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ARAŞTIRMA MAKALESİ **RESEARCH ARTICLE**

Comparative analysis of fatty acid profiles, phytochemical and mineral contents of pepper spice types in Türkiye

Türkiye'deki biber baharat tiplerinin yağ asidi profilleri, fitokimyasal ve mineral madde içeriklerinin karşılaştırmalı analizi

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ABSTRACT

Peppers are significant crops frequently used in cooking or as spice. Numerous phytochemicals, including capsaicinoids, phenolics, and carotenoids are found in peppers. Capsaicinoids are responsible for the distinctively pungent flavor. A comparative study of the proximate, mineral, fatty acid composition, and phytochemical components of 15 types of pepper spices (1 Isot Pepper Flake, 4 Chili Powders, and 10 Chili Pepper Flakes) were investigated. Analysis of the proximate composition included moisture content (6.54-19.49%), ash content (6.53-22.48%) and acid insoluble ash content (0.41-1.12%). Total phenolic content ranged from 9.72 to 20.05 mg GAE g^{-1}. The lowest and highest capsaicinoid content were found in S15 (10247.6 Scoville Heat Unit (SHU)) and S9 (38861.7 SHU) samples, respectively. Total carotenoid content ranged from 739.8–1941.7 mg kg^{-1}. Phytochemical analyses revealed that these spices are high in phytonutrients such as carotenoid and capsaicinoid. Mineral elements such as calcium, magnesium, potassium, copper, iron, manganese, and sodium were also present in the spices, which are essential for human nutrition.

ÖZET

Biber, yemeklerde veya baharat olarak sıklıkla kullanılan önemli bir üründür. Biberlerde kapsaisinoidler, fenolikler ve karotenoidler de dahil olmak üzere çok sayıda fitokimyasal bulunmaktadır. Kapsaisinoidler, belirgin acı tat özelliğinden sorumludurlar. Bu çalışmada, 15 çeşit biber baharatının (1 İsot, 4 Toz Biber ve 10 Pul Biber) proksimat, mineral, yağ asidi kompozisyonu ve fitokimyasal bileşenleri karşılaştırmalı olarak incelenmiştir. Proksimat bileşimi analizi şunları ortaya koymuştur: nem (%6.54-19.49), kül (%6.53-22.48) ve asitte çözünmeyen kül (%0.41-1.12). Toplam fenolik madde içeriği 9.72 ile 20.05 mg GAE g^{-1} arasında değişmiştir. En düşük ve en yüksek kapsaisinoid içeriği sırasıyla S15 (10247.6 SHU) ve S9 (38861.7 SHU) örneklerinde bulunmuştur. Toplam karotenoid içeriğinin 739.8-1941.7 mg kg^{-1} arasında değiştiği belirlenmiştir. Fitokimyasal analizler, bu baharatların karotenoid ve kapsaisinoid gibi fitokimyasallar bakımından zengin olduğunu ortaya koymuştur. Baharatlarda insan beslenmesi için gerekli olan kalsiyum, magnezyum, potasyum, bakır, demir, mangan ve sodyum gibi mineral elementler de mevcut olarak bulunmuştur.

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INTRODUCTION

Pepper is a member of the *Solanaceae* family and belongs to the genus *Capsicum*. Peppers (*Capsicum annuum* L.), grown around the world, are very popular and crucial to the consumers, producers, and processing industries. Along with Mexico, peppers are also widely grown in China, Türkiye, and other countries. According to the 2021 data presented by the Food and Agriculture Organization (FAO) of the United Nations, the total production of various pepper varieties amounted to 36 million and 286 thousand tons. In this production, China ranked first with 16 million and 721 thousand tons, while Türkiye ranked second with 3 million and 91 thousand tons, and Indonesia ranked third with a production of 2 million and 747 thousand tons (FAOSTAT, 2021). Mexico and Spain occupied the fourth and fifth positions, respectively. Pepper is one of the main elements in most cuisines throughout the world and one of the most extensively produced spices in the world. As an important agricultural crop, pepper has a distinctive color, flavor, and aroma, as well as economic and nutritional value. It is also one of the most significant sources of essential nutrients, including protein, oil, dietary fiber, and fat-soluble vitamins. Researchers highlight the antibacterial, anti-cancerogenic, and antioxidant properties of their bioactive and functional components (Kim et al., 2018; Romero-Luna et al., 2023). Isot is the name of the most well-known pepper variety grown in Anatolia's South East region. Isot pepper is often consumed in powder form after being dried (Minguez-Mosquera et al., 1994; Koc et al., 2008).

Peppers (*Capsicum* spp.) are considered to have a high nutritional value containing phytochemicals. They can be consumed fresh, canned, roasted, or dried as a spice. Phytochemicals such as carotenoids, capsaicinoids, flavonoids, ascorbic acids, anthocyanins, vitamins, phenolic acids, and tocopherols are abundant in peppers. These substances are known to prevent inflammatory diseases caused by oxidative damage to preserve good health. Capsaicinoids are the constituents in peppers that are responsible for pungency. Scoville heat units (SHU), which are based on the concentrations of capsaicinoid chemicals in the fruit, are used to describe the level of pungency (Kim et al., 2019; Carvalho Lemos et al., 2019; Atalay & Inanc, 2020).

While the moisture and ash contents of spices play a decisive role in the shelf life and stability of the product, the pungency and color values are also crucial in terms of consumer preferences and usage. Additionally, total carotenoid and phenolic contents play a significant role in explaining the nutritional value and health benefits of spices. Fatty acid esters and element contents are critical for understanding the individual components of spices. In this context, a comprehensive analysis of 15 different pepper samples (red pepper, paprika, and isot pepper) sold in Türkiye was conducted, covering moisture, ash amounts, color values, capsaicinoid, total carotenoid and phenolic contents, fatty acid esters, and mineral contents. This study provides important data to assess the quality and nutritional content of pepper spices produced in Türkiye.

