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Saygılarımla.

**Prof. Dr. Oğuz Gürsoy**
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**3. Uluslararası Geleneksel Gıdalar ve Sürdürülebilir Beslenme Sempozyumu**

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**7. Uluslararası Malzeme ve Polimer Bilimi Kongresi**

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**5. YABITED Fats and Oils Congress**

Yağ Bilimi ve Teknolojisi Derneği tarafından 7-9 Kasım 2024 tarihleri arasında Antalya'da gerçekleştirilecek olan kongre ile ilgili bilgilere **https://yabited.org/5th-yabited-fats-and-oils-congress/** adresinden ulaşılabilir.

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**Research Paper / Araştırma Makalesi**

# Effect of Different Heat-Treated Ultrafiltration (UF) Retentates with Altered Protein-to-Fat Ratios on Chemical, Physicochemical and Sensory Properties of UF White Cheese

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## ABSTRACT

In this study, ultrafiltration (UF) white cheese samples were manufactured using UF retentate, which had been heat-treated at different temperatures (75°C and 85°C for 15 s) and had altered ratios of protein-to-fat (P/F) (0.8, 0.7, and 0.6). The physicochemical and sensory properties of UF white cheeses during storage (90 days) were determined. White cheese manufactured from UF retentate with a P/F ratio of 0.6 had higher fat and lower protein contents than those with the P/F ratios of 0.7 and 0.8. Treatment temperature significantly influenced the pH and titratable acidity values of UF white cheeses. The highest hardness and chewiness values and the lowest adhesiveness values were determined in UF white cheese with P/F ratios of 0.8 and heat-treated at 85°C. During storage, the L*, a*, b*, and ΔE* values of white cheeses varied between 94.51 and 98.34, -0.77 and -0.06, 7.78 and 10.13, and 10.61 and 13.00, respectively. The use of high-fat-content ultrafiltered retentate subjected to high temperatures in the manufacture of white cheeses had a positive effect on overall acceptability scores.

**Keywords:** Cheese, Membrane filtration, Texture

## Farklı Isıl İşlem Görmüş ve Protein-Yağ Oranları Değiştirilmiş Ultrafiltrasyon Retentatlarının Süzme Beyaz Peynirin Kimyasal, Fizikokimyasal ve Duyusal Özellikleri Üzerine Etkisi

## ÖZ

Bu çalışmada farklı sıcaklıklarda ısıl işlem uygulanan (75°C ve 85°C'de 15 s) ve protein yağ oranı değiştirilmiş ultrafiltrasyon (UF) retentatları kullanılarak beyaz peynir örnekleri üretilmiştir. UF beyaz peynir örneklerinin fizikokimyasal özellikleri ve tüketici kabulü 90 günlük depolama süresince belirlenmiştir. Protein/yağ oranı 0.6 olan UF retentattan üretilen peynirler, protein/yağ oranı 0.7 ve 0.8 olan UF retentatlardan üretilen örneklere göre yüksek yağ ve düşük protein içeriğine sahiptir. Isıl işlem sıcaklığı, beyaz peynirlerin pH ve titrasyon asitliği değerlerini istatistiksel olarak önemli düzeyde etkilemiştir. En yüksek sertlik ve çiğnenebilirlik ile en düşük yapışkanlık değerleri protein/yağ oranı 0.8 olan 85° C'de ısıl işlem görmüş UF retentantlardan üretilen beyaz peynirlerde belirlenmiştir. Depolama süresince, beyaz peynirlerin L*, a*, b* ve ΔE* değerleri sırasıyla 94.51-98.34, -0.77 - -0,06, 7.78-10.13 ve 10.61-13.00 arasında değişmiştir. Beyaz peynirlerin üretiminde yüksek ısıl işlem sıcaklıklarına tabi tutulan yüksek yağ içeren UF retentant kullanımı genel kabul edilebilirlik puanları üzerine olumlu bir etkiye sahiptir.

**Anahtar Kelimeler:** Peynir, Membran filtrasyon, Tekstür

## INTRODUCTION

The use of concentrated milk through ultrafiltration (UF) membranes in its production has created a new production process for white cheese. The use of the UF process for milk concentration enables the selective separation of molecules with a molecular weight ranging from 1 to 200 kDa from the milk serum through cross-flow processing on the membrane surface under pressure [1]. In the UF process, the milk is divided into two parts: the diluted part, known as permeate, and the concentrated part, referred to as retentate. In the production of white cheese by the UF process, the retentate is utilized, and unlike traditional white cheese, whey is not expelled from the curd [2]. The salt, starter culture, and rennet could be added to the UF retentate in the production of white cheese by the UF process [1]. The UF process can increase the cheese yield and textural quality and decrease protein, fat, and total solids loss compared to traditional cheese production [3].

Different strategies, such as full concentration (approximately 7.5 times), 4 to 6 times concentration, a maximum of two-fold concentration, and concentration plus evaporation, can be employed in cheese production by the UF process. In the production of white cheese, as well as feta, mozzarella, havarti, danbo, and cheddar cheeses with the UF process, milk can be concentrated four to six times [4-6]. Alterations in the composition and structure of the UF retentate create differences in casein aggregation compared to milk. Casein micelles move more closely together because of the reduced distance between them, which likely increases the aggregation rate and directly influences the curd structure. The protein network becomes coarser with increasing casein concentration in cheeses produced by the UF process [7, 8]. The fat content, distribution of fat globules, and protein-fat interactions within cheese are important for the textural and structural properties of cheese [9]. The concentration of fat by the UF process appears to enhance the contact surface among fat globules, which causes them to aggregate and then fuse. When the fat globules remain intact within the protein matrix, the plasticizing influence of fat and water restricts the interactions among casein chains. Moreover, the retention of whey proteins in cheeses produced by the UF process results in the entrapment of water within the cheese matrix due to the hydrophilic characteristics of whey proteins [10]. The degree of whey protein denaturation may also impede rennet-induced aggregation of the casein micelles. Consequently, the manufacture of semi-hard and hard cheeses should not be achieved by subjecting milk to severe heat treatment. These lead to textural defects, such as softness and openness in cheeses produced by the UF process [11-13]. The heat treatment temperature and time significantly influence the degree of whey protein denaturation [14]. The milk composition and processing parameters affect the properties of cheeses [15].

Although there are many studies in the literature on the use of the UF process in cheese production, to our knowledge, there is no study evaluating the combined effects of the P/F ratio of UF retentate and the heat treatment applied to UF retentate on the quality and sensory characteristics of white cheese. The aim of this study was to ascertain how three different P/F ratios in UF retentate, along with two different heat treatments, affected the physicochemical and sensory properties of white cheese.

## MATERIALS and METHODS

For the manufacture of white cheese by the UF process, raw cow milk was obtained from the Cattle Farms of BBB, Türkiye. Rennet (600 International Milk Clotting Units $mL^{-1}$) and starter culture (R-704-DVS, including *Lactococcus lactis* subsp. *lactis* and *Lactococcus lactis* subsp. *cremoris*) were purchased from Maysa Food (İstanbul, Türkiye) and Peyma-Hansen Cheese Rennet Industry and Trade Inc. (İstanbul, Türkiye), respectively. The commercial salt (NaCl), polyethylene boxes (500 mL), and lids were supplied from local markets of BBB. All the chemicals were of analytical grade, produced by Sigma Aldrich (Steinheim, Germany).

### Manufacture of White Cheese by Ultrafiltration

The raw milk was heat treated at 72°C for 15 s and then standardized to about 3% fat using a separator (Tetra Pak, Alfa Laval Tumba AB, Eskilstuna, Sweden) at 50°C. Standardized milk was concentrated using an UF unit (UF 2000, Teknoproses Engineering Consulting Industry Trade Ltd. Co., Ankara, Türkiye) with a spiral-type membrane module (GR73PP-UF-pHt, Alfa Laval) at a pressure of 4.5 bar and a temperature of 50°C. After concentration, UF retentate was divided into three parts to adjust the protein-to-fat ratios of each differently as 0.6, 0.7, and 0.8 using milk cream (with 65% fat). The UF retentates with different P/F ratios were homogenized at 80 bar with an MG4-140B model homogenizer (BOS Homogenizers BV, Hilversum, Netherlands). The homogenized ultrafiltered retentates were heat treated at 75°C for 15 s and at 85°C for 15 s with a plate heat exchanger (Alfa Laval, PHE, Lund, Sweden) to denature whey proteins, respectively, in 1% and 10% ratios according to the kinetic data of Kessler [16]. After heat treatment, the UF retentates with varying P/F ratios were cooled at 32°C, followed by the addition of salt (1.0% w/v) and inoculation with starter culture (0.1% w/v). Then, the UF retentates were mixed with rennet solution in the filler machine (RPK-SBPD 2000, Ropak Machine Process Automation Industry and Trade Inc., Bursa, Türkiye). Then, the polyethylene boxes were filled immediately with the UF retentates and sent to a coagulation tunnel (Tünel 2000, Teknoproses Engineering Consulting Industry Trade Ltd. Co.). After moving through the tunnel at 32°C for 30 min, boxes were then sealed with aluminum foil and closed with lids. The UF retentates were incubated at 32°C for 24 hours until the pH reached about 4.7 and then cooled to 4°C. The white cheese manufactured by the UF process, named UF white cheese, was stored at 4°C for 90 days.

### Physicochemical Analysis

Total solids, fat, and protein contents were determined in the raw milk, UF retentates, and UF white cheeses according to the Association of Official Analytical Chemists (AOAC) methods by the gravimetric, Gerber, and Kjeldahl, respectively [17-19]. The pH values of the raw milk, UF retentates, and UF white cheeses were measured with a pH meter (Thermo Scientific Orion 2 Star, Bremen, Germany). The titratable acidity and salt (NaCl) content of the UF white cheeses were determined by the Soxhlet-Henkel and Volhard methods, respectively [20, 21]. The CIE Lab color parameters (L*, a*, and b*) of each UF white cheese were measured by using a Konica Minolta Chroma Meter CR300 (Minolta Co., Ltd., Osaka, Japan) [22]. ΔE* values were calculated using the equation 1 below [18]:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

The device was calibrated using a white calibration plate (L=95.14, a=-0.13, b=2.71, and ΔE=0.57) before use. The TA-XT2 Texture Analyzer (Stable Micro Systems, Haselmere, Surrey, UK), equipped with a cylinder probe (P/2, 25 mm diameter), was used to measure the texture quality attributes (hardness, cohesiveness, resilience, and chewiness) of the UF white cheese samples [23].

