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Journal of Agroalimentary Processes and Technologies 2024, 30 (3), pp 293-299

Journal of Agroalimentary Processes and Technologies

Determination of the distribution of macro-, micro- and toxic element contents in different halva samples produced by grinding sesame seeds

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Abstract

The element assigned in the highest amounts in halva samples was P, followed by K, Mg and Ca in decreasing order. Phosphorus and K quantities of halva samples were assessed to be beter 5.82 (foam halwa) and 2387 mg/kg (plain tehina halva) to 49.80 (foam halva) and 3049 mg/kg (halva with boiled grape juice), respectively. In general, the most abundant microelements in halva and special dessert samples were Fe and Zn, followed by Cu, Mn and B in decreasing order. Iron and Zinc quantities of the halva and special sweets (Sultan sarması and Höşmerim) were recorded to be between 0.96 (foam halva) and 26.37 mg/kg (tehina halwa with cacao) to 2.42 (Höşmerim) and 25.59 mg/kg (Plain tehina halwa), respectively. Depending on the halwa and dessert varieties, the toxic elements detected in the highest amounts in the samples were Al, As, Ba and Pb. However, the amounts of other analyzed toxic elements are also thought to be above the consumable limit values. Al and As quantities of the halva and special sweets were assessed to be between 3.24 (Çekme halva) and 256.3 mg/kg (Tehina halwa with cacao) to 17.16 (Summer halva) and 3107 mg/kg (foam halva), respectively. Also, Ba amounts of the halwa and special sweet samples changed between 1.76 (foam halva) and 67.26 mg/kg (Tehina halva with cacao). Also, Pb contents of summer halwa, çekme halwa, tehina halva with cacao, foam halva and höşmerim sweet ranged between 9.53 and 15.28 mg/kg. Pb contents of other halva samples were found below 9.53 mg/kg. The toxic elements that contaminated halva and special dessert samples in the highest amounts were Al, Asi Ba and Pb.

Keywords: Halva types, sweets, macro and micro elements, toxivc elements, ICP-OES.

1. Introduction

The halva is a traditional dessert type that is prepared by mixing roasted and ground sesame (tahini), Saponaria officinalis root, sugar (sucrose) and citric acid and is widely consumed in the Mediterranean Region (Elleuch et al. 2007). Tahini halva is a product obtained by kneading the sugar syrup obtained by cooking after adding sugar, drinking water, citric acid or tartaric acid and, when necessary, edible glucose syrup, after being thickened and whitened with soapwort extract, and mixed with tahini in accordance with the technique (Anonymous, 2004). The dessert is called halva in Turkish and is known by various names such as halva in English, halava in Sanskrit, halawi in Arabic (Davidson, 2002). Tahini halva can be consumed plain or with ingredients such as walnuts, pistachios, cocoa and vanilla. Halva contains approximately 500-550 kcal/100g energy, 30-35% fat, 40-47% carbohydrate, 10-12% protein (Güneşer, 2009). Tahini Halva, which is among the traditional Turkish foods, is also known as Turkish Honey, Turkish Dessert or Turkish Halva in the Western world (Anonim, 2001b; Güven,1928) and has been a food item consumed by the people living in Asian lands for many years. It is stated by exporting companies that tahini halva is consumed outside our country, as well as in the Balkan countries, Israel, Middle Eastern countries, Poland, Russia, England and America (Karakahya and Yılmaz, 2006).Apart from tahini and sugar, a