MATERIALS and METHODS

Pepper spice materials and sample preparation

Fifteen spice samples (1 Isot Pepper Flake, 10 Chili Pepper Flakes, and 4 Chili Powders) with five brands offered for sale in Türkiye (Southeastern Anatolia Region) were supplied from trade centers (Spice shops in Kilis, Gaziantep, Kahramanmaraş, Şanlıurfa provinces). All chemicals used in the analyses were obtained from Sigma and Merck. The names, coding, and characteristics of the spice samples are presented in Table 1. These samples were stored in an oven at 40 ^{o}C for 24 hours before being analyzed.

Table 1. Pepper spices types and characteristics

Çizelge 1. Biber baharat tipleri ve karakteristik özellikleri

Sample Code	Spice Types	Characteristics	Brand
S1	Isot Pepper	Isot Pepper Flakes	A
S2	Oily Hot Chili Pepper Flakes	Chili Pepper Flakes	A
S3	Hot Chili Pepper Flakes	Chili Pepper Flakes	A
S4	Hot Chili Pepper Flakes	Chili Pepper Flakes	A
S5	Sweet Chili Pepper Flakes	Chili Pepper Flakes	B
S6	Sweet Chili Pepper Flakes	Chili Pepper Flakes	C
S7	Oven Dried Chili Pepper Flakes	Chili Pepper Flakes	C
S8	Sun Dried Chili Pepper Flakes	Chili Pepper Flakes	C
S9	Fresh Chili Pepper Flakes	Chili Pepper Flakes	C
S10	Extra Chili Pepper Flakes	Chili Pepper Flakes	C
S11	Medium Chili Pepper Flakes	Chili Pepper Flakes	C
S12	Hot Chili Powder	Chili Powder	C
S13	Hot Chili Powder	Chili Powder	D
S14	Sweet Chili Powder	Chili Powder	D
S15	Sweet Chili Powder	Chili Powder	E

Determination of moisture content

The moisture content of the samples was measured according to the method of AOAC, 2005: 930.15, based on the principle of evaporating the water in the spice under a certain temperature and finding the amount of moisture by utilizing weight loss. The moisture content of 2 g spice samples was determined from the difference in the initial and final weights of the samples, which were kept in the Memmert brand UN55 model oven at 135 °C for two hours. The following equation was used for the moisture content calculation (AOAC Official Method 930.15, 2005).

$$Moisture\ Content\ \% = \left(\frac{Final\ Weights}{Initial\ Weights}\right) \times 100 \quad \textbf{Eq.(1)}$$

Determination of ash content

The weighed pepper spice sample was placed into a pre-weighed crucible. The sample was placed into the muffle furnace. 2 grams of spice samples were allowed to ash in a tared porcelain crucible by a Thermomac brand CMF7 model muffle furnace which was set to 550 °C for two hours. The amount of inorganic ash remaining without combustion was calculated according to the equation below (Anonymous, 1974).

Ash Content %= ((Weight of Ash) / (Dry Matter)) × 100 **Eq.(2)**

Determination of acid insoluble ash

25 ml of 10% HCl solution was added to 3 g of spice sample weighed in a porcelain crucible. The HCl solution, which was boiled for 10 minutes, was cooled and filtered through filter paper. The residue on the paper was washed with warm distilled water until the acid was removed. The filter paper, together with the residue inside, was combusted in a porcelain crucible at 525 °C for one hour. At the end of the combustion process, the crucible cooled in the desiccator was weighed with a precision balance. The amount of % acid insoluble ash was calculated according to the TS 1128 ISO 763 method with the following equation (Anonymous, 2000; Erol and Arpaci, 2023b).

$$Acid\ Insoluble\ Ash\ Content\ \% = m \times (100\ /\ M) \times (100\ /\ D) \quad \textbf{Eq.(3)}$$

where; M = Weight of the sample taken from the ground sample used in the determination of total ash (g)
m = Weight of acid insoluble ash (g)
D = Percent dry matter content of the ground sample by weight

Color analyses

Color analyses were performed using colorimetric and spectrophotometric methods. Colorimetric measurements were made by using a Techkon brand SpectroDens model colorimeter by taking L, a, and b values. The spectrophotometric method was applied according to the method of AOAC, 2005: 971,26. Spice samples of 0.1 g were kept in 100 ml acetone for 16 hours at room temperature. At the end of 16 hours, the absorbance against acetone was read with a Biocrome brand Libra S70 model spectrophotometer at a wavelength of 460 nm. The calculation was made according to the following equation (Horwitz, 2002).

$$ASTA = Abs \times 16.4 \times (If\ /\ m) \quad \textbf{Eq.(4)}$$

where; "m" is the weight of the sample, "If" is the deviation factor of the spectrophotometer calculated by dividing the absorbance by the true absorbance of the standard color solution (0.001 M $K_2Cr_2O_7$ and 0.09 M $(NH_4)_2Co(SO4)_2.6H_2O$; 1.8M H_2SO_4) at 460 nm.

Capsaicinoid measurements

The total amount of capsaicinoids in the spice samples was calculated by measuring capsaicin and dihydrocapsaicin (Zaki et al., 2013). The extraction steps of the capsaicinoids from spices are as follows: 1.0 g of spice sample was made up to 100 ml with 95% ethyl alcohol saturated with sodium acetate. It was kept in a water bath at 60 °C for three hours. It was passed through a 0.45 µm PTFE filter and placed in HPLC vials with a volume of 2 ml. HPLC (Shimadzu, Prominence Modular LC20A HPLC) system containing ultraviolet (UV) detector, C18 column (Hypersil ODS, C18, 5 µl, 4.6×250 mm) was used for the analysis. An isocratic mixture of 48.4% methanol, 30.2% water, 13.3% dioxane, 7.9% acetonitrile, and 0.2% perchloric acid (2%) was used as the mobile phase (Topaloglu, 2010; Firat et al., 2021). The injection volume was 10 µl, the detection wavelength was 280 nm, the analysis time was 20 min, and the mobile phase flow rate was 1 ml min^{-1}. The analysis process consisted of first reading the capsaicin and dihydrocapsaicin standard solutions into the device and then reading the spice extraction samples. The obtained data were converted into SHU (scoville heat unit) pungency unit according to the following equation.

