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Preliminary investigations of ice creams for the determination of the physico-chemical properties and aroma compounds by GC-MS produced from cow, sheep, goat, and buffalo milk

Voruntersuchungen an Speiseeis, hergestellt aus Kuh-, Schaf-, Ziegen- und Büffelmilch, zur Bestimmung der physikalisch-chemischen Eigenschaften und Aromastoffe

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Summary

In the present study, four different ice creams were produced using cow, buffalo, goat, and sheep milk. The physicochemical, sensory properties and aroma profile analyzes of ice creams were carried out and the differences were evaluated. Dry matter rates of cow ice cream (CIC), goat ice cream (GIC), sheep ice cream (SIC) and buffalo ice cream (BIC) ranged from 27.96% to 30.51% and SIC had the highest content of dry matter (30.51%). Ash values of ice creams were determined between 0.61% and 0.75%. Titratable acidity values varied between 0.11% and 0.18%. pH values of ice creams ranged from 6.49 to 7.04. Fat values of ice creams were determined between 4.05 (g/100g) and 5.10 (g/100g). The lowest overrun value (20.61%) was determined in SIC. The highest first dripping time (1980 s) and complete melting time (5970 s) were found in GIC. A total of 41 aroma compounds were determined by gas chromatography-mass spectrometry (GC-MS) and the BIC was the most aromatic product containing 36 aroma compounds. After the BIC, 15, 13 and 8 aroma compounds were detected in CIC, GIC and SIC, respectively. Additionally, aroma compounds 2-hexanone, 2-heptanone, limonene, 2-nonanone, 2-undecanone, 2-tridecanone, 2-pentadecanone were detected in all ice cream types. To evaluate the correlations between the aroma compounds, the principal component analysis (PCA) method was applied to ice cream samples and a strong positive relation was detected between CIC and GIC. In terms of sensory analysis, general acceptability properties were not statistically different ($p > 0.05$) between all ice creams. SIC had the lowest flavor score and CIC had the highest one as sensory property.

Keywords: ice cream, physicochemical properties, aroma compounds, sensory properties

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Introduction

Ice cream is a frozen milk dessert consumed tastefully by consumers. Ice cream has nutritional and energizing features due to the high content of milk and sugar in it and consumption is possible by all age groups all over the world (Robins et al., 2019). The stability and quality of ice cream can be changed due to the used milk, sugar, fat, stabilizers, sweeteners, and other materials. (Fiol et al., 2017). According to OECD and FAO (2020) data, global milk production was 852 million tons in 2019 and cattle milk constitutes 81% of the world's total milk production. Additionally, buffalo milk (15%), goat, and sheep milk (4% combined with camel milk) also have important places in the world economy. Cow milk is generally used in ice cream production because of its high economic value. At the same time, ice cream can be produced by sheep, goat, and buffalo milk or mixing certain amounts of these.

The type of milk used in ice cream production plays a major role in consumer demands, appearance of the products as well as their unique taste/aroma, texture, physical and chemical properties. Cow, goat, sheep, and buffalo milk may have different effects on people in terms of nutrition and health. For example, goat milk has better digestibility, more alkalinity, higher buffering capability, lower allergic properties, and stronger antimicrobial properties than cow's milk (Bhattarai, 2014; Robins et al., 2019). Sheep milk has a higher density and viscosity with a lower freezing point compared to cow's milk. On the other hand, cow milk has a more yellowish color caused by its carotene content in comparison to sheep and goat milk (Folch et al., 1993; Jooyandeh and Aberoumand, 2010).

In this study, salep was used as a stabilizer in ice cream production as it generally is in Turkey. Salep, special to Maraş type ice cream which is specific to Turkey, enhances flavor and aroma (Mehmet Güven et al., 2010). It has high starch and glucomannan content and is obtained by a series of processes from various plants (from the *Orchidaceae* family) (Tekinşen, 2010; Yaşar et al., 2009). Its healthy effect due to its glucomannan content has been indicated in several studies (Ece Tamer et al., 2006; Tester and Al-Ghazzewi, 2013, 2016).

As in many foods, taste, smell, and aroma compounds are major properties to get consumer acceptance of ice cream. Formation of aroma compounds is closely related to various biochemical compounds such as aldehydes, ketones, amino acids, peptides, and fatty acids in the food. Enzymatic reactions and food production processes such as heat treatment may play a role in the release of aroma compounds. In addition, some flavor compounds can be formed during the oxidative reduction of carotenoids (Belitz et al., 2011).

A limited number of studies have been found comparing ice creams produced from cow, sheep, goat, and buffalo milk at the same time. The aim of this study is to reveal the differences between flavor compounds in the ice creams produced with different animals' milk. Also, the physicochemical properties, viscosity, color parameters, and sensory evaluation of ice creams produced from cow, sheep, goat, and buffalo milk were compared.

Materials and methods

Each type of milk (cow, sheep, goat, and buffalo) was collected from bulk tanks in different dairy farms in Şiran,

Gümüşhane. Other ingredients used in ice cream production such as salep, sugar, and emulsifier were obtained from local markets.

Ice cream manufacturing

Ice cream made from cow, goat, sheep, and buffalo milk were manufactured in the laboratory of Şiran Mustafa Beyaz Vocational School, Gümüşhane, Turkey. 1 kg of ice cream were produced for each milk type by adding 16% (w/w) sugar, 0.7% (w/w) salep, and 0.2% (w/w) emulsifier (mono- and di-glycerides). Pasteurization was carried out at 85°C for 25 s and mixtures were cooled (4 °C) immediately. Produced mixtures were stored in the refrigerator at 4 °C for 24 h to mature. An ice cream machine (Sage BCI 600 BSS., Australia) was used to ice (−5 °C) samples. Then, hardened samples were stored in a deep freezer at −22 °C for 24 h. The ice creams were stored at −18 °C during analyses. All ice cream samples were produced in duplicate batches.

Physical and chemical analyses

The physicochemical properties such as dry matter (% w/w), ash (% w/w), fat (g/100g), titratable acidity (% lactic acid, w/w), and pH of ice cream samples were determined as explained by Güven and Karaca (2002). Dry matter (% w/w) and ash (% w/w) contents were determined gravimetrically. The Gerber method was used to get fat (g/100g) contents of ice cream samples. The acidity of mixtures was evaluated with the titrimetric method and expressed in % lactic acid (w/w) (Metin, 2012). pH values were obtained via pH meter (WTW 3110, Weilheim, Germany). To express overrun (%) of the products the following equation was used (Akbari et al., 2016).

$$\text{Overrun (\%)} = \frac{(\text{weight of mix}) - (\text{weight of ice cream})}{\text{weight of ice cream}} \times 100$$

First dripping and complete melting time of samples were determined according to the method of Güven and Karaca (2002). 25 g ice cream samples were kept on a mesh (mesh size 2 mm) on a flask (about 20 °C) for melting. First dripping times and complete melting times were noted as seconds.