### Sensory Analysis

For the sensory analysis, 100 participants (59 females and 41 males) between the ages of 20 and 50 who did not have any specific health problems that would have an impact on the sensory evaluation were selected from the Department of Food Engineering at the Akdeniz University (Antalya, Türkiye) and from Yörükoğlu Dairy Product Inc., Co. (Antalya, Türkiye). The selected participants were informed about the sensory evaluation for approximately 1 h to become familiar with the descriptors. All samples were presented with a three-digit code. The hedonic scale ranging from 1 = “dislike extremely” to 7 = “like extremely” was used to evaluate the sensory quality parameters (color, texture, taste, and overall acceptability) for the sensory properties of the UF white cheese samples during the stored period. The participants evaluated a total of six UF white cheeses after 1, 45, and 90 days of storage [24].

### Statistical Analysis

Each test was done in triplicate. SAS Statistical Software (release for Windows, SAS Institute Inc., Cary, NC, USA) was used to analyze the data. A three-factor ANOVA was performed to determine the effects of P/F ratio, heat treatment temperature, and storage time on the physicochemical and sensory properties of the UF white cheese samples. To identify differences among treatment means, Duncan's multiple range test was used.

## RESULTS

Table 1 presents the chemical composition of the UF retentate with varying P/F ratios alongside raw milk. The total solids and protein contents of the UF retentates increased more than 2.6 and 4.1 times compared to raw cow milk, respectively. The highest fat content was found in the UF retentate with a P/F of 0.6. Similar pH values were observed for the raw cow milk and in UF retentates.

Table 1. The chemical composition of the UF retentate with varying P/F ratios alongside raw cow milk

| Components | Raw cow milk | UFR0.8 P/F * | UFR0.7 P/F | UFR0.6 P/F |
|---|---|---|---|---|
| Total solids (%) | 12.02±0.22 | 31.98±0.72 | 31.78±0.29 | 32.01±0.23 |
| Fat (%) | 3.76±0.08 | 16.12±0.11 | 17.85±0.15 | 19.81±0.09 |
| Protein (%) | 2.95±0.08 | 12.95±0.12 | 12.54±0.06 | 12.10±0.14 |
| pH | 6.71±0.06 | 6.66±0.51 | 6.71±0.36 | 6.68±0.44 |

Values are the means ± standard deviations. * UFR0.8 P/F: UF retentate with a P/F ratio of 0.8, UFR0.7 P/F: UF retentate with a P/F ratio of 0.7, * UFR0.6P/F: UF retentate with a P/F ratio of 0.6.

Total solids, fat, protein, and salt contents as well as pH and titratable acidity of the UF white cheeses are shown in Table 2. The results of ANOVA analysis showed that only storage time significantly ($P<0.05$) affected the total solids content of the UF white cheeses. The total solids content of the UF white cheese samples decreased from day 1 until day 45 and then slightly increased up to day 90 (Table 3). This may be explained by the rearrangement of peptide bonds in cheeses and the osmotic pressure difference between the cheese and the environment during storage [25, 26]. The UF white cheese sample manufactured from the UF retentate with a P/F ratio of 0.6 had a higher fat content and a lower protein content than that of other UF white cheese samples (Table 3). It might be due to the higher fat content and lower protein content of the UF retentate with a P/F ratio of 0.6 comparing the UF retentates with P/F ratios of 0.7 and 0.8. During the storage period, both the fat and protein contents of the UF white cheeses decreased. Gholamhosseinpour et al. [27] also found a notable decrease in fat content in cheese with a prolonged storage period. This phenomenon is attributed to the lipolysis of fats, resulting in the production of glycerol and free fatty acids. Soltani et al. [28] reported that changes in protein contents related to protein proteolysis and diffusion of water-soluble nitrogen into brine in UF Iranian white cheese. In the current study, the P/F ratio and heat treatment temperature did not significantly affect the salt content of the UF white cheeses. Salt content increased at the end of the storage period related to the osmotic pressure difference between the cheese moisture and the brine. This trend was similar to results reported by Al-Otaibi and Wilbey [29] and Guven et al. [30].

Table 2. Some chemical characteristics of the UF white cheese

| Parameters | Storage (day) | 0.8 P/F-75* | 0.7 P/F-75 | 0.6 P/F-75 | 0.8 P/F-85 | 0.7 P/F-85 | 0.6 P/F-85 |
|---|---|---|---|---|---|---|---|
| Total solids (%) | 1 | 35.95±0.29 | 35.63±0.39 | 35.44±0.69 | 36.02±0.42 | 35.63±0.32 | 35.33±0.32 |
| | 45 | 35.24±0.10 | 35.06±0.22 | 35.11±0.09 | 35.48±0.10 | 35.56±0.48 | 35.13±0.37 |
| | 90 | 35.43±0.18 | 35.12±0.08 | 35.21±0.07 | 35.56±0.27 | 35.62±0.10 | 35.21±0.25 |
| Fat (%) | 1 | 17.00±0.28 | 19.05±0.35 | 20.65±0.14 | 17.25±0.07 | 18.75±1.06 | 20.20±0.99 |
| | 45 | 16.40±0.57 | 18.75±0.35 | 20.25±0.21 | 16.90±0.42 | 18.00±0.28 | 19.50±0.71 |
| | 90 | 16.15±0.21 | 18.70±0.28 | 19.77±0.32 | 16.81±0.84 | 17.52±0.11 | 19.29±0.34 |
| Protein (%) | 1 | 13.43±0.46 | 12.96±0.13 | 12.46±0.18 | 13.51±0.25 | 12.98±0.06 | 12.56±0.19 |
| | 45 | 13.00±0.30 | 12.88±0.20 | 12.37±0.35 | 13.16±0.11 | 12.87±0.04 | 12.43±0.11 |
| | 90 | 12.64±0.21 | 12.32±0.28 | 12.18±0.18 | 12.84±0.08 | 12.65±0.25 | 12.21±0.49 |
| Salt (%) | 1 | 1.79±0.03 | 1.85±0.09 | 1.86±0.12 | 1.82±0.04 | 1.83±0.11 | 1.85±0.08 |
| | 45 | 1.88±0.03 | 1.87±0.05 | 1.86±0.09 | 1.89±0.07 | 1.96±0.09 | 1.88±0.06 |
| | 90 | 1.93±0.12 | 1.89±0.04 | 1.94±0.02 | 1.89±0.03 | 1.99±0.07 | 1.95±0.09 |
| pH | 1 | 4.74±0.04 | 4.78±0.03 | 4.75±0.03 | 4.65±0.04 | 4.66±0.02 | 4.66±0.01 |
| | 45 | 4.63±0.03 | 4.65±0.04 | 4.68±0.04 | 4.58±0.02 | 4.55±0.01 | 4.57±0.03 |
| | 90 | 4.68±0.01 | 4.72±0.03 | 4.71±0.04 | 4.69±0.03 | 4.69±0.04 | 4.69±0.03 |
| Titratable acidity (SH) | 1 | 74.05±2.47 | 68.15±1.91 | 72.90±3.82 | 80.95±1.63 | 71.10±3.54 | 74.85±3.46 |
| | 45 | 79.75±2.19 | 79.05±2.05 | 82.10±0.42 | 89.25±1.91 | 80.15±3.61 | 84.95±1.91 |
| | 90 | 90.75±2.05 | 82.95±1.91 | 85.00±0.99 | 82.00±0.42 | 85.95±1.91 | 86.85±2.19 |

Values are the means ± standard deviations. * 0.8 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.8, 0.7 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.7, 0.6 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.6, 0.8 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.8, 0.7 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.7, 0.6 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.6

The pH and titratable acidity values of the UF white cheeses were determined to be 4.55-4.78 and 68.15-90.75 SH, respectively. The effect of the P/F ratio of the UF retentate on pH and titratable acidity values of the UF white cheese was not significant. The UF white cheeses manufactured from the UF retentate heat-treated at 85°C had lower pH and higher titratable acidity than those manufactured from the UF retentate heat-treated at 75°C. Because of their increased moisture content and consequent lactic acid, as well as their lower protein content (casein as a proportion of total protein) and buffering capacity, high-temperature cheeses have a lower pH [31, 32]. An increasing heating temperature leads to the denaturation of whey proteins, and the insolubilization of calcium phosphate causes changes in buffering capacity [33]. The pH values of all UF white cheese samples decreased during the first 45 days, after which a significant increase was observed. The increase in pH at the end of the storage is ascribed to the utilization of lactic acid, the formation of non-acidic decomposition by-products, and the release of alkaline substances resulting from the hydrolysis of proteins [34].

Table 3. The effects of the P/F ratio, heat treatment temperature, and storage time on some chemical and physicochemical characteristics of the UF white cheese

| Experimental factors | Total solids (%) | Fat (%) | Protein (%) | Salt (%) | pH | Titratable acidity (SH) |
|---|---|---|---|---|---|---|
| P/F ratio | ns | *** | *** | ns | ns | ns |
| 0.8 | 35.61±0.38 a* | 16.75±0.50 c | 13.09 ±0.38 a | 1.87±0.08 a | 4.66±0.06 a | 82.79±6.11 a |
| 0.7 | 35.44±0.40 a | 18.46±0.67 b | 12.78±0.28 b | 1.90±0.10 a | 4.68±0.08 a | 81.75±6.85 a |
| 0.6 | 35.24±0.40 a | 19.94±0.63 a | 12.37±0.25 c | 1.89±0.10 a | 4.68±0.06 a | 81.11±5.85 a |
| Heat treatment temperature (°C) | ns | ns | ns | ns | *** | *** |
| 75 | 35.50±0.42 a | 18.53±1.62 a | 12.69±0.44 a | 1.88±0.09 a | 4.70±0.05 a | 78.77±5.96 b |
| 85 | 35.35±0.39 a | 18.25±1.28 a | 12.80±0.41 a | 1.90±0.10 a | 4.64±0.06 b | 84.99±6.55 a |
| Storage time (day) | * | ** | *** | * | *** | *** |
| 1 | 35.67±0.53 a | 18.82±1.49 a | 12.98±0.45 a | 1.83±0.09 b | 4.71±0.06 a | 75.32±4.63 c |
| 45 | 35.26±0.36 b | 18.30±1.46 b | 12.78±0.34 a | 1.89±0.08 ba | 4.61±0.05 b | 83.61±4.08 b |
| 90 | 35.36±0.27 ba | 18.04±1.41 b | 12.47±0.33 b | 1.93±0.08 a | 4.70±0.03 a | 86.71±3.23 a |

Values are the means ± standard deviations; different letters for each parameter in a column show significant differences using Duncan's multiple range test (P<0.05). Significant effects at * P<0.05, ** P<0.01, ***P<0.001. ns; not significant.

