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ARAŞTIRMA MAKALESİ **RESEARCH ARTICLE**

Investigation of The Physicochemical Properties of Propolis Added Ice Creams During Storage*

Propolis İlaveli Dondurmaların Depolama Süresince Fizikokimyasal Özelliklerinin Belirlenmesi

Serdar MEHMETOĞLU[1], Zekai TARAKÇI[2]*

Abstract

The aim of this study is to add functional food feature to ice cream, which is a popular food, by enriching it with propolis. In addition, another aim of the study is to provide a widespread consumption potential through ice cream to propolis, which cannot be consumed raw and whose benefits and functional properties are unknown to most consumers. A mixture consisting of a total of 6 sample groups containing 0.0%, 0.1%, 0.2%, 0.3%, 0.4% and 0.5% propolis powder was prepared for the ice cream mix. Ice cream samples were prepared from these ice cream mixes. Different analyses were executed for propolis, ice cream mix and ice cream samples. While only antioxidant analysis was executed for propolis samples, Dry matter, pH, titration acidity analyzes were executed in ice cream mix samples. Volume increase index, texture analysis, melting rate, antioxidant activity and sensory analyzes in propolis added ice cream samples were carried out on different days during 2 months of storage. According to the findings, while the volume increase of the ice cream samples was not affected by the storage time, the difference between the propolis concentrations was found. It was observed that the first dripping times increased with storage, while the melting rate of the ice cream decreased. There was no significant change in the meltıng rate and first drip times depending on the propolis concentrations. The texture properties of ice cream samples have changed with the addition of propolis. The hardness and stickiness values of ice cream samples changed depending on the storage time. The addition of propolis significantly increased the antioxidant activity. Antioxidant activity was changed with the addition of propolis. The phenolic content and of ice cream did not change with storage, but FRAP value decreased slightly after 60 days of storage. Storage time had a limited effect on the physicochemical and sensory properties of ice cream. Although the addition of propolis negatively affected the physical and sensory properties of ice cream, it contributed significantly to the antioxidant activity even at the lowest concentrations. With this study, the potential of propolis-added ice cream as a functional new food for consumers of all ages has been demonstrated. In line with these results, new studies should be conducted by trying different propolis extracts and different concentrations, by revealing the functionality of propolis and adding it to new other foods.

Keywords: Antioxidant activity, Functional food, Ice cream, Propolis, Sensory, Texture.

[1]Serdar Mehmetoğlu, Apiculture Research Institute, Ordu, Turkey. E-mail: mehmetogluserdar@hotmail.com OrcID: 0000-0001-7787-0707.
[2]***Sorumlu Yazar/Corresponding Author:** Zekai Tarakçı, Ordu University, Faculty of Agriculture, Department of Food Engineering, Ordu, Turkey. E-mail: ztarakci@odu.edu.tr OrcID: 0000-0002-3828-3232.
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Öz

Bu çalışmanın amacı, propolis ile zenginleştirilerek popüler bir gıda olan dondurmaya fonksiyonel gıda özelliği kazandırmaktır. Ayrıca çalışmanın bir diğer amacı çiğ olarak tüketilmesi mümkün olmayan, faydaları ve fonksiyonel özellikleri çoğu tüketici tarafından bilinmeyen propolise dondurma yoluyla yaygın bir tüketim potansiyeli sağlamaktır. Dondurma miksine % 0.0, % 0.1, % 0.2, % 0.3, % 0.4 ve % 0.5 propolis tozu içeren toplam 6 adet örnek gruplarından oluşan miks hazırlanmıştır. Propolis örneğinde antioksidan analizi; dondurma misklerinde kuru madde, pH, titrasyon asitliği analizleri ile propolis katkılı dondurma örneklerinde hacim artış indeksi, tekstür analizi, erime oranı, antioksidan aktivite ve duyusal analizler 2 aylık depolama süresince farklı günlerde gerçekleştirilmiştir. Bulgulara göre, dondurmaların hacim artışını depolama süresi etkilemezken, dondurma çeşitleri arasında fark bulunmuştur. İlk damlama sürelerinde depolama ile artmış, dondurmaların erime oranlarında ise azalma gözlemlenmiştir. Propolis konsantrasyonlarına bağlı olarak erime oranı ve ilk damlama sürelerinde önemli değişiklik olmamıştır. Dondurmaların tekstür özellikleri propolis ilavesi ile değişmiştir. Dondurmanın sertlik ve yapışkanlık değerleri depolama süresine bağlı olarak değişmiştir. Propolis ilavesi antioksidan aktiviteyi önemli düzeyde arttırmıştır. Antioksidan aktivitede propolis ilavesi ile değişimler olmuştur. Dondurmanın fenolik madde miktarı değeri depolama ile değişmezken, FRAP değeri 60 günlük depolama sonrası bir miktar azalmıştır. Depolama süresinin dondurmanın fizikokimyasal ve duyusal özelliklerine sınırlı bir etkisi olmuştur. Propolis ilavesi, dondurmanın fiziksel ve duyusal özelliklerini olumsuz etkilemesine rağmen, en düşük konsantrasyonlarda bile, antioksidan aktiviteye önemli katkılar sağlamıştır. Bu çalışmayla, propolis katkılı dondurmanın her yaştan tüketici için fonksiyonel yeni bir gıda olarak potansiyeli ortaya konmuştur. Bu sonuçlar doğrultusunda farklı propolis ekstraktları ve bunların farklı konsantrasyonları gıdalar üzerinde denenerek yeni çalışmalar yapılmalı ve propolisin fonksiyonelliği araştırılmalıdır.

