

DETERMINATION OF SOME QUALITY CHARACTERISTICS OF DULCE DE LECHE'S PRODUCED INDUSTRIALLY IN TURKEY

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ABSTRACT

In this study, nine different Dulce de leche (DL) samples produced by industrial methods were investigated for their physicochemical, textural, sensory properties, total phenolic content (TPC) and antioxidant activities. In addition, macro and micro element (Ca, K, Mg, Na, Fe, Mn, Zn, Cu) contents of the samples were determined. Dry matter, fat, protein, acidity, pH, ash and brix values of the samples varied between 48.14–77.21%, 1.45–12.35%, 0.10–9.04%, 0.41–0.65%, 5.66–6.65%, 1.15–3.18% and 59.65–79.35%, respectively. HMF was determined as 9.98–25.06 µmol/100 g, total phenolic content as 810.00–2830.83 mg GAE/kg, antioxidant activity as 12.73–71.95% in the DPPH, and 4.50–7.91 mmol TE/g in the TEAC test. In the mineral analysis, the concentration of Ca and K elements was found to be high, and a wide variation was observed in color and textural properties. Sensory evaluations showed that DL formulation and production differences significantly affected product properties.

Keywords: Dulce de leche, quality characteristics, nutritional values

TÜRKİYE'DE ENDÜSTRİYEL OLARAK ÜRETİLEN SÜT REÇELLERİNİN BAZI KALİTE ÖZELLİKLERİNİN BELİRLENMESİ

ÖZ

Bu çalışmada, endüstriyel yöntemlerle üretilen dokuz farklı süt reçeli (Dulce de leche) örneğinin fizikokimyasal, tekstürel, duyuşal özellikleri ile toplam fenolik madde (TFM) ve antioksidan aktiviteleri incelenmiştir. Ayrıca örneklerin makro ve mikro element (Ca, K, Mg, Na, Fe, Mn, Zn, Cu) içerikleri belirlenmiştir. Numunelerin kuru madde, yağ, protein, asitlik, pH, kül ve briks değerleri sırasıyla %48.14–77.21, %1.45–12.35, %0.10–9.04, %0.41–0.65, 5.66–6.65, %1.15–3.18 ve 59.65–79.35 arasında değişmiştir. HMF 9.98–25.06 µmol/100 g, toplam fenolik madde 810.00–2830.83 mg GAE/kg, antioksidan aktivite DPPH testinde %12.73–71.95, TEAK testinde 4.50–7.91 mmol TE/g olarak belirlenmiştir. Mineral analizinde Ca ve K elementlerinin konsantrasyonu yüksek bulunmuş olup, renk ve tekstürel özellikleri arasında geniş varyasyon gözlenmiştir. Duyusal değerlendirmeler,

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reçel formülasyonunun ve üretim farklılıklarının ürün özelliklerini önemli ölçüde etkilediğini göstermiştir.

Anahtar kelimeler: Süt reçeli, kalite özellikleri, besin değerleri

INTRODUCTION

Dulce de leche (DL) is a dairy product consumed as a dessert and used in sweet recipes. It is widely consumed in Latin America, particularly in Brazil, Uruguay, and Argentina, with lesser consumption in Paraguay, Bolivia, and Chile (Gaze et al., 2015). Also, it has been exported to the United States and the European Union (Ranalli et al., 2017). In recent years, DL products have started to be on the market, increasingly, with the name of “milk jam” in Turkey (Yuksel Onur, 2018).

In Turkey, DL is particularly consumed as a spread, dessert, and at breakfast, and is also used in the manufacture of confectionery and ice cream. The production of DL involves concentrating a mixture of milk and sucrose by boiling it at atmospheric pressure until it reaches a solids content of approximately 70%. Sometimes, sucrose is partially replaced with glucose to prevent crystallization. The maillard browning reaction, which occurs between sugars and milk proteins, is a key process in DL production (Malec et al., 1999). Sodium bicarbonate (NaHCO_3) is added during production to prevent casein coagulation and to promote the Maillard reaction, which is responsible for DL's characteristic brown color and flavor (Yuksel Onur, 2018).

While DL consists of milk, sucrose, sodium bicarbonate, and various additives, its characteristics can significantly differ across industries and production regions. Factors such as cow management, genetics, good agricultural practices, formulation, and final processing play a crucial role in determining these characteristics (Smit, 2000). Different processing parameters including heating time and temperature, intensity of negative pressure, and mass balance directly impact the physicochemical and sensory properties of the final product (Perrone et al., 2011). These characteristics are closely linked to the nutritional, technological, and sensory quality of DL, which in turn influences consumer purchasing decisions, opens new market

opportunities, reduces production costs, and enhances quality control (Giménez et al., 2008). Therefore, the quality characteristics and nutritional values of DL must be consistently monitored.