$$Total\ Pungency\ (SHU) = (Capsaicin + (0.82 \times Dihydrocapsaicin) \times 16 \quad \textbf{Eq.(5)}$$

Total carotenoid content

Total carotenoid content analysis was performed using the spectrophotometric method (Martín-Diana et al., 2009). 1 g dry spice sample was taken and extracted with 10 ml acetone/water (90/10, v/v) mixture. After the extraction, the supernatant part of the samples, which was centrifuged at 5000 rpm, 4 °C, and 10 minutes, was taken. The extraction process was repeated five times until the colored supernatant part became colorless. Each time, colorless extracts were collected and analyzed by reading the absorbance in a spectrophotometer (Biochrom, Libra S70) at a wavelength of 471 nm against acetone. Total carotenoid content was calculated according to the following equation. β-carotene was used as the standard substance, and total carotenoid content was calculated from the calibration curve and expressed as milligrams of β-carotene equivalent per gram of fruit (mg β-carotene g^{-1} dry weight).

$$Total\ Carotenoid\ (mg/ml) = (\ Absmax\ \times\ 250 \times\ 10\ ml\ Aceton\ \times\ dilution\ factor\ )\ /\ (\ sample\ \ volume\ (ml))$$ **Eq.(6)**

Total phenolic content

The total phenolic content of the spice samples was determined by using the Folin–Ciocalteu method according to the methodology proposed by Castro-Concha et al. (2014). Briefly, 2.5 ml of diluted Folin-Ciocalteu reagent (1:10 v/v) was added to 0.5 ml of the extract solution in methanol. After 5 minutes, 2 ml of Sodium carbonate solution (7.5%) was added and the mixture was kept at room temperature in the dark for two hours. Absorbance was measured at 760 nm using a spectrophotometer (Biochrom brand, Libra S70 model). Gallic acid was used as a standard and the results were expressed in milligrams of gallic acid equivalent (GAE) per gram of spice.

Fatty acid composition

Extraction and esterification processes were performed to determine the fatty acid ester content of spice samples (Pérez-Gálvez et al., 1998). 10 g of spice sample was extracted with hexane by Soxhlet extraction (Buchi brand, model E-816) for four hours. After oil extraction, extracted spice samples were filtered and separated from the dry matter. Esterification was performed using 0.1N KOH and analyzed in a GC/MS (Agilent, 7890B GC-5977MSD) instrument. Components were separated by a DB-WAX (30m, 0.32mm, 0.25µm) column and analyzed on a mass detector (5977MSD). The temperature program was applied by modifying it according to the reference method. The oven temperature was first increased from 110 ^{o}C to 175 ^{o}C by increasing 10 ^{o}C per minute and kept at this temperature for 20 minutes. Then, the temperature was increased to 240 ^{o}C by increasing 10 ^{o}C per minute and kept at this temperature for 5 minutes. Detector and injection temperature were kept constant at 250 ^{o}C.

Mineral content

The copper, magnesium, potassium, iron, sodium, calcium, and manganese contents of the spice samples were determined using Atomic Absorption Spectrometer (Perkin Elmer brand, 240 FS AA model) and Flame Photometer (Jenway brand, PFP 7 model). The spice samples were dried in a compressed air oven at 72°C, and dried samples were pulverized by using a pestle. 0.25 g of the sample was mixed with 9 ml of HNO_3 and 3 ml of H_2O_2 and burned in a Cem brand Mars 6 model microwave oven under 200W power for 30 minutes. The burned samples were filtered on Whatman filter paper and diluted by adding distilled water to a final volume of 25 ml. Calcium (Ca), Magnesium (Mg), Iron (Fe), Manganese (Mn), and Copper (Cu) contents were determined by Atomic Absorption Spectrophotometer while Sodium (Na) and Potassium (K) contents were determined by Flame Photometer (Tefera and Chandravanshi, 2018; Mengel et al., 1984).

Statistical analysis

Statistical analyses of the data were performed for all parameters. Analysis of variance and multiple comparison tests in all spice samples were performed with the JMP (15.1.0) program. Student's t-test was used for multiple comparisons. For the parameters in the analysis of variation, any p-value below 0.05 was considered significant.

RESULTS and DISCUSSIONS

Ash, moisture, and acid insoluble ash content

Moisture, ash, and acid insoluble ash amounts were measured and calculated for 15 different pepper spices and are given in Table 2. According to the results of chemical analysis; it was determined that moisture content ranged between 6.95% and 8.11% in chili powder samples, between 6.54%-19.49% in chili pepper samples and it was 9.88% in the isot pepper sample. High moisture content may be an indicator of spice freshness. Spices have a longer shelf

life due to their low moisture content and can be stored at normal room temperature without refrigeration, making them safe for consumption. However, high moisture content can promote the growth of microorganisms (Ene-Obong et al., 2018). Ash content (in dry matter) was found as 9.86-14.25% in chili powder, 6.53-22.48% in chili peppers, and 12.89% in isot pepper spices. The low or high amount of ash indicates the presence of inorganic materials left unburned. The amount of ash insoluble in HCl in the spice samples was determined as 0.97%-1.12% in chili powder samples, 0.41%-0.96% in chili pepper samples, and 0.98% in isot pepper sample. The amount of ash remaining undissolved in HCl is an indicator of the mineral substances added to the spice from the outside. Considering the data obtained from these analyses, it was observed that the moisture content of some samples (S2, S3, S4, S8, S9, S11) (>15%, m/m) and ash content of one sample (S7) (>17%, m/m) were not in accordance with the Spice Report of the Turkish Food Codex Spice Report (Turkish Food Codex, 2002); while the percentage of ash dissolved in HCl and the spices except chili powder (S12, S13, S14) (>1%, m/m) were in accordance with the Turkish Food Codex Spice Report.