Anahtar kelimeler: Antioksidan aktivite, Fonksiyonel gıda, Dondurma, Duyusal, Propolis, Tekstür.

1. Introduction

Functional foods are foods that have beneficial effects on human health and nutritious properties. They are foods that help protect human health thanks to their bioactive components such as antioxidants, dietary fibers, probiotics, prebiotics, cholines, proteins, oligosaccharides, and phytochemicals (Yaşlı, 2010). Due to the relationship that consumers establish between food and health, not only the functional food market is becoming an increasingly growing industry but also studies on functional foods have increased in recent years (Aliyev, 2006; Yucel et al., 2017). Propolis is a functional food ingredient that has attracted a lot of attention in recent years. It is a natural resinous substance with a unique aromatic fragrance created by bees over collecting some sections of plants, plant buds, and plant secretions (Ghisalberti, 1979). It may have diverse colors depending on the source and level of maturity (Brown, 1989). Propolis has an overly complex chemical structure with more than 300 different compounds within. The chemical composition of propolis varies by factors such as environmental factors, climate, secretion source, (Cheng and Wong, 1996), and its approximate proximate composition is 50% resin, 30% wax, 10% essential oils, 5% pollen, and 5% other organic components. Flavanoids, phenolic compounds, esters, aromatic aldehydes, terpenes, sesquiterpenes, beta-steroids, alcohols, and caffeic acid phenyl ester (CAPE) are some of the organic compounds that propolis contains (Yucel et al., 2017). Propolis was found to contain a variety of vitamins (B_1, B_2, B_6, C, and E) and micro- and macro-elements (silver, cesium, antimony, mercury, calcium, copper, manganese, iron, aluminum, and vanadium) (Deblock-Bostyn, 1982). The main use of propolis by bees is to provide insulation inside the hive (Marcucci, 1995). In addition to the insulation function of propolis applied in layers of the hive, it is also used by bees for sealing the hive, repairing, and bonding the honeycomb, diminishing the access to the hive, fighting disease factors. Furthermore, propolis helps prevent microbial growth in the hive (Kumova, 2002) owing to its region and strain dependent antimicrobial activity (Apaydin and Gümüş, 2018). It was also applied as a coating material to enhance shelf life of food products due to its antimicrobial properties (Güler et al., 2022). Researchers utilized antimicrobial properties of propolis at different foodstuffs such as milk, meat and sausages and used propolis for enhancing antioxidative and pharmalogical properties of foods such as yoghurt, soups, and dairy beverages (Irigoiti et al., 2021).

Development of a new functional food by adding propolis to ice cream is aimed in this study. Providing a wıdespread consumption potential to propolis which cannot be consumed in raw form, by adding popular foodstuff ice cream was also aimed. In this context, propolis powder was prepared and included in the ice cream formulation at different concentrations, and its effect on the physicochemical, functional, textural, and sensory properties was determined for two months of storage.

2. Materials and Methods

2.1. Propolis powder preparation

For the preparation of propolis powder, raw propolis collected from the apiaries of the Apiculture Research Institute in Ordu, Turkey in the summer of 2018 was used. Crude propolis was shaken in an ethanol-water solution (70/30%) for the propolis pre-extraction process for 10 days. The propolis extract was obtained after the removal of the precipitate by passing the mixture through filter paper. The extract was kept at 4°C for one day and filtered again and the precipitate was again removed. A rotary evaporator system (Buchi R300) at the Apiculture Research Institute Food Technology Laboratory was used to remove ethanol from the extract. Aqueous propolis extract was kept in a freezer at -18°C until the lyophilization process, which was carried out at the Ordu University Central Research Laboratory. The propolis powder obtained (83.4 g of powder was obtained from 212.37 g of crude propolis) was stored at -18°C until its utilization for ice cream production. Total phenolic content and antioxidant activity of propolis powder were determined according to the method explained in the sections of 2.4.2 and 2.4.3, respectively.