The textural properties of the UF white cheese samples are shown in Table 4. The UF retentate with a P/F ratio of 0.8 resulted in the UF white cheese having significantly (P<0.05) higher hardness and lower adhesiveness compared to those samples manufactured from the UF retentates with P/F ratios of 0.6 and 0.7. However, non-significant effects of the P/F ratio of the UF retentate were recorded on the springiness and chewiness of the UF white cheese samples (Table 5). Similar to our results, Lepesioti et al.

[35] determined higher hardness and lower adhesiveness in fat-reduced Quark-type cheese. When the P/F ratio decreased, the fat content of the UF white cheese increased, and its hardness decreased. This may be explained by the effect of fat, which disrupts the protein matrix and acts as a lubricant, resulting in smoothness and a softer texture [36]. Moreover, the UF white cheeses manufactured from the UF retentate with a P/F of 0.8 had the highest protein content, which may be related to adhesiveness [35].

Table 4. Some textural properties of the UF white cheese

| Parameters | Storage (day) | 0.8 P/F-75 | 0.7 P/F-75 | 0.6 P/F-75 | 0.8 P/F-85 | 0.7 P/F-85 | 0.6 P/F-85 |
|---|---|---|---|---|---|---|---|
| Hardness (g) | 1 | 795.13±90.31 | 745.51±90.23 | 640.07±70.08 | 874.78±66.63 | 821.63±41.81 | 769.21±55.05 |
| | 45 | 682.64±51.53 | 544.34±62.67 | 599.31±86.78 | 837.06±64.39 | 756.21±64.24 | 719.82±80.09 |
| | 90 | 606.72±82.05 | 435.17±79.57 | 486.60±63.04 | 627.94±51.08 | 615.64±16.78 | 647.29±64.54 |
| Adhesiveness (g.mm) | 1 | -113.09±33.25 | -77.79±24.80 | -60.57±19.88 | -121.01±48.83 | -82.73±6.56 | -79.67±11.89 |
| | 45 | -71.15±9.98 | -64.79±6.34 | -53.66±18.03 | -86.31±19.90 | -69.65±7.71 | -60.27±18.04 |
| | 90 | -48.50±11.57 | -46.57±8.44 | -40.91±7.75 | -64.87±9.04 | -56.04±15.97 | -54.51±21.19 |
| Springiness (mm) | 1 | 0.69±0.15 | 0.75±0.13 | 1.16±0.48 | 0.59±0.20 | 0.71±0.15 | 1.08±0.59 |
| | 45 | 0.78±0.10 | 0.75±0.09 | 0.72±0.15 | 0.94±0.36 | 1.08±0.58 | 0.81±0.11 |
| | 90 | 0.88±0.40 | 0.82±0.41 | 0.69±0.18 | 1.13±0.65 | 0.92±0.46 | 0.74±0.23 |
| Chewiness (g.mm) | 1 | 122.86±14.88 | 109.90±22.71 | 105.80±35.73 | 140.06±33.79 | 130.66±13.92 | 125.82±35.74 |
| | 45 | 106.02±16.45 | 100.90±22.14 | 99.49±15.00 | 111.89±41.27 | 108.92±17.35 | 107.67±31.78 |
| | 90 | 97.07±30.76 | 94.72±26.23 | 91.27±23.15 | 102.99±4.39 | 96.71±31.90 | 86.68±15.17 |

Values are the means ± standard deviations. * 0.8 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.8, 0.7 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.7, 0.6 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.6, 0.8 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.8, 0.7 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.7, 0.6 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.6.

The increasing heat treatment temperature increased the hardness and chewiness values of the UF white cheese samples. Aydemir and Kurt [37] observed that the highest hardness value was determined in white cheeses produced from milk heated at 85°C for 5 min, followed by white cheeses made from milk at 75°C for 5 min and at 65°C for 20 min, respectively. The authors attributed this to the fact that intramolecular and intermolecular interactions of casein occur more intensely with higher heat treatment temperatures. Moreover, the percentage of denatured protein in cheese increases with temperature, which leads to an improvement in the overall cheese structure. In the UF white cheese samples, chewiness demonstrated a similar trend to hardness, which was in accordance with the study of Jia et al. [38]. The results of the ANOVA analysis showed that heat treatment temperature and storage time had no significant effect on the springiness of the UF white cheeses (Table 5), which may be because of the constant and high moisture content of the samples [39]. The hardness and chewiness of the UF white cheeses decreased during storage. Aydemir and Kurt [37] and Fathollahi et al. [40] explained the decrease in hardness value caused by protein proteolysis in white cheeses during storage.

Table 5. The effects of the P/F ratio, heat treatment temperature, and storage time on some textural properties of the UF white cheese

| Experimental factors | Hardness (g) | Adhesiveness (g.mm) | Springiness (mm) | Chewiness (g.mm) |
|---|---|---|---|---|
| P/F ratio | *** | *** | ns | ns |
| 0.8 | 737.36±122.74 a* | -84.16±37.00 b | 0.85±0.42 a | 113.48±30.37 a |
| 0.7 | 653.06±147.21 b | -66.27±18.23 a | 0.83±0.36 a | 106.97±26.05 a |
| 0.6 | 644.89±114.00 b | -58.27±20.22 a | 0.87±0.35 a | 102.79±30.34 a |
| Heat-treatment temperature (°C) | *** | ** | ns | * |
| 75 | 615.05±131.81 b | -64.12±27.17 a | 0.82±0.30 a | 103.11±25.58 b |
| 85 | 741.82±166.49 a | -75.01±29.04 b | 0.89±0.36 a | 112.38±31.98 a |
| Storage time (day) | *** | *** | ns | *** |
| 1 | 773.49±98.01 a | -89.15±35.00 c | 0.83±0.36 a | 122.52±30.14 a |
| 45 | 691.93±118.48 b | -67.64±17.46 b | 0.85±0.31 a | 105.82±26.19 b |
| 90 | 569.89±101.64 c | -51.90±15.26 a | 0.88±0.44 a | 94.91±24.47 b |

Values are the means ± standard deviations; different letters for each parameter in a column show significant differences using Duncan's multiple range test (P<0.05). Significant effects at * P<0.05, ** P<0.01, ***P<0.001. ns; not significant.

The color parameters, such as L* (lightness), a* (+redness/-greenness), and b* (+yellowness/-blueness), and ΔE* (total color differences) values [41] are shown in Table 6. The highest L*, a*, b*, and ΔE* values were found in the UF white cheeses manufactured from the UF retentate with a P/F of 0.6. Sánchez-Macías et al. [42] also determined that during 28 days of ripening, the L*, a*, and b* values of the full-fat goat cheeses were higher than those of reduced-fat and low-fat goat cheeses. An increasing fat content leads to lightness due to the high light scattering ability of fat globules [43]. In cheese, light penetrates the outer layers and is dispersed by milk fat globules and the boundaries of whey pockets [44]. According to the ΔE value ranges,

the color difference between samples can be estimated as recognizable (1.5-3.0) and well visible (3.0-6.0) [18]. In the current study, ΔE* values were found above 6.0 in all UF white cheeses, which is noticeable to the consumer.

Table 6. Color characteristics of the UF white cheese

| Parameters | Storage (day) | 0.8 P/F-75 | 0.7 P/F-75 | 0.6 P/F-75 | 0.8 P/F-85 | 0.7 P/F-85 | 0.6 P/F-85 |
|---|---|---|---|---|---|---|---|
| L* | 1 | 96.54±0.45 | 96.07±0.80 | 97.15±1.20 | 97.20±0.48 | 97.46±0.80 | 98.34±0.52 |
| | 45 | 95.45±0.37 | 95.02±1.25 | 95.80±0.66 | 96.45±0.27 | 96.52±0.38 | 96.56±0.41 |
| | 90 | 94.71±0.78 | 94.87±0.24 | 94.94±0.66 | 94.57±2.09 | 94.51±2.16 | 94.64±2.04 |
| a* | 1 | -0.35±0.19 | -0.35±0.16 | -0.30±0.11 | -0.77±0.20 | -0.64±0.05 | -0.51±0.05 |
| | 45 | -0.29±0.11 | -0.20±0.12 | -0.18±0.10 | -0.68±0.12 | -0.64±0.09 | -0.54±0.06 |
| | 90 | -0.19±0.14 | -0.10±0.13 | -0.06±0.05 | -0.60±0.23 | -0.43±0.13 | -0.41±0.08 |
| b* | 1 | 8.02±0.20 | 8.30±0.29 | 8.45±0.19 | 7.81±0.41 | 8.19±0.35 | 8.23±0.05 |
| | 45 | 8.69±0.27 | 8.84±0.27 | 8.84±0.19 | 7.78±0.36 | 8.23±0.26 | 9.44±0.72 |
| | 90 | 8.97±0.57 | 9.97±0.87 | 10.00±0.38 | 8.25±0.15 | 9.21±0.42 | 10.13±0.91 |
| ΔE* | 1 | 10.84±0.20 | 11.08±0.25 | 11.41±0.32 | 10.76±0.35 | 11.20±0.20 | 11.43±0.13 |
| | 45 | 11.42±0.26 | 11.52±0.34 | 11.59±0.18 | 10.61±0.37 | 11.06±0.24 | 12.25±0.71 |
| | 90 | 11.71±0.61 | 12.68±0.17 | 12.74±0.40 | 11.19±0.15 | 12.14±0.58 | 13.00±1.05 |

Values are the means ± standard deviations. * 0.8 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.8, 0.7 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.7, 0.6 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.6, 0.8 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.8, 0.7 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.7, 0.6 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.6.

As seen in Table 7, high heat treatment temperatures caused an increase in L* values and a decrease in the a* and b* values of the UF white cheeses. Darnay et al. [22] compared the color parameters of cheese made from raw or pasteurized (72°C, 15 s) buffalo milk and observed higher L* values and lighter yellow color in buffalo cheese made from pasteurized milk. The color properties of cheeses may be related to the changes in milk during heat treatment. As an example, the larger size of casein micelles during heat treatment results in milk whitening, which subsequently influences reflectance and reduces the yellow index [45]. The storage time was found to be significantly effective on the L*, a*, b*, and ΔE* values of the UF white cheese samples (P<0.001). Although lipolysis and amounts of carotenoid compounds were not measured in this study, alteration of color parameters in cheeses during storage may be due to lipolysis of fats and oxidation of carotenoids. Sabbagh et al. [46] also noticed a decrease in the L* value, probably due to the degree of lipolysis during the storage period. Moreover, Sabetsolat et al. [2] explained that the color alterations in the cheese samples are likely attributable to the oxidation of the carotenoid compounds that contribute to red color during storage.

