

Determination of cadmium, lead, iron, nickel and chromium in selected food matrices by plasma spectrometric techniques

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Abstract

In the frame of a study aimed at investigating the transfer of metal contaminants through the food chain and the effects of food processing, five elements, namely Cd, Pb, Fe, Ni and Cr, were accurately determined in (i) durum wheat grain and derived products, (ii) wheat-based reference materials, (iii) drinking water, used both as an ingredient and for technological purposes in the investigated industrial process. Microwave closed vessel digestion was selected as the dissolution technique for solid samples, whereas water samples were acidified with ultrapure nitric acid and analysed directly. As several analytes had to be quantified at trace or ultratrace levels, inductively coupled plasma mass spectrometry (ICP-MS) was resorted to for analytical determinations. Overall, this straightforward analytical approach enabled to detect the often small changes in element concentration associated with the different technological steps of processing. Nevertheless, detection of heavily interfered elements, especially Cr, as well as analyte quantification at ultratrace-level level in water, posed analytical challenges that required suited analytical solutions.

Changes in the sample introduction system and complementary use of inductively coupled plasma atomic emission spectrometry (ICP-AES) straightforwardly overcame the difficulties in determining the analytes in the selected food matrixes. The benefits of ultrasonic nebulization in reducing the effects of problematic spectral interferences were demonstrated. Overall, a robust and high-throughput analytical method was outlined.

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1. Introduction

The studies on the transfer of chemical contaminants through the food chain provide useful information for the development of surveillance programs aimed at ensuring the safety of the food supply and minimising human exposure to toxic agents. Knowledge of the routes enabling the carrying on of environmental contaminants such as cadmium and lead in the food chain from staples to consumer is an essential component of an effective system for the prevention and control of food risks. For this purpose, the identification of any possible source of food contamination during processing is also required.

In the frame of a research activity on metal contaminants in the durum wheat food chain, a study on the effects of processing on five selected elements, namely Cd, Cr, Fe, Ni and Pb, was carried out by our group [1]. Cd and Pb are well-known toxic elements regulated by the European legislation on food contaminants [2], while Cr, Fe and Ni were included in the study as markers of metal release from equipment during processing. The above-mentioned elements have been determined in cereal matrixes (durum wheat grain and derived products) and in the drinking water used both as an ingredient and for technological purposes in the investigated industrial process.

The need of quantifying several analytes at trace or ultratrace levels required a highly sensitive multielemental technique and inductively coupled plasma mass spectrometry (ICP-MS) was resorted to for analytical determinations.

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However, even though the sensitivity of ICP-MS was adequate to trace the often small changes in element concentration associated with the different technological steps of processing, the detection of heavily interfered elements, especially Cr, as well as analyte quantification at ultratrace-level level in water, posed analytical challenges that required suited analytical solutions. Various authors addressed the analytical issues connected with ICP-MS determination of low-level and/or spectrally interfered elements in cereal matrixes [3–8] and water [9–11]. Time-consuming analyte separation/preconcentration procedures [3,7,9], the employment of ‘cold’ plasma conditions [3], or the use of more complex and expensive sector field instruments [5,8,10,11] have been proposed in most of the studies carried out so far.

In the present work, the use of ultrasonic nebulization (UN) as a viable alternative to conventional nebulization not only for increasing sample introduction efficiency and thus improving analytical sensitivity, but also for the alleviation of problematic spectral interferences, was investigated. It is shown that changes in the sample introduction system and complementary use of another fast and simple atomic detector, namely inductively coupled plasma atomic emission spectrometry (ICP-AES), straightforwardly overcome the difficulties in determining the analytes in the selected matrixes.

2. Experimental

2.1. Samples and reagents

Samples of durum wheat grain, semolina (durum wheat flour), pasta and water were collected at an industrial plant for milling and pasta making as described in a separate paper [1]. Two reference materials—RM 8436 durum wheat flour, by NIST (Gaithersburg, MD), and BCR CRM 189 wholemeal flour, by IRMM (Geel, Belgium)—were included in the analytical batches to assess the accuracy of the analytical procedure.

Standard solutions were prepared by diluting commercially available mono-element stock solutions (1 g l^{-1}) (Merck, Darmstadt, Germany) with high purity deionized water (Milli-Q, Millipore, Molsheim, France). Calibration standards were 1% v/v (water analyses) or 2% (other matrixes) in ultrapure concentrated HNO_3 (Carlo Erba Reagenti, Milan, Italy). The reagents used in sample digestion were ultrapure concentrated HNO_3 and H_2O_2 (Merck). In the study of spectral interferences, standard solutions of Mo and Ca and ultrapure Na_2CO_3 (Merck) were used.

2.2. Sample preparation

Grain and pasta were homogenized for subsampling by an automatic mortar grinder (RM100, Retsch, Haan, Germany). The device bears a mortar and a pestle made of agate, which avoids the risk of sample contamination [12]. Pasta was

manually broken in small pieces before being submitted to grinding. Two aliquots of each sample were ground until a fine powder was obtained (15 min). The first aliquot was discarded, while the second one was submitted to closed vessel microwave digestion. The same dissolution technique was used for semolina. Each sample was digested in duplicate. For sample digestion, a Milestone MLS-1200 Mega MW oven (FKV, Bergamo, Italy) was used with the following irradiation program: 1 min at 250 W, 2 min at 0 W, 5 min at 250 W, 6 min at 400 W and 6 min at 650 W. About 0.35 g of fresh matter were placed in the digestion vessels with 3 and 1 ml of ultrapure concentrated HNO_3 and H_2O_2 , respectively. All samples were made up to 30 ml in polypropylene disposable tubes with high purity deionized water. Water samples were analysed as such following acidification with ultrapure HNO_3 (1% v/v).

2.3. Analyses

ICP-MS measurements were performed by a quadruple Sciex Elan 6000 spectrometer (Perkin-Elmer, Norwalk, CT, USA), equipped with an ASX-500 autosampler model 510 and an ADX-500 autodilutor (both from CETAC Technologies, Omaha, NE, USA). Two sample introduction systems were employed in this study: a cross-flow nebulizer with a Scott type spray chamber and a U-5000AT⁺ ultrasonic nebulizer (CETAC Technologies). The U-5000AT⁺ features a desolvation system consisting of a heated tube and a Peltier condenser. Temperature settings were 142 °C for the heater and 3 °C for the cooler.

Calibration was performed with external standards or the method of standard addition, depending on the matrix. Rhodium was selected as the internal standard at a concentration of $2 \mu\text{g l}^{-1}$ with UN and $20 \mu\text{g l}^{-1}$ with pneumatic nebulization (PN). Details on the instrumentation and the operating conditions are summarized in Table 1.

Table 1
Instrumental operating conditions for ICP-MS

Spectrometer	Sciex Elan 6000 (Perkin-Elmer, Norwalk, CT, USA)
Plasma	
RF generator	Frequency: 40 MHz, power output 1050–1150 W
Ar flow rate (1 min^{-1})	Plasma: 16, auxiliary: 0.9, nebulizer: 0.90–1.00
Solution uptake rate	1.8 ml min^{-1}
Interface	Ni sampler and skimmer cones, i.d. 1.1 and 0.9 mm, respectively
Data acquisition	Peak hopping; dwell time 200 ms, sweeps/reading 20, number of replicates 3
Internal standard	^{103}Rh
Analytical masses	^{114}Cd , ^{53}Cr , ^{57}Fe , ^{60}Ni , 206 , 207 , ^{208}Pb
Masses for interference correction	^{13}C , ^{43}Ca , ^{98}Mo

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