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Chemical Properties of Tahini Halva Marketed in The Southern Marmara Region of Turkey and Their Compliance with Turkish Food Codex

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ABSTRACT

In this study, three different tahini halva types (plain, cocoa and pistachio) from 15 different brands were obtained from various manufacturers or commercial markets in the Southern Marmara Region provinces of Balikesir, Bilecik, Bursa, and Canakkale. For each manufacturer, samples were obtained from two different lot numbers. The results of chemical analysis in tahini halva samples were as follows; the total amount of sugar was 39.45-43.78%, the total amount of tahini was 51.88-76.84%, the amount of oil was 33.1-38.9%, the amount of peroxide in the extracted oil was 0-3.67 meq/kg, the amount of copper was 5.88-7.98 mg/kg, the amount of iron varied between 6.98 and 22.33 mg/kg, the amount of tin ranged between 0 and 1.12 mg/kg, the amount of lead was 0.019 mg/kg, and the amount of salt was between 0 and 0.96 g/100g. Arsenic was not detected in halva samples. When the fatty acid composition of oils extracted from tahini halva was analyzed using gas chromatography, a profile high in oleic acid (43%) and linoleic acid (40%) was found. When all of these results were taken into consideration, tahini halva from one of the examined enterprises was inconsistent with the Turkish Food Codex Communique on Tahini Halva (2015/28) in terms of the total amount of tahini.

Keywords: Tahini halva, Chemical quality, Southern Marmara Region

Güney Marmara Bölgesi'nde Tüketime Sunulan Tahin Helvalarının Kimyasal Kalitesi ve Standartlara Uygunluğu

ÖZ

Bu çalışmada Güney Marmara Bölgesi'nde yer alan Balıkesir, Bilecik, Bursa ve Çanakkale illerindeki üreticilerden ve ticari olarak satış yapılan marketlerden 15 farklı markaya ait sade, kakaolu, Antep fıstıklı olmak üzere üç farklı çeşit tahin helvası örnekleri toplanmıştır. Numuneler her bir firma için iki farklı parti numarasından olacak şekilde temin edilmiştir. Tahin helvası numunelerine yapılan analizler neticesinde toplam şeker miktarının %39.45-43.78, toplam tahin miktarının %51.88-76.84, yağ miktarının %33.1-38.9, ekstrakte edilen yağda peroksit sayısının 0-3.67 meq/kg, Bakır miktarının 5.88-7.98 mg/kg, Demir miktarının 6.98-22.33 mg/kg, Kalay miktarının 0-1.12 mg/kg, Kurşun miktarının 0-0.019 mg/kg, tuz miktarının 0-0.96 g/100g aralığında değiştiği tespit edilirken numunelerde Arsenik ölçülmemiştir. Tahin helvalardan ekstrakte edilen yağ, yağ asitleri bileşeni yönünden kromatografik analize tabi tutulduğunda oleik asit (~%43) ve linoleik asit (~%40) açısından zengin bir profille karşılaşılmıştır. Tüm bu sonuçlar değerlendirildiğinde numune alınan firmalardan bir tanesinin toplam tahin miktarı açısından Türk Gıda Kodeksi Tahin Helvası Tebliği (2015/28)'ne uygun üretim yapmadığı belirlenmiştir.

Anahtar Kelimeler: Tahin helva, Kimyasal kalite, Güney Marmara Bölgesi

INTRODUCTION

The term "halva" refers to a kind of traditional dessert prepared by mixing roasted and ground white sesame seeds (tahini), *Saponaria officinalis root*, sugar (sucrose), and citric acid, reaching the peak of its popularity in the Mediterranean Region [1]. The dessert, which is expressed as halva in Turkish, has various names such as halva in English, halava in Sanskrit, halawi in Arabic [2]. Tahini halva can be consumed plain-type or with added ingredients like walnuts, pistachios, cocoa, and vanilla. When its nutritional composition is examined, it is discovered that when consumed in 100 g, it provides approximately 500-550 kcal of energy. While the high amount of fat (30-35%) plays the leading role in reaching these high energy values, it is also stated as an important carbohydrate (40-47%), protein (10-12%), and mineral source [3].