The aim of this study was to determine the quality characteristics and nutritional values of industrially produced DL in Turkey. By analysing the physicochemical and sensory characteristics of DL, we aim to provide the necessary data to understand the impact of this product on consumer acceptability and to improve quality control processes. In addition, the results are expected to contribute to the improvement of DL production standards in Turkey and increase its competitiveness in local and international markets.

MATERIALS AND METHODS

Materials

Nine DL samples belonging to different brands sold in grocery stores in Istanbul in 2022 were obtained as research material. These samples were stored in their original glass packages at 4 ± 1 °C during the analyses. The ingredient information and codes given on the labels of the milk jams are shown in Table 1.

Methods

Physicochemical Analyses

The dry matter, protein, fat, ash and Brix was determined according to the recommendations of the Association of Official Analytical Chemists (AOAC, 2005). The pH and the percentage of lactic acid were determined according to Adolfo Lutz Institute methodology (Instituto Adolfo Lutz, IAL, 2008).

Hunter L^* , a^* and b^* values of milk jam samples were determined by Konica Minolta Chroma Meter CR-400 (Osaka, Japan) colour meter. L^* value shows 100 lightness /0 darkness, a^* value shows +redness/greenness and b^* value shows +yellowness/blueness (Elgün, 2002).

Table 1. Contents and sample numbers of industrially produced milk jam samples

| Sample No | Content of DL Samples |
|-----------|---|
| 1. | Milk, sugar and lemon juice |
| 2. | Sheep milk, sugar, baking soda |
| 3. | Whole cow's milk, sugar, sodium carbonate, and flavour (vanilla) |
| 4. | Milk, sugar, natural fruit pectin |
| 5. | Sweetened condensed milk, glucose syrup, sugar, water, and acidity regulator: sodium citrates, salt, gelling agent (fruit pectin) |
| 6. | Cow's milk, beetroot sugar, baking soda and vanilla |
| 7. | Pasteurised cow's milk, sugar, baking soda, vanilla |
| 8. | Goat's milk, beetroot sugar, food soda (baking soda) |
| 9. | Pasteurised milk, beet sugar, milk powder, baking soda |

HMF Analyses

Spectrophotometric HMF analysis as determined by Mistry and Pulgar (1996) method was made by modification. 10 g of DL samples were weighed and completed to 100 ml with pure water and homogenized in ultraturax at 12500 rpm for 1 minute. 10 ml of the diluted sample was taken into a test tube and 5 ml of 0.3 N oxalic acid was added and mixed, then kept in a water bath at 100°C for 1 hour. The tubes removed from the water bath were cooled to 25°C. Then 5 ml of 40% TCA solution was added and mixed in a vortex device, then filtered with Whatman 42 filter papers. 4 ml of the filtrate was taken into another tube and 1 ml of 0.05 M TBA solution was added, mixed and kept in a water bath at 40°C for 35 minutes. Then the tubes were cooled to 25°C. Absorption values of samples were measured at 443 nm wavelength in the spectrophotometer. HMF values were calculated according to the formula.

HMF in milk jam ($\mu\text{mol/l}$) = $(\text{Absorbance} - 0.055) \times 87.5$

Textural Analyses

Texture profile analyses of DL samples were performed using a texture analyzer (Texture Technologies Corp., Hamilton, MA, USA) equipped with a 5 kg load cell, cylindrical probe (25.4 mm diameter) and software program (Exponent, Version 6.0.6.0., Texture Technologies Corp.). Before analysis, samples were kept at room temperature. Texture profile analysis was performed by compressing twice with a cylindrical probe at a speed of 5 mm/s to provide 10 mm penetration. Texture parameters were measured as hardness, springiness,

resilience, chewiness, external adhesiveness, gumminess and internal adhesiveness (Erim-Köse et al., 2018).

Sensory Analyses

The sensory analysis panel form adapted by Cebeci (2020) from (TSE, 1982) for DL was used. Sensory analyses were carried out by fifteen panellists and the samples were scored in the range of 1-5.