Table 7. The effects of the P/F ratio, heat treatment temperature, and storage time on color parameters of the UF white cheese

| Experimental factors | L* | a* | b* | ΔE* |
|---|---|---|---|---|
| P/F ratio | * | *** | *** | *** |
| 0.8 | 95.81±1.38 ba | -0.48±0.27 c | 8.25±0.57 c | 11.09±0.53 c |
| 0.7 | 95.73±1.54 b | -0.39±0.24 b | 8.79±0.79 b | 11.63±0.77 b |
| 0.6 | 96.23±1.67 a | -0.33±0.19 a | 9.18±0.89 a | 12.07±0.85 a |
| Heat treatment temperature (°C) | * | *** | *** | ns |
| 75 | 95.61±1.11 b | -0.22±0.16 a | 8.89±0.77 a | 11.68±0.75 a |
| 85 | 96.24±1.83 a | -0.57±0.17 b | 8.54±0.90 b | 11.52±0.90 a |
| Storage time (day) | *** | *** | *** | *** |
| 1 | 97.12±1.04 a | -0.48±0.22 c | 8.16±0.34 c | 11.12±0.36 c |
| 45 | 95.96±0.88 b | -0.42±0.23 b | 8.63±0.65 b | 11.43±0.64 b |
| 90 | 94.70±1.55 c | -0.29±0.24 a | 9.42±0.91 a | 12.25±0.93 a |

Values are the means ± standard deviations; different letters for each parameter in a column show significant differences using Duncan's multiple range test (P<0.05). Significant effects at * P<0.05, ** P<0.01, ***P<0.001. ns; not significant.

Table 8 shows the scores of panelists for the UF white cheeses during 90 days of storage. Farah et al. [47] reported that acceptance of food depends mostly on three attributes: appearance, taste, and texture, and is independent of the nutritional content of the food. The effect of the P/F ratio and heat treatment temperature on the appearance, texture, and taste scores of the UF white cheese samples was not significant (P>0.05), whereas the lowest overall acceptability scores were found in the samples manufactured from the UF retentate with a P/F of 0.8 (Table 9). Sánchez-Macías et al. [42] demonstrated that most of the 50 untrained consumers were not accepting of lower-fat goat cheese because of its hard, dry, and rough texture. Besides,

Kaczyński et al. [48] showed that the fat content, which affects the texture and color of cheeses, determines its acceptability.

Table 8. Sensory properties of the UF white cheese

| Parameters | Storage (day) | 0.8 P/F-75* | 0.7 P/F-75 | 0.6 P/F-75 | 0.8 P/F-85 | 0.7 P/F-85 | 0.6 P/F-85 |
|---|---|---|---|---|---|---|---|
| Appearance | 1 | 3.79±1.40 | 3.77±1.30 | 3.63±1.43 | 3.78±1.50 | 3.73±1.55 | 3.67±1.54 |
| | 45 | 3.20±0.94 | 3.33±1.34 | 3.34±1.27 | 3.23±0.95 | 2.69±0.85 | 3.03±0.96 |
| | 90 | 3.92±1.54 | 4.00±1.25 | 3.87±1.50 | 4.65±1.36 | 4.14±1.36 | 3.81±1.20 |
| Texture | 1 | 3.36±0.98 | 3.55±0.81 | 3.37±0.78 | 3.38±0.80 | 3.15±0.80 | 3.51±0.70 |
| | 45 | 3.29±0.98 | 3.69±0.70 | 3.79±0.84 | 3.64±0.81 | 3.45±0.76 | 3.31±0.80 |
| | 90 | 3.51±0.82 | 3.34±0.84 | 3.70±0.72 | 3.74±0.86 | 3.70±0.81 | 3.68±0.92 |
| Taste | 1 | 2.73±0.71 | 3.03±0.84 | 3.31±0.82 | 2.99±0.85 | 3.06±0.83 | 3.33±0.61 |
| | 45 | 2.53±0.61 | 2.78±0.63 | 2.99±0.55 | 2.98±0.85 | 2.71±0.64 | 2.78±0.70 |
| | 90 | 3.52±0.96 | 3.44±0.78 | 3.59±0.85 | 3.35±0.80 | 3.22±0.62 | 3.22±0.69 |
| Overall acceptability | 1 | 3.97±1.45 | 3.63±1.83 | 4.40±1.72 | 3.73±1.98 | 3.83±1.73 | 4.23±1.78 |
| | 45 | 4.50±1.37 | 3.88±1.80 | 4.13±1.22 | 3.94±1.71 | 4.06±1.43 | 4.25±1.25 |
| | 90 | 2.44±1.79 | 3.24±1.82 | 3.40±2.00 | 3.32±1.67 | 4.80±1.55 | 4.32±1.62 |

Values are the means ± standard deviations. * 0.8 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.8, 0.7 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.7, 0.6 P/F-75: UF white cheese manufactured from UF retentate heat treated at 75°C and having a P/F ratio of 0.6, 0.8 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.8, 0.7 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.7, 0.6 P/F-85: UF white cheese manufactured from UF retentate heat treated at 85°C and having a P/F ratio of 0.6.

Overall acceptability scores increased when the heat treatment temperature was increased from 75 to 85°C (Table 9). Similar to our results, Frau et al. [49] demonstrated that spreadable cheese obtained from milk pasteurized at 75°C had higher general acceptance scores than those obtained from milk pasteurized at 65°C because of the change in the water-holding capacity of protein. Miloradoviç et al. [50] found that as the heat treatment temperature of milk increased, the sensory acceptability in cheeses increased and reported that after high heat treatment, components of the milk fat globule membrane engaging with milk proteins, which resulted in the microstructure of cheese produced from milk subjected to high heat treatment having fewer spherical voids. The storage time significantly affected the appearance, taste, and overall acceptability scores of the UF white cheeses. The appearance and taste scores of the UF white cheese samples decreased from day 1 to day 45 and then increased until day 90. The overall acceptability scores increased on the 45th day and then decreased at the end of the storage period (Table 9). The research conducted by Soltani et al. [28] demonstrated no correlation among appearance scores, texture scores, and total scores noted in UF cheeses during storage time. Al-Otaibi and Wilbey [39] reported that consumers' appreciation of UF cheeses on a hedonic scale did not change during 12 weeks of storage and explained that the breakdown of proteins and release of proteolysis products at moderate levels may cause the formation of an acceptable taste in cheeses.

Table 9. The effects of the P/F ratio, heat treatment temperature, and storage time on sensory properties of the UF white cheese

| Experimental factors | Appearance scores | Texture scores | Taste scores | Overall acceptability scores |
|---|---|---|---|---|
| P/F ratio | ns | ns | ns | * |
| 0.8 | 3.87±1.45 a* | 3.54±0.87 a | 3.20±0.94 a | 3.35±1.93 b |
| 0.7 | 3.69±1.38 a | 3.46±0.82 a | 3.07±0.78 a | 3.88±1.78 a |
| 0.6 | 3.61±1.39 a | 3.56±0.80 a | 3.25±0.76 a | 4.14±1.69 a |
| Heat-treatment temperature (°C) | ns | ns | ns | *** |
| 75 | 3.73±1.40 a | 3.54±0.83 a | 3.24±0.89 a | 3.54±1.90 b |
| 85 | 3.72±1.43 a | 3.50±0.83 a | 3.10±0.76 a | 4.04±1.73 a |
| Storage time (day) | *** | ns | *** | ** |
| 1 | 3.75±1.47 a | 3.43±0.82 a | 3.20±0.85 a | 3.77±1.91 ba |
| 45 | 3.13±1.08 b | 3.52±0.84 a | 2.79±0.68 b | 4.12±1.49 a |
| 90 | 4.08±1.40 a | 3.63±0.83 a | 3.39±0.80 a | 3.60±1.90 b |

Values are the means ± standard deviations; different letters for each parameter in a column show significant differences using Duncan's multiple range test (P<0.05). Significant effects at * P<0.05, ** P<0.01, ***P<0.001. ns; not significant.

## CONCLUSION

The findings of this study indicated that the P/F ratio of UF retentate and the temperature of applied heat treatment to UF retentate led to substantial differences in the physicochemical and sensory properties of the UF white cheese. The decreasing P/F ratio and heat treatment temperature decreased the hardness value

and increased the adhesiveness, a*, and b* values in the UF white cheese. The overall acceptability scores were lower for the UF white cheeses, which were manufactured from the UF retentate heat-treated at 75°C for 15 s and had a P/F of 0.8. In conclusion, when the physicochemical and sensory properties of the UF white cheese are taken into account, experiments conducted within the scope of this study showed that the UF retentate, which was heat treated at 85°C for 15 s and has a P/F ratio of 0.7, is more suitable for use in the manufacture of UF white cheese.