Tahini, one of the main components in the production of tahini halva, is produced only from sesame seeds and does not contain additives. In the Turkish Food Codex, tahini is defined as “the product obtained by separating the sesame (*Sesamum indicum L.*) seeds suitable for tahini production from their shells in accordance with the technique, drying in the oven, roasting and then crushing in the mill.” Furthermore, as stated in the Codex, tahini should contain at least 50% fat, 20% protein, a maximum of 1.5% moisture, and 3.2% ash [23]. When the chemical composition of tahini, one of the primary ingredients in halva, is examined in more detail; it mainly consists of 57-65% oil (oleic and linoleic acid), 23-27% protein (rich in methionine, cystine, and tryptophan), and some minerals such as calcium, phosphorus, potassium, and magnesium [5].

Despite the fact that tahini halva is produced in industrial quantities, there are still traditional and minimal productions with regional variations using a wide range of materials [6].

Tahini halva has a reasonably extended shelf life of two years starting from the date of manufacturing because of its low moisture content (3%). Nevertheless, depending on the temperature and humidity levels, changes that occur throughout manufacturing, storage, distribution, and use can result in various condensation problems in the product, and this circumstance paves the way for numerous developments that will harm the food [8]. While there are various studies on tahini halva in countries such as Egypt and Greece [9, 10]; Turkey has lagged in tahini halva research despite high consumption rates.

Food production involves lots of risks during production processes. It is therefore essential to ensure, monitor, and evaluate adequate and effective controls in all phases of a food production chain. In this regard, many countries and companies meet to develop standards for safe food supply [24]. One of these developed standards is the Turkish Food Codex Communique. Based on this knowledge, the purpose of this research was to monitor and evaluate the chemical quality aspects of tahini halva samples collected from marketplaces in terms of the Turkish Codex for tahini halva. Tahini halva samples were obtained from producers and retail outlets in the Southern Marmara Region (in the provinces of Balikesir, Bilecik, Bursa, and Canakkale). The reasons for selecting the Southern Marmara Region can be summarized as having geographical significance due to its location as a transition corridor between Europe and Asia, as well as diversity in terms of both corporate businesses and small family businesses and sales points.

MATERIALS and METHODS

Materials

Samples of plain-type, cocoa, and pistachio tahini halva belonging to 15 different brands were collected from producers and commercial markets in Balikesir, Bilecik, Bursa, and Canakkale provinces located in the Southern Marmara Region between January 2022 and March 2022. From the manufacturers of the same product with two different lot numbers, a total of 90 samples were collected. All samples were collected in their original packaging and delivered to the laboratory at 4-6ºC. Until and during the analyses, halva samples were kept in a refrigerator at 4-6ºC.

Figure 1 shows the process flow diagram for the production of tahini halva [7].

Determination of Total Sugar Content

The determination of total sugar content in tahini halva samples was made according to [11]. According to the method; 5 g of the halva samples were weighed and transferred to a 250 mL balloon jug, and after being dissolved in 50 mL of distilled water, Carrez-I and Carrez-II solutions were added to make up to 250 mL with distilled water. The solution is filtered through filter paper, and the clear filtrate is titrated in a boiling solution with 5 mL of Fehling I and II solutions and a few drops of distilled water using the methylene blue indicator. The titration was completed as soon as the solution color changed from blue to brick red. Calculation made by the equation; [11].

i. Amount of Natural Invert Sugar in the Sample: g/100g= (f/M_1)*100
   f: The amount of inverted sugar determined at the adjustment, equivalent to 10 mL of Fehling's solution mixture, g
   M_1: Previously inversion, the titration's spent sample solution actually contained sample, g.
ii. Total Amount of Invert Sugar: g/100g= (f/M_2)*100
   f: The amount of invert sugar determined at the adjustment, equivalent to 10 mL of Fehling's solution mixture, g
   M_2: The amount of actual sample used in the titration of the sample following its inversion in the sample solution
iii. Amount of Sucrose, g/100g: (Total Amount of Invert Sugar- Amount of Natural Invert Sugar in the Sample)*0.95

