

Characterization of Heavy Metals in Some Edible Dried Fruits and Nuts Using Inductively Coupled Plasma Optical Emission Spectroscopy

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Abstract

The heavy metal content of dried fruits, seed and nuts show some fluctuations depending on the source of fruit, seed and nut types. The amounts of Cd and Co in air-dried fruit samples ranged from 0.05721 µg/g (white mulberry) and 0.21724 (persimmon) to 0.05101 (white mulberry) and 0.40833 µg/g (persimmon), respectively. Also, the content of Cr and Mo in dried fruit samples was found to be between 5.05 (white mulberry) and 15.85 (persimmon) and 0.01374 µg/g (white mulberry) and 0.15071 (persimmon), respectively. The heavy metal content of the fruits was different. The content of Cr, Ni and Pb in fruits is higher than other heavy metals. The content of Pb and Se in nuts and seeds is determined to be between 0.04613 (sunflower) and 0.66848 µg/g (roasted pumpkin) and 0.237 (raw almond) and 1.85 µg/g (salted peanut), respectively. The concentration order of elements in different edible seed and nuts was: $Cr > Ni > Pb > Se > Co > Cd > Mo$.

Keywords Dried fruit · Tidbits · Snack · Heavy metals · ICP-OES

Introduction

Foods with nutritional values such as minerals and vitamins, which are the basic components of human nutrition, are the most common source of toxic trace elements (Tegegne [2015\)](#page-6-0). Edible dry fruit, seed and nuts, rich in essential minerals and vitamins, are widely consumed products worldwide (Chen and Blumberg [2008;](#page-5-0) Rodushkin et al. [2008;](#page-6-1) Da Silva et al. [2014\)](#page-5-1). Edible nuts, which have positive effects on human health, are considered an important component of diets. However, contamination of metal contaminants in food through transport, food processing, and packaging contributes to the broader problem of overall food safety, with potential adverse effects from toxic elements (Roshila et al. [2007;](#page-6-2) Rodushkin et al. [2008\)](#page-6-1). Apart

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from the beneficial effect of the nutritional content of nuts and dried fruits, studies have shown protective effects on human health. Recently, there has been an increased tendency for the consumption of nuts, dried fruits and edible roasted and unroasted seeds due to their nutritional properties and beneficial effects on human health (Sabaté et al. [1996;](#page-6-3) Tošic et al. [2015\)](#page-6-4). Essential trace elements that are vital for various metabolic processes can have toxic effects if their amounts in foods are high (Gopalani et al., [2007;](#page-5-2) Nogaim et al., [2013\)](#page-5-3). Many factors such as the content of certain nutrients, their digestibility or bioavailability for an organism, the content of non-nutrients and contaminants, and the technological processes applied are the main factors affecting the quality of plant foods (Lee et al. [2010;](#page-5-4) Tighe et al. [2010\)](#page-6-5). Bioavailability of minerals can be achieved by various in vitro and in vivo methods (Skibniewska et al. [2002\)](#page-6-6). Depending on the minerals in question, various reactions and enzymatic activities have proved to be effective (Santos et al. [2013\)](#page-6-7). Some of the essential trace elements (Cr, Cu, Fe, Mn, Mo, Co, Sr, Se and Zn) act as cofactors for many physiological and metabolic functions (Yang [2009;](#page-6-8) Tošic et al. [2015;](#page-6-4) Yin et al. [2015\)](#page-6-9). Recently, studies on the risk associated with the consumption of foodstuffs containing heavy metals, pesticides and/or toxins has increased due to the growing demand for food safety (D'Mello [2003;](#page-5-5) Aydinalp and Marinova [2012\)](#page-5-6). Heavy metals, considered to be the main contaminants of food and food products,

