SİDAS MEDYA
Akademik Gıda®
ISSN Online: 2148-015X
https://dergipark.org.tr/tr/pub/akademik-gida

Akademik Gıda 22(3) (2024) 215-223, DOI: 10.24323/akademik-gida.1603870

Research Paper / Araştırma Makalesi

Quality Assessment and Bioactive Component Analysis of Honey from Different Geographical Regions in Erzurum, Türkiye

Adem Savaş[1], Halil İbrahim Binici[2] ✉, İhsan Güngör Şat[1], Mustafa Kılıç[1]

[1]Department of Food Engineering, Faculty of Agriculture, Ataturk University, 25240 Erzurum, Türkiye
[2]Department of Nutrition and Dietetics, Faculty of Health Sciences, İstanbul Esenyurt University, İstanbul, Türkiye

Received (Geliş Tarihi): 05.02.2024, Accepted (Kabul Tarihi): 28.11.2024
✉ *Corresponding author (Yazışmalardan Sorumlu Yazar): halilibrahimbinici@esenyurt.edu.tr (H.İ. Binici)*
+90 212 347 74 70 +90 212 699 09 90

ABSTRACT

In recent years, there has been an increasing interest on foods that are perceived to be healthy and functional among societies. The composition of honey, which plays an important role in human nutrition, is influenced by a number of factors. The aim of this study was to analyze fifteen different honey samples according to various quality criteria and to determine their 5-hydroxymethylfurfural (HMF) and phenolic components. In this context, honey samples were obtained from five distinct geographical regions within the Erzurum city area in Türkiye. There were statistically significant ($P<0.05$) differences in the pH, moisture, total sugar, reducing sugar, sucrose, proline, 5-hydroxymethylfurfural (HMF) and phenolic content values among the honey samples. HMF contents of samples varied between 5.20 and 108.12 mg/kg, and their phenolic contents ranged from non-detected to 202.95 mg/kg. While the HMF contents of honey samples were in accordance with the Turkish Food Codex Honey Communiqué (Communiqué No: 2020/7), with the exception of only one sample in terms of its proline and HMF contents.

Keywords: Honey, 5-Hydroxymethylfurfural, Proline, Phenolic components, Principal component analysis

Erzurum'un Farklı Coğrafi Bölgelerinden Elde Edilen Balların Kalite Değerlendirmesi ve Biyoaktif Bileşen Analizi

ÖZ

Son yıllarda, toplumlar arasında sağlıklı ve işlevsel olarak algılanan gıdalara olan ilgide kayda değer bir artış olmuştur. İnsan beslenmesinde önemli bir rol oynayan balın bileşimi çeşitli faktörlerden etkilenmektedir. Bu çalışmanın amacı, on beş farklı bal örneğini çeşitli kalite kriterlerine göre analiz etmek ve 5-hidroksimetilfurfural (HMF) ve fenolik bileşen içeriklerini belirlemektir. Bu kapsamda Erzurum ili sınırları içerisinde yer alan beş farklı coğrafi bölgeden bal örnekleri temin edilmiştir. pH, nem, toplam şeker, indirgen şeker,sükroz, prolin, 5-hidroksimetilfurfural (HMF) ve fenolik bileşen değerleri arasında istatistiksel olarak önemli ($P<0.05$) bir fark olduğu tespit edilmiştir. Bal örneklerinin HMF içeriklerinin 5.20-108.12 mg/kg arasında, fenolik bileşen içeriklerinin ise nd-202.95 mg/kg arasında değiştiği belirlenmiştir. Bal örneklerinin HMF içeriklerinin Türk Gıda Kodeksi Bal Tebliği'ne (Tebliğ No: 2020/7) uygun olduğu belirlenirken, pirolin ve HMF içerikleri bakımından sadece bir örneğin kodeksle uyumlu olmadığı tespit edilmiştir.

Anahtar Kelimeler: Bal, 5-hidroksimetilfurfural, Pirolin, Fenolik bileşen, Temel bileşen analizi

INTRODUCTION

The global population is expanding at an accelerated rate, with projections indicating that it will reach 10 billion by 2050. In light of these demographic projections, it is reasonable to anticipate an expansion in food demand in conjunction with population growth. In the era, there is a discernible inclination towards the consumption of safe food, particularly in light of the growing awareness among consumers. In order to safeguard consumer interests and facilitate the sound advancement of the food industry, the battle against food fraud represents a pivotal element of food quality control. One of the most significant challenges facing the food industry is the issue of food fraud. Fraudulent practices have the potential to disrupt the chemical composition and bioactive components of food products, thereby reducing their structural integrity and overall quality. One of the most susceptible products to counterfeiting is honey [1-7].

Honey is a sweet, natural product derived from animal sources that offers a high level of nutritional value [8, 9]. The majority of honey is composed of carbohydrates, which are further subdivided into macromolecules and micromolecules. The composition of honey, which has a viscous structure, is subject to variation depending on a number of factors. It is evident that the geographical location of the bee and the floral composition of the region are among the most significant factors [5, 7]. Honey and honey products, which have a complex chemical composition, are employed in the treatment of a range of diseases. Additionally, honey and its derivatives have been demonstrated to possess a range of beneficial properties, including antibacterial, antiviral, and antioxidant effects [8, 10-12].