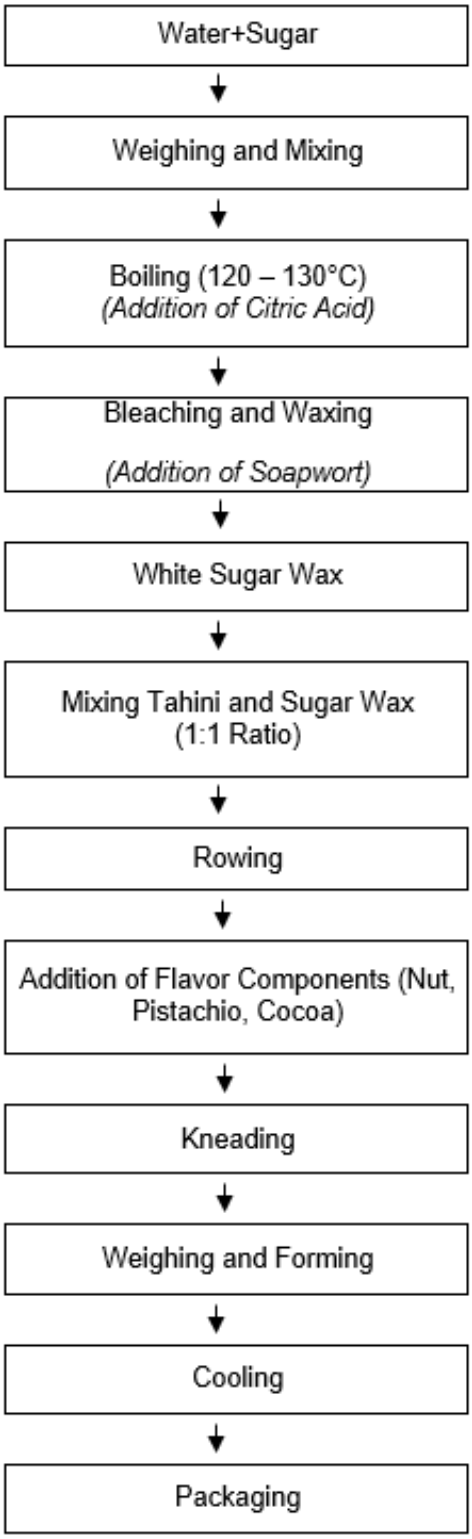

Figure 1. Tahini halva production flow chart [7]

Determination of Tahini Amount

The approach of multiplying the amount of oil in the samples by 1.9 was used to calculate the amount of tahini in the tahini halva samples according to the method in TS2590, Turkish Standards Institute Tahini Halva Standard [12].

Determination of Total Oil Content

The oil content of the tahini halva was determined gravimetrically as a result of extraction with the soxhlet method. 10 g of halva sample were weighed and added to the Soxhlet cartridge for analysis. For the extraction process to be more effective, sand was also added to the cartridge and mixed with the sample to make it homogeneous. Petroleum ether was placed in the cooler compartment, and after the extraction process, which lasted for about 6 hours, the remaining petroleum ether in the soxhlet balloon was removed, and the cartridge was kept at 100°C until it reached a constant weight [11].

Determination of Peroxide Value in Extracted Oil

Peroxide value determination was made as specified in TS-4964, "Animal and Vegetable Oils-Peroxide Number Determination Standard". Accordingly, approximately 2 g of oil extracted from tahini halva samples was weighed and put into a 250 mL flask. Oil was dissolved by adding 10 mL of chloroform, and then potassium iodide solution was added, shaken for 1 minute, and kept in the dark for 5 minutes. After adding approximately 75 mL of distilled water, a few drops of the starch solution were added to the flask, which was shaken well, and iodine was released. Titration was carried out with 0.01 N sodium thiosulfate solution. All these processes were also repeated without a sample, as in the witness experiment [13].

Determination of Transmitted Minerals Content

The Turkish Food Codex Communique on Tahini Halva specifies maximum levels for arsenic, copper, iron, lead and tin. Tahini halva samples were analyzed for the relevant contaminants. Analyses were made with an ICP-MS device according to the international standard of NMKL–186 (Nordic Committee on Food Analysis) [14].

Determination of Total Salt Content

The total salt in the tahini halva samples was determined by the Mohr method. In the analysis, tahini halva samples weighing 10 g were titrated with a solution of silver nitrate, the normality of which was predetermined using a potassium chromate indicator [15].