Table 2. Moisture, ash, and acid-insoluble ash content results in the pepper spice samples
Çizelge 2. Biber baharat örneklerinde nem, kül ve asitte çözünmeyen kül içeriği sonuçları

Sample Code	Moisture (m/m%)	Ash (m/m%)	Acid Insoluble Ash (m/m%)
S1	9.88±0.52^{g}	12.89±0.17^{e}	0.98±0.06^{c}
S2	19.49±4.10^{a}	16.52±0.53^{b}	0.79±0.24^{d}
S3	15.75±2.04^{d}	10.32±1.65^{i}	0.96±0.09^{c}
S4	18.71±0.67^{b}	12.32±1.62^{f}	0.59±0.12^{g}
S5	6.54±0.57^{k}	6.53±2.26^{l}	0.48±0.73^{i}
S6	8.16±0.64^{h}	8.59±1.79^{k}	0.76±0.31^{e}
S7	10.41±0.95^{f}	22.48±1.83^{a}	0.96±0.16^{c}
S8	16.03±0.67^{d}	13.58±2.14^{d}	0.96±0.25^{c}
S9	16.38±0.74^{c}	12.95±2.01^{e}	0.56±0.27^{h}
S10	12.27±0.96^{e}	10.78±1.79^{h}	0.73±0.32^{f}
S11	15.86±1.27^{d}	11.96±0.37^{g}	0.41±0.14^{j}
S12	8.11±0.97^{hi}	14.25±2.12^{c}	1.11±0.46^{a}
S13	7.79±1.14^{i}	10.05±1.27^{ij}	1.12±0.54^{a}
S14	6.95±0.42^{j}	9.86±0.51^{j}	1.06±0.19^{b}
S15	8.02±1.17^{hi}	11.13±1.73^{h}	0.97±0.14^{c}

Means with different superscripts in the same column indicate statistical significance ($p<0.05$).

Color and capsaicinoid content

The ASTA value, a parameter defined by the American Spice Trade Association to evaluate the color of red peppers, ranged from 10.32 to 70.01 ASTA ($p < 0.001$) for the spice samples studied. The ASTA values of the samples are shown in Table 3. It has been reported that the color ASTA value can be used as an indicator of the total carotenoid content in the quality evaluation of the pepper spices (Mínguez-Mosquera & Pérez-Gálvez, 1998). In this regard, a very strong correlation was found between the values of ASTA and the total carotenoid content of the samples ($r = 0.846$, $p < 0.05$). These results indicated that the ASTA values are a reliable measure for estimating the carotenoid content of the pepper spice samples. It was reported in the literature that the ASTA values of 52 different pepper spices of different origins ranged from 101 ± 28.3 to 140 ± 35.4 (Molnár et al., 2018). Chatterjee et al. (1999) studied the changes in color values of three red pepper cultivars as a function of storage time and abiotic stress conditions and they observed significant ($p<0.05$) storage time-dependent decreases in color values with advancing storage in both stressed and non-stressed samples of all cultivars. Reddy et al. (2023) have also reported that the same

product dried by different methods may have different product qualities and that chemical constituents and color changes may vary with the drying method. It was emphasized that drying at low temperatures and humidity is necessary to maintain the color of the product. In the colorimetric color measurements, the L, a, and b values varied between 11.41-37.77 ($p < 0.05$), 11.40-36.48 ($p < 0.05$), and 3.98-43.02 ($p < 0.05$), respectively in the present study (Table 3). The smallest L-value was measured for the S7 spice sample, while the highest L-value was for the spice with the sample S5. In the analysis of the a-values, the lowest value was found in sample S5, and the highest a-value was found in the sample S7, while the lowest and highest b-values were obtained in the spice samples of S5 and S7, respectively. It was observed that the color of the spice samples became lighter and shifted to red with decreasing particle size; a dark or black final product was obtained with increasing particle size. Cervantes-Paz et al. (2014) studied antioxidant, chlorophyll, and carotenoid content in maturing jalapeño pepper cultivars. They concluded that the degradation and browning of carotenoids caused the discoloration of red pepper fruits with a heat effect. Horváth and Hodúr (2007) found that the changes in L, a, and b values in color measurements in spices made from red pepper could vary depending on the moisture content. It was also found that the a-values were shaped as a function of the organic acids contained in the fruit and that the a-values decrease with increasing amounts of acetic and citric acids in the fruit.

Table 3. Color results in the pepper spice samples
Çizelge 3. Biber baharat örneklerinde renk sonuçları

Sample Code	ASTA Values	Color Measurements		
		L	a	b
S1	12.89±1.51^{k}	30.94±4.25^{d}	17.74±4.52^{d}	37.94±0.22^{c}
S2	16.52±1.72^{j}	11.47±5.17^{k}	19.02±0.74^{c}	12.61±6.28^{l}
S3	10.32±1.28^{m}	23.54±1.35^{g}	14.25±0.47^{g}	39.05±0.34^{b}
S4	12.32±0.17^{l}	21.71±2.78^{h}	17.19±1.05^{e}	27.89±0.23^{g}
S5	22.12±0.53^{i}	37.77±3.33^{a}	11.40±0.52^{j}	3.98±0.44^{n}
S6	23.75±1.65^{h}	12.48±0.64^{j}	12.78±0.25^{i}	19.41±0.57^{j}
S7	70.01±1.62^{a}	11.41±0.61^{k}	36.48±3.56^{a}	43.02±0.35^{a}
S8	34.08±2.26^{c}	31.93±0.33^{c}	17.99±1.22^{d}	33.92±0.30^{d}
S9	27.42±1.79^{e}	36.77±0.40^{b}	17.76±3.94^{d}	16.15±0.83^{k}
S10	28.92±1.83^{d}	24.09±2.42^{f}	14.29±4.11^{g}	27.52±0.44^{g}
S11	38.19±2.14^{b}	25.10±3.08^{e}	15.07±4.53^{f}	28.36±0.19^{f}
S12	26.56±2.01^{f}	21.45±2.28^{h}	19.92±1.18^{b}	31.72±1.39^{e}
S13	24.89±1.79^{g}	32.27±2.67^{c}	13.84±3.76^{h}	24.16±0.71^{i}
S14	28.84±0.37^{d}	12.97±2.27^{i}	12.97±4.41^{i}	10.27±0.17^{m}
S15	34.08±2.12^{c}	24.36±2.55^{f}	15.03±0.51^{f}	27.11±0.32^{h}

Means with different superscripts in the same column indicate statistical significance ($p<0.05$).

