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Physicochemical and sensory properties of mulberry products: Gümüşhane pestil and köme

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Abstract: 'Pestil' and 'köme', well known as traditional Turkish foods, are prepared from a mulberry, honey, walnut, hazelnut, and flour mixture. Although they have been produced for the last few decades on an industrial scale, the production methods and properties of the products are not standardized yet. Gümüşhane pestil and köme have been registered and attained Geographical Registration Certificates from the Turkish Patent Institute with their unique production methods and properties, among many similar products in the world markets. Thus, some biochemical, physical, and sensory properties of pestil and köme were investigated. In addition, the main raw material (mulberry) and semifinished product ('herle') were analyzed for some biochemical, physical, and rheological parameters. The characteristics of pestil and köme ranged from 9.78% to 20.06% for moisture content, 0.52% to 1.48% for crude fiber, 4.34% to 7.42% for protein, 41.04% to 62.54% for total sugar, and 37.52% to 56.23% for invert sugar, and the thickness, hydroxymethylfurfural (HMF), total phenolic content, and ferric ion reducing antioxidant power antioxidant activity ranged from 0.80 to 1.25 mm, 18.15 to 27.94 mg kg^{-1}, 25 to 32 mg GAE per 100 g, and 81 to 92 mM TE g^{-1}, respectively. Strong correlations between sugar contents and HMF content and between HMF and antioxidant activity ($r > 0.5$) were identified separately. The consistency index for herle was determined as 9.7–65.9 Pa s. Color parameters (*L*, *a*, and *b*) were read, and the difference in *L* between the product types was found to be statistically significant. Gümüşhane pestil and köme were found to be very different from other similar fruit leather products that are commercially available in terms of production method, composition, and biological and physical properties.

Key words: Antioxidant activity, köme, *Morus alba*, pestil

1. Introduction

Traditional foods, existing in the process of historical development, are mostly local foods that reflect community cultural and social features. Generally, agricultural products grown in a region diversify traditional foods. Mulberry fruits are grown in subtropical regions of the southern and northern hemispheres (Tutin, 1996; Vijayan et al., 1997; Ercişli and Orhan, 2008). A great deal of mulberry is grown in Gümüşhane, Turkey. Mulberry fruits are consumed as fresh, dried, or processed food products in Turkey. 'Pestil', an edible film (Kaya and Maskan, 2003), and 'köme', pulp-coated walnut, made from white mulberry (*Morus alba*) are special traditional foods of Gümüşhane. Pestil and köme in Turkey are produced from fruit juice such as mulberry, grape, apple, rose hip, fig, or cornelian cherry and also from concentrated fruit juice ('pekmez' or molasses) (Nas and Nas, 1987; Demir and Kalyoncu, 2003; Ercişli, 2007). Sometimes called fruit rolls, leathers, or taffies in some countries, pestil is produced in different ways and by using different ingredients. Some pestil types are produced from fruit juice, starch, and sugar (Maskan, 2001). In addition to these ingredients, honey, wheat flour, and milk are added to traditional Gümüşhane pestil and köme. Gümüşhane pestil is softer and brighter than other types of pestil. Protected geographical indications are required in traditional foods (Cayot, 2007). Due to these requirements and differences in prescribing, a Geographical Registration Certificate (GRC) was obtained for traditional Gümüşhane köme and pestil from the Turkish Patent Institute by the Gümüşhane Governor's Office (Gümüşhane Valiliği, 2004a, 2004b).

The ingredients and the production process must be studied in parallel with the properties of final products. Aflatoxins, a group of secondary metabolites, are important risks in walnuts and hazelnuts (Tabata et al., 1993; Anderson, 2007). It is thus clear that pestil and köme have an aflatoxin risk due to the content of walnuts and hazelnuts, and this must be examined. The concept of food quality includes many factors such as good taste, healthy, natural contents, and safety (Cayot, 2007). Sensory

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properties and physical and biochemical compositions of traditional foods should be investigated. Although köme and pestil of different fruits and regions are well known, Gümüşhane mulberry pestil and köme have not been researched and their specifications have not been determined yet. As far as it is known, up to now no research studies have been available about these products. In this research, it is attempted to determine some properties of Gümüşhane pestil and köme.