Determination of Fatty Acid Composition

Chromatographic analysis was used to assess the fatty acid composition of the oils recovered from tahini halva. In the analysis applied according to [16], firstly, the methyl esters of the samples were prepared, and then the methyl esters of the fatty acids were given to the gas chromatography device.

The operating conditions for the gas chromatography device were adjusted according to the following parameters:

i. Inlet Conditions; temperature: 250ºC, pressure: 21.231 psi, total flow: 42.398 mL/min, injection volume: 1µL, split ratio: 1:20.
ii. Column Conditions: A RESTEK Rtx-2330 column (fusedsilica) 30 mm x 0.25 mm x 0.2 µm (Cat. No. 10723, Serial no. 1081624) capillary GC column was used. Flow rate: 1.8761 mL/min, pressure: 21.231 psi, flow state: constant pressure.
iii. Column Oven Programme: The initial temperature was increased to 50°C for 1 min at this temperature, to 200°C at this temperature with 25°C increments per minute, to 230°C with an increase of 3°C per minute and 27 min at these temperatures. Total program duration was 45 minutes.
iv. Detector Conditions: FID temperature: 280°C, H_2 flow rate: 40 mL/min, air flow rate: 450 mL/min, make up Helium flow rate: 25 mL/min.

RESULTS and DISCUSSION

The analysis results of the samples were compared with the limit values given in the Turkish Food Codex Communique on Tahini Halva in this study, which determined the chemical quality features of tahini halva samples acquired from various sales locations. The limit values in the Turkish Food Codex regarding the chemical composition of tahini halva are given in Table 1.

Table 1. Chemical properties of tahini halva [4]

Components	Value
Sesame oil (at least, %)	26
Tahini (at least, %)	52
Protein (at least, %)	10
Total sugar (in terms of sucrose) (at most, %)	47
Moisture (at most, %)	3
Ash (at most, %)	2
Peroxide value (in the extracted oil) (at most, meq/kg)	10
Acidity (as oleic acid in the extracted oil) (at most, %)	2
Saponin (at most, %)	0.1

There is a provision in the Turkish Food Codex Communiqué on Tahini Halva, "Dry-shelled fruits such as hazelnuts and pistachios, among the seasoning materials used in Tahini Halva, should be added as whole or in separable pieces, and their ratio in the net mass of the product should be at least 8%.". In the analysis of whether the relevant rule is fulfilled in tahini halva with pistachio varieties, the flavor ratio detected in the range of 9.87-12.53% is compatible with the codex.

The findings of the chemical analysis of the samples of the plain, cocoa, and pistachio halva varieties are compiled in Tables 2, 3, and 4. In terms of the quantity of tahini they contain, it has been determined that the samples from a brand included in the halva samples are not acceptable for the Turkish Food Codex when compared to the limit values of the pertinent analyses in the codex.

Table 2. Chemical analysis results of plain-type tahini halva samples

Parameter	Min	Max	Mean	Turkish Food Codex Limit Value
Sugar (%)	39.45	42.57	40.10	≤ 47
Tahini (%)	53.82	73.17	69.35	≥ 52
Oil (%)	33.60	35.30	34.70	≥26
Peroxide Value (meq/kg)	1.71	3.67	2.40	≤ 10
Arsenic (mg/kg)	0.00	0.00	0.00	≤ 0.2
Copper (mg/kg)	5.94	7.84	7.13	≤ 10
Iron (mg/kg)	14.27	17.39	15.06	≤ 40
Tin (mg/kg)	0.00	0.98	0.62	≤ 200
Lead (mg/kg)	0.00	0.017	0.013	≤ 0.3
Salt (g/100 g)	0.35	0.96	0.40	-

Table 3. Chemical analysis results of tahini halva samples with cocoa

Parameter	Min	Max	Mean	Turkish Food Codex Limit Value
Sugar (%)	40.06	43.78	41.80	≤ 47
Tahini (%)	51.88	57.53	54.72	≥ 52
Oil (%)	33.10	38.90	36.10	≥26
Peroxide value (meq/kg)	0.00	0.00	0.00	≤ 10
Arsenic (mg/kg)	0.00	0.00	0.00	≤ 0.2
Copper (mg/kg)	6.34	7.98	7.50	≤ 10
Iron (mg/kg)	15.78	22.33	20.25	≤ 40
Tin (mg/kg)	0.11	0.34	0.28	≤ 200
Lead (mg/kg)	0.00	0.02	0.01	≤ 0.3
Salt (g/100 g)	0.12	0.64	0.50	-