2.2. Preparation of ice cream mix

Pasteurized cow milk (Ak Gıda, Sakarya) with 10.7% dry matter, 2.9% protein, and 3.1% fat contents and a pH value of 6.57 was used for producing ice cream mix. The company also provided cow milk cream (35% fat, 3% lactose, and 1.5% protein). Skimmed milk powder was obtained from the Pınar Company (İzmir, Turkey) and powdered beet sugar was from the Konya Sugar Company (Konya, Turkey). An emulsifier/stabilizer mix was obtained from MEC3 Company (İzmir, Turkey).

Ice cream mix (ICM) consisted of 63.07% milk, 16% sucrose, 15.17% cream, %5.17 skimmed milk powder, 0.3% emulsifier, and 0.3% stabilizer. For an efficient pasteurization process, all ingredients were stirred in milk and homogenized. Prior to ice cream production, ice cream mix was aged for 24 h, at 4°C. Propolis powder content of ICM samples were adjusted to 0%, 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% and their analyzes were performed following 24 h of aging.

2.3. Ice cream production

The six groups of propolis added ice cream were produced (Delonghi, ICK 5000) in two replicates. Ice cream samples (ICM) were analyzed on the 3rd, 30th, and 60th days of storage. Ice cream samples and ice cream formulations were grouped as in *Table 1*.

Table 1. The trial pattern of ice cream mix and ice cream samples

Ice cream mix	Application level	Storage time (days)		
		3	30	60
Control	Mix group without propolis			
ICM1	Mix group with 0.1% propolis added			
ICM2	Mix group with 0.2% propolis added			
ICM3	Mix group with 0.3% propolis added			
ICM4	Mix group with 0.4% propolis added			
ICM5	Mix group with 0.5% propolis added			

2.4. Analyzes of ice cream mix

2.4.1. Physicochemical analysis

Ice cream samples (5 g) were dried in an oven at 105°C and the results were presented (% of sample weight) (AOAC, 2013). The pH values of samples were determined using a pH meter (Ohaus Starter 3100, USA). Solution from pH analysis was filtered, and the titratable acidity analysis was carried out by calculating as lactic acid equivalent.

2.5. Analyses of ice cream

2.5.1. Volume increase index (overrun), first dripping time, and melting rate

The overrun rate was detected by method given by Ahmad et al. (2020). Ice cream samples were weighed as 15 g and used for analysis of melting rate and first dripping time. The time of first drop (in seconds) is recorded and melting rate was calculated according to melted amount after 30 minutes (Kavaz et al., 2016).

2.5.2. Total phenolic content

To determine the total phenolic content of propolis powder and ice cream samples, 200 µL of the solution was taken from the solution, 0.1 mL of Folin-Ciocalteu reagent and 0.3 mL of Na_2CO_3 (2%) were added and it was completed to the final volume of 5 ml with distilled water. Different concentrations of gallic acid were used to obtain the standard curve. The samples that will react with Folin-Ciocalteu were read against blank (pure water) at 760 nm and the results were expressed as gallic acid equivalents (Gülçin, et al., 2004).

2.5.3. Antioxidant activity

For antioxidant activity analysis, propolis powder and ice cream samples were extracted with ethanol. Ferric Reducing Antioxidant Power (FRAP) and analysis were performed to determine antioxidant activity of samples.

For the FRAP analysis, Trolox was used to plot the standard curve, and analysis was performed by reading the colored liquid formed as a result of the reaction between the sample solutions and Trolox $FeCl_3$, against the blank at 700 nm. Briefly, 1.17 mL sodium phosphate buffer (0.2 M, pH 6.6) and 1.25 mL of potassium Ferro cyanide, Trolox standard solution (80-240 µL), stock sample solution (80 µL) and $K_3Fe\ (CN)\ _6]$ (1%) were added at tubes and the solutions were incubated at 50ºC for 20 minutes. After incubation, 1.25 mL of 10% trichloroacetic acid is added to the solutions. Then 0.25 mL of 0.1% $FeCl_3$ was added and the tubes were vortexed and read in the spectrophotometer (Oyaizu, 1986).

2.5.4. Texture analysis

Following storage of ice creams at -18ºC for 24 hours, the hardness and stickiness values of ice creams were measured at 20ºC by a texture analyzer (TA-TX plus, Stable Microsystem, Reading, UK).

2.6. Sensory analysis

Ice cream samples were evaluated by 10 panelists. The panelists evaluated color-appearance, taste-aroma, structure-texture, meltability, and overall acceptability of samples on a 5 point hedonic scale (1: unacceptable; 5: very good).

2.7. Statistical analysis

All ice cream and propolis analyses were performed as two replicates. The SPSS statistical software was used to analyze the obtained data. Analysis of variance (ANOVA) was executed for statistical differences between propolis added ice cream groups and storage periods. Then, the statistically significant (α=0.05) differences were subjected to the Tukey's Multiple Range Test.