DOI: 10.59463/JAPT.2024.2.25

small amount of citric acid, soapwort extract, a certain amount of vanilla, cocoa and pistachios for flavored halvas, dried fruits, and some gum for gummy and honey halvas are added to tahini halva. The most important ingredient ingredient in summer halva with walnuts is walnuts. According to the sensory elements specified in the TS-10913 standard, summer halva with walnuts must have a variety-specific shine, not have significant oil leakage, and the taste and smell of the added seasoning and tahini must be perceptible (Anonim, 2001a). Apart from tahini halva, another product that maintains its tradition, especially in our country, is summer halva, another type of halva made with tahini. This product, which has been popularly known as summer halva for many years and is widely consumed, is actually a form of tahini halva with walnuts and high cocoa, produced with several different ingredients (Anonim, 2001a). Halva (sweetened sesame paste) is a famous sweet food widely used in several countries of Europea and Middle East.Halva, which is one of the delicious and various desserts of Turkish cuisine, has an important place on our tables for many years, and this flavor dating back to ancient times is among the most consumed foods today (Güneser and Zorba, 2014; Aloui *et al*., 2016). The tahini used in the production of tahini halva is produced only from sesame seeds and does not contain any additives. According to the Turkish Food Codex, tahini is defined as "the product obtained by separating sesame (*Sesamum indicum* L.) seeds suitable for tahini production from their shells in accordance with the technique, drying them in the oven, roasting them and then grinding them in the mill." Additionally, as stated in the Codex, tahini must not exceed 3.2% ashes (TGK, 2015). The chemical composition of tahini, the main ingredient of halva, consists of approximately 57-65% fat, 23-27% protein and some minerals such as calcium, phosphorus, potassium and magnesium (Yamani and Isa, 2006). Although tahini halva is produced in industrial quantities, there are still traditional productions with regional differences using a wide variety of ingredients such as hazelnuts, pistachios, cocoa, vanilla and walnuts (Catargiu *et al.*, 2017). Tahini halva has a very long shelf life (about 2 years) due to its low moisture content (3%). Changes in the production, storage, distribution and use stages depending on temperature and humidity values may cause various condensation problems in the product (Sengun *et al.*, 2005). While various studies have been conducted on tahini halva in countries such as Egypt and Greece (Eissa and Zohair, 2006; Kotzekidou, 1998), limited studies on tahini halva have been carried out in Turkey despite the high consumption rates (Kilci and Çetin, 2023). The aim of the study was to evaluate the content of macro, micro and toxic elements in different varieties of halva.

2. Materials and method

Material

Halva samples (Fig.1) were provided from Konya province in 2023 (Fig. 2). Before the analysis, the samples were crushed by a blender. HNO_3 and H_2O_2 are analytical grade and Merck company (Darmstadt, Germany).

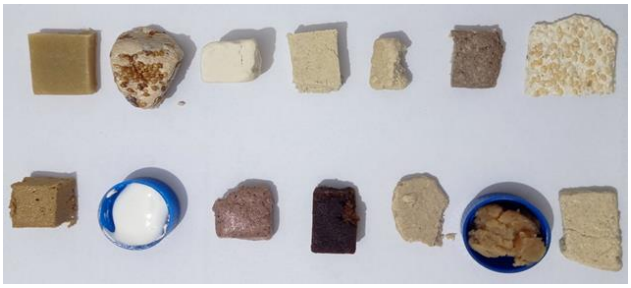

Figure 1. Halva samples provided from Konya province.

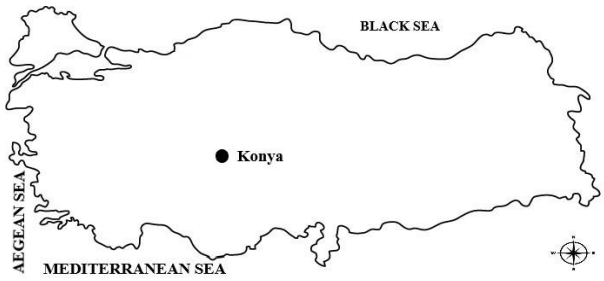

Figure 2. Locations where the halva samplesused in this study were collected

Methods

Element analysis

Samples were taken homogeneously and the weights of these samples to be used during burning were determined in grams. The samples were dissolved with 5 ml concentrated HNO_3 and 2 ml H_2O_2 (30% w/v) in a microwave device (Cem MARSXpress) under high temperature (210°C) and pressure (200 PSI). To ensure the reliability of the analysis, 1 blank and 1 certified reference material (Tomato Leaves, NIST) will be added to the 40-cell microwave set. The volumes of the dissolved samples were completed to 20 ml with deionized water, filtered with blue banded filter paper and stored in the refrigerator at +4°C until the reading was taken. After