One of the key elements in the evaluation of honey quality is the measurement of HMF. HMF can be formed as a result of the Maillard reaction or the dehydration of hexoses. The formation of HMF in honey products is dependent on a number of factors. The aforementioned factors include the composition of the honey, the temperature at which it is stored, the processing techniques employed, and numerous other variables [13, 14].

Honey is a natural food product obtained by bees from the nectar of flowers or honeydew. The composition and content of honey is directly related to environmental conditions under which it is produced. It is stated that honey has been used as a source of healing since ancient times. Especially honey has many positive effects on health. In this context, antioxidants assume particular significance in honey products. One of the important antioxidant compounds in honey products is phenolic components [15, 16].

Honey and its derivatives have been employed for a multitude of purposes throughout history. As a natural food product, honey is of significant nutritional value. However, the quality of honey is susceptible to a number of factors, which can impact its nutrition.The quality of honey and its products can vary considerably. In particular, the geographical location and floral flora directly affect the quality of honey. In the evaluation of the quality parameters of honey, a range of analytical techniques are employed, including pH measurement, electrical conductivity, colour assessment, sugar analysis, diastase activity determination, water content estimation, ash analysis, proline quantification and 5-hydroxymethylfurfural detection [17]. The present study analysed fifteen samples of flower honey obtained from different regions of Erzurum in terms of pH, moisture, total sugar, reducing sugar, sucrose, HMF, proline and phenolic components contents. The objective was to determine the quality criteria and qualities of honey obtained from various regions of Erzurum.

MATERIALS and METHODS

Fifteen flower honey samples from the 2022 flower season were obtained from beekeepers in Erzurum, Türkiye. The honeys used in the analysis were obtained from five different locations in Erzurum. Honeys obtained from different regions of Erzurum were stored at 24±1°C and dark environment until analysis.

Some Physicochemical Parameters

pH and moisture analysis were performed according to Cemeroğlu [18]. While Ohaus-Starter 3100 (Switzerland) pH meter device was used for pH analysis, Binder BD53 (Tuttlingen, Germany) oven device was used for moisture analysis.

Determination of Proline Contents

Proline analysis of honey samples was determined per International Honey Commission IHC [19]. Five g of honey sample was made homogeneous by dissolving in distilled water. Then, it was taken into 3 test tubes and sample, water and proline (0.5 mL of each) were added to each tube, respectively. And 1 mL each of formic and ninhydrin was added to each tube, and shaken in a water bath for 15 minutes. Then, it is kept in a 70°C water bath for another 10 minutes. At the end of the period, 5 mL of 2-propanol (50%) was added to each tube and subjected to a water bath at 70°C for 45 minutes. Then absornance reading was obtanied at 510 nm. Using Equation 1, proline contents were calculated:

$$\text{Proline (mg/kg)}=((E_s/E_a)\times(E_1/E_2))\times 80 \quad (1)$$

where E_s: sample absorbance, E_a: Absorbance value of proline standard solution, E_1: mg proline standard solution, E_2: value of honey in g, and 80: dilution factor

Determination of Total Sugar, Reducing Sugar and Sucrose

The total, reducing and sucrose sugars were determined in accordance with the methodology set forth by Cemeroğlu [18], which employs the Lane-Eynon method. The determination of sugar by this method is based on the reduction of $CuSO_4$ in Fehling's solution of invert sugar to $Cu(OH)_2$ (insoluble in water), in an

alkaline medium and at boiling temperature. Five gram of honey samples were collected and transferred to a 250-milliliter measuring balloon. Two mL of a saturated neutral lead acetate solution were added, and the solution was completed with distilled water up to the balloon line. Subsequently, sodium oxalate was added and filtered once more, following the filtration of the sample through ordinary filter paper. A volume of 50 mL of the filtrate was then transferred to two separate 250 mL flasks. The initial balloon was filled to the 250 mL line with distilled water, while the second was combined with 10 mL of 1/1 HCl and inverted for five minutes in a water bath maintained at a temperature between 67 and 70 °C. Following inversion, neutralisation was achieved through the addition of a few drops of phenol-phthalene and 4N sodium hydroxide, with the solution completed to the line with distilled water. The prepared sample solutions were subjected to titration by boiling with Fehling-I copper sulfate pentahydrate ($Cu_2SO_4 \bullet 5H_2O$) and Fehling-II sodium potassium tartrate tetrahydrate ($KNaC_4H_4O_6 \bullet 4H_2O$) solutions. The results were calculated in accordance with the following formula:

$$\text{Total sugar (g/100g)} = (f/M_1) \times 100 \quad (2)$$

$$\text{Reducing sugar (g/100g)} = (f/M_2) \times 100 \quad (3)$$

$$\text{Sucrose (\%)} = (\text{Total sugar} - \text{reducing sugar}) \times 100 \quad (4)$$

where f: The amount of invert sugar determined in the adjustment, equivalent to 10 mL of Fehling's solution mixture, g, M_1: The actual sample amount contained in the spent sample solution in the titration before inversion, g, and M_2: The actual amount of sample contained in the amount of sample solution spent in the titration after sample inversion, g.

Extractions and HPLC Conditions

The phenolic component and HMF contents of the samples were determined in accordance with the methodology outlined by Alwazeer et al. [20]. The honey samples were subjected to analysis for a range of phenolic components, including epicatechin, caffeic, rutin, *p*-coumaric, *e*-ferrulic, syringic, gallic, chlorogenic acid, and catechin.