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**Research Paper / Araştırma Makalesi**

# Quality Characteristics of Cookies Made with Red Rice Flour Composite Flour

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## ABSTRACT

Red rice flour (RRF) is renowned for its nutritional richness, particularly in terms of total dietary fiber and essential minerals. This flour is derived from red rice, a variety of rice known for its distinctive red husk and bran layer. The study aimed to identify the best formulation through sensory evaluation, determine nutritional composition, physical characteristics, and assess shelf life compared to a control cookie. Five formulations of red rice cookies were prepared with RRF substitution levels ranging from 20% to 100%. The F2 red rice cookie with 40% RRF substitution was chosen as the best formulation based on sensory evaluation. It showed similarities to the control cookie in terms of sensory attributes. Nutritional analysis revealed lower moisture and crude protein content but higher crude fiber and total dietary fiber in the F2 compared to the control. Physical analysis showed lower hardness and different color characteristics for the F2. Consumer study results indicated high acceptability for the F2 red rice cookie. In conclusion, this study offers a promising strategy to improve the nutritional profile of cookies by boosting their dietary fiber content, effectively addressing a common deficiency observed in cookies made with conventional wheat flour

**Keywords:** Cookies, Pigmented rice, Rice flour, Sensory evaluation, Proximate composition

## Kırmızı Pirinç Unu Karışımıyla Yapılan Kurabiyelerin Kalite Özellikleri

## ÖZ

Kırmızı pirinç unu (KPU), özellikle toplam diyet lifi ve temel mineraller açısından besin zenginliği ile tanınır. Bu un, kendine özgü kırmızı kabuk ve kepek tabakasıyla bilinen bir pirinç çeşidi olan kırmızı pirinçten elde edilir. Çalışma, duyusal değerlendirme yoluyla en iyi formülasyonu belirlemeyi, besin bileşimini, fiziksel özellikleri incelemeyi ve kontrol kurabiyesine kıyasla raf ömrünü değerlendirmeyi amaçlamıştır. Kırmızı pirinç kurabiyelerinin beş farklı formülasyonu, %20 ile %100 arasında değişen KPU ikame seviyeleriyle hazırlanmıştır. Duyusal değerlendirme sonuçlarına göre, %40 KPU ikamesi ile hazırlanan F2 kırmızı pirinç kurabiyesi en iyi formülasyon olarak seçilmiştir. Duyusal özellikler açısından kontrol kurabiyesine benzerlik göstermiştir. Besin değeri analizi, F2'nin kontrol kurabiyesine kıyasla daha düşük nem ve ham protein içeriğine, ancak daha yüksek ham lif ve toplam diyet lifi içeriğine sahip olduğunu ortaya koymuştur. Fiziksel analiz, F2'nin daha düşük sertlik ve farklı renk özelliklerine sahip olduğunu göstermiştir. Tüketici çalışması sonuçları, F2 kırmızı pirinç kurabiyesinin yüksek kabul gördüğünü göstermiştir. Sonuç

olarak, bu çalışma, geleneksel buğday unu ile yapılan kurabiyelerde yaygın olarak görülen diyet lifi eksikliğini etkili bir şekilde ele alarak, kurabiyelerin besin profilini iyileştirmek için umut verici bir strateji sunmaktadır.

**Anahtar Kelimeler:** Kurabiye, Pigmentli pirinç, Pirinç unu, Duyusal değerlendirme, Yaklaşık bileşim

## INTRODUCTION

Cookies are a popular convenience snack favored by individuals with busy lifestyles due to their grab-and-go nature and extended shelf life compared to other baked goods [1, 2]. Cookies provide significant amounts of nutrients such as minerals, protein, fiber, and some vitamins, contributing to our dietary intake and meeting daily nutritional needs [3, 4]. However, traditional cookies, primarily composed of wheat flour, eggs, sugar, and butter, are often considered unhealthy due to their high starch content, low dietary fiber, and gluten intolerance issues [5, 6].

In efforts to develop healthier cookies with enhanced nutritional and textural attributes, various studies have explored the substitution of wheat flour with different rice flour, offering gluten-free options with added nutritional benefits [7-10]. Bolarinwa et al. [5] demonstrated that a blend formulation of germinated brown rice flour and potato starch yielded quality gluten-free cookies. Similarly, Klunklin and Savage [11] investigated the effects of substituting purple rice flour for wheat flour in biscuits, resulting in improved nutritional properties and reduced starch digestibility. Pigmented rice varieties, including black, purple, and red rice, are rich in pigments and nutrients deposited in their bran layers [12].

This research focuses on utilizing red rice flour (RRF) as a substitute for wheat flour in various ratios to develop healthier cookies. Red rice, characterized by its red bran layer, is rich in iron, fiber, vitamins, and other essential nutrients [13-15]. Previous studies have shown that pigmented rice varieties, including red rice, have higher dietary fiber content than non-pigmented rice [16, 17]. However, the substitution of RRF in cookies is expected to alter their textural qualities due to the higher content of dietary fiber [18].

The lack of awareness and limited market access for pigmented rice pose challenges to its production and consumption. Moreover, there is limited research on the nutritional composition of pigmented rice in Sabah, Malaysia, where upland rice cultivation predominates due to geographical constraints [19]. Addressing these gaps, this study aims to produce cookies from RRF to increase market awareness of pigmented rice in Sabah, promote its cultivation, and contribute to achieving self-sufficiency in rice production as outlined in the National Agrofood Policy (NAP) 2021-2030. By producing cookies from RRF, this study aims to align with NAP 2.0's objectives of strengthening the domestic market, developing demand-driven products, and capitalizing on local specialty rice varieties to drive growth in the agri-food sector. Furthermore, this research responds to changing consumer dietary preferences by offering healthier, nutrient-rich cookie options while showcasing the unique attributes of pigmented rice to the consumer.

## MATERIALS and METHODS

### Raw Materials

The raw materials used in preparing the cookies included all-purpose flour (Bake with Yen, 9.8% protein content, 11% moisture content), red rice (Pasar Besar, Kota Kinabalu), unsalted butter (Ausicows, Bake with Yen), granulated sugar (Central Sugar Refinery, Bake with Yen), icing sugar (Namye, Bataras), eggs, and salt (E&G Food Ingredient, 99 Speedmart).

### Preparation of Red Rice Flour

The procedure for preparing RRF was adapted from Bolarinwa et al. [5]. Initially, red rice grains were thoroughly washed and cleaned under tap water for five minutes to ensure the removal of dust and dirt particles. After draining, rice grains were dried in a drying cabinet (Thermoline Scientific TD-78T-SD, Australia) at 40°C for a minimum of 12 hours until the moisture content reached below 10%. Once dried, red rice grains were ground into RRF using a Waring blender (Panasonic MX-898, Malaysia) at low speed for five minutes, with intermittent scraping of residue from the blender walls at one-minute intervals. Subsequently, RRF was sifted using a sieve shaker (Endecotts Ltd., UK) to achieve a consistent particle size of less than 250 µm. Figure 1 depicts the photographs of RRP after washing, drying and milling processes.


Figure 1. The photographs of RRP after washing, drying and milling processes

### Formulation of Cookies

The cookie formulation incorporating RRF was adapted from Klunklin and Savage [11] with minor adjustments. The experimental cookies were prepared by replacing all-purpose flour with RRF at various levels: 20%, 40%, 60%, 80%, and 100% of the total flour weight. Table 1 presents the formulation of cookies substituted with RRF.

Table 1. Rice flour cookies formulations

| Ingredient | Control | F1 | F2 | F3 | F4 | F5 |
|---|---|---|---|---|---|---|
| All-purpose flour (%) | 100 | 80 | 60 | 40 | 20 | 0 |
| Red rice flour (%) | 0 | 20 | 40 | 60 | 80 | 100 |
| Butter$^{+}$ | 50 | 50 | 50 | 50 | 50 | 50 |
| Granulated sugar$^{+}$ | 30 | 30 | 30 | 30 | 30 | 30 |
| Icing sugar$^{+}$ | 20 | 20 | 20 | 20 | 20 | 20 |
| Egg$^{+}$ | 20 | 20 | 20 | 20 | 20 | 20 |
| Salt$^{+}$ | 1 | 1 | 1 | 1 | 1 | 1 |

$^{+}$Baker's percentage

### Preparation of Cookies

The procedure for preparing red rice cookies was adapted from Zouari et al. [20] with slight modifications. Firstly, the butter was softened at room temperature and creamed with granulated sugar and icing sugar using a kitchen mixer (Panasonic, Malaysia) until smooth and pale in color. Beaten egg liquid was then added gradually, ensuring thorough mixing between each addition. Sifted flour and salt were gently folded into the mixture to prevent gluten formation. The dough was rolled out to a thickness of 6 mm, chilled in the freezer for 20 minutes, and then cut into shapes using a 5cm diameter cookie cutter. The cookies were baked in a commercial oven (Sinmag, Malaysia) at 150°C for 15 minutes, cooled on a wire rack for 30 minutes, and finally stored in zip-lock polyethylene bags at room temperature before analysis.

### Sensory Evaluation

The sensory evaluation of the cookies was conducted at the Sensory Laboratory within the Faculty of Food Science and Nutrition, UMS, Malaysia. Panellists were recruited from the faculty staff and students through advertisements, and all participants provided informed consent. To ensure anonymity, the cookies were coded with three-digit numbers, permutated, and randomly served to panellists on trays. The panel evaluated the six formulations, with a total of 60 untrained panellists participating in the hedonic tests. Panellists assessed product attributes including appearance, color, aroma, taste, crispness, and overall acceptability using a nine-point hedonic scale ranging from 1 ("dislike extremely") to 9 ("like extremely") [21]. Sensory evaluation was conducted using printed questionnaires

### Proximate Composition, Dietary Fibre and Total Energy

The proximate analysis of the samples was conducted according to the AOAC method [22] to determine their moisture, crude protein, crude fat, crude fiber, and ash content. Moisture content was determined by subjecting the samples to oven drying at 105°C until a constant weight was achieved. Crude protein content was assessed using Kjeldhal method (Kjeltex System-Texator), where the nitrogen value was converted to protein using a factor of 6.25. The crude fat content was determined using the Soxhlet system (Soxtec System-Texator), while carbohydrate content was calculated using the difference method. Ash content was determined by dry-ashing the samples in a furnace at 550°C for 24 hours. Total dietary fiber was evaluated using a Megazyme TDF kit [23]. The calorific content (kcal/100g) of the samples was calculated by multiplying the crude protein, crude fat, and available carbohydrate contents by factors of 4, 9, and 4, respectively.

### Hardness Measurement

The hardness test to assess the breaking strength of the cookies was adapted from the methodology outlined by Jauharah et al. [24]. For this test, a texture analyzer (Stable Micro Systems, UK) was equipped with a 3-point bending rig (HDP/3PB) was utilized, operating at specific parameters: a pre-test speed of 1mms$^{-1}$, a test speed of 3mms$^{-1}$, with force measured at a distance of 5 mm and a trigger force of 50g was set to ensure accurate measurement of the cookies' hardness.

### Color Analysis

The color characteristics of the cookies were assessed using a colorimeter (Hunterlab ColorFlex EZ, Reston) following the method described by Nielsen [25]. The Hunter color solid measures three dimensions: *L, a,* and *b.* The *L* value represents lightness, where a value of 100 indicates white color and a value of 0 indicates dark color. The *a* value indicates the red (+) or green (−) coordinate, while the *b* value indicates the yellow (+) or blue (−) coordinate.

### Aspect Ratio and Bulk Density Determination

The aspect ratio of the cookies was determined using the length or diameter and the width or thickness of the cookies, following the methods described by AACC [23]. The bulk volume of the cookies was measured using the green beans displacement method, adapted from Mir et al. [26].

### Consumer Study

In this study, 100 respondents were randomly selected from the general public, representing users or frequent users of the product. The consumer study was conducted at Padang Merdeka Kota Kinabalu (Sabah, Malaysia). Participants were asked to evaluate sensory attributes such as color, aroma, taste, texture, and overall acceptability.