Viscosity measurements were performed with a digital Brookfield Viscometer, Model DV-II (Brookfield Engineering Laboratories, Stoughton, MA, USA). After removing air bubbles by gently stirring the samples (Ozer et al., 1998) analysis was applied with spindle number 4 at different rpms (from 2.5 rpm to 100 rpm) at 4 °C. The rheological properties were modeled by the Power-law model and the apparent viscosity, the flow behavior index, the consistency coefficient, and the shear rate were equated with η (Pa s), n , K (Pa sⁿ), and γ (rpm), respectively (Steffe, 1967).

$$\eta = K \gamma^{(n-1)}$$

Color measurements

The color parameters (L^* , a^* , b^*) were analyzed with a CR-200 Minolta colorimeter (Minolta Camera Co., Osaka, Japan). Before the analysis calibration of the machine was made by a standard white plate. Hue angle (H°) value was calculated as reported in a method of McLellan et al. (1995). The colors (red, yellow, green, blue, and red) were defined with 0° 90° 180°, 270°, 360°, respectively. Croma (C^*) was calculated according to the formula $C^* = [a^{*2} + b^{*2}]^{1/2}$ (Cecchini et al., 2011). White Index (WI) was determined according to Kurt and Atalar (2018) method.

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Aroma profile analysis with gas chromatography/mass spectrometry (GC/MS)

Aroma components of ice cream samples were determined by Gümüşhane University Faculty of Engineering and Natural Science Department of Food Engineering. The method of Chen et al. (1998) with some modifications was used for aroma profile analyses. Aroma compounds were obtained using a micro-steam distillation-solvent extraction (SDE) device developed in Gümüşhane University Central Research and Application Centre. The design of the SDE device allows both steam and liquid phase extraction to be performed. Also, continuous water and solvent reflux were provided during the extraction period. 50 g of homogenized ice cream sample was weighed into a 2000 ml flask. The sample was placed in a circulating water bath with cooling water at $-1\text{ }^{\circ}\text{C}$ in a modified double skin cooled Clevenger device. It was diluted with a saturated NaCl solution to a ratio of 1:3. Samples were placed in a modified glass collection device with a special cooler and boiled for 3 hours. Evaporated aroma compounds were kept in 2 mL of hexane. Water in hexane was dried with anhydrous Na_2SO_4 . After drying samples were taken into a 1.8 mL vial and given to the GC-MS / FID Detector (Agilent 5975, Agilent Technologies Inc., USA) device for analysis.

GC-MS Conditions; Device: Agilent 5975 GC-MS Detector. Column: 30 m \times 0.25 mm ID, 0.2 μm HP-5MS. Oven: 50 $^{\circ}\text{C}$, 4 $^{\circ}\text{C}/\text{min}$ 260 $^{\circ}\text{C}$, 15 min. Carrier Gas: Helium, constant flow 1.2 mL/min. Injection: 250 $^{\circ}\text{C}$. Detector: MS, 230 $^{\circ}\text{C}$. Split: 1:25. Injection volume: 1 μL . H2: 40 mL/min. Dry Air: 400 mL/min.

The amount of each compound was calculated as % area by the formula below. Results are given as % area.

$$\% \text{ Area} = (A_x / A_T) \times 100 \quad (A_x: \text{Area of aroma compound}, A_T: \text{Total area of aroma compounds})$$

Sensory analysis

The sensorial features of ice cream samples were analyzed by the results of 50 consumer evaluations. The parameters for analysis were color, gumming, texture, flavor, resistance to melting, and overall acceptability of samples (Meilgaard et al., 2016). The scale between 1 (poor) and 9 (excellent) was used for assessment. The panelists tasted the samples in separated compartments. There were 29 males and 21 females, and their ages were between 20 and 40. The panelists were trained and experienced about sensorial analysis.

Statistical analysis

SPSS software program version 17 (SPSS Inc., Chicago, IL, USA) was used for analyzing the data. Differences were stated out with Analyses of variance (ANOVA) and Duncan's Multiple Range Tests in the presented study. PCA and agglomerative hierarchical clustering (AHC) were performed to evaluate the possible relationship between the studied parameters using the software package (XLSTAT Addinsoft SARL 2019). All analyses were carried out in triplicates and results were given as mean \pm standard deviation (SD).

Results and discussion

With this study, it is aimed to observe the differences between ice creams produced with cow, sheep, goat, and buffalo milk. There is not any standardization on milk

TABLE 1: Physico-chemical properties of used milk for producing cow, buffalo, goat, and sheep ice creams.

	Cow	Buffalo	Goat	Sheep
Dry matter (% w/w)	14.15	14.60	14.92	16.73
Ash (% w/w)	0.71	0.66	0.86	0.83
Titrateable acidity (% lactic acid, w/w)	0.14	0.19	0.21	0.24
pH	6.82	6.51	6.59	6.62
Fat (g/100g)	5.4	5.9	5.1	6.0

* Results are shown by means.

used in ice cream production. After milking, milk was kept under suitable conditions and ice cream was produced afterwards. The average of dry matter (% w/w), fat (g/100g), ash (% w/w), pH, and titrateable acidity values of milk used in ice cream production are given in Table 1. It is seen that dry matter ratio of sheep milk is higher than other milk (Table 1). Sheep and buffalo milk are expected to have higher dry matter and fat content than cow and goat milk. However, seasonal factors, nutrition, and animal strain may decrease the differences. In previous research, Kanwal et al. (2004) reported dry matter ratio in sheep, cow, goat, and buffalo milk as 18.53%, 13.73%, 13.55%, and 14.04%, respectively, as in parallel to our results.