preliminary analytical procedures, P, K, Ca, Mg, Fe, Zn, Mn, Cu, B, Al, As, Ba, Cd, Co, Cr, Ni and Pb element contents in halva samples were determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) (Agilent-5110) device. The raw results (reading values) obtained as a result of the reading were multiplied by the coefficient determined for each sample (completed volume after combustion/ initial sample weight) and thus the element amounts in the sample were determined in mg kg-1 unit. Study wavelengths, LOD and LOQ values of elements analyzed with ICP-OES are assigned in Table 1.

Table 1. Study wavelengths, LOD and LOQ values of elements analyzed with ICP-OES

Nutrient elements	Wavelengths (nm)	LOD (mg/kg)	LOQ (mg/kg)	R^2
P	213.618	4.346591	14.48864	0.999
K	766.491	131.6843	438.9477	0.999
Ca	317.933	259.2863	864.2877	0.999
Mg	279.553	58.98845	196.6282	0.999
Fe	238.204	1.411773	4.705909	0.999
Zn	213.857	0.208636	0.695455	0.999
Cu	327.395	0.027818	0.092727	0.999
Mn	257.610	0.424227	1.414091	0.999
B	249.772	0.118227	0.394091	0.999
Al	167.019	415.8818	1386.273	0.999
As	175.800	0.729991	2.433303	0.999
Ba	614.171	43.81364	146.0455	0.999
Cd	214.439	0.006955	0.023182	0.999
Co	238.892	0.006955	0.023182	0.999
Cr	267.716	0.000695	0.002318	0.999
Ni	231.604	1.578189	5.260631	0.999
Pb	220.353	5.276961	17.58987	0.999

Statistical Analysis

Statistical analysis of the data obtained in the study was performed through the JMP statistical program (JMP, SAS Institute, Cary, NC). The significance level of application effects was tested through analysis of variance and Student's t method was used to compare mean values (Jones and Sall, 2011). In order to examine the correlation between heavy metal contents of halva varieties, a multivariate cluster analysis was carried out using the PAST statistical program to perform Principal Component Analysis (PCA) (Pulliainen and Wallin, 1996).

3. Results and Discussion

Macro element quantities of differenthalva samples

Macro element contents of different types of halva and special desserts are displayed in Table 2. Macro element contents of halva samples differed depending on the halva types. The element assigned in the highest amounts in halva samples was P, followed by K, Mg and Ca in decreasing order. Phosphorus and K quantities of halva samples were assessed to be beter 5.82 (foam halwa) and 2387 mg/kg (plain tehina halva) to 49.80 (foam halva) and 3049 mg/kg (halva with boiled grape juice), respectively. In addition, while Ca amounts of the halwa samples vary between 17.71 (foam halwa) and 1650 mg/kg (Summer halva with cacao), Mg quantities of the halwa samples were assigned to be between 5.56 (foam halwa) and 1012 mg/kg (Halva with boiled grape juice). The lowest macro element contents in halwa samples were detected in the foam halva sample. In general, halva samples contained high amounts of P, K, Ca and Mg (except drawn halva and summer halva). In halva samples, the highest amounts of K and Mg were detected in grape molasses halwa, while P and Ca were determined in plain tahini halwa and cocoa summer halwa. However, fluctuations were observed in the mineral