### Statistical Analysis

The experimental data collected from the nine-point hedonic test for the best formulation of red rice cookies were statistically analyzed using a one-way ANOVA with a completely randomized design. The mean values obtained were further analyzed using Tukey's honestly significant difference (HSD) test for a multicomparison of means. A t-test was used to analyze the results of the two samples. A significance level of $p<0.05$ was applied. Frequency analysis was utilized to analyze the data obtained from the consumer study. All statistical analyses were conducted using SPSS Statistics version 28.

## RESULTS and DISCUSSION

### Sensory Evaluation

Table 2 shows the sensory characteristics of the control cookie and different formulations of red rice cookies. Cookies achieving a score of 5 or higher were deemed acceptable, as noted by Yildiz and Gocmen [27], while a mean score of 7 or above typically indicates highly satisfactory sensory quality, making it a reliable benchmark for the "target" attribute [28]. Color plays a pivotal role in food acceptability and palatability [29], significantly influencing consumer preference and purchase decisions for baked goods [30]. Consumers often gauge food quality based on its color, which forms their initial impression and serves as a quality indicator [31].

Table 2. Sensory characteristics of control cookies and different formulations of red rice cookies

| Attributes | Control | F1 | F2 | F3 | F4 | F5 |
|---|---|---|---|---|---|---|
| Color | 7.11±2.14$^{a}$ | 6.79±2.01$^{a}$ | 6.57±1.87$^{a}$ | 6.48±1.83$^{a}$ | 6.41±2.01$^{a}$ | 6.16±2.01$^{a}$ |
| Aroma | 6.56±2.17$^{a}$ | 6.67±2.07$^{a}$ | 6.63±1.91$^{a}$ | 6.48±2.04$^{a}$ | 6.19±1.93$^{a}$ | 6.22±1.97$^{a}$ |
| Taste | 7.22±1.94$^{a}$ | 6.90±1.94$^{ab}$ | 7.02±2.07$^{a}$ | 6.84±2.15$^{abc}$ | 5.84±2.26$^{bc}$ | 5.79±2.20$^{c}$ |
| Crispness | 7.16±2.02$^{a}$ | 7.10±2.04$^{a}$ | 7.21±1.83$^{a}$ | 6.78±1.98$^{ab}$ | 5.89±2.28$^{bc}$ | 5.70±2.15$^{c}$ |
| Overall acceptability | 7.11±1.86$^{a}$ | 6.90±1.88$^{a}$ | 7.00±1.92$^{a}$ | 6.75±2.03$^{ab}$ | 5.86±2.10$^{b}$ | 5.87±2.18$^{b}$ |

Data expressed as mean ± standard deviation, (n=63). Mean values in the same row with different superscripts are significantly different with $p<0.05$. Sample details: A = Control, 100% wheat flour; B = 20% substitution red rice flour, C = 40% substitution red rice flour, D = 60% substitution red rice flour, E = 80% substitution red rice flour, F = 100% substitution red rice flour.

Based on the mean scores for color presented in Table 2, no notable differences were found in color acceptability between the control cookie and any of the red rice cookie samples. This differs from Klunklin and Savage's [11] findings, where substituting up to 50% resulted in significant color variation. However, in our investigation, even with a complete substitution of RRF, color acceptability remained unchanged. The absence of a distinct red hue in the natural beige color of RRF could indeed contribute to this observation. Washing raw rice grains before processing is a common practice for hygiene, but it also removes aleurone layers containing red pigments like anthocyanins and proanthocyanins [32]. Thus, the resulting RRF may closely resemble unbleached white flour in color. Consequently, it can be inferred that RRF substitution does not significantly alter cookie color acceptability.

Aroma, along with taste, texture, color, and warmth, significantly influences food acceptance and appreciation [33]. However, based on the mean scores obtained for aroma in Table 2, RRF substitution levels from 20% to 100% did not significantly affect aroma acceptability compared to the control cookie. This indicates high acceptability among panelists for various levels of wheat flour substitution with RRF. This contrasts with Klunklin and Savage's [11] study, where all levels of purple rice flour substitution (20%, 40%, 60%, 80% and 100%) resulted in significant aroma differences compared to the control wheat cookie. The lack of aroma impact in our study may stem from the aromatic compound content in RRF itself. Processing methods, including rice washing, roasting, milling, and storage, can affect aromatic volatile compounds in rice flour. Washing rice grains before drying and grinding significantly reduces volatile content [34]. Additionally, the removal of the lipid-rich bran layer during milling reduces lipid breakdown products, impacting aroma. Storage conditions can also alter rice aroma over time.

Taste is a critical factor in consumer acceptance, with the four primary taste qualities being sweet, salty, sour, and bitter [31]. In our study, formulations F4 and F5 showed a significant taste difference compared to the control sample and F2. However, these formulations received the lowest taste scores, indicating lesser preference among panelists. Formulations F1, F2, and F3 did not significantly differ from the control, with F2 being the closest to the control sample.

Crispness, describing cookie hardness, affects textural preference [35]. Mean scores for F4 and F5 were significantly lower than the control and F1 – F3, indicating undesired crispness. Additionally, crispness decreased significantly as RRF substitution levels exceeded 60%. This trend is consistent with previous studies on flour substitutions [20]. Overall acceptability reflects a blend of sensory attributes in a product [36]. Mean scores for overall acceptability decreased as RRF substitution levels exceeded 60%. However,

formulations F1, F2, and F3 did not significantly differ from the control, with F2 being the closest to the control sample.

### Selection of Best Formulation

Color, flavor, aroma, and texture, including hardness, crispiness, and dryness, are critical sensory attributes defining the quality of cookies [35]. Identifying the optimal formulation involves comparing it to the control cookie, aiming for similarity and achieving high mean scores across sensory attributes. Analysis of Figure 2 and preceding discussions reveals that formulation F2 of the red rice cookie closely resembles the control cookie in terms of taste, crispness, and overall acceptability. Notably, these attributes attained mean scores exceeding seven, indicating exceptionally satisfactory sensory quality. Additionally, feedback from the hedonic test highlighted that the cookie with a 40% substitution level was perceived as the most palatable by the majority of respondents. Therefore, based on these findings, formulation F2 with a 40% substitution level of RRF emerges as the optimal choice among the various substitution levels tested. Thus, formulation F2 was chosen for further evaluation through proximate analysis, physical assessment, and consumer study.

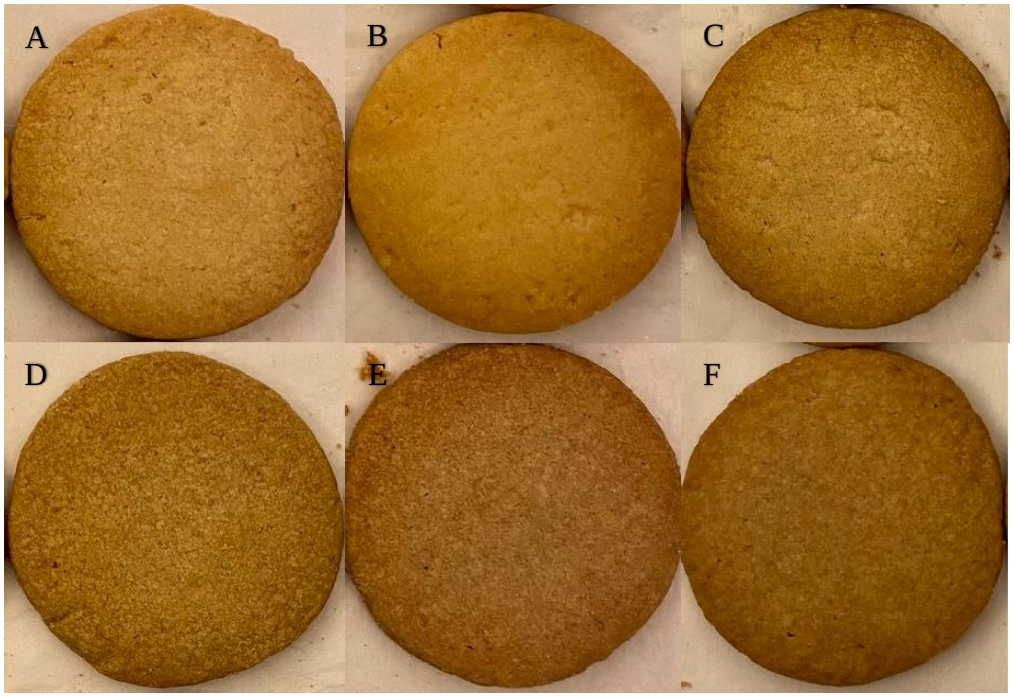


Figure 2. Cookies made of wheat flour and red rice flour (RRF) with different levels of substitution (A: control, B: 20% substitution RRF, C: 40% substitution RRF, D: 60% substitution RRF, E: 80% substitution RRF and F: 100% substitution RRF)

### Proximate Composition

Table 3 presents the nutritional compositions of red rice flour, the control cookie, and formulation F2. A notable finding from Table 3 is the significantly higher moisture content in the control cookie compared to the F2 red rice cookie. This disparity can be attributed to the inherently lower moisture content of RRF. Consequently, the reduced moisture content in F2 can be attributed to the characteristics of RRF itself. Contrary to the findings in this study, Klunklin and Savage [11] reported a linear increase in moisture content with higher concentrations of purple rice flour. This discrepancy could be due to differences in flour particle size, with coarser particles, as observed in RRF, possessing lower water-holding capacity. Additionally, the dryland soil conditions of upland rice cultivation may limit mineral availability, possibly contributing to the lower ash content in RRF [37]. Although it was anticipated that RRF would exhibit higher mineral content, including iron and potassium, the ash content did not significantly differ from that of refined wheat flour.

Moreover, the crude protein content in the control cookie was significantly higher than that of F2. This difference is attributable to the lower protein of the rice flour than wheat flour. Furthermore, the crude fat content did not significantly differ between the control cookie and F2, despite RRF exhibiting a higher fat content than refined wheat flour. Regarding crude fibre content, F2 displayed significantly higher levels compared to the control cookie. This discrepancy is consistent with the higher fibre content of RRF compared to refined wheat flour [37]. Furthermore, available carbohydrate content did not significantly differ between the control cookie and F2. However, RRF exhibited higher available carbohydrate content compared to refined wheat flour, aligning with previous research [38, 39]. This higher carbohydrate content in RRF may contribute to the carbohydrate content of F2.

