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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)

for 24 h, then rinsed thoroughly with doubly deionized water, and dried in a dust free area before use.

2.4. Statistical analyses

The one-way analysis of variance was applied for the statistical evaluation of the results. It was designed by using the pocket program of the Minitab version 14.0. (Minitab Inc., State College, PA, USA). LSD ($p < 0.05$) was used in the tests.

3. Results and discussion

Generally the major matrix problems are shown in the quantification of trace metals in biological sample because of effective dissolution. The edible portion of olive fruit is mainly constituted by fatty matter (the fatty matter of our samples was between 18.85% and 49.62% in the fruits), which represents a problem in the simplification of the matrix for further quantification of the metals. In previously studies, we determined optimum digestion methods and showed recovery values (99.1–100.2%) of each metal for table olive samples (Sahan, Cetinoğlu, Basoglu, & Gücer, 2004, 2005). The detection and quantification limits of elements determined are shown in Table 4.

In this study, 92 table olive samples were analyzed for ten metals. The mean (±standard deviation) and range of the concentrations of the metals in black and green olives are given in Table 5.

The Mg levels were found to be higher than other metals. The amount decreased with the order of Sn, Fe, Zn, Cu, Pb, Cr, Ni, Cd and Co, respectively. In the comparison of the concentration of trace elements among black and green olives, differences were observed. These variations could be from olive varieties, distribution of elements in the soil, maturation and processing method of olives, packing material, as well as environmental and weather conditions.

Mg was the most abundant among the elements quantified. In the black table olives levels between 36.12 ± 3.46 and 125.11 ± 5.02 were determined, whereas in green table olives were 21.61 ± 3.24 and 57.91 ± 8.62. Our results were indicated that Mg contents of olives changed according to maturation and processing method. Nergiz and Engez (2000) and Yasar and Gücer (2004) reported that Mg concentrations in green olive were in the range of 114–372 and 132.0–223.3 mg/kg, respectively. When these levels were compared with the results obtained in this study, Mg concentration in green olives was different; however different species and processed olives were examined. In addition, the metal exhibited significant differences in content among the three processing types of the black table olives. These differences are related to the differences in the processes. Thus, the use of NaOH in the Californian style may decrease the mineral concentration in these black olives.

The contents of Cr, Co and Ni were similar in all table olive samples. Also no significant differences of these metal levels were found between olive types ($p > 0.05$). Nergiz and Engez (2000) reported that the concentration of Cr decreases the maturation period in Domat variety olives. On the other hand, there were no differences in the level of Co in the same olives. Ni values (1–3 mg/kg) in wild olives were higher than the values in this study reported by Madejan, Moranon, and Murillo (2006). These differences might be explained by differences values in air and soil composition (Konarski, Hałuszka, & Ćwil, 2006; Micó, Recatalá, Peris, & Sánchez, 2006; Tasdemir, Kural, Cindoruk, & Vardar, 2006).

Table 4
Performance characteristics of the method

Metals	Detection limit (μg/kg)	Quantification limit (μg/kg)
Mg	0.110	0.170
Cr	0.003	0.020
Fe	0.120	0.200
Co	0.007	0.013
Ni	0.030	0.040
Cu	0.030	0.070
Zn	0.160	0.280
Cd	0.004	0.006
Sn	0.230	0.290
Pb	0.022	0.043

Table 5
Average values of metals on tables olive samples

Metals	Black olives (mg/kg)			Green olives (mg/kg)		
	Minimum	Maximum	Mean value	Minimum	Maximum	Mean value
Mg	36.12 ± 3.46*	125.11 ± 5.02	79.28 ± 19.58	21.61 ± 3.24	57.91 ± 8.62	37.64 ± 13.14
Cr	0.35 ± 0.13	0.88 ± 0.14	0.56 ± 0.11	0.39 ± 0.18	1.30 ± 0.43	0.59 ± 0.20
Fe	6.11 ± 1.45	48.58 ± 2.53	12.65 ± 8.44	4.45 ± 1.36	12.27± 4.67	7.08 ± 1.56
Co	0.05 ± 0.01	0.08 ± 0.01	0.06 ± 0.01	0.05 ± 0.01	0.09 ± 0.01	0.06 ± 0.03
Ni	0.18 ± 0.06	0.53 ± 0.04	0.30 ± 0.06	0.20 ± 0.09	0.50 ± 0.05	0.37 ± 0.06
Cu	0.73 ± 0.08	2.55 ± 0.22	1.48 ± 0.38	0.54 ± 0.53	1.37 ± 0.46	0.78 ± 0.20
Zn	4.25 ± 1.03	13.33 ± 1.94	8.50 ± 1.74	5.59 ± 2.01	14.30 ± 1.87	10.58 ± 2.01
Cd	0.08 ± 0.01	0.15 ± 0.01	0.11 ± 0.01	0.09 ± 0.01	0.16 ± 0.02	0.12 ± 0.04
Sn	14.40 ± 2.31	53.62 ± 3.56	35.48± 7.65	33.34 ± 4.21	47.58 ± 3.85	39.06 ± 6.42
Pb	0.57 ± 0.04	0.91 ± 0.06	0.71 ± 0.07	0.56 ± 0.03	0.86 ± 0.06	0.75 ± 0.12

* Mean value ($n = 6$) of standard division ($p < 0.05$).