Mineral Analyses

The dry incineration method specified in TS 3606 was used for the analysis of mineral substances (Anonymous, 1995). The milk jam sample was weighed into a porcelain crucible and dried in an oven and then subjected to incineration at a temperature of 500-550 °C in a gradually increasing muffle furnace. The ash obtained as a result of the incineration process was dissolved with nitric acid solution and then quantitatively transferred to 100 ml plastic filters with 1 N nitric acid solution. Appropriate dilutions were made from the stock solutions prepared in this way and analysed samples were prepared. Na, Ca, Mg, K, Zn, Fe, Cu and Mn concentrations of the samples were determined by using ICP-OES device at Van Yuzuncu Yil University Science Research and Application Centre.

Preparation of Water Soluble Extracts for Determination of Total Phenolic Substance and Antioxidant Activity

The samples were diluted 1:1 (w/v) with 95% ethanol and homogenised in a homogeniser for 1 min. They were mixed in an ultrasonic water bath

for 10 min and in a mechanical shaker for 15 min at room temperature. After centrifugation in a refrigerated centrifuge (NÜVE NF 1200 R) at 8500 rpm for 15 min at 4°C, the clear part remaining on the tubes was collected and stored in a cooler until analysis (Selcuk and Yilmaz, 2009).

Total Phenolic Substance

Determination of total phenolic content according to Folin & Ciocalteu's method was performed. For this purpose, 150 µL of sample and 3 mL of 2% Na₂CO₃ solution were added to the test tubes. After approximately 2 min. 150 µL of Folin-Ciocalteu's marker diluted 1:1 with ultrapure water was added to the tubes. The tubes were mixed in a vortex device and kept at room temperature and in a dark place for 45 min. The absorbance values were then read at 765 nm in a spectrophotometer (UV Mini-1240, Shimadzu, Japan). The total phenolic content of the samples was calculated from the calibration graph with gallic acid and expressed as gallic acid equivalent (Bae and Suh, 2007).

Antioxidant Activity

DPPH Test

DPPH radical scavenging power was determined according to the method of Brand-Williams et al. (1995). DPPH solution (25 mg DPPH/L methanol) was prepared daily and the absorbance of the solution was diluted to 0.700 ± 0.020 at 520 nm. 100 µL of the extracts of the obtained DL samples were placed in tubes and 2.4 mL of DPPH solution was added. After 30 min in the dark, the absorbance values of the samples were analysed in a spectrophotometer (UV Mini-1240, Shimadzu, Japan) at 520 nm wavelength. The % inhibition rate of DPPH radical was calculated according to the formula.

$$\text{Inhibition \%} = (\text{Control absorbance} - \text{sample absorbance}) / \text{Control absorbance} \times 100$$

TEAC Test

In order to determine the TEAC values of the samples, 7 mM ABTS+ radical solution containing 2.45 mM potassium persulfate was first prepared. This solution was kept at room temperature and in a dark environment for 12-16

hours. At the end of the waiting time, the radical solution was diluted with 80% ethanol to give an absorbance of 0.700 ± 0.2 at 734 nm in the spectrophotometer. 2.9 mL of the diluted radical solution was added to the test tube. 100 µL of the DL sample extract was added. This mixture was mixed rapidly on a vortex for 6 minutes and read at 734 nm on a spectrophotometer (UV Mini-1240, Shimadzu, Japan). The results using the Trolox standard curve calculated according to the formula (Kirca and Ozkan, 2007).

$$\text{TEAC (mM Trolox/g sample)} = \text{Slope of the inhibition curve of the sample} / \text{Trolox standard curve} \times \text{Dilution Factor}$$

Statistical Analyses

In the statistical analysis using IBM SPSS Statistic 20 package programme, the differences between the results obtained in the study were subjected to Duncan multiple comparison test according to 95% significance level.

RESULTS AND DISCUSSIONS

As seen in Table 2, the chemical analysis results of the DL samples varied within a wide range. Yuksel-Onur (2018) determined that the chemical composition of 6 DL's obtained from the market varied within a similarly wide range. It is thought that the biggest reason for these differences is the lack of legal regulation on DL in Turkey.

In the present study, the lowest L* value was determined in sample number 4 (28.71) and the highest in sample number 2 (41.40). The parameter a* ranged from 4.28 to 11.28 in the samples 5 and 1, respectively. The samples 6 and 1 had the lowest (-1.28) and the highest (18.02) b* values, respectively. The obtained data (Table 3) were lower than the L*, a* and b* values of seven milk jams obtained from the Brazilian market by Gaze et al. (2015). The differences between the samples in terms of colour parameters are probably due to differences in protein and sugar composition, as well as changes in time, temperature and pressure, according to the protocol of each industry (Oliveira et al., 2009).