Physicochemical analysis of ice creams

Table 2 shows the results of dry matter, ash, acidity, pH, fat, overrun, first dripping time, and complete melting time of CIC, GIC, SIC, and BIC. Dry matter ratios of CIC, GIC, SIC, and BIC range from 27.96 to 30.51% (Table 2). When statistically evaluated, there are differences ($p < 0.05$) between the dry matter of ice creams. However, according to multiple comparison results, there were no statistical differences between CIC-BIC and likewise between GIC-SIC samples. SIC had the highest content of dry matter (30.51%). Nadelman et al. (2017) reported moisture content of 67.61% in conventional SIC and 72.73% in probiotic SIC and Balthazar et al. (2017) found moisture content as 69.5% in SIC which are closed results with SIC in our study. Kurt et al. (2016) determined total solids between 28.10% and 28.66% in salep-based ice cream and Erkaya et al. (2012) found total solids as 29.31% in ice cream which were similar to CIC. These differences of dry matter may arise from the type of milk used in production, production method, and other factors.

CIC, SIC, GIC, and BIC samples are observed to have statistical differences ($p < 0.05$) in ash, acidity, pH, and fat. Ash values of ice creams were determined between 0.61% and 0.75% and GIC had the highest ash content. The ash amount of ice creams may differ according to ice cream mix content (Dervisoglu and Yazici, 2006). Although variable ash ratios were found in the literature (Açu et al., 2017; Gupta and Park, 2015; Pankiewicz et al., 2020; Yanglar, 2015), the ash values found by Silva et al. (2015) in goat's milk ice cream were similar with GIC. Titrateable acidity values varied between 0.11% and 0.18% and SIC had the highest percentage. Similar results were found in literature range from 0.13 to 0.19 for CIC (Kurt et al., 2016; Ozdemir et al., 2015) and GIC (Robins et al., 2019; Senaka Ranadheera et al., 2013). pH values of ice creams ranged from 6.49 to 7.04. Güzeler et al. (2017) found pH value as 6.44 for goat ice cream which was close to our results and cow ice cream's pH value (6.66) was different from ours. Similarly, close pH results are available in literature for BIC

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TABLE 2: Physico-chemical properties of ice cream samples produced from different milk types.

	CIC ¹	BIC ¹	GIC ¹	SIC ¹
Dry matter (% w/w)	27.96 ± 0.01 ^A	28.09 ± 0.04 ^A	29.94 ± 0.01 ^B	30.51 ± 0.55 ^B
Ash (% w/w)	0.66 ± 0.01 ^A	0.61 ± 0.01 ^B	0.75 ± 0.02 ^C	0.68 ± 0.02 ^A
Titrate acidity (% lactic acid, w/w)	0.15 ± 0.00 ^A	0.11 ± 0.01 ^B	0.16 ± 0.02 ^{AC}	0.18 ± 0.00 ^C
pH	7.04 ± 0.01 ^A	6.49 ± 0.01 ^B	6.53 ± 0.03 ^B	6.62 ± 0.01 ^C
Fat (g/100g)	4.05 ± 0.0 ^A	4.40 ± 0.14 ^{AB}	4.65 ± 0.21 ^B	5.10 ± 0.14 ^C
Overrun (%)	36.47 ± 3.79 ^A	38.84 ± 6.20 ^A	30.18 ± 5.90 ^{AB}	20.61 ± 0.91 ^B
First dripping time (s)	1470 ± 127.28 ^A	1350 ± 42.43 ^A	1980 ± 0.00 ^B	1890 ± 127.28 ^B
Complete melting time (s)	5670 ± 636.40 ^A	3870 ± 212.13 ^B	5970 ± 212.13 ^A	3390 ± 42.43 ^B

* Results are shown by means ± standard deviation. Different letters used in the same line show the statistical difference ($p < 0.05$) between the results. ¹CIC: Cow ice cream, BIC: Buffalo ice cream, GIC: Goat ice cream, SIC: Sheep ice cream

(Bekiroğlu and Özdemir, 2020) and incompatible results for SIC (Balthazar et al., 2018). CIC had the highest pH when compared with other ice creams. This may be due to the high pH value of cow milk. On the other hand, BIC had the lowest pH and titrate acidity values. Fat values of ice creams were determined between 4.05 g/100g and 5.10 g/100g and the fat content of SIC was higher than the other ice creams (Table 2). Helena et al. (2014) found fat content of CIC as 4% which was similar to our CIC.

Overrun is an expression of change in volume with air entering the ice cream mix. Although it is a technical parameter, it may vary depending on the production process and ingredients of ice cream (Karaca et al., 2009; Sun-Waterhouse et al., 2013; Silva et al., 2015). There are statistical differences ($p < 0.05$) between the overrun values of ice creams. While BIC had the highest overrun value as 38.84%, the lowest value was determined with SIC as 20.61%. It is understood that the highest volume increase occurred in BIC. It can be concluded that BIC contains more air than the other ice creams. The overrun value of the CIC was in agreement with the studies of Güzeler et al. (2017), Karaca and Güven (2016) and the study of Bekiroğlu and Özdemir (2020) for the BIC.

Melting properties have an important place among the quality parameters of ice cream (Ercaya et al. 2012). Overrun (Sofjan and Hartel, 2004), fat content (Karaca et al., 2009), and milk type (Pandya and Ghodke, 2007) can affect the melting properties of ice cream. There were statistical differences ($p < 0.05$) between the melting properties of ice creams. BIC had the lowest first dripping time but there was no statistical difference with CIC (Table 2). It could be deduced from the results that BIC and SIC had lower resistance to melting compared to CIC and GIC. However, it is interesting that the first dripping time of SIC was longer than CIC and BIC while complete melting time was shorter than the other ice creams. GIC showed a longer complete melting time and first dripping. But there was no statistical difference between GIC and CIC for complete melting time and between GIC and SIC for the first dripping time. It is also seen in Table 2 that GIC has the highest first dripping time and complete melting time. It was observed GIC had the highest resistance to melting. Karagözlü and Ayhan (2019) found first dripping time of ice cream made from goat milk was higher than cow milk ice cream according to first day analysis which was a similar result with our study.

For color measurement, parameter a^* refers to reddish colors for positive values and greenish colors for negative values, b^* refers to yellowish colors for positive values and bluish colors for negative values. The L^* parameter is an approximate brightness measurement between black and white. C^* value includes calculations that express the degree of colorfulness, and WI expresses the degree of whiteness. H° is an attribute that defines reddish, yellowish, greenish, and bluish colors. An angle of 0° or 360° , 90° , 180° , and 270° represent red, yellow, green, and blue hues, respectively (Pathare et al., 2013). Color values (L^* , a^* , b^* , H° , C^* , and WI) of ice creams were given in Table 3. When color measurements of ice cream samples were compared, there were significant differences ($p < 0.05$) between all color parameters except WI values. The highest values of L^* , a^* , b^* , and C^* parameters were measured for CIC. There was no statistical difference between SIC and GIC, and they had the highest H° value. When compared GIC and SIC from Table 3, it was understood that there were no significant differences between the color values. Balthazar et al. (2018) found similar WI values about SIC as like our study.