The process of HMF extraction was initiated by weighing 2 g of honey and subsequently adding 20 mL of pure water. Subsequently, the solution was mixed in a shaker until dissolution was complete, after which it was filtered through a 0.45 µm filter. In order to ascertain the phenolic components, 15 g of honey was shaken with methanol at room temperature and filtered using filter paper. Once the filtrate had been completed with methanol, it was filtered through a 0.45 µm filter and placed in vials. The Dioder Array Detector (DAD) was employed for HPLC analysis (Agilent 1260 Infinity series, USA). The flow rate employed for the analysis of HMF was 0.6 mL/min, with a total analysis time of 25 minutes. For the phenolic components, a flow rate of 0.8 mL/min was used, with a total analysis time of 40 minutes.

Statistical Analysis

One-way analysis of variance (ANOVA) to determine the significance difference ($P<0.05$) of honey samples. It was also subjected to PCA and Pearsen Correlation analyzes to evaluate differences between honey samples.

RESULTS and DISCUSSION

The pH values and moisture contents of the honey samples are presented in Table 1. Their pH values exhibited considerable variation, with a range of 3.55 to 4.19. These findings were statistically significant ($P<0.05$). The highest pH value was observed in the "g" sample, while the lowest was observed in the "c" sample. Furthermore, it was established that all the honeys subjected to analysis exhibited an acidic structure. It is stated that the acidic structure observed in these honeys is a consequence of the fermentation of sugars and organic acids [21]. Conversely, while there is no restriction on pH value in the legal legislation, it is stated that the total amount of acidity can be 50 meq/kg at most [22]. These findings are corroborated by the literature [21, 23-25, 27].

Table 1. pH and moisture content (%) results of different flower honey samples

Code of Honey Samples	pH±SD	Moisture Content ±SD (%)
a	$3.95\pm0.01^{d*}$	15.77 ± 0.01^{b}
b	3.75 ± 0.04^{j}	19.50 ± 0.00^{gh}
c	3.55 ± 0.01^{k}	16.65 ± 0.21^{c}
d	3.87 ± 0.01^{e}	19.04 ± 0.05^{fg}
e	3.76 ± 0.01^{j}	20.55 ± 0.07^{i}
f	3.86 ± 0.02^{ef}	17.60 ± 0.14^{e}
g	4.19 ± 0.01^{a}	17.35 ± 0.21^{de}
h	3.78 ± 0.01^{ij}	22.25 ± 0.07^{j}
i	3.95 ± 0.01^{d}	17.00 ± 0.71^{cd}
j	3.83 ± 0.01^{gh}	19.75 ± 0.35^{h}
k	4.05 ± 0.01^{c}	18.75 ± 0.07^{f}
l	4.14 ± 0.02^{b}	14.75 ± 0.04^{a}
m	3.81 ± 0.01^{hi}	23.20 ± 0.14^{k}
n	3.84 ± 0.02^{fgh}	19.00 ± 0.28^{fg}
o	3.86 ± 0.01^{efg}	18.57 ± 0.04^{f}

*Different letters (a-l) in the same column are significantly different ($P<0.05$)
Abbreviations: SD: standard deviation.

The moisture content of the honeys varied ranged between 15.77 and 23.20% (Table 1) and were statistically very significant (P<0.05). In addition, the highest moisture content was determined in the “m” sample, while the lowest was determined in the “a” sample. It was detected that only three of the analyzed honey samples did not comply with the Turkish Food Codex (20%). Kayacıer and Karaman [21] reported that the moisture values of different honeys varied ranged beetwen 16.30 and 17.90. Nouri [28] determined that the moisture values of different honeys varied among 16.16-19.08%.

The total sugar content of the honeys varied between 70.57-80.24 g/100g (P<0.05) (Table 2). In honey samples, the highest total sugar content was determined in the “l” sample, while the lowest was determined in the “o” sample. In another study on total sugar, they reported that the total sugar ratio varied between 68.1 and 86 g/100g [24]. Khalil et al. [25] found the total sugar values in Algerian honeys between 62.80 and 70 g/mL. Nouri [28], in his study on different honey, determined that the total sugar content of the samples varied between 72.74 and 73.63%.

The reducing sugar contents of honeys were determined to vary between 56.30 and 78.00 g/100g (P<0.05) (Table 2). In honeys, the highest reducing sugar ratio was determined in the “l” sample, while the lowest was determined in the “o” sample. According to the Turkish Food Codex, it is indicated that the reducing sugar ratio for flower honey should be at least 60 g in 100 g. Ajlouni and Sujirapinyokul [24] determined that the reducing sugar level in honey samples varied between 57.3 and 73.6 g/100g. Oroian et al. [29] reported that the reducing sugar level in different honey samples varied between 65.00 and 70.52 g/100g. Again, Gültekin-Özgüven et al. [30] reported that the honey obtained from various regions of Turkey varies between 56.31 and 81.61%. Ucar and others detected that the reducing sugar ratios in honey samples varied between 63.72 and 71.94%.