Table 4. Chemical analysis results of tahini halva samples with pistachio

Parameter	Min	Max	Mean	Turkish Food Codex Limit Value
Sugar (%)	39.98	41.12	40.60	≤ 47
Tahini (%)	54.01	76.84	68.02	≥ 52
Oil (%)	34.40	36.10	35.20	≥26
Peroxide value (meq/kg)	1.23	2.32	1.98	≤ 10
Arsenic (mg/kg)	0.00	0.00	0.00	≤ 0.2
Copper (mg/kg)	5.88	6.44	6.00	≤ 10
Iron (mg/kg)	6.98	7.78	7.5	≤ 40
Tin (mg/kg)	0.87	1.12	0.95	≤ 200
Lead (mg/kg)	0.00	0.00	0.00	≤ 0.3
Salt (g/100 g)	0.00	0.05	0.02	-

The table shows that for plain-type halva samples, the lowest amount of sugar is 39.45% and the highest is 43.78%; for tahini halva samples with cocoa the lowest value is 40.06% and the highest is 43.78%; the lowest and highest amount of sugar values were found in tahini halva samples with pistachio, respectively at 39.98% and 41.12%. The Turkish Food Codex Communiqué on Tahini Halva (2015/28) states that the amount of sugar that should be present in halvah need to be at least 47% in percentage terms. When evaluated from this point of view, it can be said that the halva samples of all three varieties are compatible with the TFC in terms of sugar content. In a study examining the properties of plain-type tahini halva produced in Samsun, it was stated that the sugar content of halva samples was measured in the range of 31.06-46.79% [17]. In another study in which the chemical and microbiological properties of tahini halvah offered for sale in Bursa were determined, it was stated that the sugar content of the samples was determined as 41.99-48.23% [18]. The results for all three types are similar as compared to research with a corresponding scope.

Tahini, one of the most critical ingredients in halva, is crucial for the best possible development of the product's nutritional and textural qualities [19]. The amount of tahini in the samples was 53.82-73.17% for plain-type halva, 51.88-57.53% for halva with cocoa, and 54.01-76.84% for tahini halva with pistachio. Halva must include a minimum of 52 percent tahini, according to the Turkish Food Codex Communiqué on Tahini Halva (2015/28). From this current perspective, the samples of cocoa tahini halva samples belonging to a company are slightly below the limit value specified in the Codex. When the literature on the subject is researched, it is seen that the tahini content values of the samples are similar to the studies conducted in previous years [17, 19, 20].

Once Table 2, Table 3 and Table 4 given above are examined, the lowest amount of fat for plain-type halva samples is 33.6%, the highest 35.3%; for cocoa halva samples the lowest fat value was 33.1%, the highest was 38.9%; the lowest fat value 34.4% and the highest 36.1% were determined for the pistachio halva samples. The Turkish Food Codex Communiqué on Tahini Halva (2015/28) states that the amount of sesame oil that should be in halvah needs to be at least 26% in percentage terms. From this current perspective, it is possible to claim that all three varieties of halva samples have values that are in accordance with the Codex. The oil content values of the tahini halva samples were indicated to be in the range of 27.76-38.48 in the research done by [7], and it was observed that the results were consistent with the products examined within the scope of this study. Similar findings may also be noticed in the study carried out by [21].

In oil and foods containing oil, oxidation begins when the oil comes into contact with the oxygen in the air; this process is referred to as “autooxidation”, and it progresses autocatalytically, forming first peroxides and later hydroperoxides. Hydroperoxides produce aldehydes, ketones, and derivatives of polymers since they are unstable substances. According to reports, these have a negative impact on the appearance, flavor, and aroma of food [25]. The peroxide number, which measures the amount of active oxygen in oils, is the milligram amount of peroxide oxygen present in 1 kg of oil [26]. The peroxide value measures the amount of active oxygen present in oils, and it is correlated positively with the rancidity level of oil [22]. Tahini halva's peroxide value is restricted by the Turkish Food Codex at 10 meq/kg [4]. When evaluated from this point of view, all of the plain-type, cocoa, and pistachio tahini halva samples had values compatible with the codex and their peroxide values were determined below the limit of 10 meq/kg. The varying oxygen permeability of various packing techniques can be demonstrated as one of the factors for different peroxide value measurements to be made in tahini halva variants. The change and increase in the peroxide number are expected results given this permeability difference [26]. Furthermore, search of the relevant literature reveals that the samples' peroxide content levels are comparable to the studies conducted in previous years [17, 20].