Viscosity is an important factor and feature in terms of the melting of ice cream and the taste left in the mouth. When the viscosity values of ice cream samples were compared, it was observed that GIC had the highest result for rpm 20 and rpm 50 (Table 4). BIC had the second-highest result but was not statistically different ($p < 0.05$) from the highest one. Kaya and Tekin (2001) stated the increase of viscosity increases resistance against melting. It was expected that GIC may have had a high resistance to melting due to its high viscosity values. Factors such as the amount of air penetrating, structure of ice crystals, form of fat globules can be effective against melting resistance. Additionally, if ice cream contains more air bubbles, it may cause slower heat conduction (Moeenfarid and Tehrani, 2008). When the ice creams were compared, it was seen that SIC had the lowest overrun value. These factors may have reduced the SIC's resistance to melting. The average

TABLE 3: Color parameters of ice cream samples produced from different milk types.

Color parameters	CIC ¹	BIC ¹	GIC ¹	SIC ¹
L^*	95,16 ± 0.45 ^A	88,47 ± 0.31 ^{AB}	82,91 ± 0.26 ^B	88,59 ± 6.73 ^{AB}
a^*	-3,24 ± 0.13 ^A	-3,32 ± 0.20 ^A	-4,80 ± 0.18 ^B	-4,52 ± 0.16 ^B
b^*	14,47 ± 1.33 ^A	9,14 ± 0.70 ^B	10,07 ± 0.72 ^B	9,84 ± 1.50 ^B
H°	102,64 ± 0.64 ^A	109,97 ± 0.32 ^B	115,53 ± 0.81 ^C	114,82 ± 2.54 ^C
C^*	14,83 ± 1.33 ^A	9,73 ± 0.73 ^B	11,15 ± 0.73 ^B	10,84 ± 1.42 ^B
WI	84,40 ± 1.40 ^A	84,72 ± 0.62 ^A	79,54 ± 0.21 ^A	83,62 ± 3.83 ^A

* Results are shown by means ± standard deviation. Different letters used in the same line show the statistical difference ($p < 0.05$) between the results. ¹CIC: Cow ice cream, BIC: Buffalo ice cream, GIC: Goat ice cream, SIC: Sheep ice cream

TABLE 4: Rheological properties of ice cream samples produced from different milk types.

Viscosity (cP)	CIC ¹	BIC ¹	GIC ¹	SIC ¹
rpm20	4949.79 ± 4322.48 ^A	13831.11 ± 1538.15 ^B	15553.38 ± 146.20 ^B	10741.40 ± 442.87 ^{AB}
rpm50	3166.22 ± 2533.64 ^A	8668.26 ± 322.81 ^{BC}	9520.36 ± 133.85 ^C	5598.32 ± 12.18 ^{AB}
K	12.00 ± 2.60 ^A	37.06 ± 8.00 ^B	70.69 ± 3.05 ^C	55.85 ± 10.83 ^{BC}
n	0.60 ± 0.17 ^A	0.62 ± 0.06 ^A	0.36 ± 0.03 ^A	0.57 ± 0.05 ^A

* Results are shown by means ± standard deviation. Different letters used in the same line show the statistical difference ($p < 0.05$) between the results. ¹CIC: Cow ice cream, BIC: Buffalo ice cream, GIC: Goat ice cream, SIC: Sheep ice cream

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viscosity value of the non-newtonian fluid is expressed as K and the flow behavior index, which measures the deviation of fluid from Newtonian flow, is expressed by n (Pang et al., 2020). Therefore, the K value is a parameter related to viscosity that affects structure and texture (Balthazar et al., 2017). K values of samples ranged from 12.00 to 70.69 Pa sⁿ (Table 4). K value of GIC (70.69 Pa sⁿ) was higher than other samples ($p < 0.05$). Javidi et al. (2016) stated that the K values related to fat type and concentration and found the ice cream with low fat content had the lower K value as similar with CIC in our study. n values of all ice creams ranged from 0.36 to 0.62 and there were no significant differences between them ($p > 0.05$). Depending on the n values, all ice creams showed pseudoplastic behavior (Table 4). Similar results were determined by Kurt and Atalar (2018).

Aroma profile of ice creams

Aroma profiles of CIC, SIC, GIC, and BIC are shown in Table 5. The presence and amount of aroma compounds determined in ice creams are expressed as % area. To indicate differences in ice cream samples AHC, PCA, and partial least squares (PLS) analyzes were performed. Multiple analyzes are shown in Figure 1 in a combined form. The further analyzes via AHC resulted in a distinguished red, black, and green cluster. As seen in Figure 1, the red cluster was BIC, the black cluster was CIC and GIC and finally, the green cluster was SIC. According to PLS analysis, 41 aroma compounds were detected in the red cluster containing BIC. Additionally, approximately 15 flavor compounds were determined in the black cluster containing CIC and GIC. There were approximately 8 flavor compounds in the green cluster demonstrating SIC. Another analysis of data was PCA and it was used to show correlations between the amounts of flavor components. These correlations can also be seen in Figure 1 and strong positive relation was obtained for flavor components in CIC and GIC.