Table 2. Macro element contents of different halva types

Sweet Samples	**P**	**K**	**Ca**	**Mg**
	---------- (mg kg^{-1}) ----------			
Summer halva	628.1±24.62E	772.1±8.35 HI	187.3±1.63 G	223.7±1.22 F
Sultan Sarması	2221±167.08 BC	1654±3.84 D	614.6±4.40 C	753.5±16.88 C
Çekme Halva	349.1±8.34 F	623.9±7.33 J	66.98±0.634H	80.82±1.47 G
Plain tehina halva withoy gluten	2139±13.52 C	1249±25.50 F	502.6±5.69 D	855.9±23.89 B
Plain tehina halwa	2387±85.96 AB	1253±16.28 F	757.1±13.29 B	864.1±14.74 B
Tehina halva with cacao	2443±7.13 A	1962±15.01 B	761.1±24.98 B	960.5±21.40 A
Kıtır Halwa	1339±30.25 D	749.2±20.34 I	155.1±0.86 G	558.3±13.101D
Halwa with boiled grape juice	2448±14.63 A	3049±29.78 A	485.7±1.07 D	1012±15.80 A
Tehina halva with elderberry without sugar	2247±44.05 ABC	1694±3.244D	465.4±11.67 D	873.8±1130 B
Summer halva with cacao	1432±27.85 D	1837±21.62 C	1650±13.68 A	459.8±18.21 E
Kürek Halva	2057±7.20 C	942.4±1.54 G	491.2±5.61 D	829.1±11.12 B
Foam Halva	5.82±0.11 G	49.80±1.62J	17.71±0.24 I	5.56±0.284 H
Höşmerim	421.1±0.56 EF	830.5±11.83 H	228.4±0.96 F	65.20±1.22 G
Diabetic Halva	2115±40.12 C	1458±31.81 E	376.0±9.01 E	879.2±12.34 B

A-J; p<0.01

contents of halva samples. These differences may be due to the ingredients (sesame tehina, grape juice, cacao, blackberry) and their concentrations added during the production of each type of halva.

Micro element quantities of different halva types

The microelement distributions analyzed in various halva and special desserts are depicted in Table 3. In general, the most abundant microelements in halva and special dessert samples were Fe and Zn, followed by Cu, Mn and B in decreasing order. Iron and Zinc quantities of the halva and special sweets (Sultan sarması and Höşmerim) were recorded to be between

Table 3. Micro element contents of different halva types

Sweet Samples	Fe	Zn	Cu	Mn	B
	---------- (mg kg^{-1}) ----------				
Summer halva	6.34±0.15 H	6.41±0.12 H	2.77±0.02E	2.73±0.07 F	0.87±0.00 E
Sultan Sarması	20.54±0.01 B	15.67±0.07 E	6.41±0.24 C	6.34±0.26 BC	2.49±0.23 BC
Çekme Halva	4.44±0.16 I	3.58±0.11 I	1.74±0.01 F	1.79±0.06 G	0.86±0.01 E
Plain tehina halva withoy gluten	20.26±0.35 B	16.22±0.12 E	6.52±0.12 C	3.71±0.08 E	1.84±0.022D
Plain tehina halwa	19.49±0.25 C	25.59±0.17 A	8.70±0.09 A	6.51±0.23 B	1.86±0.02 D
Tehina halva with cacao	26.37±0.29 A	20.56±0.24 B	8.50±0.06 A	6.86±0.00 B	2.43±0.19 BC
Kıtır Halwa	9.25±0.06 G	11.68±0.31 F	4.39±0.14 D	2.60±0.08 F	0.86±0.01 E
Halwa with boiled grape juice	17.64±0.06 D	20.56±0.15 BC	8.34±0.23 A	4.57±0.15 D	5.41±0.24 A
Tehina halva with elderberry without sugar	19.39±0.10 C	17.65±0.09 D	7.43±0.13 B	8.81±0.30 A	2.60±0.13 BC
Summer halva with cacao	15.33±0.18 F	9.35±0.13 G	4.62±0.04 D	6.43±0.12 BC	1.71±0.15D
Kürek Halva	16.86±0.03 E	16.04±0.51 E	6.68±0.24 C	0.001±0.11 H	2.12±0.09 CD
Foam Halva	0.96±0.02 K	2.95±0.02 IJ	0.99±0.00 G	4.80±0.00 D	1.85±0.09 D
Höşmerim	3.31±0.09 J	2.42±0.22 J	0.75±0.02 G	1.35±0.10 G	1.66±0.14 D
Diabetic Halva	17.39±0.26 DE	19.26±0.35 C	7.76±0.09 B	5.89±0.01 C	2.81±0.14 B