Table 2. Total sugar, reducing sugar, sucrose, proline and 5-HMF contents of different flower honey samples

Code of Honey Samples	Total sugar (g/100g)	Reducing sugar (g/100g)	Sucrose (%)	Proline (mg/kg)	5-HMF (mg/kg)
a	73.03 ± 0.37^{f}*	70.370 ± 0.00^{fg}	2.52 ± 0.35^{fgh}	459.54 ± 8.94^{g}	6.25 ± 1.76^{f}
b	73.67 ± 0.20^{ef}	69.76 ± 0.18^{g}	3.72 ± 0.01^{d}	390.15 ± 6.33^{ij}	11.70 ± 1.55^{e}
c	74.10 ± 0.30^{ef}	71.83 ± 0.28^{ef}	2.16 ± 0.01^{ghi}	505.80 ± 4.10^{d}	7.50 ± 1.41^{f}
d	74.15 ± 0.23^{ef}	69.78 ± 0.13^{g}	4.15 ± 0.08^{cd}	412.26 ± 2.69^{h}	18.60 ± 2.54^{c}
e	70.59 ± 0.21^{g}	67.36 ± 0.19^{h}	3.07 ± 0.01^{e}	467.91 ± 2.89^{fg}	13.55 ± 1.55^{de}
f	77.61 ± 0.21^{cd}	72.36 ± 0.16^{de}	4.98 ±0.04^{b}	379.57 ± 9.73^{j}	23.5 ± 0.70^{b}
g	76.52 ± 0.40^{d}	74.70 ± 0.03^{c}	1.72 ± 0.35^{i}	479.25 ± 4.86^{ef}	15.75 ± 1.06^{d}
h	76.68 ± 0.76^{cd}	72.03 ± 0.54^{de}	4.42 ± 0.21^{c}	403.66 ± 6.63^{hi}	5.70 ± 0.14^{f}
i	79.37 ± 0.55^{ab}	76.32 ± 0.59^{b}	2.90 ± 0.04^{ef}	576.02 ± 6.99^{c}	6.45 ± 0.70^{f}
j	76.84 ±1.39^{cd}	73.58 ± 1.27^{cd}	3.09 ± 0.11^{e}	486.93 ± 1.38^{e}	11.50 ±0.71^{e}
k	74.72 ± 0.79^{e}	72.51 ± 1.00^{de}	2.11 ± 0.19^{hi}	636.85 ± 2.62^{b}	6.30 ± 0.71^{f}
l	80.24 ± 0.58^{a}	78.00 ± 0.91^{a}	2.13 ± 0.30^{hi}	363.62 ± 3.06^{k}	19.15 ± 0.49^{c}
m	78.22 ± 0.84^{bc}	75.13 ± 0.61^{bc}	2.94 ± 0.22^{ef}	396.91 ± 2.18^{i}	6.40 ± 0.14^{f}
n	73.03 ± 0.37^{f}	70.28 ± 0.82^{fg}	2.62 ± 0.43^{efg}	726.00 ± 5.23^{a}	5.20 ± 0.56^{f}
o	70.57 ± 1.53^{g}	56.30 ± 1.44^{i}	13.56 ± 0.08^{a}	244.79 ± 13.61^{l}	108.12 ±1.48^{a}
Significance	**	**	**	**	**

*Different letters (a-l) in the same column are significantly different (P<0.05) Abbreviations: SD: standard deviation.

It was detected that the sucrose contents of honey samples varied between 1.72-13.56% (P<0.05) (Table 2). As per Turkish Food Codex, it is stated that 5g/100g should be used for flower honey. According to Khalil and others the sucrose content of Algerian honey samples was 1.80-2.54%, Oroian et al. [29] determined the sucrose content of different honey samples as 1.76 g/100g, while Gültekin-Özgüven et al. [30] determined the sucrose content of the samples between non-detected and 3.43% in honey obtained from various regions of Turkey, and Nouri [28] determined that the sucrose content of the samples in his study on different honeys varied between 0.5 and 3.48%.

Proline, one of the important amino acids, is mostly derived from the salivary secretions of *Apis mellifera* during the transformation of nectar into honey [23]. The main amino acid in honey products is proline [15]. Therefore, the amount of proline in honey samples has an important role in determining sugar adulteration. Proline constitutes the largest part of the amino acid composition of honey (~ 85%). It is stated that the proline value of adulterated honey is low [15, 29]. As per the TFC, the proline content of honey varies depending on the honey types. It is stated that the proline content of flower honey should be at least 300 mg/kg [30]. In the study, it was determined that the proline value of honeys varied between 244.79 and 726 mg/kg (Table 2). It is also observed that the “n” group with the highest proline value has the lowest 5-HMF level. Similarly, Gültekin-Özgüven et al. [30] determined the proline value of 271-928.2 mg/kg in honey obtained from various regions of Turkey. Ucar et al. [14] detected that the values of proline in honeys varied between 657.39 and 1974.23 mg/ kg. Machado and others determined that the proline values of honey samples varied ranged between 0.2 and 2.2 mg/g. Ecem Bayram et al. [31], in his study, determined that the proline content of forty different honeys varied between 384.41 and 1271.56 mg/kg.

HMF is one of the important parameters in point of purity and quality of honey. HMF, which can be found at very

low levels even in fresh honey, is known to increase with storage and heat treatment of honey [23]. Due to high exposure to HMF, it can cause various health problems [33]. It is stated that the recommended level of HMF in all fresh honey samples is 40 mg/kg [24]. As per the results of the research, it was determined that the HMF values of honey samples varied between 5.20 and 108.12 mg/kg and were within the limits determined by the Turkish Food Codex except for only one sample (P<0.05) (Table 2). As a matter of fact, there are studies in the literature with similar and different results. Silva et al. [23] found HMF values between 17 and 51.5 mg/kg in honey samples. In their study, Ajlouni and Sujirapinyokul [24] determined that the HMF values of commercial and fresh honeys in which they applied different temperatures (65.75 and 85 °C) varied between 0.36 and 74.9 mg/kg. Khalil et al. [25] found HMF values between 15.23 and 24.21 mg/kg in Algerian honey samples. In another study, they determined that HMF values varied between 8.8 and 400 mg/kg [8]. Tomczyk et al. [34] determined the HMF levels of honey samples as 5.03-22.98 mg/kg in his study.