The aroma compounds 2-hexanone, 2-heptanone, limonene, 2-nonanone, 2-undecanone, 2-tridecanone, 2-pentadecanone and 3,7,11,15-tetramethyl-2-hexadecene were detected in all types of ice creams. Limonene had the highest ratio among the aroma compounds detected (Table 5). Limonene with a lemon-like odor is a monocyclic monoterpene. Limonene is an essential ingredient of citrus oil such as orange, lemon, tangerine, and

grapefruit widely used as a flavoring due to its pleasant citrus scent (Sun, 2007). Limonene amounts were determined as 28.60% in sample CIC and 26.75% in GIC. The second highest aroma compound was 2-pentadecanone and it was detected in SIC with an area of 26.92%. The compounds

TABLE 5: *Aroma compounds identified by GC-MS in cow, sheep, goat, and buffalo milk ice creams.*

Aroma compounds	Experimental Kovats Index	Literature Kovats Index	Ice creams made with different milk (% Area)			
			CIC ¹	SIC ¹	GIC ¹	BIC ¹
2-hexanone	789	789	2.90	7.33	2.56	1.57
2-heptanone	890	890	8.18	8.01	11.96	4.66
α -pinene	933	933			0.75	
Limonene	1029	1029	28.60	10.52	26.75	2.37
γ -terpinen	1059	1059			5.80	
2-nonanone	1092	1092	5.72	4.94	9.55	3.00
2-undecanone	1294	1294	7.60	8.12	4.58	4.00
α -guaiene	1443	1444				1.28
α -farnesene	1465	1462				0.61
(+) γ -selinene	1480	1480				0.86
(-)-zingiberene	1483	1484				1.74
α -curcumene	1486	1486				4.42
(+)- β -selinene	1490	1493				0.31
2-tridecanone	1496	1496	8.12	21.96	6.68	7.26
Zingiberene	1498	1498				7.63
Eremophilene	1512	1504				3.54
2,4-di-tert-butylphenol	1514	1513	1.22		0.99	
Cubenene	1515	1515				0.66
β -sesquiphellandrene	1528	1528				2.20
α -bisabolene	1541	1543				1.03
Selina-3.7(11)-diene	1549	1548				0.34
β -oplopenone	1571	1574				0.93
Humulene epoxide	1604	1604				1.13
Caryophylladienol II	1639	1639				0.02
3-oxo- α -ionol	1662	1665				1.11
Eudesm-4(15).7-dien-1 β -ol	1674	1670				0.51
Δ -dodecalactone	1682	1681	1.93			
γ -dodecalactone	1683	1684	1.90			2.04
2-tetradecanone	1597	1597	8.20			
2-pentadecanone	1699	1699	7.40	26.92	19.69	5.03
δ -dodecalactone	1712	1711	3.74		1.30	3.87
9-octadecene	1788	1771				1.12
Octadecane	1799	1800	2.30			1.39
1,2-tetradecanediol	1818	1826			2.39	
3,7,11,15-tetramethyl-2-hexadecene	1847	1847	3.82	8.14	3.51	3.00
Phenethyl octanoate	1857	1857				5.16
Methyl hexadecanoate	1927	1927	6.72			11.94
Hexadecanoic acid	1970	1970				6.11
Ethyl palmitate	1995	1995				1.43
Methyl linoleate	2096	2096				0.52
Not detected	2107	-				1.56
Methyl octadecanoate	2128	2128				3.50

* Ice creams containing aroma compounds are expressed as %area. ¹CIC: Cow ice cream, BIC: Buffalo ice cream, GIC: Goat ice cream, SIC: Sheep ice cream

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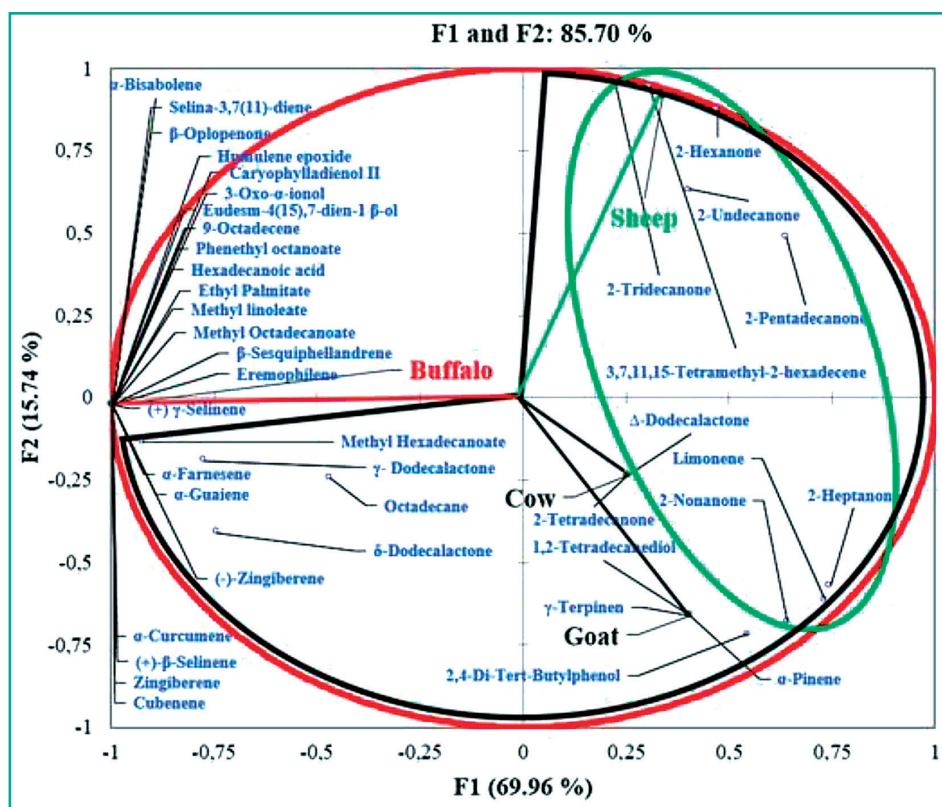


FIGURE 1: To compare ice creams samples, the AHC, PCA and PLS methods were applied. The first two main components explained 85.70% of the total variance, with the first component (F1) 69.16% and the second one (F2) 15.74%.

α -pinene, γ -terpinene, and 1,2-tetradecanediol were detected only in GIC. However, 6-heptyl-tetrahydropyran-2-one and 2-tetradecanone were found only in CIC. On the other hand, all aroma compounds in the SIC were also detected in all types of ice creams. In other words, SIC had no specific aroma compounds. Since BIC had much more aromatic compounds in comparison to other ice creams, it could be expected as more variable in taste and aromas. Bennato et al. (2020) reported dodecanoic acid, 2-heptanone and 2-nonanone compounds in goat cheeses ripened for 60 days and these results were similar to our study. Besides, in another SIC research, 2-heptanone, limonene, 2-nonanone, 2-undecanone, 2-tridecanone, which are aroma compounds were detected as in the case of our study. In the same study, reported γ -terpinene, dodecanoic acid, and hexadecanoic acid were also obtained in our study (Balthazar et al., 2018). In addition, γ -terpinene in GIC, dodecanoic acid in GIC, BIC, and CIC, and hexadecanoic acid in BIC were detected in this research.