A-I; p<0.01

0.96 (foam halva) and 26.37 mg/kg (tehina halwa with cacao) to 2.42 (Höşmerim) and 25.59 mg/kg (Plain tehina halwa), respectively. In addition, the highest Cu, Mn and B quantities were assessed in plain tehina halva, tehina halva with blackberry (without sugar) and halva with boiled grape juice, respectively. In general, the microelements detected in the lowest amounts in halwa samples were determined in foam halwa, Höşmerim, Çekme halwa and summer halva. It was determined that plain tahini halva had high Zn and Cu contents. Some differences in the microelement contents of halva and special dessert varieties were observed. These differences may probably be due to the ingredients used in the production of halva and dessert, the genetic structure and growing conditions of sesame, which is the main ingredient of halva, whether the sesame is shelled or not, halwa production conditions and the composition and solubility of the metal materials used in production.

Toxic element quantities of different halva samples

Toxic element contents of different types of halva and special desserts are assigned in Table 4.

Table 4. Toxic element contents of different halva types

Sweet Samples	Al	As	Ba	Cd	Co	Cr	Ni	Pb
	---------- (mg/kg) ----------							
Summer halva	126.5±0.16 B	17.16±1.18 I	13.11±0.86 F	1.67±0.12 BC	0.19±0.01 E	8.40±0.08 D	6.48±0.15 E	9.58±0.28 CD
Sultan Sarması	3.44±0.08 E	348.7±16.25 H	50.69±3.61 C	2.44±0.13 A	0.54±0.02 E	8.50±0.21 D	9.73±0.22 B	4.37±0.23 H
Çekme Halva	3.24±0.17 E	2350±52.58 B	20.64±0.32 E	2.70±0.06 A	1.57±0.12 C	7.70±0.29 E	1.51±0.21 I	10.57±0.16 C
Plain tehina halva with soy gluten	143.5±0.16 B	330.5±10.24 H	7.96±0.32 GH	1.34±0.16 C	1.63±0.12 BC	10.24±0.06 AB	7.62±0.25 D	5.49±0.22 GH
Plain tehina halwa	152.0±0.83 B	94.59±2.30 I	66.10±0.73 A	2.63±0.15 A	0.74±0.06 DE	9.59±0.05 BC	9.41±0.18 B	8.42±0.21 DE
Tehina halva with cacao	265.3±17.55 A	23.24±0.09 I	67.26±0.32 A	2.48±0.0 A	1.64±0.20 BC	10.31±0.23 A	7.47±0.27 D	10.61±0.21 C
Kıtır Halwa	139.4±12.55 B	878.4±2.26 F	11.50±0.19 FG	2.29±0.13 AB	2.23±0.08 AB	9.37±0.27 C	4.70±0.10 GH	10.62±0.20 C
Halwa with boiled grape juice	160.7±14.52 B	642.9±19.77 G	20.56±0.06 E	2.47±0.24 A	2.57±0.13 A	3.21±0.18 F	5.64±0.05 F	6.75±0.13 FG
Tehina halva with elderberry without sugar	139.9±.20.56 B	657.6±12.05 G	58.83±0.59 B	2.48±0.09 A	1.71±0.22 BC	2.27±0.08 GH	5.16±0.07 FG	7.32±0.20 EF
Summer halva with cacao	66.36±1.81 CD	1710±113.24 C	24.56±1.25 E	2.22±0.18 AB	2.37±0.24 A	2.53±0.16 G	10.57±0.28 A	9.56±0.120 CD
Kürek Halva	33.48±2.11 DE	962.1±22.42 EF	43.53±1.04 D	2.28±0.19 AB	1.39±0.10 C	2.60±0.19 FG	4.44±0.05 H	12.56±0.13 B
Foam Halva	44.01±1.55 D	3106±138.61 A	1.76±0.06 I	1.20±0.06 C	2.35±0.29 A	3.24±0.17 F	9.82±0.09 B	15.28±0.14 A
Höşmerim	85.69±1.97 C	1116.2±51.04 E	5.49±0.26 HI	2.42±0.272 A	1.30±0.15 CD	1.25±0.06 I	5.73±0.10 F	9.53±0.16 CD
Diabetic Halva	143.44±0.91 B	1394±61.71 D	56.30±2.10 B	2.44±0.08 A	1.41±0.14 C	1.83±0.06 HI	8.53±0.22 C	0.95±0.02 I