Table 2. Chemical analysis results of DL samples

| Sample no | Drymatter (%) | Ash (%) | Fat (%) | Protein (%) | pH | Acidity (%) | Brix |
|-----------|--------------------------|-------------------------|-------------------------|------------------------|-------------------------|-------------------------|-------------------------|
| 1 | 65.17±0.11 ^{CD} | 1.15±0.03 ^A | 4.45±0.07 ^D | 4.20±0.01 ^D | 6.35±0.07 ^D | 0.43±0.01 ^A | 67.45±0.07 ^C |
| 2 | 65.91±0.27 ^{CD} | 1.85±0.14 ^C | 12.35±0.07 ^H | 9.04±0.06 ^G | 6.24±0.03 ^C | 0.55±0.01 ^{BC} | 66.45±0.01 ^B |
| 3 | 48.14±1.83 ^A | 2.76±0.21 ^D | 1.45±0.07 ^A | 0.10±0.03 ^A | 5.98±0.04 ^B | 0.64±0.01 ^D | 69.75±0.07 ^F |
| 4 | 64.63±0.45 ^{CD} | 3.18±0.02 ^E | 3.15±0.07 ^B | 2.90±0.14 ^B | 6.28±0.04 ^{CD} | 0.65±0.01 ^D | 68.55±0.03 ^D |
| 5 | 71.35±1.02 ^{DE} | 1.47±0.02 ^B | 3.75±0.07 ^C | 3.40±0.14 ^C | 5.95±0.02 ^B | 0.41±0.01 ^A | 71.85±0.07 ^H |
| 6 | 67.10±1.65 ^{CD} | 1.98±0.01 ^C | 7.25±0.07 ^F | 6.46±0.08 ^F | 5.70±0.01 ^A | 0.57±0.01 ^C | 69.05±0.01 ^E |
| 7 | 51.74±1.2 ^{AB} | 1.76±0.04 ^C | 8.15±0.07 ^G | 6.39±0.01 ^F | 5.66±0.03 ^A | 0.52±0.02 ^B | 70.05±0.07 ^G |
| 8 | 58.34±1.21 ^{BC} | 1.69±0.24 ^{BC} | 7.20±0.14 ^F | 6.44±0.06 ^F | 6.23±0.01 ^C | 0.44±0.01 ^A | 59.65±0.07 ^A |
| 9 | 77.21±1.03 ^E | 1.69±0.04 ^{BC} | 6.05±0.07 ^E | 5.80±0.42 ^E | 6.65±0.07 ^E | 0.42±0.03 ^A | 79.35±0.06 ^I |

A,B,C,D,E,F,G,H,I Indicates the difference ($p < 0.05$) between samples.

Table 3. Color values of DL samples

| Sample no | L* | a* | b* |
|-----------|-------------------------|-------------------------|--------------------------|
| 1 | 37.66±0.85 ^E | 11.28±0.54 ^G | 18.02±1.01 ^F |
| 2 | 41.40±0.57 ^F | 9.68±0.40 ^E | 16.52±0.25 ^E |
| 3 | 33.24±0.95 ^C | 14.39±0.21 ^H | 11.42±1.58 ^{CD} |
| 4 | 28.71±0.28 ^A | 5.56±0.03 ^C | 6.20±0.06 ^B |
| 5 | 31.04±1.29 ^B | 4.28±0.13 ^A | 6.46±0.13 ^B |
| 6 | 29.45±0.25 ^A | 4.43±0.01 ^{AB} | -1.28±0.11 ^A |
| 7 | 32.70±0.09 ^C | 10.55±0.01 ^F | 10.14±0.37 ^C |
| 8 | 35.74±0.06 ^D | 6.84±0.01 ^D | 12.52±0.04 ^D |
| 9 | 35.69±0.14 ^D | 4.88±0.02 ^B | 12.15±0.06 ^D |

A,B,C,D,E,F shows the difference ($p < 0.05$) between samples.

The Maillard reaction, known as a non-enzymatic browning reaction in foods, is based on the interaction of protein and sugar with heat. The Maillard reaction, which is not desired to occur in drinking milk, is desired for the formation of characteristic color and aroma in DL. HMF is an intermediate product formed during the Maillard reaction and shows the intensity of the applied heat treatment (Akal et al., 2018). HMF values were found to be 9.98-25.06 $\mu\text{mol/l}$ (Table 4).

