Determination of Some Trace Elements in Dried Red Plum Using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

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ABSTRACT: *This study aims to determine the trace metals of (Mg, Fe, Cu, Pb, Cr, As, Ni, Zn, and Se) in some dried red plum specimens from Turkey. The metals in the dried red plum samples were determined using ICP-OES after solubilization in microwave digestion methods. With this technique, fast and high-precision determination of trace elements was made. Validation of the proposed method was carried out by using a NIST-SRM 1515-Apple Leaves certified reference material. Element concentrations in dried red plum samples were 44–47 (Mg), 15-18 (Fe), 03.1–3.2 (Cu), 4.1–4.4 (Pb), 1.4–1.6 (Cr), 1.1–1.3 (As), 2.5–2.8 (Ni), 3.4–3.7 (Zn) and 4.2–4.5 (Se) µg/g. The results were compared with the literature values.*

KEYWORDS: *Dried red plum; Microwave digestion; Trace elements; ICP-OES.*

INTRODUCTION

Trace elements are essential for vitamins, enzymes, and proteins in many biological activities in the human body. People get trace elements from fruit, vegetables, and meat products. Dried red plum, Turkey has gained importance in the analysis of the work because it is the most consumed fruit element.

Although elements such as Ca, K, Mg, and Na are in high amounts, others are found in trace amounts. Metals such as Cu, Fe, Zn, and Mn are effectively used in biochemical processes in the human body. These elements, if they are in high amounts, show toxic effects and negatively affect human health. In addition, Pb, Cd, Ni, and As show toxic effects [1-4].

Trace elements in fruit juices sold in Portugal were determined by AAS. It was decided to reduce the amount of metal in it [5]. In one study, metals were determined by ICP-OES of certain dried fruit in Turkey. The effects of different dissolving techniques on element quantities were examined [6]. Trace element determinations with or without toxic effects on fruits and vegetables in Saudi Arabia were made by ICP-OES method [7].

Iron (Fe) is an important element for oxygen transport and hemoglobin [8]. Copper (Cu) is an important coenzyme and cofactor in biochemical functions [9]. Magnesium (Mg) forms the structural tissue of the bone. It provides absorption of many enzymes and proteins [10]. Nickel (Ni) is an essential element in ensuring the absorption of iron in the body [11]. Removal of nickel and cadmium from wastewater was carried out [12].

In another study, toxic elements in fruits and vegetables were measured due to environmental and

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industrial pollution and it was found to be caused by contaminated irrigation and wastewater [13-17].

The purpose of this study to determine some metal ions such as Mg, Fe, Cu, Pb, Ni, Zn, As, and Se in dried red plum from Turkey which is consumed directly or as compote. The ICP-OES method was used for the determination.

EXPERIMENTAL SECTION

Materials and apparatuses

All chemicals used throughout the experiments were of analytical-reagent grade (Merck, Germany). HNO_3 (65%), H_2O_2 (30%) and HCI (37%) were of analytical pure quality (Merck, Germany).

The most consumed dried red plum examples from Turkey, obtained in September 2019 commercially sourced from local market. All determinations were performed by Perkin Elmer Optima 5300 DV model inductively coupled plasma optical emission spectrometry (ICP- OES).

Samples

The dried red plum samples were brought to a fixed scale in the oven. Dried red plum samples were washed and dried at 100 ^oC for 48 hours. The cleaned samples were ground in the grinding machine. Before the amount of metals was determined, a microwave digestion procedure was performed by a Milestone microwave digestion system in order to achieve a total digestion in a short time, thus avoiding loss of metals by volatilization and minimizing the amount of added acid. The digestion procedure was as follows: 0.5 g of sample was placed in a polytetrafluoethylene (PTFE) digestion vessel, 1.5 mL of nitric acid and 2 mL of hydrogen peroxide were added. The samples were then kept at room temperature for 6 hours for homogenization and slow digestion. Afterwards, the containers were closed and the digestion process was carried out by applying a temperature program of 170 °C for 18 minutes to the samples inside.

After the digestion procedure, all digestion vessels were left closed overnight for cooling. The next day, container contents were transferred to vials and the final volume adjusted to 10 mL with ultrapure water. Colorless solutions obtained were read for further analysis. The excitation wavelengths used in the qualitative and quantitative analysis of each metal are shown in Table 1. As can be seen from the table, the characteristic light of its wavelength is used for which metal to measure.

Table 1: Magnesium, iron, copper, lead, chromium, arsenic, nickel, zinc, and selenium of selected wavelength for metals quantified by ICP-OES.

Element	Wavelength (nm)
Mg	285.23
Fe	238.20
Cu	327.39
Pb	220.35
Cr	267.71
As	228.81
Ni	231.60
Zn	206.20
Se	196.02

Table 2: The operating parameters of determination of elements by ICP-OES.

Instrument	SPECTRO ARCOS
Viewing height (mm)	12
Wavelength	nm
Replicates	3
RF Power (W)	1450
Spray chamber	Cyclonic
Nebulizer	Modified Lichte
Nebulizer flow (L/min)	0.8
Plasma Torc	Quartz, fixed, 3.0 mm injector tube
Replicate read time	50 s per replicate
Plasma Gas Flow (L/min)	13
Auxiliary Gas Flow (L/min)	0.7
Sample aspiration rate (mL/min)	2.0
Sample Pump Rate (rpm)	25

The reduction procedure was performed for the As and Se determinations only. For this, 2 mL of dissolved sample was added to 1 mL of hydrochloric acid and its volume was completed with 10 mL of ultrapure water.