One of the important bioactive compounds in honey products is their phenolic components. In honey products, phenolic compounds has an affect sensory properties as well as antioxdidant activity. Phenolic components commonly found in honey products are gallic acid, *p*-coumaric acid, caffeic acid, chlorogenic acid, vanillic acid and syringic acid [35]. Phenolic substance contents vary in honey products. It is stated that these differences are especially due to climatic conditions, geography and honey types [6, 36]. Phenolic contents of the analyzed honey samples are given in Table 3. Epicatechin, caffeic, routine, *p*-coumaric, *e*-ferrulic and syringic acid were not detected in the study. However, gallic acid was determined as 194.72 µg/g in all samples, chlorogenic acid as 21.47 µg/g in only one sample, and catechin as 202.96 µg/g in three samples.

Table 3. Phenolic components of different flower honey samples (µg/g)

Code of Honey Samples	Gallic acid	Chlorogenic	Catechin
a	58.42±1.16^{g*}	21.47±0.5	nd
b	53.66±1.38^{i}	nd	nd
c	88.73±0.73^{d}	nd	160.86±0.01^{c}
d	53.65±2.85^{i}	nd	nd
e	58.15±0.65^{gh}	nd	nd
f	49.33±1.94^{jk}	nd	nd
g	41.00±0.50^{l}	nd	nd
h	45.44±2.01^{k}	nd	nd
i	103.59±2.27^{c}	nd	190.23±9.76^{b}
j	85.82±0.46^{e}	nd	nd
k	150.85±3.47^{b}	nd	202.95±7.49^{a}
l	54.03±5.55^{h}	nd	nd
m	49.79±5.21^{jk}	nd	nd
n	194.72±0.48^{a}	nd	nd
o	84.92±1.37^{f}	nd	nd

*Different letters (a-l) in the same column are significantly different (P<0.05), Abbreviations: SD: standard deviation.nd: not determined.

Gallic acid was the most abundant phenolic component in the analyzed honey samples. The gallic acid contents of the samples were determined between 41.00 and 194.72 µg/g. Different levels of gallic acid contents have been determined in the studies conducted in the literature. Alshammari et al. [37] determined the gallic acid contents of the samples between non-detected and 1.14 mg/100g, Andrade et al. [38] determined 237.20 mg phenolic acid /100g in different honey samples. In another study, gallic acid contents in different Turkish honey samples were reported to be between non-detected and 82.49 µg/g [39]. Pham et al. [40] determined gallic acid between 0.28 and 12.50 mg/100g in honey samples.

Chlorogenic acid contents of honeys were determined between non-detected and 21.47 µg/g (Table 3). Pham et al. [40] determined chlorogenic acid between 0.28-12.50 mg/100g in honey samples. On the other hand, Can et al. [39] reported that they could not detect chlorogenic acid in any sample in different Turkish honeys. Catechin contents of the samples were determined between non-detected and 202.95 µg/g. Pham et al. [40] determined catechin contents between 9.51 and 104.40 mg/100g in honey samples. In another study, catechin contents of different honey samples were found to vary between non-detected and 23.07 µg/g [39].

Correlation Among Quality Characteristics

When the results are analyzed, there are negative and positive correlations between pH, total sugar, reducing sugar and sucrose. On the other hand, 5-HMF, which is very important for honey samples, was negatively correlated with total sugar (P<0.05, r=-0,38*), reducing sugar (P<0.01, r=-0.80**) and pyroline (P<0.01, r=-0.60**), while it was positively correlated with sucrose (P<0.01, r= 0,95**). In addition, it was determined that there was a positive correlation between proline and reducing sugar (P<0.05, r=0.365*), while there was a negative correlation with sucrose (P<0.01, r=-0.60**) and HMF (P<0.01, r=-0.60**). Among the phenolic components, gallic acid (P<0.01, r=0.78**) and catechin (P<0.01, r=0.84**) showed a positive correlation with proline (Figure 1).

	Moisture (%)	pH	Total Sugar (g/100g)	Reducing sugar (g/100g)	Sucrose (%)	Pyroline (mg/kg)	HMF (mg/kg)	Gallic acid	Catechin	Chlorogenic
Moisture (%)	1,00	0,41*	0,13	0,15	-0,12	0,07	0,08	0,07	0,18	0,35
pH	0,41*	1,00	0,39*	0,32	-0,15	0,06	0,03	-0,02	-0,07	0,11
Total sugar (g/100g)	0,13	0,39*	1,00	0,84**	-0,41*	0,00	-0,38*	-0,22	-0,03	-0,21
Reducing sugar (g/100g)	0,15	0,32	0,84**	1,00	-0,84**	0,37*	-0,80**	-0,09	0,15	-0,05
Sucrose (%)	-0,12	-0,15	-0,41*	-0,84**	1,00	-0,61**	0,95**	-0,07	-0,27	-0,12
Pyroline (mg/kg)	0,07	0,06	0,00	0,37*	-0,61**	1,00	-0,60**	0,78**	0,84**	-0,01
HMF (mg/kg)	0,08	0,03	-0,38*	-0,80**	0,95**	-0,60**	1,00	-0,07	-0,27	-0,12
Gallic acid	0,07	-0,02	-0,22	-0,09	-0,07	0,78**	-0,07	1,00	0,89**	-0,13
Catechin	0,18	-0,07	-0,03	0,15	-0,27	0,84**	-0,27	0,89**	1,00	-0,16
Chlorogenic acid	0,35	0,11	-0,21	-0,05	-0,12	-0,01	-0,12	-0,13	-0,16	1,00