The differences between HMF values of samples were found statistically different ($p < 0.05$). It is thought that different production methods of the producers and different heat treatment-duration combination applications and the composition of the milk used cause the difference in HMF values. It is also thought that storage conditions, milk type and ratio, sugar amount and sodium bicarbonate amount may also cause the difference in HMF values.

Table 4. HMF values of DL samples

| Sample no | HMF ($\mu\text{mol/l}$) |
|-----------|---------------------------|
| 1 | 14.44±0.74 ^{CDE} |
| 2 | 9.98±0.12 ^A |
| 3 | 23.63±1.86 ^F |
| 4 | 10.29±3.53 ^{AB} |
| 5 | 17.07±1.11 ^E |
| 6 | 25.06±0.04 ^F |
| 7 | 16.66±0.04 ^{DE} |
| 8 | 11.82±0.12 ^{ABC} |
| 9 | 13.52±1.17 ^{BCD} |

A,B,C,D,E,F shows the difference ($p < 0.05$) between samples

The hardness of the DL samples varied between 15.78 and 19.00 g, the adhesiveness between -2662.78 and -253.45 g.sec, the resilience between 1.87 and 50.85%, the cohesiveness between 0.81 and 1.03, the springiness between 46 and 95.33%, the gumminess between 14.02 and 18.45 and the chewiness between 7.97 and 16.85 (Table 5). In order for DL to be spreadable and used as a filler, it is neither too soft nor too viscous. Therefore, the product should not show high gumminess, adhesiveness and hardness values (Silva et al., 2015). Bruno et al. (2018) evaluated the textural properties of six different brands of milk jam and reported that the hardness value of the samples was between 106.39 and 145.66, the adhesiveness value was between -416.07 and -201.37, the cohesiveness value was between 0.667 and 0.730, the gumminess value was between 71.02 and 102.14 and the chewiness value was between 64.03 and 86.14. Chacon-Villalobos et al. (2013) observed that hardness, adhesiveness and

springiness values increased as the proportion of cow's milk in the mixture increased in their study on DL samples prepared with different milk combinations of cow's milk and goat's milk. Similar to the findings of this study, the hardness, adhesiveness and springiness values of the sample number eight made from goat's milk in our study were found to be lower than the other samples. Lima et al. (2020) found that the gumminess value of milk jam samples obtained from different ratios of cow's milk and sheep's milk mixtures was between 0.16 and 0.24 and stated that DL samples with higher sheep's milk ratio had higher gumminess and springiness values because sheep's milk contains more lipids than cow's milk. The data obtained in our study were similar to this study and it was observed that the gumminess and springiness values of the sample number two produced from sheep milk were higher than the other samples.

Table 5. Textural profile analysis results of DL samples

| Sample no | Hardness (g) | Adhesiveness (g.s) | Resilience (%) | Cohesiveness | Springiness (%) | Gumminess | Chewiness |
|-----------|-------------------------|------------------------------|--------------------------|-------------------------|--------------------------|--------------------------|---------------------------|
| 1 | 19.00±3.62 ^A | -336.55±147.79 ^{DE} | 24.26±4.99 ^C | 0.93±0.01 ^{BC} | 95.33±6.75 ^E | 17.55±3.52 ^{AB} | 16.85±4.55 ^D |
| 2 | 18.11±0.16 ^A | -870.98±59.35 ^B | 16.57±1.79 ^B | 0.98±0.01 ^{CD} | 86.06±1.91 ^D | 17.71±0.38 ^{AB} | 15.24±0.01 ^{CD} |
| 3 | 18.22±1.57 ^A | -366.88±6.39 ^{DE} | 41.50±0.32 ^E | 0.93±0.01 ^{BC} | 46.98±1.63 ^A | 16.99±1.75 ^{AB} | 7.97±0.54 ^A |
| 4 | 17.17±0.23 ^A | -2662.78±18.07 ^A | 1.87±0.09 ^A | 0.81±0.01 ^A | 70.67±0.07 ^{BC} | 14.27±0.03 ^A | 10.11±0.01 ^{AB} |
| 5 | 18.56±0.47 ^A | -340.32±31.71 ^{DE} | 50.85±2.79 ^F | 0.89±0.06 ^B | 79.36±0.78 ^{CD} | 16.56±1.51 ^{AB} | 13.15±1.32 ^{BCD} |
| 6 | 16.00±0.31 ^A | -510.06±19.62 ^C | 21.83±1.12 ^C | 1.03±0.02 ^D | 72.39±1.66 ^{BC} | 16.40±0.69 ^{AB} | 11.88±0.78 ^{ABC} |
| 7 | 18.45±0.32 ^A | -472.30±14.7 ^{CD} | 21.22±0.49 ^{BC} | 0.91±0.05 ^B | 81.59±7.04 ^D | 16.65±1.26 ^{AB} | 13.54±0.15 ^{BCD} |
| 8 | 15.78±0.31 ^A | -303.86±24.85 ^E | 31.49±1.29 ^D | 0.89±0.01 ^B | 69.54±3.63 ^B | 14.02±0.03 ^A | 9.76±0.53 ^{AB} |
| 9 | 18.89±2.20 ^A | -253.45±26.13 ^E | 23.30±1.93 ^C | 0.98±0.01 ^{CD} | 78.61±1.41 ^{CD} | 18.45±2.04 ^B | 14.49±1.34 ^{CD} |