Capsaicinoids are the chemical substances that determine the pungency and sweetness of red pepper. The capsaicin and dihydrocapsaicin content of all spice samples was determined by high-pressure liquid chromatography (HPLC). Table 4 shows the capsaicinoid contents of the samples converted to SHU pungency units. The hottest sample analyzed was S9 with a value of 38861.7 SHU ($p>0.05$), while the least pungent spice was S15 with a value of 10247.6 SHU ($p > 0.05$). In almost all studies, capsaicinoids were found to accumulate in the early stages of fruit development and continued to accumulate to their maximum during ripening. It is reported that pepper varieties have different degrees of pungency (Gaur et al., 2016). Several studies have been conducted on the accumulation of capsaicinoids in Capsicum fruits with maturity and developmental stage (Olguín-Rojas et al., 2019; Vázquez-Espinosa et al., 2023). The differences in the amount of capsaicinoids in the spice types studied were

thought to depend on the bell pepper varieties/species and the ripening periods in which they were collected (Duman et al., 2021). The different levels of pungency in spice samples of the same or different brands suggest that the red bell pepper fruits were harvested and processed at different ripening times. In addition, it has been reported that losses of capsaicinoids may occur during the drying period of red peppers, possibly due to oxidation by peroxidase activity (Bernal et al., 1993). Temperature and spice size are also thought to play a role in oxidation and drying time may also be an important factor affecting pungency.

Total carotenoid and total phenolic content

The total phenolic content of the spice samples produced from red pepper was measured between 9.7 mg GAE g^{-1} and 20.0 mg GAE g^{-1} ($p<0.05$) (Table 4). It can be said that the S1 coded isot sample was the spice with the highest phenolic content with 20.05 mg GAE g^{-1}. The spice sample with the least amount of phenolic substances was chili powder with the S13 code. In the study in which spices from isot and four different red peppers in the market in Türkiye were investigated, the total phenolic content was found as 10.4 and 11.9 mg GAE g^{-1} (Coskun & Unsal, 2020). Papathanasiou et al. (2021) reported that the amount of phenolic compounds contained in different types of peppers may vary depending on the ripeness stage of the pepper fruits and emphasized the importance of the ripeness stage at which the fruits were harvested.

Table 4. Capsaisinoid and phytochemical property results in pepper spice samples
Çizelge 4. Biber baharat örneklerinde kapsoisinoid ve fitokimyasal özellik sonuçları

Code	Capsaicinoids (SHU)	Total Carotenoid (mg kg^{-1})	Total Phenolic Content (mg GAE g^{-1})
S1	32 850.6±10.25^{b}	739.8±13.09^{j}	20.0±2.24^{a}
S2	29 347.2±13.56^{e}	1 941.7±10.29^{a}	18.5±1.14^{c}
S3	32 167.2±17.22^{c}	1 694.4±16.89^{d}	16.5±1.84^{d}
S4	30 290.3±23.94^{d}	1 390.6±12.88^{f}	15.7±1.75^{e}
S5	23 726.6±14.11^{i}	1 099.7±11.23^{i}	19.4±0.94^{b}
S6	13 158.6±24.53^{k}	1 478.3±10.97^{e}	16.7±1.15^{d}
S7	30 253.5±11.18^{d}	1 740.3±9.99^{d}	18.6±1.01^{c}
S8	27 574.8±10.74^{f}	1 907.2±10.22^{ab}	10.1±0.69^{i}
S9	38 861.7±17.46^{a}	1 826.9±19.14^{bc}	12.7±1.02^{g}
S10	12 131.5±12.30^{l}	1 309.4±8.02^{gh}	19.1±0.65^{bc}
S11	24 343.5±10.78^{h}	1 751.0±35.18^{cd}	14.9±3.76^{f}
S12	24 761.1±13.17^{g}	1 369.2±5.21^{fg}	11.6±0.65^{h}
S13	14 261.9±20.39^{j}	1 263.7±12.21^{h}	9.7±1.13^{i}
S14	12 153.2±13.76^{l}	1 856.9±7.73^{b}	12.0±1.83^{h}
S15	10 247.6±24.09^{m}	1 845.4±5.90^{b}	12.2±0.20^{gh}

Means with different superscripts in the same column indicate statistical significance ($p<0.05$).

The carotenoid contents of the spice samples are also given in Table 4. The data show that there were significant differences between the carotenoid contents of the spice samples ($p<0.05$). It was found that the carotenoid levels of the spice samples used in our study ranged from 739.8 to 1 941.7 mg kg^{-1}. While the total carotenoid content in the S1-coded isot sample was the lowest at 739.8 mg kg^{-1}, the highest carotenoid content was in the S2-coded spice sample at 1 941.7 mg kg^{-1}. The total carotenoid content of the S7 oven-dried spice sample and the S8 sun-dried spice sample was 1 740.3 mg kg^{-1} ($p > 0.05$) and 1 907.2 mg kg^{-1} ($p > 0.05$), respectively. It is known that heat treatments and different preservation methods for the spices cause changes in carotenoid levels. Korkmaz et al. (2021) stated that the carotenoid content of isot and chili pepper samples decreased due to the oxidation of carotenoids during the sun-drying process in carotenoid measurements using the HPLC system. Similarly, it was

reported that the greater decrease in some spices could be mainly due to heat treatment, which can also cause thermal degradation of carotenoids (Daood et al., 2006). Topuz and Ozdemir (2003) reported a decrease of about 81.11% in total carotenoid concentration in red pepper subjected to sun drying. The determination of different levels of carotenoids in spice products of the same brand indicated that thermal oxidation changes with the decrease in product particle size and causes a decrease in carotenoids. It was concluded that the lowest total carotenoid content in S1-encoded isot spice was due to the different processes of thermal applications of isot.