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pose a potential danger to human health and the environment due to their great toxicity and are deemed to be one of the major problems for our environment; furthermore, they are not biodegradable, have a long biological half-life and can bioaccumulate through biological chains (Radwan and Salama [2006;](#page-5-7) Bempah et al. [2011\)](#page-5-8). Therefore, it was considered imperative to assess the level of heavy metals in edible fruits, seeds and nuts and to report possible contamination that could pose a health hazard. Fruits and seednuts, including grapes, figs, prunes, peaches and apricots, are typically dried in whole form or in slices (fruits), either under direct sun or by natural or forced convection dryers heated by solar and/or electric energy (Ratti and Mujumdar [1996\)](#page-5-9). It is necessary to determine the risk levels in terms of food safety by measuring the heavy metal content of the dried fruits and nuts consumed by humans according to various positive or negative features such as the basic composition, toxicity, pollution, and geographical origin of herbal products. The aim of present study was to profile the heavy metals of important dried fruits and nut products consumed by humans and originating from Turkey.

Material and Methods

Nut, seed and snack fruits were purchased from a market in Konya Province in Turkey. These fruits are air-dried and belong to products from 2021. Samples were transferred to the laboratory in paper bags. Hulls were broken, and the nuts, seeds and dried fruits obtained were kept until analysis.

Heavy Metal Analysis of Nut, Seed and Dried Fruit Samples

Nut, seed and dried fruit, weighing 0.2 g per sample, were dissolved in a microwave device (Cem MARSXpress 6 One Touch model, USA) under high temperature (210 °C) and pressure (200 PSI) with 5 mL concentrated $HNO₃$ and $2mL H₂O₂$ (30% w/v). A 40-cell microwave was used to ensure the reliability of the analysis. One blank and one certified reference material (1547a Wheat Flour, 8346 Condition Wheat Flour, 1547 Peach Leaves, NIST) was added to the set. The volumes of the dissolved samples were complemented to 20mL with deionized water and the concentrations of the heavy metals in the samples were determined by inductively coupled plasma optical emission spectrometry (ICP-OES; Varian-Vista Model; Tošic et al. [2015\)](#page-6-4).

Analysis of Se in Samples

Snack and dried fruit samples were dissolved in a microwave device (Cem MARSXpress) with 5mL concentrated HNO₃ and $2 \text{ mL H}_2\text{O}_2$ (30% w/v) under high temperature $(210\degree C)$ and pressure (200 PSI) to ensure the reliability of the analysis. One blank and one certified reference material (1547a Wheat Flour, 1547 Peach Leaves, 1573a Tomato Leaves, NIST) was added to the 40-cell microwave set. The volumes of the dissolved samples were filled to 30mL with deionized water and the Se concentration in the samples was measured with the ICP-OES device to which the ETC-60 (Electro Thermal Temperature Controller) and HS 60 (Hydride System) apparatus were connected. First, 10mL of the samples was taken and treated with 10mL hydrochloric acid; then the Se (+ VI) form was reduced to the Se $(+ IV)$ form by keeping it in a water bath at 90 °C for 20min, with a hydride generator installed in front of the sample inlet system of the ICP-OES device Using this module, Se measurements were made.

Statistical Analysis

Statistical analysis of the data obtained from the snacks and dried fruit samples was performed using the ANOVA test in the JMP statistics program (JMP, SAS Institute, Cary, NC, UA; Savaşlı et al. [2019\)](#page-6-10).

Results and Discussion

The heavy metal content of several dried fruits consumed as tidbits is presented in Table [1.](#page-2-0) The heavy metal content of dried fruits showed some differences depending on fruit types. While the amount of Cd in air-dried fruit samples ranged between 0.05721 µg/g (white mulberry) and 0.21724 µg/g (persimmon), the amount of Co in fruit samples was between 0.05101 µg/g (white mulberry) and 0.40833 µg/g (persimmon). Also, the amount of Cr and Mo in dried fruit samples was identified between 5.05 µg/g (white mulberry) and 15.85 µg/g (persimmon) and 0.01374μ g/g (white mulberry) and 0.15071μ g/g (persimmon), respectively. In addition, the Ni content in fruits was found to be between 1.73 (Mürdüm plum) and 3.68 (yellow raisin). While the amount of Pb in air-dried fruits was determined as 0.87 (white mulberry) and $2.52 \mu g/g$ (persimmon), the content of Se in fruit samples was between 0.184 (black raisin) and $0.910 \mu g/g$ (chestnut).