A,B,C,D,E Indicates the difference between samples ($p < 0.05$).

Although DL is a semi-solid product, its gel-like texture justifies the inclusion of additional parameters such as chewiness and springiness in the texture profile analysis. These parameters are widely used in gel-structured semi-solid foods such as yogurt, milk-based desserts, and custards. In our study, chewiness and springiness varied meaningfully among samples and provided valuable information about the mechanical structure and elasticity of DL, which may influence consumer perception and the product's applicability as a spread or filling.

Texture is one of the most important quality characteristics in DL because it defines the

characteristics of the product in subsequent applications, especially for doughy products. Texture defects can negatively affect consumer acceptance, which is generally caused by poor production (Ranalli et al., 2012).

When the samples were examined in terms of sensory analysis (Table 6), they received scores between 3.65 and 4.95 in terms of color, with sample number one receiving the highest score and sample number eight receiving the lowest score. The odour of the DL samples was scored between 3.20 and 4.60 and the highest score was obtained by sample number one and three and the lowest score was obtained by sample number six.

The consistency of the DL's was scored between 3.20 and 4.61 and the highest score was obtained by sample number 2 and the lowest score was obtained by sample number 1. In terms of taste characteristics, milk jams scored between 2.40 and 4.85 and the highest score was given to sample

number 2 and the lowest score was given to sample number 9. According to the results of sensory analysis, the different materials used in jam making caused different results to be obtained among the samples.

Table 6. Sensory properties of DL samples

| Sample no | Colour | Odour | Consistency | Taste/Aroma |
|-----------|-------------------------|-------------------------|-------------------------|------------------------|
| 1 | 4.94±0.06 ^D | 4.60±0.07 ^E | 3.20±0.16 ^A | 4.60±0.14 ^H |
| 2 | 4.40±0.14 ^B | 3.60±0.14 ^{BC} | 4.61±0.14 ^D | 4.85±0.04 ^I |
| 3 | 4.40±0.03 ^B | 4.60±0.06 ^E | 4.60±0.07 ^D | 2.80±0.03 ^B |
| 4 | 4.60±0.07 ^{BC} | 3.80±0.17 ^C | 3.80±0.14 ^{BC} | 3.40±0.07 ^D |
| 5 | 4.80±0.14 ^{CD} | 3.40±0.14 ^{AB} | 3.60±0.07 ^B | 3.80±0.06 ^E |
| 6 | 4.95±0.5 ^D | 3.20±0.21 ^A | 4.40±0.14 ^D | 4.00±0.07 ^F |
| 7 | 4.00±0.1 ^A | 4.20±0.14 ^D | 3.65±0.17 ^B | 4.40±0.1 ^G |
| 8 | 3.65±0.14 ^A | 3.80±0.08 ^C | 4.00±0.06 ^C | 3.00±0.07 ^C |
| 9 | 4.00±0.11 ^A | 3.60±0.11 ^{BC} | 3.60±0.07 ^B | 2.40±0.03 ^A |

A,B,C,D,E,F,G,H,I Indicates the difference between samples ($p < 0.05$).

Cebeci (2020) examined the sensory properties of milk jam samples produced using different milk combinations and found that the taste and odour appreciation of the samples increased as the proportion of cow's milk in the mixture increased. It was found that the consistency properties increased with increasing cow milk ratio in cow milk and sheep milk combinations and decreased with increasing cow milk ratio in goat milk combinations. Chacón-Villalobos et al. (2013) reported that the general liking decreased as the goat milk ratio increased in milk jam samples prepared with different combinations of cow milk and goat milk. When we compared the obtained data with the studies in the literature, contrary to the findings of Cebeci (2020), it was observed that the taste characteristics of sample number two containing sheep milk received higher scores compared to the other samples, similar to the study of Chacón-Villalobos et al. (2013), and the taste appreciation of sample number eight made from goat milk was lower than the other samples.