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 [4]. In light of this analysis, it can be stated that each of the plain-type, cocoa and pistachio tahini halva samples have values in line with the codex.

When the tables containing the results of the chemical analysis are examined, it is seen that the amount of salt differs in halva varieties. For plain-type tahini halva samples, the lowest salt content was 0.45%, the highest 0.96%; while the lowest salt amount was calculated as 0.12%, the highest 0.64% for the cocoa tahini halva samples; whereas the highest salt amount was measured as 0.05%, the average salt value of all halva with pistachio samples’ was determined as 0.02%.

Table 5. Analysis of the fatty acid methyl esters in samples of the plain, cocoa, and pistachio halva types

Fatty Acid Name	Plain-Type Halva	Halva with Cocoa	Halva with Pistachio	Turkish Food Codex Interpretation
	Average Amount (%)			
Palmitic acid (C16:0)	9.2	9.3	9.2	Proper
Palmitoleic acid (C16:1)	0.1	0.2	0.2	Proper
Heptadecanoic acid (C17:0)	-	0.06	-	Proper
Stearic acid (C18:0)	5.5	5.7	5.3	Proper
Oleic acid (C18:1n9c)	43.1	43.2	43.7	Proper
Linoleic acid (C18:2n6c)	40.7	40.4	40.4	Proper
Linolenic acid (C18:3n3)	0.3	0.2	0.2	Proper
Arachidic acid (C20:0)	0.6	0.6	0.6	Proper
Cis-11-Eicosenoic acid (C20:1n9)	0.2	0.2	0.2	Proper
Behenic acid (C22:0)	0.1	0.1	0.1	Proper
Lignoseric acid (C24:0)	0.08	0.08	0.07	Proper

Table 5 shows information on the ratios and fatty acid composition of plain-type, cocoa, and pistachio tahini halva samples. The results of all three types of tahini halva samples show that productions are made by the Turkish Food Codex Communique on Tahini Halva (2015/28) [4]. One of the most crucial and critical ingredients in the production of tahini halva is sesame oil. The use of oils from other grains, such as sunflower and soybean, in place of sesame oil, has been found to produce imitation and adulteration recently. At this point, a chromatographic analysis can be applied to the tahini halva samples and more robust predictions can be made about the oil used in production. For example, it can be assumed that an oil rich in stearic acid, such as peanut oil, is adulterated if the stearic acid level is higher than the values listed in the TFC. Nevertheless, gas chromatography gives an idea of the components, and an advanced diagnostic analysis such as Principal Component Analysis (PCA) should be applied to samples suspected of counterfeiting or adulteration.

It can be concluded from the fatty acid composition analysis results of a total of 90 samples (15 different brands * 3 varieties of tahini halva * 2 different lot numbers) examined as part of this study that there is no evidence that these samples are adulterated or counterfeited.

CONCLUSION

In this study, some chemical quality characteristics of tahini halva, which is commercially produced and offered for sale in our country, were tried to be determined. In this case, 15 different brands of tahini halva in 3 different types (plain, cocoa, and pistachio) were analyzed. The investigation revealed that the tahini halva sample with cocoa from a particular manufacturer did not adhere to the Turkish Food Codex Communique on Tahini Halva (2015/28) in terms of tahini content. The amount of tahini should be at least 52% according to the TFC, but the relevant sample is slightly below this limit value. In addition, when the chemical analysis results are examined, no tahini halva sample shows results incompatible with the Turkish Food Codex. When the outcomes of an analysis using gas chromatography are evaluated, no inappropriate fatty acid component was found in the samples after the fatty acid components were identified using gas chromatography analysis, which would indicate imitation and/or adulteration. As a result, it may be inferred that the tahini halva manufacturers in the Southern Marmara Region adhere to the general criteria for chemical quality given that it is a popular dessert in our nation.

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