3. Results and Discussion

3.1. Total phenolic content and antioxidant activity of propolis powder

The antioxidant activity values of the propolis powder sample were as" FRAP 396.84±6.52 mg TE in powder propolis sample. The total phenolic content was as total phenolic 136.19±3.35 GAE mg/mL. Sowmya et al. (2019), found the value of the total phenolic substance amount of the propolis sample they obtained in their study in India as 18.6 mg/mL GAE. The value that we found in this study is higher than this value. Ozdal et al. (2018), collected propolis samples from various parts of Turkey and determined their antioxidant activity. While the total amount of phenolic substance was higher than the value we found in our study, the FRAP value was found to be lower than the value we obtained in our study. The difference may be due to regional differences and seasonal factors to propolis content.

3.2. Dry matter, pH and titratable acidity of ice cream mix samples

The dry matter analysis, pH, titratable acidity results of the ice cream mix samples were shown in *Table 2*. In general, a higher dry matter content in an ice cream mix ensures a greater resistance to melting (Öztürk, 1969; Tekinsen and Karacabey, 1984). There was no significant difference between the ice cream mixes in terms of dry matter (p>0.05). The dry matter concentrations of the have been determined between 36.9 and 39.3%. While Şen (2016) found the dry matter ratio of ice cream mixes between 35.25-37.35 g/100g, Or (2009) found the dry matter ratios of ice cream samples between 32.43-45.71 g/100g, and. These dry matter ratios are similar to the dry matter ratios we found in our study. However, dry matter content of propolis-added ice creams ranged between 32.43 and 33.37% in the study done by Demir Özer (2021) and it was lower than the results found in this study.

Table 2. Dry matter, pH and titratable acidity of ice cream mixes

Ice Cream Mix Type	Analyses		
	Dry Matter (%)	pH	Titratable Acidity (Lactic acid %)
Control	39.1±1.7	6.45±0.02	1.20±0.03
ICM1	36.9±1.2	6.50±0.02	1.31±0.09
ICM2	38.5±4.1	6.29±0.04	1.11±0.01
ICM3	39.3±0.5	6.42±0.03	1.29±0.15
ICM4	38.4±3.1	6.36±0.06	1.39±0.18
ICM5	38.5±1.8	6.40±0.05	1.29±0.01

Control (0% propolis powder), ICM1 (0.1% propolis powder), ICM2 (0.2% propolis powder), ICM3 (0.3% propolis powder), ICM4 (0.4% propolis powder), ICM5 (0.5% propolis powder)

It can be seen from *Table 2* that the difference between the pH values of the ice cream mix samples was not statistically significant according to the analysis of variance (p>0.05). The pH values have been found in the range of 6.29 to 6.50. The fact that the only difference between the compositions of the ice cream mixes is propolis concentration made the pH values very close to each other. Kurultay et al. (2010), found the pH values of the samples as 6.55 and 6.57, Tekinşen et al. (2011) found it between 6.35 and 6.41, and Demir Özer (2021) determined the pH values as 6.59-6.61. Similar pH readings to these values were recorded in our study.

According to the calculations, titratable acidity values of propolis-added ice cream mixes were between 1.11 and 1.39% as lactic acid equivalents. There was no statistical difference between the calculated values ($p>0.05$). Titratable acidity value in ice cream depends on the amount of fat-free dry matter in the formulation (Gürsel and Karacabey, 1998). Dağlı (2006) and Demir Özer (2021) determined the acidity of ice cream samples as 0.190-0.198% and 0.20-0.24% lactic acid equivalent, respectively while Açu et al. (2017) determined as 1.25-1.47%. The reason for the acidity differences between studies is thought to be the use of different ingredients and formulations.

3.3. Ice cream analyses

3.3.1. Volume increase index (overrun)

The volume increase index in ice cream is due to the air entering the mixture while it is partially frozen by mixing. Too much air entering to the mixture causes a granular structure while a low amount of air causes too hard texture. A volume increase between 15% and 50% is required in good quality ice creams (Tekinşen, 2008). The change in the volume increase values of the propolis-added ice cream samples in the 60-day storage period is given in the *Table 3*.

Table 3. Overrun values (%) of ice cream samples

Ice Cream Type	Storage Time (days)		
	3	30	60
Control	20.84±0.75b	23.50±0.77a	22.49±0.49ab
ICM1	23.26±0.16a	23.00±0.10a	23.37±0.36a
ICM2	23.45±0.15a	21.50±0.20b	23.41±0.32a
ICM3	20.83±1.64b	21.90±1.50b	21.65±0.75b
ICM4	22.57±0.58ab	23.50±0.46a	23.67±0.24a
ICM5	22.57±0.59ab	22.90±0.37ab	22.65±0.09ab

Control (0% propolis powder), ICM1 (0.1% propolis powder), ICM2 (0.2% propolis powder), ICM3 (0.3% propolis powder), ICM4 (0.4% propolis powder), ICM5 (0.5% propolis powder). a-b: There is a statistical difference between ice cream groups shown with different letters in the same column ($P<0.05$).