2. Materials and methods

2.1. Fruits

Mulberry fruits (*Morus alba*) were harvested up from Gümüşhane in June and July 2011. Freshly harvested fruits were brought to a processing plant (Kral pestil factory, Gümüşhane, Turkey) as fresh and stored in a cold room at around 4 °C. Filtered flower honey, shelled walnuts, and semiskimmed cow's milk were supplied from a local village bazaar. Beet sugar (crystalline sucrose) was produced by Sugar Factory Inc. in Erzincan, Turkey; wheat flour (Type 1) was milled by Dervişoğlu Flour Factory Inc. in Trabzon, Turkey; whole "grain" hazelnuts, which are roasted and separated from their membranes, were packed by Demirciler Hazelnut Factory Inc. (Giresun, Turkey) and they were purchased from their wholesalers.

2.2. Chemicals and reagents

All the chemicals were of analytical grade and were used without further purification. Methanol, ethanol, acetonitrile, and standard solutions of aflatoxins (mix kit) were procured from Sigma-Aldrich (St. Louis, MO, USA). The other chemicals in the study were obtained from Merck Co. (Darmstadt, Germany).

2.3. Production of pestil and köme

Foreign materials such as stones, grass, insects, and leaves were removed from the mulberry fruits. Fruits were washed in a washing pool (washing machine, Kurtsan, Bursa, Turkey), transferred to a boiler where fruit were covered with a 2× volume of water (boiler, Kurtsan), and boiled for 20 minutes. The mixture was then pressed with a hydraulic press (Kermak, Trabzon, Turkey) and filtered using cheesecloth. The obtained liquid product after pressing is traditionally called mulberry 'şıra'. The next step was the preparation of 'herle', which is the traditional name of the mixture of boiled mulberry juice, honey, flour, crystalline sucrose, and milk. Approximately half of the liquid product was mixed together with milk and flour in a mechanical mixer (mixer, 2800 rpm, Kermak). The rest of the liquid products were heated together with flower honey, which is used to get the typical aroma, and crystalline sucrose in the boiler. It is known that to get the typical aroma, only flower honey is used, not honeydew honey. When the mixture was boiled, the first mixture was added to the boiler under vacuum (–560 mmHg). The final mixture was boiled for about 30 min. The obtained herle can be eaten as a dessert, as well. In this study, the following quantities were used according to the GRC: 80 kg fresh fruits, 25 kg flower honey, 15 kg milk, 25 kg crystalline sucrose, and 15 kg flour. Other manufacturers in the area use similar quantities in accordance with local preferences.

The herle was divided into 3 equal parts. Ground hazelnuts and walnuts, 3–6 mm in sieve diameter, were added to the first and the second herle parts separately (hazelnuts or walnuts were used in a 1:5 nut:herle ratio). Nothing was added to the third herle part. Each herle part was spread over the clothes; their thicknesses were minimized. They were kept in a ventilated glasshouse under the sun to dry. The drying temperature should be 40 ± 5 °C. These degrees are not basic, but the thorough-dry time directly changes in parallel with the temperature, which must not exceed 55 °C. After 6 h, the pestil cloth was inverted and the opposite sides were wetted. The pestil was peeled away from the cloth and dried again under the sun for 10 min.

During the production of the köme, walnuts, sliced in half, were strung on ropes, and then the strings of walnuts were kept in the drying room for 30 min at 70 ± 3 °C in order to kill larvae and achieve crispiness. They were dipped into herle, kept there for 10–15 s, hung from a high place, and allowed to dry in a thermostatic drying room at 50 ± 5 °C. Temperature control was ensured by specially designed fans. After 9 h, the strung walnuts were removed from the drying room. Gümüşhane köme was produced by repeating the same process for 3 repetitions.