Fatty acid composition

The fatty acid methyl esters found in the oils extracted from spices are shown in Table 5. The most abundant major fatty acids in the oil samples were linoleic acid, palmitic acid, oleic acid, linolenic acid, stearic acid, and myristic acid. Minor fatty acids included lauric acid, palmitoleic acid, arachidic acid, and behenic acid. Different amounts of SFAs, MUFAs, and PUFAs were detected in all spice samples. When the saturated and unsaturated fatty acids are compared, it was seen that the ratio of unsaturated fatty acids was considerably higher than the ratio of saturated fatty acids.

Table 5. Fatty acid composition in the pepper spice samples
Çizelge 5. Biber aharat örneklerinde yağ asidi kompozisyonu

Sample Code	(%) Lauric A. (C12:0)	(%) Myristic A. (C14:0)	(%) Palmitic A. (C16:0)	(%) Palmitoleic A. (C16:1)	(%) Stearic A. (C18:0)	(%) Oleic A. (C18:1)	(%) Linoleic A. (C18:2)
S1	0.81±0.18^{j}	1.86±0.69^{a}	11.95±0.44^{bc}	0.42±0.25^{de}	2.73±0.09^{d}	8.43±0.13^{a}	69.87±7.46^{d}
S2	1.31±0.43^{f}	1.67±0.46^{de}	11.63±0.79^{cd}	0.41±0.13^{ef}	2.87±0.69^{bcd}	8.50±0.06^{a}	70.17±3.95^{d}
S3	1.40±0.36^{de}	1.81±0.69^{abc}	13.06±0.96^{a}	0.45±0.17^{bc}	2.13±0.46^{f}	7.36±0.22^{b}	70.17±4.62^{d}
S4	1.41±0.23^{d}	1.72±0.13^{bcde}	11.81±0.59^{bc}	0.48±0.14^{a}	3.07±0.10^{a}	7.09±0.18^{bc}	68.38±7.54^{de}
S5	1.42±0.51^{cd}	1.71±0.34^{cde}	12.31±0.48^{b}	0.48±0.33^{a}	2.73±0.17^{d}	8.69±0.05^{a}	65.51±3.39^{e}
S6	1.30±0.25^{f}	1.48±0.13^{f}	10.94±0.76^{ef}	0.34±0.22^{h}	2.47±0.26^{e}	8.71±0.11^{a}	78.22±4.54^{a}
S7	1.55±0.12^{ab}	1.82±0.44^{ab}	11.69±0.96^{bcd}	0.34±0.29^{h}	2.93±0.29^{abc}	6.79±0.36^{cd}	71.96±1.79^{bcd}
S8	1.49±1.02^{bc}	1.80±0.57^{abc}	10.94±0.96^{ef}	0.48±0.05^{a}	2.10±0.01^{f}	6.72±0.27^{cd}	76.79±0.26^{a}
S9	1.62±0.59^{a}	1.85±0.35^{a}	10.88±0.56^{ef}	0.38±0.16^{g}	3.07±0.22^{a}	6.13±0.17^{f}	70.17±2.92^{d}
S10	1.22±0.32^{g}	1.49±0.48^{f}	11.31±0.73^{cde}	0.48±0.06^{a}	2.84±0.22^{cd}	8.79±0.04^{a}	71.78±7.35^{bcd}
S11	1.55±0.48^{ab}	1.74±0.83^{bcd}	11.31±0.41^{cde}	0.41±0.21^{ef}	2.87±0.24^{bcd}	6.44±0.09^{de}	75.36±3.47^{ab}
S12	1.33±0.18^{ef}	1.63±0.44^{e}	10.19±0.53^{g}	0.34±0.27^{h}	2.93±0.27^{abc}	6.26±0.29^{ef}	75.01±5.46^{abc}
S13	0.75±0.29^{i}	1.76±0.19^{abcd}	10.58±0.15^{fg}	0.39±0.17^{fg}	2.81±0.20^{cd}	6.79±0.89^{cd}	72.19±1.83^{bcd}
S14	0.58±0.17^{j}	1.81±0.88^{abc}	10.95±0.81^{ef}	0.47±0.14^{ab}	2.96±0.17^{abc}	6.68±0.88^{cde}	70.86±4.08^{cd}
S15	1.07±0.34^{h}	1.67±0.41^{de}	11.09±0.12^{def}	0.44±0.21^{cd}	3.01±0.18^{ab}	7.08±0.23^{bc}	71.95±3.26^{bcd}

MUFA: Monounsaturated fatty acids, SFA: Saturated fatty acids, UFA: Unsaturated fatty acids and PUFA: Polyunsaturated fatty acids

Means with different superscripts in the same column indicate significant differences ($p<0.05$).