The three elements Fe, Zn and Cu are essential in human nutrition and were quantified in olives. As shown in Table 5, the mean Fe levels of olive samples were varied between 4.45 and 48.58 mg/kg depending on the type, processing method, brand and packing type. Several studies have monitored the levels of Fe in olives. Our results were close to those obtained by Vavoulidou, Avramides, Papadopoulos, and Dimirkou (2004) (26–31 mg/kg), but higher than by Ziena, Yousef, and Aman (1997) (10.76–180.06 mg/kg). According to the results of other studies, maturation, variety properties and production condition influenced the value of Fe in olives (Madejan et al., 2006; Nergiz & Engez, 2000; Ziena et al., 1997). The Zn levels in analyzed olive samples are shown in Table 5. The mean Zn values belonging to black and green type were found to be 8.50 ± 1.74 mg/kg and 10.58 ± 2.01 mg/kg, respectively. In statistical evaluation, it was found that the differences were important ($p < 0.05$) among types. It might be that raw material (variety, origin of the fruit and maturation period) and different processing methods caused these differences. Among several metals, Cu is of great importance because formulations containing this element are largely used as fungicides to fight fungal disease of olive trees. Furthermore Cu is a transition metal that even in small concentrations is a very potent oxidation catalyst. Taking into account the high contents of lipids in olives, their Cu residues should be controlled to ascertain their influence in the final product quality (Soares et al., 2006). All the 92 commercial olives studied had a Cu concentration below 6 mg/kg, the limit recommended by the Turkish local food standards (TSE, 2003).

For 75 of the 92 olives (81.52%), Sn concentration was below 45 mg/kg. None of the olive samples were found to be above the limit recommended by Codex Alimentarius (1987) and Turkish local food standards (250 mg/kg). It was observed that Sn values showed relationship to packing material. This result supported the findings of another study reported that increasing concentration of Sn in food with respect to use of tinplate for food packaging (Blunden & Wallace, 2003).

The levels of the toxic metals, Cd and Pb were low, being much less than or just about 1 mg/kg in almost all samples. Cd values in the black olives were found to range between 0.08 and 0.15, similar to in green olives (0.09 and 0.16 mg/kg). The statistical evaluation shows that the variation in the results was insignificant ($p > 0.05$). Cd levels in olives were much lower from those obtained in Egypt (3.2–8.1 mg/kg) but slightly higher than those obtained in Greece (0.06 mg/kg) (Madejan et al., 2006; Ziena et al., 1997). The mean value of Pb measured in this study was 0.71 mg/kg in black olives and 0.75 mg/kg in green olives, which were below the safe limits specified for table olives by Codex Alimentarius. Contact between food and metal, such as processing equipment, storage and packaging container, is a significant source of metal in food. Once metals are present in food, their concentrations are rarely modified by traditional preparation and processing techniques, although in some cases washing may decrease the metal content (Morgan, 1999). In addition it has been reported that olives treated with chemicals contain more Pb than natural processed olives (Ziena et al., 1997). This case might be explained the Pb concentration in Californian and Spanish style olives.

The olive consumption of our country is higher than that of many countries (IOOC, 2005). Therefore this product can contribute to the overall intakes of these metals. The daily consumption of olive for an adult living in these provinces is about 15–45 g per capita. The results of the calculation (Table 6) show that dietary intake values and rates in mean toxic metal level basis olive has no significant role. The levels of the various metals are mostly below the safe limits specified for table olives by the Codex Alimentarius and Turkish local food standards.

We can thus conclude that the levels of the ten metals studied are within safety limits. The data here obtained will be valuable in complementing available food composition data, and estimating dietary intakes of heavy metals in Tur-

Table 6
Estimated intake values of metals based on consumption of table olives

Metal (mg/kg)	Mean value (mg/kg)	Daily estimated intake values*	The daily intake value established from JECFA and EVM** (mg)	Rate (%)***
		Calculated value from mean (mg)		Calculated value from mean
Mg	79.28	1.182–3.561	280	0.42–1.27
Cr	0.56	0.010–0.023	0.10	10.00–20.00
Fe	12.65	0.180–0.564	20	0.90–2.80
Co	0.06	0.001–0.002	0.012	8.30–16.60
Ni	0.30	0.004–0.013	0.13	3.07–10.00
Cu	1.48	0.022–0.073	30	0.06–0.23
Zn	8.50	0.131–0.380	22	0.59–1.72
Cd	0.11	0.002–0.005	0.06	3.33–8.33
Sn	35.48	0.534–1.601	1.8	29.44–88.88
Pb	0.71	0.010–0.030	0.21	4.76–14.28

* The dietary intake of each metal was calculated by multiplying the concentration of these metals in olive by the weights of that group consumed per capita.
** Anonymous (1984, 1993, 1994) and EVM (2003).
*** The rate (%) of each metal was calculated by division the estimated intake value to the daily intake value establish from JECFA and EVM and by multiplying by 100.

key. The metals Mg, Fe, Zn, Sn and Pb presented significant differences in content between two type, hence processing method, brand and packing material could be influence their content. In order to explain these differences in details, new studies must be performing with the same sample, which means with difference species varying the soil composition as well as studying with different processing method.

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