A-I; $p<0.01$

Depending on the halwa and dessert varieties, the toxic elements detected in the highest amounts in the samples were Al, As, Ba and Pb. However, the amounts of other analyzed toxic elements are also thought to be above the consumable limit values. Al and As quantities of the halva and special sweets were assessed to be between 3.24 (Çekme halva) and 256.3 mg/kg (Tehina halwa with cacao) to 17.16 (Summer halva) and 3106.78 mg/kg (foam halva), respectively. Also, Ba amounts of the halwa and special sweet samples changed between 1.76 (foam halva) and 67.26 mg/kg (Tehina halwa with cacao). Ni and Pb quantities of the halva and special sweets were assigned to be between 1.51 (Çekme halwa) and

10.57 mg/kg (summer halva with cacao) to 0.95 (diabetic halwa) and 15.28 mg/kg (foam halva), respectively. The As contents of Çekme halwa, summer halva with cacao, foam halva, kürek halva, höşmerim and diabetic halva ranged between 962.07 and 3106.78 mg/kg. The highest Cd (2.70), Co (2.57) and Cr (10.31 mg/kg) were detected in Çekme halva, halva with boiled grape juicea and tehina halwa with cacao samples. Also, Pb contents of summer halwa, çekme halwa, tehina halva with cacao, foam halva and höşmerim sweet ranged between 9.53 and 15.28 mg/kg. Pb contents of other halva samples were found below 9.53 mg/kg. The toxic elements that contaminated halva and special dessert samples in the highest amounts were Al, Asi Ba and Pb. The main sources of this contamination are probably thought to arise from factors such as the materials added to halva and special dessert samples and their compositions, production materials, packaging types, chemical composition of metal materials used in production, transportation and storage.

Kilci and Çetin (2023) determined 5.88-7.98 mg/kg Cu, 6.98 and 22.33 mg/kg Fe, 0 and 1.12 mg/kg tin, 0.019 mg/kg Pb in halva samples. Also, Arsenic was not detected in halva samples (Kilci and Çetin, 2023). According to the Turkish Food Codex Communique on Tahini Halva (2015/28), tahini halva may include a maximum to 0.2 mg/kg of arsenic, 10 mg/kg of copper, 40 mg/kg of iron, 200 mg/kg of tin, and 0.3 mg/kg of lead (TGK, 2015). The heavy metal contents of the halva samples analyzed in this study were well above the values stated by Kilci and Çetin (2023) and TGK (2015). It is thought that the ingredients and additives used in halva samples, metal materials used in production, packaging types and storage conditions may be the factors causing this increase.

Principal Companent Analysis (PCA)

Halva types (Summer halwa, Sultan Sarması, Pulled halwa, Plain tehina halwa withoy gluten, Plain tehina halwa, Tehina halwa with cacao, Kıtır Halwa, Halwa with boiled grape juice, Tehina halwa with elderberry without sugar, Summer halwa with cacao, Kürek Halwa The Pearson correlation (r) between heavy metal contents (Al, As, Ba, Cd, Co, Cr, Ni and Pb) is given in Figure 3.