The amount of heavy metal in nuts and seeds (tidbits) consumed as tidbits is displayed in Table [2.](#page-3-0) Results showed some fluctuations depending on the source of the seeds and nuts. Cr and Ni in most seeds and nuts were found at the highest concentrations. Cd in nuts and seeds ranged between 0.00220 µg/g (sunflower) and 0.09204 µg/g (yellow roasted chickpea). Also, while the Co content in nuts and seeds was found to be between 0.01798 µg/g (sauced roasted chickpea) and 0.42943 µg/g (salted peanut), the

amount of Cr in tidbits (nuts and seeds) varied between $0.96 \mu g/g$ (Corn with oil) and $2.00 \mu g/g$ (peanut with skin and salted peanut). In addition, the content of Mo in tidbits was measured between 0.00650 (sunflower) and 0.38949 (while roasted chickpea), while the amount of Ni in nuts and seeds was recorded between 0.346 µg/g (sunflower) and $5.94 \mu g/g$ (roasted pumpkin). The Pb content in nuts and seeds was determined to be between 0.04613 µg/g (sunflower) and 0.66848 µg/g (roasted pumpkin), while the Se content in tidbit (nuts and seeds) samples was between 0.237 (raw almond) and 1.85 (salted peanut).

In general, the heavy metal content of the fruits was different: Cr, Ni and Pb was higher than the other heavy metals (Fig. [1\)](#page-4-0). This may be due to the fact that fruits with high levels of heavy metals may grow close to industrial areas and roadsides. In addition, it may have been caused by the air pollution during the drying. Also, the soil element composition and the pollution of the irrigation water used during the growing of fruits can be another source of heavy metal contamination. Cr was detected in the highest amounts, followed by Ni, Pb, Se, Co, Cd and Mo in decreasing order. Statistically significant differences were observed between the amount of heavy metals in dried fruits $(p>0.01)$. According to the results obtained from the ICP-OES analysis, in general, the highest levels of heavy metals were found in persimmon and fig fruits.

The high or low heavy metal content of raw and roasted nuts and seeds is likely due to the soil element content of the location in which the material is grown, the degree of pollution of the irrigation water and the air, the content of pesticides used for agricultural control, and processing conditions such as roasting, coating, and additives used. In general, some of the nuts and seeds are consumed raw and some of them are roasted. In addition, it is thought that the salt and spices used as coating material in nuts and seeds, and whether the material is shelled or not, affects the heavy metal content of the food products. The Cr and Ni content in nuts and seeds was higher than the other elements. The Cr, Ni and Pb concentrations showed greater variation among different types of nuts and seeds. The order of the element concentrations for different edible seeds and nuts was: Cr >Ni > Pb > Se >Co >Cd >Mo (Fig. [2\)](#page-4-1). Statistically significant fluctuations were observed between the amounts of heavy metals in seeds and nuts $(p<0.01)$.

An excessive intake of Co may cause the overproduc-tion of red blood cells (Kalagbor and Diri [2014\)](#page-5-10). Saraçoğlu et al. [\(2009\)](#page-6-11) found 0.92–6.49 µg/g Cu, 1.57–7.53 µg/g Mn, 10.4–80.1 µg/g Fe, 0.32–0.64 µg/g Se, 0.02–0.72 µg/g Cd, 0.72–3.77 µg/g Pb, 2.30–5.83 µg/g Ni, 4.76–28.9 µg/g Cr and 0.08–0.22 µg/g Al in ten apricot samples harvested from gardens in Kayseri, Turkey. Zahoor et al. [\(2003\)](#page-6-12) reported that the concentration of Cd (II) ion and Pb in apricot grown in Pakistan ranged between 0.09 and

Table 2 Heavy metal content in seed and nuts consumed as tidbits or snacks (µg/g) Table 2 Heavy metal content in seed and nuts