+1

-1

Figure 1. Correlation among quality parameters of honey samples (significant at *P<0.05 and **P<0.01)

Discrimination of Honey Samples by Principal Component Analysis

PCA has recently been widely used in the literature for the discrimination of different food samples [4, 41, 42, 43]. PCA analysis was used to determine the differences between honey samples. In addition, the differences between the applied analyzes were determined. The score scatterplot, loading scatter plot, biplot and hierarchical clustering are shown in Figure 2 A–D, it can be seen that two principal components accounted for 69.3 % of the variance. Honey samples were divided into 5 different groups as group1 (o), group 2 (n), group 3 (c, k, i), group 4 (l, g, m) and group 5 (a, e, j, d, b, h, f). The analyses are clustered in two regions, right and left. Moisture, 5-hydroxymethylfurfural and sucrose values clustered on the right side, while catechin, gallic acid, cholorocenic acid, proline, reducing sugar, total sugar and pH clustered on the left side.

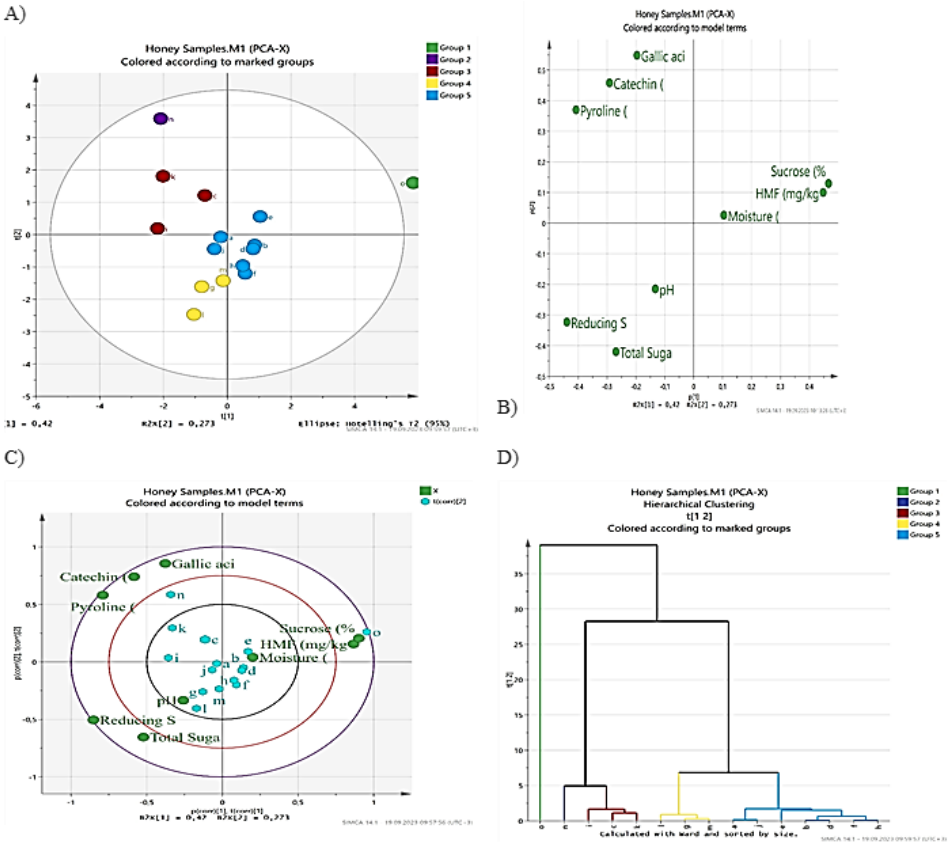

Figure 2. S-S-P (A), L-S-P (B), B (C) and H-C (D) of PCA analysis (PC1 versus PC2) for the components in the honey samples

CONCLUSIONS

Honey and its derivatives represent a significant component of the human nutrition. The present study investigated the quality parameters and the contents of HMF, proline and phenolic compounds of honey samples collected from various regions of the Erzurum province of Turkey. The results demonstrated that three of the fifteen honey samples exhibited moisture values that did not meet the national standards, while one sample displayed reducing sugar and proline values that did not align with the national standards. In general, the honey samples collected from Erzurum province were found to comply with the national standards. The adulteration of honey gives rise to unfair competition and has the potential to have adverse effects on consumer health.

Consequently, food adulteration represents not only an economic loss but also a significant threat to public health. In order to protect themselves from such adulteration, consumers should endeavour to purchase products from reliable brands, to examine product labels carefully and to avoid cheap products that are below market value. In this context, it is recommended that honey producers should be continuously inspected to maintain quality.

ACKNOWLEDGEMENTS

The author Mustafa Kılıç are supporting by the Council of Higher Education (CoHE, Yükseköğretim Kurulu, YÖK) with 100/2000 PhD Scholarship Project.