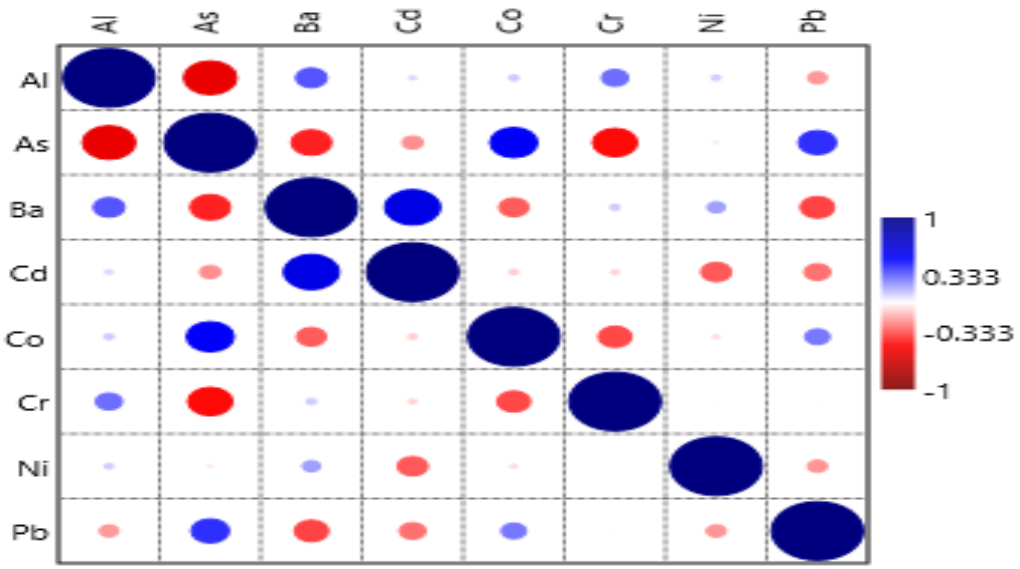

Figure 3. Summer halva, Sultan Sarması, Pulled halva, Plain tahini halva with gluten, Plain tahini halva, Cocoa tahini halva, Crispy Halva, Boiled grape halva, Elderberry halva without sugar, Summer halva with cocoa, Heavy metal contents of Kurek Halva (Al, As, Ba, Cd Pearson correlation (r) between Co, Cr, Ni and Pb)

As can be seen from examining Figure 3, which reveals the relationships between halva types and heavy metal contents; It was determined that there were both positive and negative relationships between the heavy metal contents of halva. It was determined that the relationship between Al contents and As contents of halva varieties was significant and negative (r=-0.574**). In addition, the relationship between Ba contents and Cd contents of halva varieties was shown to be significant and moderately strong positive relationships (r=0.599**). This study, using Pearson correlation analysis, aimed to determine the strength and direction of the relationship between the heavy metal contents of

variable halva varieties. Thus, Pearson correlation tried to draw the best fit line on the data of heavy metal contents of halva varieties, and the Pearson correlation coefficient (r) revealed how far all these data points were from the best fit line (Obilor and Amadi, 2018).

3. Conclusion

The lowest macro element contents in halwa samples were detected in the foam halva sample. In halva samples, the highest amounts of K and Mg were detected in grape molasses halwa, while P and Ca were determined in plain tahini halwa and cocoa summer halwa. In general, the microelements detected in the lowest amounts in halwa samples were determined in foam halwa, Höşmerim, Çekme halwa and summer halva. The toxic elements that contaminated halva and special dessert samples in the highest amounts were Al, Asi Ba and Pb.

Data availability Not applicable.

Declarations

Ethics approval Not applicable.

Competing interests No conflict of interest

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