2.4. Physical and biochemical analysis

Total phenolic content (TPC) was determined by the Folin–Ciocalteu method of Slinkard and Singleton (1977) and phenolic acid was identified as a reference. The samples and standard gallic acid were diluted to 2–20 µg in 2.0 mL of distilled water, and 2.0 mL of commercial Folin–Ciocalteu reagent was added. The content was mixed well and kept for 5 min at room temperature, followed by addition of 2.0 mL of 10% aqueous sodium carbonate and incubation at room temperature for 1 h. The absorbance was measured at 760 nm at the end of the incubation period. The concentration of total phenolic compounds was calculated as mg gallic acid equivalent (GAE) g^{-1} sample by using a standard graph. The Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) equation antioxidant capacity (TEAC) of the samples was examined using the ferric ion reducing antioxidant power (FRAP) method by comparison to the activity of known antioxidant Trolox according to the method of Benzie and Strain (1996). The FRAP assay is based on the measurement of the iron-reducing capacities of the samples. The absorbance was determined at 595 nm towards a blank that was prepared using distilled water. Hydroxymethylfurfural (HMF) was determined using a spectrophotometric method (Shimadzu UV-2450, Shimadzu Corporation, Kyoto, Japan) at 550 nm with slight modifications (Yıldız and Alpaslan, 2012). In this method, HMF reacts with *p*-toluidine and barbituric acid and forms red pigments. The intensity of the red color is dependent on the amount of 5-HMF. Total soluble solid (TSS) contents were analyzed by the method of Tanner and Brunner (1979) using a hand refractometer. TSS contents were determined by mixing 1 drop of juice from the sample into the refractometer. Total dry matter and moisture (AOAC 925.09), total protein (AOAC 920.87), ash (AOAC 923.03), pH (AOAC 981.12), fat (AOAC 922.06), total sugar and invert sugar contents (AOAC 984.22), total acidity (AOAC 962.12), ascorbic acid (AOAC 967.21), and crude fiber (AOAC 978.10) of raw materials and products were analyzed according to AOAC methods (2000). Thicknesses of the pestil were measured with a micrometer from 3 different positions after removing the walnuts and hazelnuts. Thousand-fruit mass (TFM) of white mulberry was determined from 100 fruits selected randomly; the average of 1000 fruits was calculated.

2.5. Investigation of aflatoxins

Total aflatoxin (AF) levels and aflatoxin B1 (AFB_1) levels of products were determined according to modified AOAC official method 991.31 (1994) and official method 999.07 (1999) after a 3-month shelf-life at room temperature. The extraction of AFs from products was performed using methanol–water–NaCl mixtures. First, 50 g of sample, 5 g of NaCl, 100 mL of deionized water, and 125 mL of 70% methanol were added to a blender and blended at high speed for 2 min. After the extraction of aflatoxins, a clean-up step was carried out using Whatman No. 4 filter paper, and 20 mL of deionized water was mixed with 20 mL of obtained filtrate and filtered using a microfiber glass filter. Ten milliliters of filtrate was taken into a 20-mL injection syringe equipped with an immunoaffinity column for cleaning at a flow rate of 1–2 drops s^{-1}. The column was washed twice with 15 mL of distilled water at a flow rate of less than 5 mL min^{-1}. Air was then passed through the syringe 3–5 times. The column was put into a 2.5-mL volumetric flask and AFs were slowly eluted from the column by passing 1 mL of HPLC-grade methanol through the column, allowing for dropping by gravity, and the flask was brought to volume with deionized water. After mixing in a vortex, 2 mL was transferred into a vial and 100 µL was injected into HPLC equipment (HP1100 Series, Agilent Technologies, USA). The operating conditions for the determination of aflatoxins by HPLC-FLD method were: mobile phase, water:methanol:acetonitrile (550:300:200 v:v:v); 350 µL of 4 M HNO_3 and 120 mg of KBr were added to each liter of the mobile phase, and then this mixture was filtered. The injection volume was 100 µL. Temperature of the column (C18, 4.5 × 250 mm) was 22 °C. The flow rate was 1 mL min^{-1} and the retention times of AFB_1, AFB_2, AFG_1, and AFG_2 were 12.159, 10.054, 8.951, and 7.556 min, respectively. The pressure was lower than 300 bar. The average recoveries were 84.7% for AFB1 and 85.4% for total AFs. The detection of AFs was performed by scanning in a fluorescence detector.

2.6. Color analysis

Colors of products were measured as *L*, *a*, and *b* by using a colorimeter (CR-10, Minolta, Osaka, Japan) (Alpaslan and Hayta, 2006), computed as means of at least 3 measurements from different positions and expressed as Hunter *L*; darkness (0, black) / lightness (100, white); –*a*, greenness; +*a*, redness; –*b*, blueness; and +*b*, yellowness.

2.7. Viscosity measurement and rheological behavior

The dynamic viscosity (centipoise) of herle was measured at 25, 40, 60, and 75 °C using a viscometer (Model RV DV–I +, Brookfield Engineering Labs, USA) fitted with a spindle 6 at the speeds of 0.6, 1.5, 3, 6, 12, 30, and 60 rpm. Enough herle in a 500-mL beaker was used to immerse the spindle (Sopade and Filibus, 1995). Temperature was controlled using a thermostatic water bath (Şengül et al., 2005; Alpaslan and Hayta, 2006) and the average of 3 readings was calculated at 30-s intervals.

