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ICP-MS analysis of a series of metals (Namely: Mg, Cr, Co, Ni, Fe, Cu, Zn, Sn, Cd and Pb) in black and green olive samples from Bursa, Turkey

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Abstract

Ninety-two black and green table olive samples from the Bursa, Turkey were analyzed. The olives were sampled from 56 brands, four processing methods and three packing types. The concentration of Mg, Cr, Co, Ni, Fe, Cu, Zn, Sn, Cd and Pb were measured by inductively coupled plasma mass spectrometry (ICP-MS). While the most concentrated element was Mg (125.11 ± 5.02), Co (0.09 ± 0.01) had the lowest concentration in tested olive samples. The levels of the ten metals studied are within safe limits. The data here obtained will be valuable in complementing available food composition data, and estimating dietary intakes of heavy metals in Turkey. The metals Mg, Fe, Zn, Sn and Pb presented significant differences ($p < 0.05$) in content between two types, hence processing method, brand and packing material must influence their content.

Keywords: Olive; ICP-MS; Metals

1. Introduction

The metal contents of food are gaining importance because of toxicological as well as their nutritional viewpoints. Dietary intake is considered to be the major supplier of these elements for the body (Demirözü, Sökmen, Uçak, Yılmaz, & Gülderen, 2002). Therefore, the levels of consumed food products should be investigated. Foods have been analyzed for different elements up to μg/kg levels using different techniques such as atomic emission spectrometry (AES), atomic absorption spectrometry (AAS), induced coupled plasma atomic emission (ICP-AES) and induced coupled plasma mass spectrometry (ICP-MS). Owing to the peculiar characteristics of ICP-MS (low detection limits, multi elemental capacity, wide linear range, etc.) the number of papers dealing with the analysis of food samples by ICP-MS has increased in recent years (Alam, Snow, & Tanaka, 2003; Barbaste, Medina, & Perez-Trujillo, 2003; Nikkarinen & Mertanen, 2004; Perez-Trujillo, Barbaste, & Medina, 2002; Roychowdhury, Tokunage, & Ando, 2003). Although there are many works published in the literature for olive oil samples, there are few works done for elements in table olives by ICP (Anthemidis, Arvanitidis, & Stratis, 2005; Angioni, Cabitza, Russo, & Caboni, 2006; Jimenez, Velarte, & Castillo, 2003; Zeiner, Steffan, & Cindric, 2005).

The elemental content of table olives could be originated from variety, location, environment, processing method, packing material and the chemical used (Garcıä, Romero, Brenes, & Garrıdo, 2002; Soares, Pereira, & Bastos, 2006). Fermentation is one of the oldest foods processing preservation technologies known to humankind. Table olives are the most popular fermented food in Turkey and worldwide production has been estimated to be 1,729,500 tonnes. Turkey contributes about 16.19% of this amount (IOOC, 2005; Panagou, Tassou, & Katzaboxakis, 2003).

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The table olives are prepared from specifically cultivated, olive varieties picked at the right maturation stage and whose quality, after appropriate processing, corresponds to that of an edible well preserved product. The most common sources of table olives are the Spanish for green olives, the Californian for oxidized black olives and naturally black olives (untreated black olives in brine and untreated black olives in dry salt).

In the Spanish and Californian procedures, olives are treated with a diluted aqueous NaOH solution that brings about several changes in the susceptible classes of compounds in the fruit. After the treatment the olives are rinsed to remove the alkali and the fruit is then left to ferment in brine for several months. The production of natural black olives is a simple, natural process, which does not use chemicals (Bianchi, 2003; Durán Quintana, García García, & Garrido Fernández, 1999; Tassou, Panagou, & Katsaboxakis, 2002; Uccella, 2001).

The aim of the study was to determine the Mg, Cr, Fe, Co, Ni, Cu, Zn, Cd, Sn and Pb content in the most commonly consumed table olives from different locations in Bursa, Turkey, in order to assess their contamination sources during their treatment processes as well as the harvesting conditions.

2. Materials and methods

2.1. Reagents

All reagent used were analytical grade purity. High quality water, obtained using a Milli-Q system (Millipore, Bedford, MA, USA), was used exclusively.

2.2. Apparatus and condition

An Agilent 7500 ICP mass spectrometer was used. The instrumental operating conditions for the determination of the elements are summarized in Table 1. Samples were digesting by acid assisted microwave irradiation using Perkin Elmer Multiwave 3000. The heating programmed employed is shown in Table 2.

Table 1
Operating conditions for ICP-MS

Instrument	Agilent 7500a
Nebulizer	Babington type
Spray chamber	Scott- type
Plasma	
RF generator	Frequency: 10 MHz, Power output 1300 W
Ar flow rate (l/min)	Plasma:15, auxiliary: 0.9, nebulizer: 1–1.1
Solution uptake rate	1.8 ml/min
Interface	
Sampler cone	Nickel, i.d.: 1.1 mm
Skimmer	Nickel, i.d.: 0.9 mm
Vacuum	İnterface:4 torr, quadrupole: 2×10^{-5} torr
Data acquisition	Peak hopping, replicate time 200 ms, dwell time 200 ms, sweeps/reading 3, readings/replicate 3, number of replicates 3
Analytical masses	^{24}Mg, ^{53}Cr, ^{57}Fe, ^{59}Co, ^{60}Ni, ^{63}Cu, ^{66}Zn, ^{111}Cd, ^{118}Sn, ^{208}Pb

Table 2
Heating program in Microwave digestion system

Power	Ramp	Hold	Fan
250	0:00	01:00	1
400	05:00	10:00	1
600	05:00	30:00	2
0		20:00	2

2.3. Sample preparation

Samples of black and green olives (Table 3) were obtained from local markets in Bursa, Turkey. The selections were specially made to reflect the popular types consumed by different groups. Olive seeds were removed by hand, and the flesh was cut into small portions with a plastic knife previously rinsed with 15% HNO_3 and doubly deionized water, packed in PVC decontaminated containers and stored in a deep-freezer at −20 °C until usage. Approximately 0.5 g of olives were accurately weighed and transferred to a Teflon container 5 ml of 65% HNO_3 (Merck, Darmstadt, Germany) and 1 ml 30% H_2O_2 (Merck, Darmstadt, Germany) were then added. After microwave digestion cycle, digestion solutions were added with high purity deionized water to adjust the final volume to 25 ml.

All samples were diluted and filtered using with 0.45 μm filters (Hydropinilic PVDF Millipore Millex-HV) before analysis. Standard metal solution were prepared daily from 1000 mg/l stock (Merck, Darmstadt, Germany) in 2% HNO_3 Suprapur grade (Merck). To avoid contamination of samples, all PTFE materials (Teflon vessels, pipets, micropipette tips, and auto sampler cups) were immersed in freshly prepared 15% v/v pro analysis HNO_3 (Merck)

Table 3
Sample description

Type	Processing method	Samples	Brand	Packing type
Black olives	Californian style	15	9	Glass (9) Plastic (2) Tin (4)
	Natural process in brine	24	17	Glass (8) Plastic (5) Tin (11)
	Natural Process in dry salt	7	5	Plastic (2) Tin (5)
Green olives	Spanish style	46	25	Glass (31) Plastic (11) Tin (4)