Fig. 2 The highest heavy metal content in seeds and nuts consumed as tidbits or snacks

dried fruits

0.21mg/kg to 6.6 and 9.2 µg/g in apricot fruits in Pakistan. Bennett et al. [\(2011\)](#page-5-11) reported that 'Sultana' raisin contained 247.4–528.5 µg/100 g Cu, 0.28–139.8 µg/100 g Se and $2.07-3.63 \mu g/100 g$ Fe. Bennett et al. (2011) determined 332.3-465.8 µg/100 g Cu, 2.60-4.94 µg/100 g Fe, 2.36–6.56 µg/100 g Mo, and 0.23–0.30 µg/100 g Se in apricot. Tegegne [\(2015\)](#page-6-0) determined 0.47, 3.13, 1.08, 2.79, 0.39 and 1.66mg/kg Cu; 3.82, 20.79, 9.02, 57.07, 4.06 and 33.53mg/kg Fe; 2.42, 3.99, 2.90, 6.76, 1.81, 3.78mg/kg Zn; 2.33, 2.69, 1.20, 4.04, 1.70 and 1.30mg/kg Mn; 0.40, 0.54, 0.51, 0.67, 0.30 and 0.45mg/kg Co; 0.30, 0.50, 0.22, 0.40, 0.17 and 0.33mg/kg Ni; 0.26, 0.39, 0.19, 0.36, 0.14 and 0.28mg/kg Cr; and 0.09, 0.31, 0.16, 0.37, 0.11 and 0.32mg/kg Pb in avocado, mango, papaya, pineapple, banana and orange, respectively. Our results are also similar in terms of the content of Mg, Fe, and Zn in buckwheat groats reported in an earlier study. High levels of heavy metals in some fruits can be caused by irrigation water, pesticides, contamination from farm soil or contamination from road traffic and other agronomic practices (Tegegne [2015\)](#page-6-0). Heavy metals such as Ni, Pb, Se, Cr, Co, Mo can cause toxic effects when consumed for a long time, even at low concentrations (Nogaim et al. [2013;](#page-5-3) Unak et al. [2007;](#page-6-13) Gopalani et al. [2007\)](#page-5-2). Santos et al. [\(2013\)](#page-6-7) reported 2.96 mg/100 g Fe, 1.4 mg/100 g Cu, 1.65 mg/100 g Mn, $11.48 \text{ mg}/100 \text{ g}$ Se and $3.51 \text{ mg}/100 \text{ g}$ Zn in Brazil nut kernel. Özcan et al. [\(2010\)](#page-5-12) reported that walnut kernels contained 5.676–9.333mg/kg Cu, 1.695–3.323mg/kg Cr, 17.875–21.815mg/kg Fe, 1.651–1.915mg/kg Ni and 17.981–20.623mg/kg Zn. Tošic et al. [\(2015\)](#page-6-4) found 10.72 and 11.42mg/kg B, 0.0182 and 0.0207mg/kg Co, 0.4087 and 0.4261mg/kg Cr, 12.32 and 11.64mg/kg Cu, 69.57 and 67.66mg/kg Fe, 12.54 and 12.09mg/kg Mn, 0.1728 and 0.1687mg/kg Mo, 0.1525 and 0.1362mg/kg Ni, 0.6583 and 0.7524mg/kg Se, 4.841 and 4.627mg/kg Si and 34.61 and 33.99mg/kg Zn in pistachios. Willoud et al. [\(2004\)](#page-6-14) determined 200 µg/g Cu, 4780Mn, 75 µg/g Ni and $236 \mu g/g$ Zn in almond; $309 \mu g/g$ Cu, $478 \mu g/g$ Mn, $44 \mu g/g$ Ni and $671 \mu g/g$ Zn in peanut; $223 \mu g/g$ Cu, 179 µg/g Mn, 64 µg/g Ni, and 298 µg/g Zn; 194 µg/g Cu, 2110 µg/g Mn, 93 µg/g Ni and 337 µg/g Zn in peanut kernels. Suliburska and Krejpcio [\(2014\)](#page-6-15) determined 5.4mg/100 g Fe, 3.0mg/100 g Zn in cashew (*Anacardium occidental*) kernel, 2.5mg/100 g Fe and 1.5mg/100 g Zn in hazelnut (*Corylus avellana*) and 2.1mg/100 g Fe and 1.8mg/100 g Zn in walnut (*Juglans regia*) kernels. Cosmulescu et al. [\(2009\)](#page-5-13) determined 3.815–5.927 mg/100 g Fe, 1.41–3.223 mg/100 g Cu, 0.001–0.005 mg/100 g Se, 0.103–0.525 mg/100 g Al, $0.255 - 0.692 \,\text{mg}/100 \,\text{g}$ Cr, and $1.948 - 3.613 \,\text{mg}/100 \,\text{g}$ Zn in walnut cultivars. Yin et al. [\(2015\)](#page-6-9) determined 0.15, 0.20, 0.0, 0.0019, 0.31, 0.0, 5.6, 0.22 and 2.2 µg/g Cr; 0.010, 0.060, 0.010, 0.0, 0.060, 0.020, 0.060, 0.13 and 0.13 and 0.050 µg/g Co; 7.2, 12.0, 7.1, 2.6, 7.0, 8.8, 4.1, 10.0 and 17.0 µg/g Cu; 15.0, 27.0, 38.0, 0.91, 24.0, 20.0, 9.9, 15.0, 41.0 µg/g Zn; 0.16, 1.1, 0.0, 0.0,0.57, 0.0, 0.0, 0.0, and 0.0 µg/g Se; 0.070, 0.23, 0.06, 0.10, 0.11, 0.21, 0.030, 0.010 and 0.21 µg/g Mo; 0.070, 0.17, 0.13, 0.030, 0.040, 0.020, 0.050, 0.24 and 0.040 µg/g Cd in pistachios, sunflower seeds, pine nuts, raisins, almond nut, walnuts, chestnuts, hazelnuts and cashews, respectively. Gülfer and Ozdemir [\(2016\)](#page-5-14) reported that sunflower seed, pumpkin seed, peanut and corn digested in $HNO₃+H2O₂$ contained 15.2, 6.0, 4.7 and 1.8mg/kg Cu, 49.0, 47.3, 18.9 and 16.2mg/kg Fe, 32.4, 34.2, 20.9 and 7.1mg/kg Zn and 0.70, 0.17, 0.34 and 0.35mg/kg Se, respectively.