2.8. Sensory evaluation

Sensorial properties of pestil and köme were evaluated in this study according to the methods of Alpaslan and Hayta (2006) with slight modifications. The panel consisted of 24 females and 24 males, aged 17 to 38 and nonsmokers who were familiar with pestil and köme. All panelists were selected from among the 90 candidates of a training course on the recognition of basic sensory stimuli. Samples were served under white lightning and at room temperature in porcelain plates labeled with random 3-digit codes. Water was served to the panelists to cleanse the palate between evaluations. Evaluation results were scored on a 9-point (1: dislike intensely, 5: neither dislike nor like, 9: like intensely) traditional hedonic scale (Villanueva and Da Silva, 2009).

2.9. Statistical analysis

The analyses of samples were of a completely randomized design with parallel replications. Means and standard deviations were determined for each variable in the studies. All data were tested using analysis of variance (ANOVA) followed by Duncan multiple range tests using SPSS 9.0 (SPSS Inc., Chicago, IL, USA) at the $P < 0.05$ significance level.

3. Results

White mulberry fruits were characterized before production because of being the essential fruit in pestil and köme. Physical and chemical values of white mulberry fruits are given in Table 1. As can be seen, TFM of fruits was determined as 3.554 g and total dry matter, TSS, acidity, pH, and *L*, *a*, and *b* values of mulberries were found as 23.29%, 19.89%, 0.21%, 6.1, 77.7, −13.1, and 15.5, respectively. Ash, protein, crude oil, total sugar, and invert sugar contents of fruits were about 3.18%, 2.43%, 2.71%, 17.06%, and 15.12%, respectively. Ascorbic acid values of the fruits tested were 74.86 mg per 100 g fresh fruits.

Table 2 shows the physical and biochemical properties of pestil and köme. Color parameters of the products were read and are shown in Table 3. Significant differences in the properties were recorded among all products.

The experimental data for herle are given in the Figure. The rheological behavior of herle was characterized using the power law model in Eq. (1) (Chhinnan et al., 1985).

$$\eta_a = k\gamma^{(n-1)} \quad (1)$$

η_a indicates the viscosity (Pa s), *k* indicates the consistency index (Pa s^n), γ indicates the rotational speed (s^{-1}), and dimensionless *n* indicates the flow behavior index. *n*, *k*, and the correlation coefficient (r^2) were calculated using linear regression analysis. The results are summarized in Table 4.

The consistency index can describe the variation in viscosity with temperature using the Arrhenius equation (Costaldo et al., 1990).

$$\ln k = \ln k_0 + E_a / R_g T_a \quad (2)$$

Here, k_0, E_a, R_g, and T_a indicate the Arrhenius constant (Pa s^n), the activation energy (J mol^{-1}), the universal gas constant (J mol^{-1}), and the absolute temperature (K), respectively. k_0 (Pa s^n) and E_a (J mol^{-1}) (Table 4) were calculated from the Arrhenius-type equation with linear regression analysis (Eq. (2)).

Odor and taste (aromatics associated with mulberry pekmez, honey, and milk), color evaluation (bright or undertone), hardness–softness, mouthfeel, and overall acceptability of the products were evaluated by 48 panelists. Sensory properties of products are given in Table 5.

Table 1. Physical and chemical properties of mulberry fruits (*Morus alba*).*

Properties	Values
Thousand fruit mass (g)	3.554 ± 291
Total dry matter (%)	23.29 ± 0.28
TSS (°Brix)	19.89 ± 0.3
Ash (%)	3.18 ± 0.1
Protein (%)	2.43 ± 0.08
Crude oil (%)	2.71 ± 0.04
pH	6.1 ± 0.12
Acidity (as citric acid, %)	0.21 ± 0.02
Ascorbic acid (mg per 100 g)	74.86 ± 1.22
Total sugar (%)	17.06 ± 1.05
Invert sugar (%)	15.12 ± 0.58
Total phenolic composition (mg GAE per 100 g)	978 ± 26
FRAP antioxidant capacity (mM TE g^{-1})	67 ± 3.5
L	77.7 ± 0.3
a	−13.1 ± 0.2
B	15.5 ± 0.4

*: All data represent the mean of 5 determinations ± SD.