RESULTS AND DISCUSSION

Determination of the optimum conditions of ICP-OES for trace elements

Prior to trace element quantification, conditions for sample preparation and for ICP-OES analysis were carefully chosen, in order to define the optimal settings for the analysis. These conditions are shown in Table 2. After optimization, the selected method was demonstrated to be the most selective and sensitive.

Table 3: Figures of merit of ICP-OES method showing correlation coefficient (R^2), Limits of Detection (LOD), and Limits of Quantitation (LOQ) of each element.

Element	R^2	LOD (mg/L)	LOQ (mg/L)
Mg	0.9950	0.015	0.065
Fe	0.9991	0.013	0.036
Cu	0.9995	0.004	0.007
Pb	0.9994	0.002	0.004
Cr	0.9997	0.0004	0.0015
As	0.9998	0.005	0.019
Ni	0.9999	0.0003	0.0011
Zn	0.9997	0.007	0.020
Se	0.9998	0.004	0.016

Table 4: The results of the analysis with microwave digestion procedures of NIST SRM 1515 Apple Leaves certified reference material (µg/g), N = 4.

Element	Certified value	Microwave digestion	RSD (%)
Fe	83 ± 5	81.0 ± 4.6	3.41
Cu	5.64 ± 0.24	5.56 ± 0.27	2.42
Pb	0.470 ± 0.024	0.450 ± 0.007	8.16
Ni	0.91 ± 0.12	0.88 ± 0.11	10.3
Zn	12.5 ± 0.3	12.1 ± 0.6	11.2

Quantification limits were obtained through the signal/noise (10×S/N) method. The correlation coefficient (R^2), detection limits (LOD), and quantity limits (LOQ) of each element are shown in Table 3.

According to these measurements, it was seen that the sensitivity of the method was high and the detection limit could be used easily in the quantitative analysis of metals with very low concentrations.

Analytical performance

Validation of the proposed method was carried out using certified reference material NIST-SRM 1515-Apple Leaves. The comparison of the metal content of the certified reference material with the values measured by our method is shown in Table 4.

Here, it was determined that the accuracy of our method was high. Therefore, it was decided that the metal amounts in our real sample can now be analyzed with our method.

Trace elements in dried red plum

Mg, Fe, Cu, Pb, As, Cr, Ni, Zn and Se amounts in 4 different dried red plum samples are given in Table 5. Calibration standards contained 0.01–5 mg/L Cu, Pb, As, Cr, Ni, Zn and Se; 0.1–20 mg/L Fe and 5–50 mg/L Mg. Samples that exceeded the concentration of the highest standard were diluted and re-run.

Trace elements can easily pass into various foodstuffs by contaminated ecosystems. Therefore, the determination of trace and toxic elements has gained importance for human biology. When the results obtained from the samples were examined, it was determined that there were slightly more trace elements found in prunes in the world. In addition, it was seen that it was among the values allowed in the regulation. The reason for this is estimated to be the climatic conditions of our country [18-20]. Dried fruits have the advantage of being so easy to store and distribute, they are easily incorporated into other foods and recipes at relatively low cost and provide a healthy alternative to sugar snacks.

Table 5: Trace element contents (µg/g) in dried red plum samples after microwave digestion. N= 4.

Element	Red plum 1	Red plum 2	Red plum 3	Red plum 4
Mg	45 ± 2	47 ± 3	44 ± 2	46 ± 3
Fe	15 ± 1	16 ± 1	18 ± 1	16 ± 1
Cu	3.1 ± 0.2	3.2 ± 0.2	3.1 ± 0.2	3.2 ± 0.2
Pb	4.3 ± 0.2	4.1 ± 0.3	4.2 ± 0.2	4.4 ± 0.3
Cr	1.5 ± 0.1	1.7 ± 0.2	1.4 ± 0.2	1.6 ± 0.1
As	1.2 ± 0.2	1.3 ± 0.2	1.1 ± 0.2	1.2 ± 0.1
Ni	2.8 ± 0.3	2.7 ± 0.2	2.5 ± 0.2	2.6 ± 0.2
Zn	3.5 ± 0.2	3.7 ± 0.3	3.4 ± 0.3	3.6 ± 0.3
Se	4.5 ± 0.3	4.2 ± 0.3	4.3 ± 0.3	4.5 ± 0.4

CONCLUSIONS

In this study, trace elements analysis was conducted on four dried red plum samples in Turkey with using ICP-OES. Mg, Fe, Cu, Cr, Ni, and Zn are elements that are important functions in human health. People get these elements from fruit, vegetables, and meat products. It is known that high amounts of magnesium strengthen our immune system. Thus, recently dried red plum consumption in Turkey has increased. As, Pb, Cr and Ni amounts are elements that show toxic effects when taken from food. Therefore, their analysis is extremely important. The amounts of these elements do not exceed their daily allowance [21-22]. Selenium is an essential element for many living organisms' cellular functions. The amount of selenium measured is in the daily allowable value. As a result, the amount of trace elements measured in dried red plum samples was among the values determined by the World Health Organization (WHO).

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