The important odor-active compounds 2-hexanone, 2-heptanone, 2-nonanone were identified in our study. These aroma compounds were found in all ice creams made with cow, sheep, goat, and buffalo milk. Attaie (2009) indicated that these three compounds were important odor compounds in goat cheese. In another study, these three volatile compounds and 2-undecanone were found in milk. 2-heptanone and 2-nonanone were reported as effective on off-flavor of UHT milk (Vazquez-Landaverde et al., 2005). It was stated some volatile compounds like 2-hexanone, 2-heptanone, 2-nonanone, and 2-undecanone contribute to sweet, fruity, buttery, musty, floral, and creamy flavors of dairy products (Cheng, 2010; Tian et al., 2019). These methyl-ketones with 5-13

carbon atoms that have been produced during lipid oxidation were mentioned in several studies and these volatile organic compounds were suggested to have a fruit-like flavor (Sattin et al., 2016; Schoina et al., 2020).

In our research, α -pinene and γ -terpinene were determined in only GIC. Limonene was determined in all types of milk ice creams. These monoterpene hydrocarbons have strong antimicrobial and antioxidant properties. α -bisabolene, farnesene, cubenene, zingiberene, and hexadecanoic were also determined in studies especially about goat milk and goat milk products (Bezerra et al., 2017; Palencia et al., 2014; Sabia et al., 2020). We found γ -dodecalactone which may be responsible for sweet aroma (Ozmen Togay et al., 2017) in CIC and BIC also reported in some studies of ice cream (Friedeck et al., 2003) and Circassian cheese (Guneser and Yuceer, 2011). Additionally, Herreño cheese contained dodecanoic acid and hexadecanoic acid (Palencia et al., 2014). We found octadecane in both CIC and BIC but Kınık et al. (2017) detected this compound in goat cheese unlike us.

Sensory properties

There were no significant differences between color, gumming, texture, and overall acceptability for all ice cream samples (Table 6). On the other hand, CIC got the highest score in terms of flavor and was statistically different from other ice creams. According to the panelists' evaluations about resistance to melting, BIC has the lowest value with 5.82. Helena et al. (2014) found no statistical difference between CIC and BIC about color, flavor, and texture. However, in our study, there was no statistical difference between color and texture, while aroma was statistically different. There is no statistical difference ($p > 0.05$) between ice cream samples in terms of general acceptability, but CIC had the highest score.

TABLE 6: Sensory evaluation of ice cream samples produced from different milk types.

Sensory properties	CIC ¹	BIC ¹	GIC ¹	SIC ¹
Color	7.89 ± 0.03 ^A	7.69 ± 0.11 ^A	7.57 ± 0.33 ^A	7.59 ± 0.49 ^A
Gumming	6.36 ± 0.14 ^A	6.25 ± 0.07 ^A	6.88 ± 0.57 ^A	6.38 ± 0.54 ^A
Texture	6.54 ± 0.29 ^A	6.69 ± 0.16 ^A	6.71 ± 0.11 ^A	6.85 ± 0.34 ^A
Flavor	6.78 ± 0.08 ^B	6.07 ± 0.15 ^{AB}	6.19 ± 0.32 ^{AB}	5.83 ± 0.46 ^A
Resistance to melting	6.69 ± 0.26 ^B	5.82 ± 0.02 ^A	6.23 ± 0.23 ^{AB}	6.65 ± 0.40 ^B
Overall acceptability	6.84 ± 0.05 ^A	6.23 ± 0.04 ^A	6.38 ± 0.41 ^A	6.57 ± 0.30 ^A

* Results are shown by means ± standard deviation. Different letters used in the same line show the statistical difference ($p < 0.05$) between the results. ¹CIC: Cow ice cream, BIC: Buffalo ice cream, GIC: Goat ice cream, SIC: Sheep ice cream

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Conclusion

When the differences between CIC, BIC, GIC, and SIC were evaluated, the overrun value of SIC was found lower than other ice creams. A high overrun value is desirable in the production of soft ice cream. Therefore, it can be said that using cow and buffalo milk in soft ice cream production is more appropriate. Considering the melting resistances of ice creams, it is concluded that BIC is weaker than other ice creams according to both sensory analysis and first dripping-complete melting times. But more flavoring compounds were detected in BIC than other ice creams. This may indicate that BIC has a better aromatic structure compared to other ice creams. Furthermore, there was a strong correlation between CIC and GIC in terms of aroma compounds. When viscosity, first dripping, and complete melting values are evaluated together, it is understood that GIC had the best results. Accordingly, we could say GIC was more viscous and durable than the other ice creams. In general, all ice creams were acceptable by panelists in terms of sensory. However, in terms of flavor, SIC had the least taste. After all, we could say that, although there were differences, ice creams with all milk types were acceptable for consumers. Although all ice creams were similar in general acceptance, BIC had the highest score.

Conflict of interest

The authors declare that they have no conflict of interest.