The SFAs found in the oils extracted from spices were C12:0, C14:0, C16:0, C18:0, C20:0. Palmitic acid (C16:0) was the dominant SFA in all spice samples. The highest C16:0 content was found in sample S3 (13.06±0.96%) and the lowest in sample S12 (10.19±0.53%) (Table 5). The MUFAs identified were C16:1 and C18:1. The lowest content and the highest content of oleic acid (C18:1) were S9 (6.13 %) and S10 (8.79 %), respectively. Table 5 shows that all samples contained PUFAs (C18:2 and C18:3). Linoleic acid (C18:2) as the major unsaturated fatty acid ester was measured in all spice samples. It was determined that the oils obtained from the spice samples are influenced by the oils used for spice making and externally added oils or seeds added to the spice. The spice sample with code S6

had the highest linoleic acid content of 78.22 %. In a study investigating fatty acid esters from red pepper powder using different extraction methods, the C18:2 content was 59.4% and the C18:3 content was 5.1% (Rutkowska & Stolyhwo, 2009). The ΣPUFA/ΣSFA ratio has great importance in human nutrition, a ratio higher than 1 is accepted as an edible fat with high nutritional value (Guici El Kouacheur et al., 2023; Gumus & Erol, 2023). In all spice samples, this ratio ranged between the values of 4.32 and 18.39 in the present study (Table 5). Based on these data, it can be said that all spice samples analysed in the current study are highly beneficial for human health in terms of fatty acids.

Various studies in the literature were shown that food sources rich in PUFAs reduce the risk of cardiovascular diseases (Sánchez et al., 2021). In addition, linoleic acid in the diet plays a positive role in the prevention of diseases such as coronary heart disease and cancer and has beneficial physiological effects. In particular, the fact that linoleic acid cannot be formed endogenously in the body in humans emphasizes the need to obtain this fatty acid from outside the body. The fact that linoleic acid is the most abundant unsaturated fatty acid in the oils of red pepper spices explains the importance of these oils in human nutrition (Dubois et al., 2007).

Table 5 (continued). Fatty acid composition in the pepper spice samples

Çizelge 5 (devamı). Biber aharat örneklerinde yağ asidi kompozisyonu

Sample Code	(%) Linolenic A. (C18:3)	(%) Arachidic A. (C20:0)	(%) Behenic A. (C22:0)	ΣMUFA	ΣSFA	ΣUFA	ΣPUFA	ΣPUFA/ΣSFA
S1	4.28±0.52^{de}	0.81±0.09^{ab}	0.47±0.17^{ef}	8.85	18.16	83.00	74.15	18.13
S2	4.33±2.14^{d}	0.71±0.26^{ef}	0.43±0.04^{h}	8.91	18.19	83.41	74.51	15.69
S3	4.27±0.34^{de}	0.55±0.42^{h}	0.46±0.05^{fg}	7.81	18.95	82.25	74.44	17.97
S4	4.77±0.72^{bc}	0.84±0.27^{a}	0.51±0.27^{cd}	7.57	18.85	80.72	73.15	16.93
S5	4.77±1.28^{bc}	0.72±0.16^{def}	0.54±0.34^{ab}	9.17	18.89	79.45	70.28	16.04
S6	3.68±1.73^{g}	0.65±0.09^{g}	0.49±0.33^{de}	9.05	16.84	90.95	81.90	17.21
S7	4.69±1.65^{c}	0.76±0.14^{cd}	0.55±0.26^{ab}	7.13	18.75	83.78	76.65	16.74
S8	4.12±0.28^{def}	0.72±0.23^{def}	0.44±0.05^{gh}	7.2	17.05	88.11	80.91	18.39
S9	5.17±1.42^{a}	0.77±0.13^{bc}	0.50±0.22^{d}	6.51	18.19	81.85	75.34	17.37
S10	4.26±1.46^{de}	0.74±0.22^{cde}	0.53±0.09^{bc}	9.27	17.6	85.31	76.04	4.32
S11	4.33±0.34^{d}	0.72±0.13^{def}	0.42±0.08^{h}	6.85	18.19	86.54	79.69	4.38
S12	4.97±1.52^{ab}	0.73±0.12^{cdef}	0.56±0.16^{a}	6.6	16.81	86.58	79.98	4.75
S13	3.99±1.09^{f}	0.74±0.14^{cde}	0.46±0.12^{fg}	7.18	16.64	83.36	76.18	4.57
S14	4.21±0.86^{def}	0.77±0.15^{bc}	0.42±0.19^{h}	7.15	17.07	82.22	75.07	4.39
S15	4.07±0.62^{ef}	0.69±0.15^{fg}	0.49±0.04^{de}	7.52	17.53	83.54	76.02	4.33

MUFA: Monounsaturated fatty acids, SFA: Saturated fatty acids, UFA: Unsaturated fatty acids and PUFA: Polyunsaturated fatty acids

Means with different superscripts in the same column indicate significant differences ($p<0.05$).

Mineral contents

The major and trace nutrient contents of the spice samples prepared from red pepper were measured using AAS and flame photometer in the present study. Table 6 shows that the spice samples contained calcium (Ca), magnesium (Mg), potassium (K), copper (Cu), iron (Fe), manganese (Mn), and sodium (Na). The mineral content of each spice sample varied and there were statistically significant differences between some samples ($p<0.05$). Ca values ranged between 96.81 mg kg^{-1} and 137.55 mg kg^{-1}; Mg values between 144.41 mg kg^{-1} and 173.77 mg kg^{-1}; K values between 3 137.02 mg kg^{-1} and 4 281.78 mg kg^{-1}; Cu values between 1.02 mg kg^{-1} and 1.84 mg kg^{-1}; Fe values between 6.15 mg kg^{-1} and 9.47 mg kg^{-1}; Mn values between 0.83 mg kg^{-1} and 1.14 mg kg^{-1} and Na values between

19.88 mg kg^{-1} and 38.13 mg kg^{-1}. There were significant differences between different spice samples ($p<0.05$). The highest amounts of Ca, Mg, Fe, and Mn were measured in sample S9 with 137.55 mg kg^{-1}, 173.77 mg kg^{-1}, 9.47 mg kg^{-1}, and 1.14 mg kg^{-1}, respectively, while the highest amount of K was determined in the sample S15 with 4 281.78 mg kg^{-1}, the highest amount of Cu was determined in the sample S8 with 1.84 mg kg^{-1} and the highest amount of Na was determined in the spice sample S2 with 38.13 mg kg^{-1}. The main constituents of all spice samples were K, Mg, and Ca. Guil-Guerrero et al. (1998) studied different pepper varieties and found similar results of elemental analysis. All spice types had similar nutrient content. It was determined that different processing methods and comminution processes do not affect nutrients (Bhandari et al., 2012). In the study conducted by Zou et al. (2015), it was found that the content of some macro and micro elements may vary depending on the bell pepper species and the ecological conditions of the growing region during the different ripening periods of pepper. Since spices are usually consumed in small amounts, such nutrient analysis can help consumers with nutrient intake and diet planning (Erol & Arpaci, 2023a). The mineral content of spice samples prepared from red pepper is significant and diverse. Such analysis can help us understand the nutritional value of spices in terms of health and provide consumers with a healthy dietary option.