FUNDING

There has not been any funding.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHORS' CONTRIBUTION

A. Savaş conceived and designed the evaluation and collected the data. H.İ. Binici participated in designing the evaluation, re-evaluating the data, and revising the manuscript. İ.G. Şat helped drafted the manuscript and performed parts of the statistical analysis, and revised the manuscript.M. Kılıç conceived and designed the evaluation, re-evaluated the data, and revised the manuscript.

REFERENCES

[1] Binici, H.İ., Savaş, A. (2024). The effects of lycopene on health and fields of use. *Journal of Advanced Natural Sciences and Engineering Researches*, 8(6), 158-161.

[2] Savaş, A. (2024). Fındık meyvesinin besinsel içeriği üzerine kısa bir perspektif. *Gıda Bilimi ve Mühendisliği Araştırmaları*, *3*(1), 100-103.

[3] Geana, E.I., Ciucure, C.T. (2020). Establishing authenticity of honey via comprehensive Romanian honey analysis. *Food Chemistry*, 306, 125595.

[4] Savaş, A., Oz, E., Oz, F. (2021). Is oven bag really advantageous in terms of heterocyclic aromatic amines and bisphenol-A? Chicken meat perspective. *Food Chemistry*, 355, 129646.

[5] Se, K.W., Wahab, R.A., Yaacob, S.N.S., Ghoshal, S.K. (2019). Detection techniques for adulterants in honey: Challenges and recent trends. *Journal of Food Composition and Analysis*, 80, 16-32.

[6] Wang, Y., Cai, W., Li, L., Gao, Y., Lai, K.H. (2023). Recent advances in the processing and manufacturing of plant-based meat. *Journal of Agricultural and Food Chemistry*, 71(3), 1276-1290.

[7] Zhang, G., Abdulla, W. (2022). On honey authentication and adulterant detection techniques. *Food Control*, 108992.

[8] Al-Kafaween, M.A., Alwahsh, M., Mohd Hilmi A.B., Abulebdah, D.H. (2023). Physicochemical characteristics and bioactive compounds of different types of honey and their biological and therapeutic properties: A comprehensive review. *Antibiotics*, 12(2), 337.

[9] Hossain, M.L., Lim, L.Y., Hammer, K., Hettiarachchi, D., Locher, C. (2022). A review of commonly used methodologies for assessing the antibacterial activity of honey and honey products. *Antibiotics*, 11(7), 975.

[10] Stagos, D., Soulitsiotis, N., Tsadila, C., Papaeconomou, S., Arvanitis, C. (2018). Antibacterial and antioxidant activity of different types of honey derived from Mount Olympus in Greece. *International Journal of Molecular Medicine*, 42(2), 726-734.

[11] Pauliuc, D., Dranca, F., Oroian, M. (2020). Antioxidant activity, total phenolic content, individual phenolics and physicochemical parameters suitability for Romanian honey authentication. *Foods*, 9(3), 306.

[12] Afrin, S., Giampieri, F., Gasparrini, M., Forbes-Hernández, T.Y., Cianciosi, D. (2018). The inhibitory effect of Manuka honey on human colon cancer HCT-116 and LoVo cell growth. Part 2: Induction of oxidative stress, alteration of mitochondrial respiration and glycolysis, and suppression of metastatic ability. *Food & Function*, 9(4), 2158-2170.

[13] Tosi, E., Ciappini, M., Re, E., Lucero, H. (2002). Honey thermal treatment effects on hydroxymethylfurfural content. *Food Chemistry*, 77(1), 71-74.

[14] Fallico, B., Zappala, M., Arena, E., Verzera, A. (2004). Effects of conditioning on HMF content in unifloral honeys. *Food Chemistry*, 85(2), 305-313.

[15] Cianciosi, D., Forbes-Hernández, T.Y., Afrin, S., Gasparrini, M., Reboredo-Rodriguez, P. (2018). Phenolic compounds in honey and their associated health benefits: A review. *Molecules*, 23(9), 2322.

[16] Mutlu, C., Erbaş, M., Arslan Tontul, S. (2017). Bal ve diğer arı ürünlerinin bazı özellikleri ve insan