Considering the overrun values, a statistical difference was found between the ice cream samples depending on the propolis concentrations ($p<0.05$). There was no statistical difference between the volume increase values obtained at the end of the storage periods ($p>0.05$). The highest overrun value was found in the ICM4 group, stored for 60 days, and the lowest volume increase was in the ICM3 ice cream group, stored for 3 days. Atsan and Çağlar (2008) found the overrun values between 31.13% and 41.71% in their study. The values obtained in the study in which different stabilizers were used are higher than the values we obtained in our study. Antepüzümü (2005) found an increase in volume between 16.32 and 35.95% in ice creams produced using honey and glucose syrup. Some of these results are close to the values obtained in this study. The differences between studies are thought to be due to ice cream formulations, production technique, and freezer performance.

3.3.2. Determination of first dripping time and melting rate

The first dripping times and melting rates of the propolis added ice cream samples are shown in *Table 4*. The first dripping time was not affected significantly by the concentrations of propolis ($p>0.05$). However, storage was found to have a significant effect on the first dripping time ($p<0.01$). The shortest first dripping time occurred in the ICM1 codes ice cream samples, which were stored for 3 days, and the longest was in the ICM4 codes ice cream samples, which were stored for 60 days. The first dripping time values were increased significantly after 30 days of storage. The averages of the groups with different propolis concentrations were close to each other. Şen (2016) found first dripping time values between 1288 seconds and 1044 seconds in ice cream samples produced using sahlep obtained from orchids from Turkey's different regions. Güven et al. (2010) found this value between 1285 and 2000 seconds in their study where they produced Kahramanmaraş type ice cream with low fat content. The values in these studies are higher than the values determined in this study.

Different propolis concentrations in the ice cream samples did not make a statistically significant difference in the melting rate and the first dripping time ($p>0.05$). After the 30and 60 days of storage, it was observed that the melting rate decreased, and this decrease was found to be statistically significant ($p<0.01$). The highest melting rate was calculated for the ICM3 group stored for 3 days and the lowest melting rate was in the control group ice cream stored for 60 days. Karaman et al. (2011) produced ice cream using 0.5% and 1% salep and found the melting rate at the 30th minute to be 22.70-78.89%. In this study, the melting rate of ice cream produced using 0.5% salep is close to the melting rate of ice cream produced in this study, while it is below the melting rate of ice cream produced with 1% salep. It is thought that the change in the first dripping time and melting rates during storage is because the ice cream has a harder structure during storage. The hardness values obtained in the texture analysis performed in this study also confirm this claim.

In a study evaluating the antimicrobial effect of propolis-added ice cream, it was observed that the addition of propolis did not significantly affect the first dripping time and melting rate results. In the study, the first dripping times were found to be between 421s-459s, and the melting rate was found to be between 16.4% and 16.7% (Demir Özer, 2021). The values in this study were found to be higher than these values and this difference is thought to be due to the changes in ice cream formulations.

Table 4. First dripping times (s) and melting rates (%) of samples

	Ice Cream Type	Storage Time (day)		
		3	30	60
First Drip Time (sec)	Control	759.0±36.0A	915.0±45.0B	940.0±80.0B
	ICM1	717.5±77.5A	975.0±45.0B	950.0±10.0B
	ICM2	927.5±17.5B	975.0±15.0B	900.0±60.0B
	ICM3	795.0±70.0A	960.0±0.00B	930.0±30.0B
	ICM4	865.0±20.0A	912.5±12.5B	1000±20.0B
	ICM5	845.0±45.0A	870.0±30.0A	930.0±10.0B
Melting Rate (%)	Control	63.96±1,27A	41.80±0.42B	38.10±0.80C
	ICM1	64.78±0.92A	42.05±0.67B	40.65±0.62B
	ICM2	68.10±1.12A	42.50±1.02B	39.55±0.67C
	ICM3	68.60±0.54A	39.70±0.85C	38.85±0.30C
	ICM4	66.75±1.58A	40.73±0.34B	40.40±0.20B
	ICM5	67.60±0.82A	41.30±0.29B	39.80±1.07C

Control (0% propolis powder), ICM1 (0.1% propolis powder), ICM2 (0.2% propolis powder), ICM3 (0.3% propolis powder), ICM4 (0.4% propolis powder), ICM5 (0.5% propolis powder). A-C: There is a statistically significant difference between the storage times shown in different capital letters on the same row ($P<0.05$)

3.3.3. Texture analysis in ice cream

Hardness and stickiness values of ICM samples were calculated via texture analyses. The texture analysis results of the samples are presented in *Table 5*.