Table 6. Mineral content results of the pepper spice samples
Çizelge 6. Biber baharat örneklerinde mineral içerik sonuçları

Sample Code	Ca (mg kg^{-1})	Mg (mg kg^{-1})	K (mg kg^{-1})	Cu (mg kg^{-1})	Fe (mg kg^{-1})	Mn (mg kg^{-1})	Na (mg kg^{-1})
S1	111.51±3.08^{f}	169.92±5.57^{ab}	3 375.73±10.56^{g}	1.02±0.14^{j}	7.63±0.91^{e}	0.93±0.94^{d}	34.52±0.47^{b}
S2	101.33±9.31^{g}	165.32±1.49^{bc}	4 116.93±19.09^{b}	1.04±0.19^{j}	7.26±0.59^{fg}	0.93±0.83^{d}	38.13±4.47^{a}
S3	97.44±5.50^{h}	160.29±3.95^{de}	3 137.02±32.74^{h}	1.14±0.95^{i}	6.15±1.19^{h}	0.83±0.19^{e}	24.13±0.53^{g}
S4	96.81±6.18^{h}	154.51±7.23^{g}	3 741.02±2.03^{de}	1.36±0.17^{cd}	7.13±1.09^{g}	0.93±0.42^{d}	30.48±1.24^{c}
S5	111.2±4.19^{f}	157.18±4.48^{ef}	3 965.42±1.78^{c}	1.18±0.44^{h}	7.13±1.86^{g}	1.04±0.67^{b}	24.32±0.94^{g}
S6	103.74±2.57^{g}	149.91±5.09^{g}	4 165.98±20.46^{b}	1.38±0.84^{bc}	7.38±0.6^{f}	0.93±0.81^{d}	30.69±1.24^{c}
S7	114.14±5.41^{ef}	159.11±0.08^{def}	4 142.08±1.22^{b}	1.14±0.71^{i}	8.24±0.43^{cd}	0.93±0.78^{d}	26.87±2.71^{e}
S8	121.59±6.74^{c}	159.32±0.62^{de}	3 725.62±15.82^{de}	1.84±0.26^{a}	8.61±0.96^{b}	0.93±0.65^{d}	28.67±0.74^{d}
S9	137.55±7.32^{a}	173.77±0.94^{a}	3 819.36±8.66^{d}	1.33±0.12^{de}	9.47±2.82^{a}	1.14±0.65^{a}	25.49±1.94^{f}
S10	130.62±1.11^{b}	155.69±0.83^{ef}	3 491.27±1.81^{f}	1.27±0.74^{g}	7.13±1.67^{g}	1.04±0.41^{b}	20.56±2.16^{i}
S11	116.34±7.13^{de}	157.08±1.41^{ef}	3 662.45±4.57^{e}	1.41±0.91^{b}	8.12±0.93^{d}	0.93±0.71^{d}	22.83±0.46^{h}
S12	132.62±4.28^{b}	172.16±10.19^{a}	3 975.23±1.31^{c}	1.29±0.28^{fg}	9.35±0.34^{a}	1.04±0.28^{b}	24.74±1.59^{fg}
S13	118.42±3.49^{cd}	144.41±2.34^{h}	3 212.71±1.17^{h}	1.15±0.94^{hi}	7.12±0.94^{g}	0.96±0.48^{c}	19.88±3.58^{i}
S14	130.72±0.56^{b}	170.33±3.04^{a}	4 078.24±3.58^{bc}	1.32±0.69^{ef}	7.76±0.16^{e}	1.13±0.69^{a}	24.55±2.46^{g}
S15	119.19±2.08^{cd}	163.67±1.16^{cd}	4 281.78±2.74^{a}	1.82±0.96^{a}	8.47±0.46^{bc}	1.02±0.64^{b}	29.47±1.74^{d}

Means with different superscripts in the same column indicate significant differences ($p<0.05$) analyzes.

In conclusion, this study conducted experimental analyses on 15 different red pepper spice products from various brands available and consumed in the Turkish market. The parameters investigated included moisture and ash content, spiciness and color values, total carotenoid amounts, total phenolic contents, fatty acid esters, and elemental compositions. It was observed that the products sold as spices generally did not comply with the Turkish Food Codex Spice Regulation, showing significant disparities, especially in terms of moisture, ash, and insoluble ash in acid parameters. However, in terms of spiciness, color, elemental composition, total phenolic, and total carotenoid contents, the products were found to be in harmony with the literature. The findings of this research are anticipated to make significant contributions to the spice industry and nutrition field. The results underscore that spices are rich sources of nutrients, minerals, and phytochemicals, emphasizing their potential use as

nutritional supplements. Furthermore, this study suggests that processing methods and grinding processes do not significantly affect the nutritional values of spices.

STATEMENT OF CONFLICT OF INTEREST

The authors declare that there is no conflict of interest between them.

AUTHOR'S CONTRIBUTIONS

UHE; all analyses, writing and interpretation of the article. PG; writing and control of the article. BBA; selection of the topic, procurement of materials and control of the article. The authors read and approved the final manuscript.

STATEMENT OF ETHICS CONSENT

This article does not require ethical approval as there are no experiments with human or animal subjects.

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