Table 2. Physical and biochemical properties of products.*

Physical and chemical composition	Values			
	Pestil	Pestil with hazelnuts	Pestil with walnuts	Köme
Moisture (%)	9.78 ± 0.13a	10.75 ± 0.69b	10.68 ± 0.33b	20.06 ± 1.09c
Total dry matter (%)	90.22 ± 0.13c	89.25 ± 0.69b	89.32 ± 0.33b	79.94 ± 1.09a
Ash (%)	1.2 ± 0.10c	1.00 ± 0.06b	0.70 ± 0.05a	0.66 ± 0,11a
Acidity (as citric acid, %)	0.14 ± 0.02a	0.14 ± 0.04a	0.15 ± 0.01a	0.13 ± 0.02a
Crude fiber (%)	0.52 ± 0.06a	1.48 ± 0.12b	0,60 ± 0.06a	1.42 ± 0.21b
Protein (%)	4.34 ± 0.47a	7.42 ± 0.25c	6.09 ± 0.80b	5.74 ± 0.14b
Crude oil (%)	0.98 ± 0.23a	16.08 ± 0.64c	13.78 ± 0.89b	13.24 ± 0.60b
Total sugar (%)	62.54 ± 1.92d	57.08 ± 0.98c	51.34 ± 0.91b	41.04 ± 0.85a
Invert sugar (%)	56.23 ± 1.75d	53.01 ± 0.99c	47.64 ± 0.90b	37.52 ± 0.86a
Thickness (mm)	0.80 ± 0.10a	1.12 ± 0.20b	1.25 ± 0.26b	NM
HMF (mg kg^{-1})	27.94 ± 1.54c	21.42 ± 0.61b	18.15 ± 1.20a	17.00 ± 1.58a
Aflatoxin B_1 (µg kg^{-1})	ND	ND	ND	ND
Total aflatoxin (µg kg^{-1})	ND	ND	ND	ND
Total phenolic composition (mg GAE per 100 g)	32.24 ± 21.58d	27.42 ± 0.82c	29.21 ± 1.55b	25.32 ± 1.05a
FRAP antioxidant capacity (mM TE g^{-1})	92.3 ± 2.24c	86.2 ± 1.58b	88.4 ± 1.58b	81.2 ± 2.55a

*: All data represent mean of 5 determinations ± SD; means followed by the same letter are not statistically different at P < 0.05. ND: not detectable; NM: was not measured.

Table 3. Color values of products.*

Sample	*L*	*a*	*b*
Herle	40.17 ± 0.50d	7.17 ± 0.95d	16.17 ± 1.1d
Pestil	38.54 ± 0.55c	6.23 ± 0.30c	9.58 ± 0.66c
Pestil with hazelnuts	37.71 ± 0.60c	5.44 ± 0.02c	6.68 ± 0.04b
Pestil with walnuts	35.85 ± 0.77b	3.56 ± 0.49b	5.75 ± 0.18b
Köme	32.42 ± 0.05a	2.70 ± 0.01a	1.90 ± 0.02a

*: All data represent mean of 5 determinations ± SD; means followed by the same letter are not statistically different at P < 0.05. *L*, darkness/lightness (0, black; 100, white); *a* (–*a*, greenness; +*a*, redness), and *b* (–*b*, blueness; +*b*, yellowness).

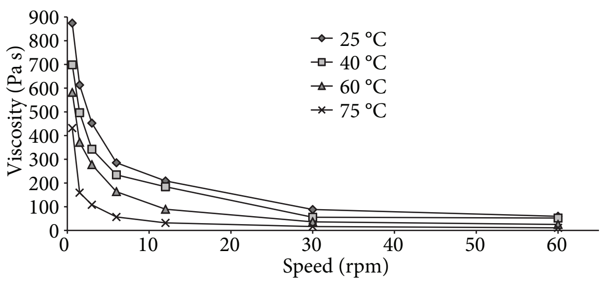

Figure. The rheological behavior of herle at different temperatures.

4. Discussion

4.1. Properties of mulberry fruits (*Morus alba*)

The main ingredient, mulberry fruit, had high antioxidant activity. TPC and FRAP antioxidant capacity tests were used to determine the antioxidant capacity. As can be seen in Table 1, TPC and the FRAP antioxidant capacity of fruits were 978 mg GAE per 100 g fresh mass and 67 mM TE g^{-1} fresh mass. TPCs of mulberries were lower than those of black mulberry fruits but higher than those of mango, guava, papaya, mangosteen, banana, and maluod

Table 4. The consistency index (k) and flow behavior index (n)* of herle at different temperatures and Arrhenius-type equation parameters** for herle.