References

- Açu M, Kinik Ö, Yerlikaya O (2017):** Functional properties of probiotic ice cream produced from goat's milk. *Carpathian Journal of Food Science and Technology* 9(4): 86–100
- Akbari M, Eskandari MH, Niakosari M, Bedeltavana A (2016):** The effect of inulin on the physicochemical properties and sensory attributes of low-fat ice cream. *International Dairy Journal* 57: 52–55
- Attaie R (2009):** Quantification of volatile compounds in goat milk Jack cheese using static headspace gas chromatography. *Journal of Dairy Science* 92(6): 2435–2443
- Balthazar CF, Silva HLA, Cavalcanti RN, Esmerino EA, Cappato LP, Abud YKD Moraes J, Andrade MM, Freitas MQ, Sant'Anna C, Raices RSL, Silva, MC, Cruz AG (2017):** Prebiotics addition in sheep milk ice cream: A rheological, microstructural and sensory study. *Journal of Functional Foods* 35: 564–573
- Balthazar CF, Silva HLA, Esmerino EA, Rocha RS, Moraes J, Carmo, MAV, Azevedo L, Camps I, Abud YKD, Sant'Anna C, Franco RM, Freitas MQ, Silva MC, Raices RSL, Escher GB, Granato D, Senaka RC, Nazarro F, Cruz AG (2018):** The addition of inulin and *Lactobacillus casei* 01 in sheep milk ice cream. *Food Chemistry* 246: 464–472
- Bekiroğlu H, Özdemir S (2020):** The quality of ice cream samples made from buffalo milk. *Food and Health* 6(1): 20–26
- Belitz HD, Grosch W, Schieberle P (2011):** *Food Chemistry*. Springer Berlin Heidelberg, Berlin, Heidelberg
- Bennato F, Ianni A, Innosa D, Grotta L, D'Onofrio A, Martino G (2020):** Chemical-nutritional characteristics and aromatic profile of milk and related dairy products obtained from goats fed with extruded linseed. *Asian-Australasian Journal of Animal Sciences* 33(1): 148–156
- Bezerra TKA, Arcanjo NMO, Araújo, ARR, Queiroz ALM, Oliveira MEG, Gomes AMP, Madruga MS (2017):** Volatile profile in goat coalho cheese supplemented with probiotic lactic acid bacteria. *LWT – Food Science and Technology* 76: 209–215
- Bhattarai RR (2014):** Importance of Goat Milk. *Journal of Food Science and Technology Nepal* 7: 107–111
- Cecchini M, Contini M, Massantini R, Monarca D, Moschetti R (2011):** Effects of controlled atmospheres and low temperature on storability of chestnuts manually and mechanically harvested. *Postharvest Biology and Technology* 61(2–3): 131–136
- Chen SH, Huang TC, Ho CT, Tsai PJ (1998):** Extraction, Analysis, and Study on the Volatiles in Roselle Tea. *Journal of Agricultural and Food Chemistry* 46(3): 1101–1105
- Cheng H (2010):** Volatile flavor compounds in yogurt: A review. *Critical Reviews in Food Science and Nutrition* 50(10): 938–950
- Dervisoglu M, Yazici F (2006):** Note The effect of citrus fibre on the physical, chemical and sensory properties of ice cream. *Food Science and Technology International* 12(2): 159–164
- Ece Tamer C, Karaman B, Utku Copur O (2006):** A Traditional Turkish Beverage: Salep. *Food Reviews International* 22(1): 43–50
- Erkaya, Dağdemir E, Şengül M (2012):** Influence of Cape gooseberry (*Physalis peruviana* L.) addition on the chemical and sensory characteristics and mineral concentrations of ice cream. *Food Research International* 45(1): 331–335
- Fiol C, Prado D, Romero C, Laburu N, Mora M, Iñaki Alava J (2017):** Introduction of a new family of ice creams. *International Journal of Gastronomy and Food Science* 7: 5–10
- Folch JM, Coll A, Sánchez A (1993):** Cloning and sequencing of the cDNA encoding goat beta-lactoglobulin. *Journal of animal science* 71(10): 2832
- Friedeck KG, Karagul-Yuceer Y, Drake MA (2003):** Soy Protein Fortification of a Low-fat Dairy-based Ice Cream. *Journal of Food Science* 68(9): 2651–2657
- Guneser O, Yuceer YK (2011):** Characterisation of aroma-active compounds, chemical and sensory properties of acid-coagulated cheese: Circassian cheese. *International Journal of Dairy Technology* 64(4): 517–525
- Gupta CE, Park BP (2015):** Evaluation of Total Fatty Acid Profiles of Two Types of Low-Fat Goat Milk Ice Creams. *Open Journal of Animal Sciences* 5: 21–29
- Güven M, Karaca OB (2002):** The effects of varying sugar content and fruit concentration on the physical properties of vanilla and fruit ice-cream-type frozen yogurts. *International Journal of Dairy Technology* 55(1): 27–31
- Güven M, Karaca OB, Yaşar K (2010):** Düşük Yağ Oranlı Kahramanmaraş Tipi Dondurma Üretiminde Farklı Emülgatörlerin Kullanımının Dondurmaların Özellikleri Üzerine Etkileri. *GIDA* 35(2): 97–104
- Güzeler N, Doğu L, Kalender M, Özbek Ç (2017):** Some Physicochemical and Sensory Properties of Ice Cream Samples Produced by Different Milk Types with Different Amounts: 2nd International Energy & Engineering Conference, Gaziantep, Turkey 2017, 820–825
- Pelegrine DHG, Silva FRSR (2014):** Dairy Products Production with Buffalo Milk. *International Journal of Applied Science and Technology* 4(3): 14–19
- Javidi F, Razavi SMA, Behrouzian F, Alghooneh A (2016):** The influence of basil seed gum, guar gum and their blend on the rheological, physical and sensory properties of low fat ice cream. *Food Hydrocolloids* 52: 625–633
- Jooyandeh H, Aberoumand A (2010):** Physico-Chemical, Nutritional, Heat Treatment Effects and Dairy Products Aspects of Goat and Sheep Milks. *World Applied Sciences Journal* 11(11): 1316–1322
- Kanwal R, Ahmed T, Mirza B (2004):** Comparative Analysis of Quality of Milk Collected from Buffalo, Cow, Goat and Sheep of Rawalpindi/Islamabad Region in Pakistan. *Asian Journal of Plant Sciences* 3(3): 300–305
- Karaca OB, Güven M (2016):** The effects of the combined use of salep and locust bean gum with some stabilizers on the properties of Kahramanmaraş type ice creams. *Agriculture & Food* 4: 327–338
- Karaca OB, Güven M, Yasar K, Kaya S, Kahyaoglu T (2009):** The functional, rheological and sensory characteristics of ice creams with various fat replacers. *International Journal of Dairy Technology* 62(1): 93–99
- Karagözli C, Ayhan E (2019):** Farklı Oranlarda Keçi Sütü İle Üretilmiş Probiyotik Dondurmaların Kalite Özellikleri Üzerine Bir Araştırma. *Ege Üniversitesi Ziraat Fakültesi Dergisi* 56(1): 131–140