When the sensory results were interpreted in conjunction with instrumental data, a partial relationship was observed between consumer preferences and key textural properties. Sample 2, which received the highest scores in taste and consistency, also exhibited relatively high

cohesiveness (0.98) and springiness (86.06%), along with moderate hardness (18.11 g) and adhesiveness (-870.98 g.s) values. These characteristics may have contributed to its pleasant mouthfeel and ease of spreadability. On the other hand, Sample 9, which received the lowest score in taste (2.40), had the highest gumminess (18.45) and one of the highest hardness values (18.89 g). Interestingly, it also showed a high cohesiveness value (0.98), indicating that despite some favorable texture traits, excessive hardness and gumminess may have negatively affected consumer acceptance. These findings suggest that certain instrumental texture parameters—particularly gumminess and hardness—are aligned with sensory perceptions and may influence the acceptability of dulce de leche samples.

When the mineral contents of the DL samples were compared, it was observed that the amount of potassium (K) and calcium (Ca) contained in the samples were higher than the other minerals. It was determined that Ca content of DL samples varied between 839.47 and 3286.16 mg/kg, Mg content between 106.70 and 318.58 mg/kg, K content between 821.59 and 3499.90 mg/kg, Na content between 488.76 and 2399.82 mg/kg, Fe content between 27.52 and 75.90 mg/kg, Cu

content between 3.64 and 12.86 mg/kg, Mn content between 0.98 and 3.27 mg/kg, Zn content between 69.98 and 319.30 mg/kg (Table 7). As can be seen, the mineral content of DL showed statistical differences among the samples except for the Mn element. This situation is thought to be due to the addition of a technological food additive during the production

process rather than the difference in the mineral content of the milk affecting the raw material in different DL samples. It was determined that the highest concentration was in mineral Ca. However, Gaze et al. (2015) found that K had the highest concentration among the minerals examined in their study.

Table 7. Mineral content of DL samples (mg/kg)

| Sample no | Ca | Mg | K | Na | Fe | Cu | Mn | Zn |
|-----------|------------------------------|---------------------------|-----------------------------|------------------------------|---------------------------|--------------------------|------------------------|-----------------------------|
| 1 | 1431.97±83.12 ^B | 210.09±29.13 ^B | 1349.97±35.33 ^B | 912.24±182.02 ^B | 34.85±46.97 ^{AB} | 3.64±4.60 ^A | 0.98±1.12 ^A | 69.98±85.74 ^A |
| 2 | 2829.57±142.67 ^E | 299.74±5.16 ^{CD} | 1502.82±64 ^B | 1248.15±17.44 ^{BC} | 41.78±0.00 ^{ABC} | 12.86±4.03 ^B | 3.27±1.06 ^A | 271.72±35.84 ^{AB} |
| 3 | 3286.16±339.8 ^F | 318.58±28.53 ^D | 3222.05±355.93 ^D | 2259.51±334.08 ^D | 40.09±5.62 ^{ABC} | 10.32±1.82 ^{AB} | 1.38±0.21 ^A | 291.20±36.32 ^B |
| 4 | 2673.87±196.78 ^{DE} | 312.66±27.2 ^{CD} | 3499.90±140.95 ^D | 2399.82±70.43 ^D | 75.90±8.97 ^{BC} | 6.57±0.84 ^{AB} | 2.71±0.46 ^A | 275.26±125.67 ^{AB} |
| 5 | 839.47±174.49 ^A | 106.70±17.59 ^A | 821.59±90.76 ^A | 1488.96±91.32 ^C | 46.13±8.01 ^{ABC} | 5.51±1.92 ^A | 1.15±0.07 ^A | 244.54±29.18 ^{AB} |
| 6 | 1520.63±115.53 ^B | 209.83±74.9 ^B | 1507.17±39.73 ^B | 1349.70±368.03 ^{BC} | 81.06±12.49 ^C | 8.55±3.52 ^{AB} | 3.06±2.30 ^A | 162.44±34.63 ^{AB} |
| 7 | 932.05±142.49 ^A | 122.24±20.68 ^A | 1029.98±125.35 ^A | 488.76±73.71 ^A | 28.31±8.39 ^A | 4.37±0.81 ^A | 1.52±0.33 ^A | 187.66±20.77 ^{AB} |
| 8 | 2411.19±44.43 ^{CD} | 237.88±26.3 ^{BC} | 2206.66±24.47 ^C | 1531.69±17.88 ^C | 27.52±3.09 ^A | 8.51±1.34 ^{AB} | 1.69±1.12 ^A | 319.30±188.83 ^B |
| 9 | 2043.81±86.94 ^C | 210.50±1.22 ^B | 1923.27±6.94 ^C | 1291.27±56.54 ^{BC} | 37.74±3.45 ^{AB} | 7.27±4.15 ^{AB} | 2.53±2.28 ^A | 156.19±18.46 ^{AB} |