Temperature (°C)	k	n	r^2	k_0 (Pa s^n)	Ea (J mol^{-1})	r^2
25	65.9	0.40	0.98			
40	51.7	0.40	0.96	1.88×10^{-4}	32,116	0.91
60	26.4	0.28	0.98			
75	9.7	0.21	0.97			

*: k (Pa s^n) and n (dimensionless) indices were obtained by fitting rotational speed-viscosity data to power-law model.
**: $\eta_a = k\gamma^{(n-1)}$, where γ is apparent viscosity, k is consistency index, and n is flow behavior index.

Table 5. Sensory properties of products.*

Sample	Odor and taste	Color evaluation	Hardness–softness	Mouthfeel	Overall acceptability
Pestil	7.08 ± 1.0a	7.25 ± 0.75b	5.50 ± 1.24a	5.67 ± 1.15a	6.25 ± 0.62a
Pestil with hazelnuts	7.58 ± 1.0a	5.92 ± 0.79a	7.08 ± 1.0b	6.83 ± 0.94b	7.00 ± 0.6b
Pestil with walnuts	7.83 ± 0.94a	5.75 ± 1.14a	7.25 ± 1.29b	7.08 ± 1.16bc	6.92 ± 0.51b
Köme	7.92 ± 0.79a	5.92 ± 1.51a	7.42 ± 1.24b	7.92 ± 0.79c	7.50 ± 0.67c

*: 1: dislike intensely, 5: neither dislike nor like, 9: like intensely. All data represent mean of 48 determinations ± SD; means followed by the same letter are not statistically different (P < 0.05).

fruits (Ercişli and Orhan, 2008; Patthamakanokporn et al., 2008) Earlier, TPC of mulberry fruits was reported to range from 1515 to 2570 mg GAE per 100 g fresh mass (Bae and Suh, 2007; Lin and Tang, 2007) and 181 mg GAE per 100 g fresh mass (Ercişli and Orhan, 2007). The reason could probably be the differences in cultivation location (Hakkinen and Torronen, 2003) and practices, genotypes, harvest condition, ripening stage (Raffo et al., 2002), and seasons (Wu et al., 2004).

TFM levels of white mulberry fruits were previously determined to be between 1.130 g and 4.250 g (Polat, 2004), which is similar to our results. Other physical and chemical parameters of fruits such as total dry matter, ash, protein, sugar content, TSS, acidity, pH, L, a, and b were generally close to those found by earlier researchers (İslam et al., 2003; Polat, 2004; Ercişli and Orhan, 2007). In general, mulberry is an important ingredient for the food industry for production different products, such as pekmez, pestil, and köme. Higher TSS, total sugar, invert sugar content, and total dry matter are desirable characteristics of mulberry and these parameters are strongly affected by altitude.

4.2. Physical and biochemical properties of products

Pestil samples gave the lowest moisture content (9.78%) and thickness values. Total protein contents of pestil with hazelnut (7.42%) and pestil with walnut (6.09%) were higher than those of other products. This was because of the nuts and walnuts in the structure of the products including high levels of protein. The thickness of pestil varied from 0.8 to 1.25 mm. The average film thickness of pestil was previously determined to be between 0.5 and 1.2 mm, consistent with our results (Kaya and Maskan, 2003). Crude oil and crude fiber levels of pestil were the lowest. Physical and chemical compositions of pestil were not similar to those found by Ekşi and Artık (1984), except for a few parameters. The reason for this may be different ingredients and different production methods. Total aflatoxin and aflatoxin B_1 were not detected in our samples. It can be said that aflatoxin did not occur in these products in 2 months of shelf-life at room temperature.

The total antioxidant activities of products ranged from 81 to 92 mM of Trolox equivalents g^{-1} and total phenolics ranged from 25 to 32 mg GAE equivalents per 100 g. The antioxidant value and total phenolic contents were higher in pestil than other products. Correlation coefficients and significant values (Table 6) show positive strong correlations between total phenolic composition and total sugar (r: 0.729; P: 0.000), total phenolic composition and invert sugar (r: 0.700; P: 0.001), total phenolic composition and L (r: 0.706; P: 0.001), and total

Table 6. Correlations between some parameters.