The addition of propolis powder to the ice cream formulation at different rates affected the hardness and stickiness values of the ice creams insignificantly ($p>0.05$). In addition, hardness values obtained after 3rd day of storage were significantly different from values obtained after 30 and 60t days ($p<0.05$). The stickiness value obtained on the 30th day of storage was found significantly different from the values obtained in other storage periods ($p<0.05$). The highest hardness value was obtained in the ICM1 ice cream stored for 60 days; the lowest hardness value was obtained in the control group ice cream stored for 3 days. In addition, among ice cream samples, the group with the highest stickiness was determined as the ICM3 group, which was stored for 30 days, and the group with the lowest stickiness, the control group, which was stored for 60 days. After the 30 day of storage, the hardness values increased significantly. The changes between concentrations and hardness values after 60 days of storage were not statistically significant. Karaman et al. (2014) produced ice cream by adding persimmon paste to ice cream mix in different proportions in their study. In this study, they also investigated the texture properties of ice cream groups. They found the hardness values of the samples between 44.34 N and 162.20 N. These values are higher than the values we found. It was thought that this situation caused by using different formulations and the fact that the ice cream dry matter components significantly affect the hardness. Karaman et al. (2014) found stickiness values between -7.83 and -4.11 N in their studies. These values are higher than the values we determined as well as the hardness value. As an alternative measurement to stickiness, Demir Özer (2021) indicated lower adhesiveness and cohesiveness values with the inclusion of propolis in ice cream samples.

Table 5. Texture analysis results of samples

	Ice Cream Type	Storage Time (day)		
		3	30	60
Hardness	Control	2.03±0.26A	29.02±10.76B	16.15±2.05B
	ICM1	7.40±0.24A	28.21±6.10B	41.47±0.54B
	ICM2	3.25±0.22A	24.64±3.31B	34.56±17.05B
	ICM3	5.88±1.53A	32.83±6.80B	35.77±22.94B
	ICM4	2.92±0.21A	34.04±4.78B	18.41±13.22B
	ICM5	4.44±1.25A	26.19±3.99B	26.31±6.10B
Stickiness	Control	-0.40±0.02A	-0.81±0.19B	-0.15±0.02A
	ICM1	-0.58±0.04A	-0.65±0.13A	-0.67±0.22A
	ICM2	-0.25±0.01A	-0.74±0.04B	-0.35±0.34A
	ICM3	-0.54±0.06A	-1.19±0.63B	-1.01±0.64B
	ICM4	-0.51±0.06A	-1.12±0.03B	-0.22±0.09A
	ICM5	-0.59±0.01A	-1.11±0.24B	-0.89±0.07B

Control (0% propolis powder), ICM1 (0.1% propolis powder), ICM2 (0.2% propolis powder), ICM3 (0.3% propolis powder), ICM4 (0.4% propolis powder), ICM5 (0.5% propolis powder). A-B: There is a statistically significant difference between the storage times shown in different capital letters on the same row ($P<0.05$).

3.3.4. Total phenolic content and antioxidant acidity of ice creams

In our study, the antioxidant activity of the samples was determined by calculating the FRAP value. Since phenolic components have antioxidant properties, the high amount of total phenolic substance usually indicates high antioxidant activity (Shori and Baba, 2013). The total phenolic contents and FRAP values of ice cream samples are as in *Table 6*.

The change of FRAP values was statistically significant ($p<0.05$). Propolis addition to ice cream has significantly affected total phenolic content and FRAP values ($p<0.01$) sample groups. The ICM5 group (3rd day) had the highest total phenolic content while the lowest was determined in the control group stored for 60 days. Kamiloglu et al. (2013) compared the antioxidant activities of food products containing black mulberry collected from the market in their study. In this study, the total phenolic contents of black mulberry ice cream was also calculated. It has been reported that the total phenolic content of black mulberry ice cream was calculated as 3.779 mgGAE/g. This value is higher than the values of all ice cream types in our study. The reason for this situation thought to be that although propolis added to the ice cream mix at a very low concentration in our study, the fruit ice cream analyzed in this study contained a higher rate of black mulberry. In their study, Ghosh and Bhattacharjee (2014) found the total phenolic contents of ice creams they produced by adding basil extract between 0.31 mg GAE/ml and 0.38 mgGAE/ml. The total phenolic contents of the ice creams we produced by adding propolis were higher. An increase in total phenolic content and antioxidant activity of yogurt was determined with the addition of propolis to yogurts in a study by Santos et al. (2019). This is thought to be due to the very high antioxidant activity of propolis. The highest FRAP value was calculated in the ICM5 group stored for 3 days and the lowest FRAP value in the control group stored for 3 days. The propolis concentration increases the FRAP values, which decrease a little with storage. Kamiloglu et al. (2013) found the FRAP value of black mulberry ice cream they collected from the market as 8.861 mg TE/g. This value is higher than the FRAP values of the ice creams we produce. The reason for this thought to be that black mulberry added to the mix at an exceedingly high concentration in the ice cream analyzed in the study. When the antioxidant activity analyses were examined in general, it was found that the addition of propolis increased the antioxidant activity of ice creams. It was determined that the storage period only affects the FRAP values but not the total phenolic content.