A,B,C,D,E,F Indicates the difference between samples ($p < 0.05$).

Total phenolic matter values of DL samples are given in Table 8. The lowest TPC value was 810 mg GAE/kg and the highest TPC value was 2830.83 mg GAE/kg. As can be seen in Table 8, the TPC values of the samples varied in a wide range. Tuna (2018) determined the amount of total phenolic matter in milk jams produced by enriching with plain and different fruits as 680 mg

GAE/kg on the first day of storage for plain milk jam and 480 mg GAE/kg on the 60th day of storage. It was determined that the data obtained were higher than the values found by Tuna (2018). It is thought that this may be due to the fact that Tuna (2018) used open boiler cooking method in his study.

Table 8. Total phenolic substance and antioxidant activity values of DL samples

| Sample no | TPC (mg GAE/kg) | DPPH (% Inhibition) | TEAC (mmol/g) |
|-----------|-------------------------------|-------------------------|------------------------|
| 1 | 810.00±117.85 ^A | 40.39±0.01 ^B | 4.50±1.37 ^A |
| 2 | 1941.25±103.12 ^{DE} | 40.91±4.05 ^B | 6.43±1.53 ^B |
| 3 | 2830.83±23.57 ^G | 41.47±3.26 ^B | 7.28±0.67 ^B |
| 4 | 2357.92±73.66 ^F | 54.61±1.09 ^C | 7.72±0.05 ^B |
| 5 | 1768.33±223.91 ^{CDE} | 12.73±5.34 ^A | 7.13±0.37 ^B |
| 6 | 2155.83±5.9 ^{EF} | 71.95±2.67 ^D | 7.84±0.06 ^B |
| 7 | 1437.08±32.41 ^{BC} | 43.62±0.02 ^B | 7.23±0.26 ^B |
| 8 | 1676.67±276.95 ^{CD} | 52.51±0.89 ^C | 7.91±0.00 ^B |
| 9 | 1268.33±329.98 ^B | 38.67±5.04 ^B | 7.34±0.37 ^B |

A,B,C,D,E,F Indicates the difference between samples ($p < 0.05$).

DPPH % inhibition values of DL samples are given in Table 8. The lowest DPPH inhibition value was 12.73 % and the highest DPPH inhibition value was 71.95 %.

The lowest TEAC value of DL samples was 4.50 mmol TE/g and the highest TEAC value was 7.91 mmol TE/g. Tuna (2018) determined the total

antioxidant activity in DL produced by enriching with plain and different fruits as 50 mmol TE/g on the first day of storage for plain DL and 80 mmol TE/g on the 60th day of storage. Tuna (2018) stated that the sample formulation affected the antioxidant activity and the antioxidant activity of DL produced from apricot, which is rich in carotene, was higher than plain DL. The

values obtained in our study were lower than the values in this study. It is thought that the difference between the TPC, DPPH and TEAC values of the samples is caused by the type and amount of milk used and the production method applied.

CONCLUSIONS

It has been observed that the type of milk used, the amount of milk, the additives and the production method cause differences in product features. Since there is no legal regulation in Turkey that sets the limit values of milk jam composition, the compliance of the examined samples with food legislation could not be evaluated. When the studies in the literature are examined, it has been determined that the studies on DL are limited and there is only one study on DL's produced industrially and offered for sale in Turkey. This study is a resource in terms of eliminating the deficiencies in the literature and will contribute to eliminating the lack of information in this field. However, more detailed and comprehensive studies on DL should be conducted and the standard production of this DL should be determined.

DECLARATION OF CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

AUTHOR CONTRIBUTIONS

Sibel ÖZTOK: investigation, data curation, writing – original draft; Şenol KÖSE: methodology, conceptualization, investigation, resources, supervision, project administration, funding acquisition, writing – review & editing; Yağmur ERİM KÖSE: investigation, data curation, writing – review & editing. All authors read and approved the final manuscript.

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