		Crude fiber	Total sugar	Invert sugar	Thickness	*L*	*a*	*b*	TPC	FRAP
Crude fiber	(*r*)	1	−0.481(*)	−0.448(*)				−0.596(**)	−0.775(**)	−0.687(**)
	(P)	.	0.032	0.048				0.006	0.000	0.001
Total sugar	(*r*)	−0.481(*)	1	0.994(**)	−0.672(**)	0.974(**)	0.948(**)	0.977(**)	0.729(**)	0.805(**)
	(P)	0.032	.	0.000	0.006	0.000	0.000	0.000	0.000	0.000
Invert sugar	(*r*)	−0.448(*)	0.994(**)	1	−0.626(*)	0.981(**)	0.934(**)	0.963(**)	0.700(**)	0.794(**)
	(P)	0.048	0.000	.	0.013	0.000	0.000	0.000	0.001	0.000
HMF	(*r*)		0.867(**)	0.817(**)		0.780(**)	0.877(**)	0.875(**)	0.669(**)	0.712(**)
	(P)		0.000	0.000		0.000	0.000	0.000	0.001	0.000
Thickness	(*r*)		−0.672(**)	−0.626(*)	1	−0.627(*)	−0.625(*)	−0.715(**)	−0.411	−0.395
	(P)		0.006	0.013	.	0.012	0.013	0.003	0.128	0.145
L	(*r*)		0.974(**)	0.981(**)	−0.627(*)	1	0.945(**)	0.950(**)	0.706(**)	0.770(**)
	(P)		0.000	0.000	0.012	.	0.000	0.000	0.001	0.000
a	(*r*)		0.948(**)	0.934(**)	−0.625(*)	0.945(**)	1	0.919(**)	0.642(**)	0.709(**)
	(P)		0.000	0.000	0.013	0.000	.	0.000	0.002	0.000
b	(*r*)	−0.596(**)	0.977(**)	0.963(**)	−0.715(**)	0.950(**)	0.919(**)	1	0.808(**)	0.882(**)
	(P)	0.006	0.000	0.000	0.003	0.000	0.000	.	0.000	0.000
TPC	(*r*)	−0.775(**)	0.729(**)	0.700(**)		0.706(**)	0.642(**)	0.808(**)	1	0.824(**)
	(P)	0.000	0.000	0.001		0.001	0.002	0.000	.	0.000
FRAP	(*r*)	−0.687(**)	0.805(**)	0.794(**)		0.770(**)	0.709(**)	0.882(**)	0.824(**)	1
	(P)	0.001	0.000	0.000		0.000	0.000	0.000	0.000	.

r: Pearson correlation coefficient ($P < 0.05$), P: significance.
*: Correlation is significant at the 0.05 level (2-tailed).
**: Correlation is significant at the 0.01 level (2-tailed).

phenolic composition and FRAP antioxidant capacity (*r*: 0.824; P: 0.000). Parallel to total sugar and invert sugar content, pestil had the highest HMF value (27.94 mg kg^{-1}). As the total sugar and invert sugar increased, HMF content increased for each group of products. The Pearson correlation coefficients and significant values among total sugar, invert sugar, and HMF are evaluated in Table 6. Significant values among total sugar and HMF, and invert sugar and HMF, were calculated as 0.867 and 0.817, respectively. According to these results, a positive strong relationship was observed between invert sugar and HMF values and total sugar and HMF values. In addition, a positive strong relationship between HMF and TPC (*r*: 0.669; P: 0.001) and between HMF and FRAP antioxidant activity (*r*: 0.712; P: 0.000) showed the antioxidant effect of the Maillard reactions products (Mastrocola and Munari, 2000; Manzocco et al., 2001; Turkmen et al., 2006; Yıldız, 2010). HMF contents are widely used as an indicator of the Maillard reactions and browning of some foods, such as pekmez and honey (Bozkurt et al., 1998; Zappala et al., 2005). During the heating of the herle, nonenzymatic phenol oxidation reactions that are called caramelization reactions may occur, such as formation of melanoidins. The formation of melanoidins has been frequently associated with antioxidant capacity (Manzocco et al., 2001), which may thus increase the antioxidant capacity. The HMF content also shows further stages of the Maillard reaction. This is the cause of Maillard reaction products, with the antioxidant activity showing correlation with the HMF content. Maillard reactions occur between reducing sugars and amino acids, and they depend on many factors, such as food content. The higher the level of invert sugar was, the higher the HMF content was. Our findings are in agreement with the literature (Maillard, 1912; Jing and Kitts, 2004; Spana et al., 2006; Yıldız and Alpaslan, 2012). The determined level of HMF in all products is distinctly lower than maximum values of honey (40 mg kg^{-1}) (Codex Alimentarius, 1981; Turkey Food Codex, 2000). HMF concentrations of apple juice concentrates were previously reported to be between 0.52 and 963 mg kg^{-1} apple juice concentrate (Burdurlu et al., 2006). Aliyazıcıoğlu et al. (2009) reported that HMF contents of mulberry pekmez were between 18.0 and 152.3 mg L^{-1}.

