate	18 l min ⁻¹
Spray chamber	Scott double-pass
Nebulizer	Gem tip cross flow
Sample propulsion	Peristaltic pump, three channel
Sample flow rate	Optimized
Polychromator/resolution	Echelle/0.006 nm at 200 nm
Detector	Segmented-array charge-coupled (SCD)

2. Experimental

2.1. Instrumentation

A Perkin Elmer Optima 3100 XL axial viewing inductively coupled plasma atomic emission spectrometer was used, according to the operating conditions given in Table 1. The analytical wavelengths for each analyte was, Pb: 220.353, 217.000, 261.418 nm; Cd: 214.440, 226.502, 228.802 nm; Cu: 324.752, 224.700, 327.393 nm; Cr: 283.563, 284.325, 267.716 nm; Co: 228.616, 238.892, 230.786 nm; Ni: 221.648, 232.003, 341.476 nm; Mn: 257.610, 259.372, 260.568 nm; Zn: 213.857, 202.548, 206.200 nm. For the optimization of the instrument's performance different radio frequency (rf) incident power levels and sample flow rates were investigated.

2.2. Reagents and solutions

All chemicals were of analytical reagent grade and were provided by Riedel de Haen. The chemical reagents used for preparation of matrix matched standards (glucose, fructose and sucrose) were of analytical grade, provided by Riedel de Haen. De-ionized water of MilliQ quality was used throughout. Mixed working standard solutions of the analytes (Pb, Cd, Cu, Cr, Co, Ni, Mg and Zn) were prepared by appropriate stepwise dilutions of stock standard solutions containing 1000 mg l⁻¹ of each analyte in 0.5 mol l⁻¹ HNO₃ (Riedel de Haen), according to the procedures described below.

Eight-point calibration curves were obtained using matrix matched standards prepared in aqueous (AQ), glucose/fructose in water (GF), sucrose in water (SU) and honey in water (HO) matrix. The final mixed working standard solutions had the following concentrations: 0.25, 0.50, 1.00, 2.50, 5.00, 10.0, 25.0 and 50 µg l⁻¹ for each metal. In case of the carbohydrate matrices, a 2% m/v solution of the corresponding sugar (glucose/fructose, sucrose, or honey) was used for dilution. In order to check the overall repeatability of the ICP-AES detector two different calibration procedures were made at two different time periods. The slope of the calibration curves was used to estimate the sensitivity of the method.

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