Table 6. Antioxidant activity values of ice cream samples

	Ice Cream Type	Storage Time(day)		
		3	30	60
Total Phenolic Content (mgGAE/gr)	Control	0.27±0.01a	0.43±0.10abc	0.09±0.03a
	ICM1	0.40±0.05abc	0.41±0.08abc	0.21±0.04a
	ICM2	0.51±0.05bc	0.42±0.12abc	0.35±0.07ab
	ICM3	0.56±0.05bc	0.63±0.07bc	0.54±0.11bc
	ICM4	0.74±0.03c	0.73±0.10c	0.65±0.19bc
	ICM5	1.19±0.26d	0.83±0.06c	0.99±0.18d
FRAP (mgTE/gr)	Control	1.18±0.07a,A	2.27±0.14ab,A	1.25±0.15a,A
	ICM1	2.48±0.03ab,A	2.56±0.05ab,A	2.19±0.64ab,A
	ICM2	4.89±0.24bc,AB	4.69±0.08bc,AB	2.92±0.16ab,A
	ICM3	5.88±0.28cd,BC	6.41±0.25cd,BC	4.73±0.13bc,AB
	ICM4	7.21±0.89d,BC	4.79±0.26bc,AB	5.18±1.45cd,BC
	ICM5	9.42±0.03d,BC	6.39±2.50cd,BC	5.90±0.14cd,BC

Control (0% propolis powder), ICM1 (0.1% propolis powder), ICM2 (0.2% propolis powder), ICM3 (0.3% propolis powder), ICM4 (0.4% propolis powder), ICM5 (0.5% propolis powder). a-d: There is a statistical difference between ice cream groups shown with different letters in the same column (P<0.05). A-C: There is a statistically significant difference between the storage times shown in different capital letters on the same row (P <0.05).

3.3.5. Sensory analyses of ice cream samples

The sensory properties of ice cream samples evaluated by different panelists in terms of color-appearance, structure-texture, taste-aroma, meltability, and overall acceptability scores are given in *Table 7*.

The change in color-appearance scores of ice cream samples during storage was found insignificant (p>0.05). The effect of different propolis concentrations on color-appearance scores was found statistically significant in sensory evaluations (p<0.01). The highest color-appearance scores were found in the control group, while the lowest scores were found in ICM5 group containing the highest concentration of propolis powder. Yaşar and Şahan (2008) stated that the color-appearance scores of the Kahramanmaraş type ice cream produced using honey was affected negatively with increasing honey ratio. Antepüzümü (2005) used honey and glucose syrup in the formulation of ice cream in his study and stated that the ice cream containing honey had lowest color scores.

When the structure-texture scores of the ice cream groups were analyzed statistically, it was found that the difference between the storage time and the scores obtained from different concentrations was insignificant (p>0.05). It is determined that the control group stored for 3 days with the highest score, and the ICM4 and ICM5 group ice creams stored for 60 days with the lowest score. The probiotic ice cream produced by Bakır (2015) has a higher score than the control group considering the structure-texture feature. Koyun (2009) stated that there was an insignificant difference between the texture-consistency scores of the ice cream produced by skimmed milk powder and whey protein concentrate.

It was determined that both the propolis concentration and the storage time significantly affected the scores obtained by the groups in the taste-aroma sensory evaluations (p<0.01). The highest taste aroma score was found in the ICM1 group, which was stored for 3 days, and the lowest in the ICM4 group, which was stored for 60 days. There is no statistical difference between the taste-aroma scores of the ice creams with propolis additions. ICM1 ice cream samples were determined to have a different mean from all other groups. When the averages of the ice cream samples are examined according to the storage time, it was determined that the 3-day group samples with high scores were different from the other groups. Yaşar and Şahan (2008) and Antepüzümü (2005) added honey to ice cream in their study. In both studies, it stated that the addition of honey negatively affected the taste-aroma scores of ice cream.

There was statistically significant difference (p>0.05) between the meltability scores during the storage period. However, the difference between the sensory scores of the ice creams with different propolis content was statistically significant (p<0.01). The highest scores were found in the control group, while the lowest scores were in the ICM5 group.

The overall acceptability scores of the sensory evaluations made by the panelists on different days are shown in Table 8 and the scores differed insignificantly among the storage periods (p>0.05). It was determined that the on the general acceptability scores was affected by the propolis concentration significantly (p<0.01). Like other sensory features, the control group achieved the highest scores, while the ICM5 group got the lowest. Because of the sensory evaluations, it can be said that the most liked group was the control group. The addition of propolis negatively affected all sensory properties of ice creams. Addition of propolis to ice cream up to 0.6% was found acceptable in terms of sensory in a study by Mironova et al. (2020) in which they studied the propolis concentrations between 0 and 0.9%.