4.3. Color measurements

The color value is one of the most important parameters in the quality of sugar and fruit products produced by heat treatment. Table 3 shows that herle samples had the highest *L* values (40.17), *a* values (7.17), and *b* values (16.17), whereas the köme sample had the lowest *L* values (32.42). *L* values varied from 37.31 to 39.30 in kiwifruit leathers as reported by some researchers (Vatthanakul et al., 2009). *a* values of the products ranged from 2.70 to 7.17 and *b* values ranged from 1.90 to 16.17. Significant differences were found in color values of the products. The addition of hazelnut and walnut changed the color of pestil (lower *L*, *a*, and *b* values), but colors became lighter (higher *L* values) with the addition of other ingredients. The redness occurs as a result of excessive caramelization of sugars and a high *a* value is not desired. The *L*, *a*, and *b* values of mulberry pekmez were previously determined as 19.27, 15.91, and –0.14, respectively (Şengül et al., 2005). Manufacturing processes such as dilution, drying, and baking can affect the final product color.

The Pearson correlation coefficients and significant values among *L*, *a*, *b*, and HMF are stated in Table 6. Correlation coefficients *L* – HMF, *a* – HMF, and *b* – HMF were 0.780, 0.877, and 0.875, respectively. As can be seen, a positive strong relationship was observed between *L*, *a*, and *b* and the HMF values ($0 < r < 0.5$; P: 0.00).

4.4. Rheological properties of herle

The flow behavior of herle appears to be suitable to the power-law model because of high r^2 values. The r^2, *n*, and *k* ranged from 0.96 to 0.98, 0.21 to 0.40, and 9.7 to 65.9, respectively (Table 4). Herle showed a pseudoplastic behavior because the *n* values, a measure of the departure from Newtonian flow, were less than 1 (Grigelmo-Miguel et al., 1999). Increasing the temperature from 25 to 75 °C considerably decreased the *k* value. Similar results were also obtained in some other studies (Grigelmo-Miguel et al., 1999; Alpaslan and Hayta, 2002; Kaya and Belibağlı, 2002; Şengül et al., 2005). The viscosity of liquids generally decreases with increase in temperature. The Figure shows that as temperature and speed increased, the viscosity of herle decreased in this study. In the literature, pekmez samples and some beverages had similar flow diagrams (Şengül et al., 2005; Alpaslan and Hayta, 2007; Aliyazıcıoğlu et al., 2009).

As can be seen in Table 4, E_a and k_a values of herle were calculated as 32,116 J mol^{-1} and 1.88×10^{-4} Pa s^n, respectively. The value of E_a obtained in our study was higher than those reported by other authors for pekmez–tahini (30,329 J mol^{-1}; Alpaslan and Hayta, 2002) and mulberry pekmez (17,970 J mol^{-1}; Şengül et al., 2005). Consequently, these values indicate that the herle did not have a homogeneous texture and uniform particle distribution as in these other samples.

4.5. Sensory properties

Statistical analyses within the evaluation of the sensory characteristics of food products are very important. As can be seen, significant differences were found in this study. Sensory evaluations of products (Table 5) showed that the higher hardness–softness scores are, the higher the dispersion in the mouth and overall acceptability scores are. Odors and tastes of products were found to be between 7.08 and 8.00. Parallel to walnut content, pestil with walnut and köme had higher odors and taste, hardness–softness, and dispersion in the mouth scores than those of pestil and pestil with hazelnut.

Sensory evaluation is an important quality criterion in foods. Taste and health in traditional foods are to be evaluated in parallel and clarified for the customer (Cayot, 2007). As underlined by previous authors (Pickering et al., 2001), further sensory work is needed to elucidate the relationship between the chemical composition and the perceived quality of products.

The present study shows that Gümüşhane pestil and köme are extremely rich food sources, particularly in terms of phenolics, carbohydrates, proteins, and crude fiber. The origin of these characteristics is the used ingredients and the production methods. These products are recommended as an alternative for supplementary snack foods, especially when taking children into account as the primary consumers of snack foods. These products can also be used as nutritional sources and complementary foods as they contain functional compounds. The rheological parameters of the herle may have useful implications for design and processing in the food industry. The findings also indicate that there is huge variation among sensory attributes HMF and *L*, *a*, and *b*. The results obtained in this study could be used in the development of traditional foods. Pestil and especially köme are not well known products and few studies have been published about this kind of product; most of the studied products were different from Gümüşhane pestil and köme. To our knowledge, this was the first detailed study on these traditional foods.

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