Table 3. Nutritional compositions of red rice flour, control cookie and F2 red rice cookie

| Composition (%) | Red rice flour | Control | F2 |
|---|---|---|---|
| Moisture | 8.98±0.38 | 3.6±0.14[a] | 2.05±0.07[b] |
| Ash | 0.58±0.05 | 0.47±0.03[a] | 0.55±0.05[a] |
| Crude protein | 7.58±0.30 | 7.14±0.11[a] | 6.15±0.10[b] |
| Crude fat | 1.62±0.30 | 22.30±1.58[a] | 20.35±0.34[a] |
| Crude fibre | 2.15±0.11 | 1.13±0.09[b] | 1.90±0.11[a] |
| Total dietary fibre | 3.75±0.32 | 1.90±0.14[b] | 3.02±0.21[a] |
| Carbohydrate | 75.34±0.10 | 63.46±1.77[a] | 65.97±0.29[a] |
| Energy (kcal) | 353.81±2.53 | 486.86±7.03[a] | 477.69±2.25[a] |

Data expressed as mean ± standard deviation, (n=3). Mean values in the same row with different superscripts are significantly different with $p<0.05$. F2 = 4% substitution red rice flour.

## Total Dietary Fibre

The total dietary fiber content in the F2 was significantly higher compared to that of the control cookie (Table 3). These findings align with the results reported by Klunklin and Savage [11], where the substitution of purple rice flour significantly increased the total dietary fiber content in purple rice cookies compared to control wheat cookies. Dietary fiber encompasses the indigestible components of food, primarily derived from plant cell wall material and consisting mainly of polysaccharide molecules. Major constituents include cellulose, hemicelluloses, lignin, and other non-starch polysaccharides like pectin. According to AACC [23], dietary fiber refers to edible plant parts or similar carbohydrates that resist digestion and absorption in the small intestine, undergoing complete or partial fermentation in the large intestine. Adequate dietary fiber intake from diverse sources is associated with reduced colon cancer risk and helps maintain healthy blood lipid levels, lowering the likelihood of obesity, hypertension, and cardiovascular disease. Given that the total dietary fiber content in the F2 significantly exceeds the threshold required for a nutrition claim, consideration may be given to such a claim. As per the Fifth A Schedule (Regulation 18 c) of Malaysia Food Regulations 1985, products must contain at least 3g of total dietary fiber per 100g (solids) to claim a source of total dietary fiber. With the F2 red rice cookie containing 3.02g of total dietary fiber per 100g, it meets the criteria for making such a claim.

## Total Energy Content

There was insignificant difference in the energy contents between the control and the F2 red rice cookies (Table 3). Although not statistically significant, the slight decrease in energy content of the F2 red rice cookie could be attributed to its lower crude protein and crude fat content compared to the control cookie.

## Hardness

The texture of cookies, particularly their hardness, is a crucial aspect influenced by various factors [27]. As depicted in Table 4, the hardness was significantly greater for the control cookie compared to the F2. This aligns with findings from Klunklin and Savage [11], where incorporating different levels of purple rice flour resulted in a notable reduction in cookie hardness. Additionally, cookies substituted with coarse-grained short-grain rice exhibited decreased hardness compared to the control wheat cookie, as observed by Mancebo et al. [40]. Chung et al. [41] similarly reported that all cookies containing rice flour required less force to snap compared to the control cookie.

Several factors may contribute to the decreased hardness of substituted cookies. Firstly, changes in the dough matrices due to reduced gluten content could play a role. The hardness of cookies relies on the structure of the composite matrix comprising gluten protein aggregates, lipids, sugars, and ungelatinized starch granules. Therefore, substituting wheat flour with gluten-free rice flour reduces the gluten protein content in the dough, resulting in a less rigid composite matrix and decreased cookie hardness [41]. Secondly, the use of flours with different particle sizes could influence hardness. Research by Mancebo et al. [40] indicates that cookies made from fine-grained flour require higher peak force compared to those made with coarse-grained flour. While the RRF used in this study was ground and sieved to achieve a consistent particle size of <250µm, most cookie flours typically have an average particle size of around 50 µm, with fewer than 10% larger than 130 µm. Consequently, the coarser particle size of RRF used in baking F2 may contribute to their lower hardness compared to the finer wheat flour used in the control cookies.

## Color

Color plays a pivotal role in influencing consumer preferences and purchasing decisions when it comes to bakery products. In the case of the F2, notable differences were observed compared to the control cookie. The F2 exhibited significantly lower lightness, higher redness, and lower yellowness than the control cookie (Table 4). The lightness of a cookie is not only influenced by the Maillard reactions occurring during baking but also by the color of the flour used, as noted by Mancebo et al. [40]. The darkened color observed in cookies can result from caramelization of sugars in the recipe or the Maillard reaction, both of which contribute to the browning effect during baking. The incorporation of RRF in the cookie formulation may lead to a darker surface compared to control cookies, which typically exhibit higher lightness values. Moreover, the increased redness observed in the F2 could be attributed to the presence of red pigments naturally found in the husk of red rice. Conversely, the decrease in yellowness of the F2 may be due to the degradation of unstable yellow compounds present in RRF during the baking process.

Table 4. Physical characteristics of red rice cookie

| Parameter | Control | F2 |
|---|---|---|
| Hardness (N) | 35.07±0.37[a] | 28.45±0.61[b] |
| L* | 70.67±0.04[a] | 57.22±0.02[b] |
| a* | 10.05±0.03[a] | 13.42±0.01[b] |
| b* | 32.69±0.03[a] | 30.74±0.13[b] |
| Aspect ratio | 5.70±0.10[a] | 6.43±0.15[a] |
| Bulk density (g/mL) | 0.77±0.02[a] | 1.15±0.04[b] |

Data expressed as mean ± standard deviation, (n=3). Mean values in the same row with different superscripts are significantly different with $p<0.05$. F2 = 40% substitution red rice flour.

## Aspect Ratio

The aspect ratio of cookies serves as a crucial quality indicator, with higher values generally associated with desirable cookie characteristics, such as a larger diameter and thinner thickness [42]. The aspect ratio was notably higher for the F2 compared to the control cookie (Table 4). Similarly, Chung et al. [41] reported a significant increase in the aspect ratio of cookies with rice flour incorporation. Research by Mancebo et al. [41] revealed a positive correlation between particle size and aspect ratio (r= 0.75; 99%) and a negative correlation between aspect ratio and water holding capacity (WHC) (r= 0.47; 95%). Finely ground flours demonstrated higher water binding and holding capacities compared to coarse-grained flours. This suggests that flours with finer particle sizes tend to have higher WHC and water binding capacities, resulting in lower spread ratios. The spread ratio of cookies is also affected by dough viscosity. Flours possessing high water holding capacity WHC and water binding capacities tend to limit the availability of water for sugar dissolution in the dough, consequently resulting in elevated initial viscosity.

## Bulk Density

Referring to Table 4, the bulk density exhibited a significant increase in the F2 compared to the control cookie. This trend aligns with findings from other studies [43, 44], where bulk density showed a significant increase with higher formulations of jering seed flour. Similarly, research by Zouari et al. [20] indicated a significant increase in bulk density when wheat flour was substituted with sesame flour. Bulk density determination holds significance as it serves as a parameter for determining storage, transportation, and packaging considerations for cookies [45]. The variance in bulk density can be attributed primarily to differences in particle size and flour density [43].

## Consumers Study

To evaluate consumers' acceptance towards the 40% substitution red rice cookie, a consumer study was conducted. A total of 100 respondents were chosen randomly during the event, with which 55 of them were female, and 45 of them were male. The respondents collected consisted of a wide range of ethnic groups, which include Bisaya, Bajau, Chinese, Dusun, Iban, Lundayeh, Malay, Murut, Sea Dayak and Toraja. In the consumer study conducted, respondents were presented with a range of attributes to evaluate their preferences and perceptions regarding the red rice cookie with a 40% substitution rate. These attributes included color, aroma, taste, texture, and overall acceptability. A significant proportion of respondents, constituting over 80% of the total participants, exhibited a high level of acceptance toward the red rice cookie with 40% substitution (Figure 3). This indicates that the majority of individuals who participated in the study found this particular variation of the cookie to be appealing across multiple sensory dimensions. The high level of acceptance observed among participants underscores the favorable sensory characteristics and overall palatability of the red rice cookie with 40% substitution. These findings suggest that the incorporation of red rice flour into the cookie formulation at this specific substitution rate has successfully met or exceeded consumer expectations, eliciting positive responses across various attributes such as color, aroma, taste, texture, and overall acceptability.

## CONCLUSION

The study revealed that incorporating 40% RRF led to reductions in moisture and crude protein content while increasing crude fiber and total dietary fiber content in the cookies. Physical analysis showed significant changes in hardness, color, aspect ratio, and bulk density of the best formulation (F2) compared to the control. However, the sensory evaluation indicated no significant differences between the F2 red rice cookie and the control, suggesting that the 40% substitution of RRF did not negatively impact sensory characteristics according to the panelists. Through sensory evaluation, the red rice cookie with 40% substitution emerged as the best formulation, closely resembling the taste, crispness, and overall acceptability of the control cookie, with no significant differences observed in sensory attributes. The F2 received higher palatability ratings based on comments from the sensory evaluation forms. In conclusion, this study presents a promising approach to enhance the cookies' nutritional profile by increasing dietary fiber content, addressing the deficiency typically found in cookies made with wheat flour. However, further research is needed to optimize the formulation to reduce fat content and produce lower-fat cookies.