The content of Pb was within the permissible limit compared to values reported in the literature. As is known, colic, anemia, headache, convulsions and chronic nephritis in the kidneys, brain damage and central nervous system disorders are typical symptoms of lead poisoning (Khan et al. [2008\)](#page-5-15). When the results were compared with the literature values, some minor differences were found. In addition, the amount of heavy metals in the analyzed foods was found to be at very low levels. The existing differences with the literature values may be due to different factors such as the geographical location of the products, plant type, precipitation, frequency of sunlight, soil differences, fertilization, precipitation, seasonal effects, grinding and dehulling (Santos et al. [2013\)](#page-6-7).

Conclusion

Seed and nut samples obtained from the market had a lower concentration of heavy metals compared with fruit samples. In general, the results indicate that the concentration of heavy metals in fruit samples obtained from the main market was in decreasing order as follows: $Cr > Ni > Pb$ $>$ Se $>$ Co $>$ Cd $>$ Mo; the concentration of heavy metals in seed and nut samples obtained from the market was in decreasing order as follows: $Cr > Ni > Pb > Se > Co > Cd$ >Mo. The presence of high amounts of heavy metals in the environment where agricultural products are grown and processed poses a potential danger to human health and to the environment due to their high toxicity in foods.

Conflict of interest D.A. Kulluk, F. Gökmen, N. Dursun, M.M. Özcan and V. Lemiashonak declare that they have no competing interests.

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