sağlığı üzerine etkileri. *Akademik Gıda*, 15(1), 75-83.
[17] Uçar, M., Kemal, M., Kanbur, E.D., Kara, Y., Özcelik, A.E. (2023). The botanical, physicochemical, and biochemical characteristics of Northern Cyprus honeys. European Food Research and Technology, 249(6), 1531-1541.
[18] Cemeroğlu, B.S. (2013). Meyve ve Sebze İşleme Teknolojisi. Bizim Grup Basımevi, Ankara, Türkiye, 312.
[19] International Honey Commission. (2009). Harmonised methods of the international honey commission. URL< http://www. bee-hexagon. net/en/network. htm> Accessed, 23, 16. International Honey Commission. (2009). Harmonised methods of the international honey commission. URL< http://www. bee-hexagon. net/en/network. htm> Accessed, 23, 16.
[20] Alwazeer, D., Elnasanelkasim, M.A., Çiçek, S., Engin, T., Çiğdem, A., Karaoğul, E. (2023). Comparative study of phytochemical extraction using hydrogen-rich water and supercritical fluid extraction methods. *Process Biochemistry*, 128, 218-226.
[21] Kayacier, A, Karaman, S. (2008). Rheological and some physicochemical characteristics of selected Turkish honeys. *Journal of Texture Studies*, 39(1), 17-27.
[22] Güzel, N., Bahçeci, K.S. (2020). Çorum yöresi ballarinin bazi kímyasal kalíte parametrelerínin değerlendírílmesi. *Gıda*, 45(2), 230-241.
[23] Silva, L.R., Videira, R., Monteiro, A.P., Valentão, P., Andrade, P.B. (2009). Honey from Luso region (Portugal): Physicochemical characteristics and mineral contents. *Microchemical Journal*, 93(1), 73-77.
[24] Ajlouni, S., Sujirapinyokul, P. (2010). Hydroxymethylfurfuraldehyde and amylase contents in Australian honey. *Food Chemistry*, 119(3), 1000-1005.
[25] Khalil, M.I., Moniruzzaman, M., Boukraâ, L., Benhanifia, M., Islam, M.A., (2012). Physicochemical and antioxidant properties of Algerian honey. *Molecules*, 17(9), 11199-11215.
[26] Machado, A.M., Tomás, A., Russo-Almeida, P., Duarte, A., Antunes, M. (2022). Quality assessment of Portuguese monofloral honeys. Physicochemical parameters as tools in botanical source differentiation. *Food Research International*, 157, 111362.
[27] Massous, A., Ouchbani, T., Lo Turco, V., Litrenta, F., Nava, V. (2023). Monitoring Moroccan honeys: physicochemical properties and contamination pattern. *Foods*, 12(5), 969.
[28] Nouri, M. (2023). The correlation of plant species and geographical regions on biological component and antioxidant potential of different honey. *Journal of Food Biosciences and Technology*, 13(1), 61-74.
[29] Oroian, M., Ropciuc, S., Paduret, S. (2018). Honey authentication using rheological and physicochemical properties. *Journal of food science and technology*, 55, 4711-4718.
[30] Özgüven, M., Demircan, E., Özçelik, B. (2020). Çeşitli yörelerimizde üretilen çiçek ballarının fizikokimyasal özelliklerinin belirlenmesi ve Türk Gıda Kodeksi'ne uygunluğunun değerlendirilmesi. *Avrupa Bilim ve Teknoloji Dergisi*, (20), 321-326.
[31] Ecem Bayram, B.N., Kutlu, N., Gercek, Y.C. (2023). Utilization of response surface methodology in optimization of proline extraction from *Castanea sativa* honey. *Chemistry & Biodiversity*, 20(3), e202201092.
[32] Turkish Food Codex. (2020) Turkish Food Codex Communiqué on Honey (No: 2020/7). (Access date: 10.03.2023).
[33] Altunay, N. (2022). Experimental design of magnetic ionic liquid ultrasound-assisted dispersive liquid-liquid microextraction for the determination of 5-HMF in honey samples. *Journal of Food Composition and Analysis*, 114, 104817.
[34] Tomczyk, M., Czerniecka-Kubicka, A., Miłek, M., Sidor, E., Dżugan, M. (2023). Tracking of thermal, physicochemical, and biological parameters of a long-term stored honey artificially adulterated with sugar syrups. *Molecules*, 28(4), 1736.
[35] Ayoub, W.S., Zahoor, I., Dar, A.H., Farooq, S., Mir, T.A. (2023). Exploiting the polyphenolic potential of honey in the prevention of chronic diseases. *Food Chemistry Advances*, 3, 100373.
[36] Becerril-Sánchez, A.L., Quintero-Salazar, B., Dublán-García, O., Escalona-Buendía, H.B. (2021). Phenolic compounds in honey and their relationship with antioxidant activity, botanical origin, and color. *Antioxidants*, 10(11), 1700.
[37] Alshammari, G.M., Ahmed, M.A., Alsulami, T., Hakeem, M.J., Ibraheem, M.A., Al-Nouri, D.M. (2022). Phenolic compounds, antioxidant activity, ascorbic acid, and sugars in honey from ingenious hail province of Saudi Arabia. *Applied Sciences*, 12(16), 8334.
[38] Andrade, P., Ferreres, F., Amaral, M.T. (1997). Analysis of honey phenolic acids by HPLC, its application to honey botanical characterization. *Journal of Liquid Chromatography & Related Technologies*, 20(14), 2281-2288.
[39] Can, Z., Yildiz, O., Sahin, H., Turumtay, E.A., Silici, S. (2015). An investigation of Turkish honeys: Their physico-chemical properties, antioxidant capacities and phenolic profiles. *Food Chemistry*, 180, 133-141.
[40] Pham, T.N., Nguyen, T.V., Le, D.T., Diep, L.M.N., Nguyen, K.N. (2022). Phenolic profiles, antioxidant, antibacterial activities and nutritional value of Vietnamese honey from different botanical and geographical sources. *Agri Engineering*, 4(4), 1116-1138.
[41] Oz, E., Savaş, A., Ekiz, E, Oz, F. (2021). Mangalda pişirilmiş sebzelerin polisiklik aromatik hidrokarbon içeriği. *Çukurova Tarım ve Gıda Bilimleri Dergisi*, 36(1), 13-22.
[42] Savaş A., Ekiz, E., Elbir, Z., Savaş, B.D., Proestos, C. (2023). Advantageous effects of sumac usage in meatball preparation on various quality criteria and formation of heterocyclic aromatic amines. *Separations*, 10(1), 29.

[43] Savas, A., Oz, E., Elbir, Z., Oz, F. (2024). Effect of dry aging on quality parameters, protein profile and protein oxidation level of beef. *International Journal of Food Science & Technology*, 59(10), 7598-7609.