Table 7. Effect of propolis powder concentrations on sensory properties of ice creams

	Ice Cream	Storage times (day)		
		3	30	60
Color and Appearance	Control	4.9±0.1a	4.7±0.1a	4.5±0.2a
	ICM1	4.2±0.2ab	4.3±0.2a	4.1±0.2ab
	ICM2	4.4±0.2a	4.4±0.2a	4.2±0.2ab
	ICM3	3.9±0.3bc	3.7±0.3bc	3.5±0.3bc
	ICM4	3.9±0.3bc	3.6±0.3bc	3.5±0.3bc
	ICM5	3.6±0.4bc	3.5±0.4bc	3.3±0.3c
Structure Texture	Control	4.1±0.3	4.0±0.2	3.8±0.2
	ICM1	3.9±0.3	3.9±0.3	3.8±0.2
	ICM2	3.7±0.2	3.8±0.2	3.6±0.2
	ICM3	3.7±0.2	3.6±0.3	3.4±0.3
	ICM4	3.7±0.2	3.6±0.3	3.5±0.3
	ICM5	3.6±0.3	3.7±0.2	3.5±0.2
Taste and Aroma	Control	4.6±0.2a,A	4.0±0.1b,B	4.0±0.2b,B
	ICM1	4.9±0.1a,A	3.9±0.1b,B	4.2±0.2ab,B
	ICM2	4.5±0.2a,A	3.8±0.2b,B	4.0±0.2b,B
	ICM3	4.4±0.2ab,A	3.6±0.2b,B	3.7±0.3b,B
	ICM4	4.4±0.2ab,A	3.6±0.2b,B	3.5±0.2b,B
	ICM5	4.3±0.3ab,A	3.7±0.3b,B	3.7±0.3b,B
Meltability	Control	4.1±0.3a	4.2±0.3a	4.1±0.3a
	ICM1	3.7±0.3ab	3.6±0.3ab	3.6±0.3ab
	ICM2	3.7±0.2ab	3.6±0.3ab	3.4±0.3b
	ICM3	3.6±0.3ab	3.5±0.3ab	3.3±0.2b
	ICM4	3.5±0.3ab	3.8±0.2ab	3.6±0.2ab
	ICM5	3.4±0.3b	3.2±0.1b	3.1±0.2b
Overall Acceptability	Control	4.3±0.2a	4.4±0.2a	4.2±0.2a
	ICM1	4.2±0.2a	4.2±0.2a	3.9±0.2ab
	ICM2	4.1±0.3ab	4.0±0.2ab	3.8±0.2ab
	ICM3	3.8±0.2ab	3.8±0.2ab	3.6±0.2b
	ICM4	3.8±0.2ab	3.9±0.2ab	3.8±0.2ab
	ICM5	3.7±0.2b	3.6±0.2b	3.5±0.2b

Control (0% propolis powder), ICM1 (0.1% propolis powder), ICM2 (0.2% propolis powder), ICM3 (0.3% propolis powder), ICM4 (0.4% propolis powder), ICM5 (0.5% propolis powder). a-c: There is a statistical difference between ice cream groups shown with different letters in the same column (P <0.05). A-B: There is a statistically significant difference between the storage times shown in different capital letters on the same row (P <0.05).

Santos et al. (2020) reported that the addition of red propolis, as a substitute of chemical preservative, into yogurt did not affect the sensory properties of yogurts while Korkmaz et al. (2021) indicated that the sensory acceptance of yogurts decreased when propolis was included in yogurts. However, Luis-Villaroya et al. (2015) added propolis to apple juice due to the protective properties of propolis in his study. Similar to the results in our study, it has been reported that as the propolis concentration increases, a decrease in consumer taste is observed. According to the results of sensory analysis, it was reported that the group containing the highest propolis achieved the lowest scores.

4. Conclusion

In this study, a functional ice cream product was formulated by adding propolis powder. It is physicochemical, sensory, and bioactive properties were investigated during storage. It was determined that adding propolis did not significantly affect the physicochemical properties. Propolis concentration did not affect the melting rate and first dripping times significantly but it affected overrun values. No significant change was seen in ice creams regarding propolis concentration, but hardness and stickiness values change with extended storage time. While the addition of propolis contributed significantly to the antioxidative properties, increasing propolis concentration caused a decrease in sensory analysis scores. When antioxidant activity analyzes and sensory analyzes are evaluated together, it is seen that the ice cream sample containing 0.2% and 0.3% propolis are the ideal groups to produce propolis-added ice cream. In line with these results, further studies should be carried out on different types of propolis extracts. It is thought that the propolis powder has highly promising properties for propose a novel functional food to consumers from any ages.

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