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- Kaya S, Tekin AR (2001):** The effect of salep content on the rheological characteristics of a typical ice-cream mix. *Journal of Food Engineering* 47(1): 59–62
- Kimik Ö, Kesenkaş H, Ergönül PG, Akan E (2017):** The effect of using pro and prebiotics on the aromatic compounds, textural and sensorial properties of symbiotic goat cheese. *Mljekarstvo* 67(1): 71–85
- Kurt A, Atalar I (2018):** Effects of quince seed on the rheological, structural and sensory characteristics of ice cream. *Food Hydrocolloids* 82: 186–195
- Kurt A, Cengiz A, Kahyaoglu T (2016):** The effect of gum tragacanth on the rheological properties of salep based ice cream mix. *Carbohydrate Polymers* 143: 116–123
- McLellan MR, Lind LR, Kime RW (1995):** Hue angle determinations and statistical analysis for multi-quadrant Hunter L,a,b Data. *Journal of Food Quality* 18(3): 235–240
- Meilgaard, MC, Civille GV, Carr BT (2016):** *Sensory Evaluation Techniques*. CRC Press, New York, USA
- Metin M (2012):** Süt ve Mamülleri Analiz Yöntemleri. Ege Üniversitesi Basımevi, Bornova, İzmir, Turkey
- Moenfarid M, Tehrani MM (2008):** Effects of some stabilizers on physicochemical and sensory properties of frozen yogurt. *American-Eurasian J Agric & Environ Sci* 4(5): 584–589
- Nadelman P, Frazão JV, Vieira TI, Balthazar CF, Andrade MM, Alexandria AK, Cruz AG, Fonseca-Gonçalves A, Maia, LC (2017):** The performance of probiotic fermented sheep milk and ice cream sheep milk in inhibiting enamel mineral loss. *Food Research International* 97: 184–190
- OECD, FAO (2020):** Dairy and dairy products – OECD-FAO Agricultural Outlook 2020–2029. Rome/OECD Publishing, Paris, France
- Ozdemir C, Arslaner A, Ozdemir S, Allahyari M (2015):** The production of ice cream using stevia as a sweetener. *Journal of Food Science and Technology* 52(11): 7545–7548
- Ozer BH, Bell AE, Grandison AS, Robinson RK (1998):** Rheological properties of concentrated yoghurt (labneh). *Journal of Texture Studies* 29(1): 67–79
- Palencia G, Ibargoitia ML, Fresno M, Sopelana P, Guillén MD (2014):** Complexity and uniqueness of the aromatic profile of smoked and unsmoked herreño cheese. *Molecules* 19(6): 7937–7958
- Pandya AJ, Ghodke KM (2007):** Goat and sheep milk products other than cheeses and yoghurt. *Small Ruminant Research* 68(1–2): 193–206
- Pang B, Wang S, Chen W, Hassan M, Lu H (2020):** Effects of flow behavior index and consistency coefficient on hydrodynamics of power-law fluids and particles in fluidized beds. *Powder Technology* 366: 249–260
- Pankiewicz U, Góral M, Kozłowicz K, Góral D (2020):** Application of pulsed electric field in production of ice cream enriched with probiotic bacteria (*L. rhamnosus* B 442) containing intracellular calcium ions. *Journal of Food Engineering* 275: 109876
- Pathare PB, Opara UL, Al-Said, FJ (2013):** Colour Measurement and Analysis in Fresh and Processed Foods: A Review. *Food and Bioprocess Technology* 6(1): 36–60
- Robins A, Radha K, Sathian CT, Geetha R, Beena AK (2019):** Development of low calorie goat milk ice cream by using stevia leaf powder. *The Pharma Innovation Journal* 8(1): 296–299
- Sabia E, Gauly M, Napolitano F, Cifuni GE, Claps S (2020):** The effect of different dietary treatments on volatile organic compounds and aromatic characteristics of buffalo Mozzarella cheese. *International Journal of Dairy Technology* 73(3): 594–603
- Sattin E, Andreani NA, Carraro L, Lucchini R, Fasolato L, Telfatin A, Balzan S, Novelli E, Simionati B, Cardazzo B (2016):** A multi-omics approach to evaluate the quality of milk whey used in ricotta cheese production. *Frontiers in Microbiology* 7(AUG): 1–13
- Schoina V, Terpou A, Papadaki A, Bosnea L, Kopsahelis N, Kanelaki M (2019):** Enhanced aromatic profile and functionality of cheese whey beverages by incorporation of probiotic cells immobilized on pistacia terebinthus resin. *Foods* 9(1): 1–16
- Senaka Ranadheera C, Evans CAA, Adams MCC, Baines SKK (2013):** Production of probiotic ice cream from goat's milk and effect of packaging materials on product quality. *Small Ruminant Research* 112(1–3): 174–180
- Silva PDL, Bezerra ME, Santos KMO, Correia RTP (2015):** Potentially probiotic ice cream from goat's milk: Characterization and cell viability during processing, storage and simulated gastrointestinal conditions. *LWT – Food Science and Technology* 62(1): 452–457
- Sofjan RP, Hartel RW (2004):** Effects of overrun on structural and physical characteristics of ice cream. *International Dairy Journal* 14(3): 255–262
- Steffe JF (1967):** *Rheological Methods in Food Process Engineering*. Freeman Press, MI, USA
- Sun-Waterhouse D, Edmonds L, Wadhwa SS, Wibisono R (2013):** Producing ice cream using a substantial amount of juice from kiwifruit with green, gold or red flesh. *Food Research International* 50(2): 647–656
- Sun J (2007):** D-Limonene: Safety and Clinical Applications. *Alternative Medicine Review* 12(3): 259–264
- Tekinşen KK (2010):** Lezzetin Doruğu Maraş Dondurması. *TSE Standart* 579: 57–63
- Tester RF, Al-Ghazzewi FH (2013):** Mannans and health, with a special focus on glucomannans. *Food Research International* 50(1): 384–391
- Tester RF, Al-Ghazzewi FH (2016):** Beneficial health characteristics of native and hydrolysed konjac (*Amorphophallus konjac*) glucomannan. *Journal of the Science of Food and Agriculture* 96(10): 3283–3291
- Tian H, Shi Y, Zhang Y, Yu H, Mu H, Chen C (2019):** Screening of aroma-producing lactic acid bacteria and their application in improving the aromatic profile of yogurt. *Journal of Food Biochemistry* 43(10): 1–13
- Togay SO, Guneser O, Yuceer YK (2017):** Evaluation of physicochemical, microbiological, sensory properties and aroma profiles of goat cheeses provided from Canakkale. *International Journal of Dairy Technology* 70(4): 514–525
- Vazquez-Landaverde PA, Velazquez G, Torres JA, Qian MC (2005):** Quantitative Determination of Thermally Derived Off-Flavor Compounds in Milk Using Solid-Phase Microextraction and Gas Chromatography. *Journal of Dairy Science* 88(11): 3764–3772
- Yanglar F (2015):** Effects of green banana flour on the physical, chemical and sensory properties of ice cream. *Food Technology and Biotechnology* 53(3): 315–323
- Yaşar K, Kahyaoglu T, Şahan N (2009):** Dynamic rheological characterization of salep glucomannan/galactomannan-based milk beverages. *Food Hydrocolloids* 23(5): 1305–1311

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