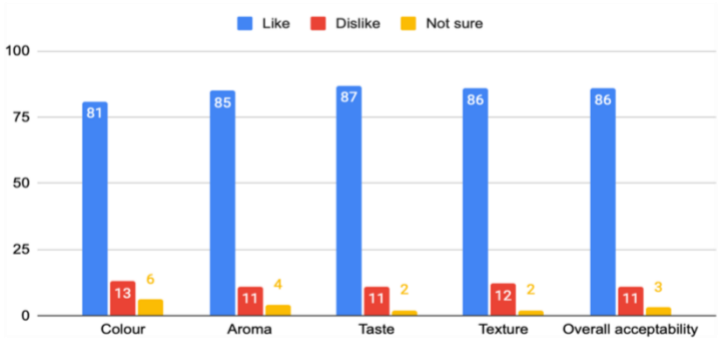


Figure 3. Consumers' acceptance towards sensory attributes of 40% substitution red rice cookie (n=100)

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**Research Paper / Araştırma Makalesi**

# Effect of Heat Treatment on Storage Stability of Sheep Tail Fat

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## ABSTRACT

In this study, the effects of various physical, chemical, and technological properties of sheep tail fat (fresh and ghee) as well as different storage durations, storage temperatures, and the use of additives on the storage stability of thermally processed sheep tail fat (ghee tail fat) were investigated. To prevent lipid oxidation and other degradation factors during use after long-term storage, salt and antioxidant additives were used. Samples were stored in glass jars at 25ºC, +4ºC, and -18ºC for 90 days. The moisture, protein, fat, and ash contents of fresh tail fat was determined as 11.2%, 3.47%, 85.0%, and 0.07%, respectively. In clarified fat, the moisture content was determined as 0.4%, protein content as 2.98%, fat content as 96.0%, and ash content as 0.05%. Additionally, the cholesterol content in tail fat was 60 mg/100 g in fresh tail fat and 58 mg/100 g in ghee tail fat. The pH values of fats were determined as 5.7 in fresh samples and 6.2 in ghee samples. The fatty acid compositions of samples revealed that the dominant fatty acid groups in both fresh and ghee sheep tail fat were palmitic, stearic, and oleic acids. Fresh tail fat stored at room temperature deteriorated in terms of chemical properties (FFA, peroxide, and TBA) within 7 days, while those stored at +4ºC deteriorated within 15 days. However, thermally processed samples were preserved without deterioration for 60 days at these storage temperatures.

**Keywords:** Sheep tail fat, Tallow, Animal fat, Storage stability

## Isıl İşlemin Koyun Kuyruk Yağının Depolama Stabilitesi Üzerine Etkisi

## ÖZ

Bu çalışmada, ısıl işlem görmüş koyun kuyruk yağının (sade) depolama stabilitesini belirlemek amacıyla, kuyruk yağının (taze ve sade) çeşitli fiziksel, kimyasal ve teknolojik özellikleri ile farklı depolama sürelerinin, depolama sıcaklıklarının ve katkı maddesi kullanımının bu özellikler üzerindeki etkileri araştırılmıştır. Uzun süreli depolamadan sonra, kullanım sırasında lipit oksidasyonu ve diğer bozulma faktörlerini önlemek için tuz ve antioksidan katkı maddesi kullanılmıştır. Örnekler cam kavanozlarda 25ºC, +4ºC ve -18ºC'de 90 gün boyunca depolanmıştır. Taze kuyruk yağında nem, protein, yağ ve kül içeriği sırasıyla %11.2, 3.47, 85.0 ve 0.07 olarak belirlenmiştir. Berraklaştırılmış yağda nem, protein, yağ ve kül oranları sırasıyla %0.4, 2.98, 96.0 ve 0.05 olarak belirlenmiştir. Ayrıca, kuyruk yağı kolesterol içeriği taze ve sade kuyruk yağlarında sırasıyla 60 ve 58 mg/100g olarak belirlenmiştir. Yağların pH değerleri taze örneklerde 5.7, sade örneklerinde ise 6.2 olarak belirlenmiştir. Örneklerin yağ asidi kompozisyonları incelenmiş ve taze ve sade koyun kuyruk yağında baskın yağ asidi gruplarının palmitik, stearik ve oleik asit olduğu belirlenmiştir. Oda sıcaklığında depolanan taze kuyruk yağları kimyasal özellikler (FFA, peroksit ve TBA) açısından 7 gün içinde bozulurken, +4ºC'de depolananlar ise 15 gün içinde bozuldu. Ancak ısıl işlem görmüş örnekler bu depolama sıcaklıklarında 60 gün boyunca bozulmadan muhafaza edilebilmiştir.

**Anahtar Kelimeler:** Koyun kuyruk yağı, İç yağ, Hayvansal yağ, Depolama stabilitesi

## INTRODUCTION

One of the products obtained from the slaughter of cattle and tailed sheep is tallow and tail fat. Sheep tail fat is the main source of flavoring, especially in the production of kebabs and some dishes. Animal fats are the second most important source of lipid raw materials produced in Turkey [1]. Fat-tailed sheep store fat in their tails for use when natural food sources are scarce [2, 3]. Sheep tail fat is used in kitchens in Turkey and Central Asian countries. In China, sheep tail fat functions as a food seasoning in cooking [4]. However, the use of sheep tail fat in the food industry is limited due to its not widely accepted flavor [5]. Sheep tail fat constitutes a large part of Turkey's animal fat production. It has been reported that as of 2022, there are approximately 44,700,000 sheep in Turkey, and most of them have fat-tails [6, 7]. The weight of sheep tail fat from individual sheep ranges from 3 to 8 kg [5]. It has also been reported that 21,500,000 sheep are slaughtered annually [6], according to a rough calculation, it can be calculated that 100,000 tons of tail tissue can be used as a source of lipid production.

The high ratio of unsaturated fatty acids in their structure causes lipids to be more susceptible to deterioration. Tail fat among animal fats is considered less risky in terms of health compared to tallow fats, since the content of unsaturated fatty acids is higher than other tallow fats [1, 8]. Animal fat can stabilize the three-dimensional network of dissolved myofibrillar proteins that increase the mouthfeel and tenderness of processed meat products. In addition, animal fat increases juiciness and yield by reducing water losses during cooking or ripening [9].

Fats with an average melting point of up to 45ºC are absorbed by the body at a rate of 95% or more. From this point of view, tail fat, which has a melting point below 45ºC, can be digested more easily by the body. Approximately 94% of the tail fat is lipid [10]. In this respect, tail fat can be considered as an important source of fat. The main fatty acids in tail fat are oleic acid (28.37-44.43%), palmitic acid (24.77-31.49%), stearic acid (16.51-30.02%), but also include different fatty acids such as margaric (4.32%), myristic (3.67-3.92%), palmitoleic (3.01-3.14%), linoleic (0.66-2.77%) and lauric acids (0.20%) [7, 11].

In addition to tail fat use as a natural lipid in dishes by melting in Turkey, it is widely used in products such as kebabs, döner, lahmacun, especially in terms of softening meat and providing flavor. Although it is thought to have a high value in terms of cholesterol, it is emphasized that it can prevent joint pain in later ages, so it should be consumed at young ages. However, since it contains omega-6 and omega-3 fatty acids, it is thought to reduce the risk of cardiovascular diseases in people while consumed at a young age, but it is recommended to be consumed occasionally in older ages [12].

Although tail fat, which is used both fresh and as edible fat after long-term storage in Turkey and has a wide range of uses in the food industry, is so important for our country, the number of studies in the literature is very limited. Especially after long-term storage, consumers may experience various problems in quality properties as a result of oxidation of lipids during use. For this reason, determining the oxidation degree and other structural changes of tail fats stored under different conditions and times are important factors in terms of food quality and technology. In this study, the effects of storage time, temperature, salt and antioxidant use on the storage stability of heat-treated tail fat (ghee tail fat) were examined, and some physical and chemical properties of tail fat and storage quality were determined and the data obtained will guide other researchers and the food industry.

## MATERIALS and METHODS

### Preparation and Storage of Tail Fat Samples

The tail fat of the sheep (Karayaka breed) used in the study was purchased fresh from a private slaughterhouse in Samsun domestic market immediately after slaughtering and then passed through a meat grinder (+4ºC) with a mirror size of 3 mm. A total of 10 sheep were used in the study, with approximately 2 kg of tail fat obtained from each animal. For fresh tail fat samples, 100 g of tail fat taken from the meat grinder was filled into glass bottles with a lid, which were sterilized by keeping them in an oven the day before. For the heat-treated tail fat (ghee tail fat) sample, 30 minutes of heat treatment was applied to the tail fat with an initial temperature of 14ºC, and when the temperature reached 100ºC, heat treatment was continued for another 5 minutes. Then, the tail fat was passed through a muslin cloth, and the cartilage tissue was separated, and the remaining fat part was filled in the same way at about 80 ºC in glass bottles with a lid. In this way, fat was obtained with 62% yield from the tail tissue used in the research. For the heat-treated salt added (salted tail fat) sample type, 2% (17.5 g) salt (sodium chloride, ≥99.8% Merck, USA) was added to 3500 g tail fat, melted in a saucepan and heat treated at 100ºC for 5 minutes. Then, it was passed through a muslin cloth and filled at approximately 80ºC at 75 g per jar. For the sample with heat-treated additive (additived tail fat), 2000 g of weighed 0.2% (2 g) antioxidant was added from the tail fat melted and filtered in the same way as the other samples, mixed and then filled into jars at about 80ºC. Butylhydroxyanisole (BHA) was preferred in the study because it is a widely used antioxidant. Each prepared sample was stored at room temperature (25ºC), +4ºC and -18ºC for 90 days.

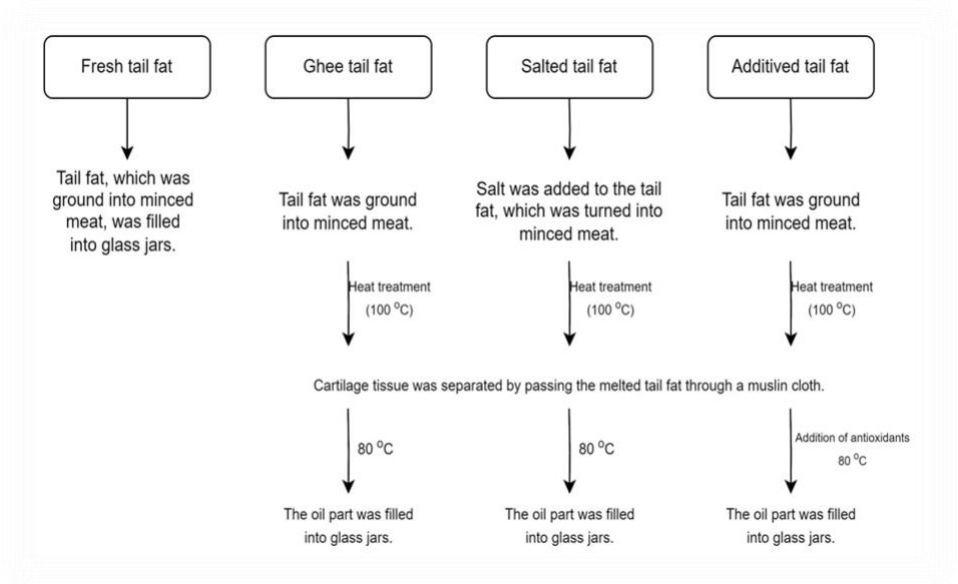


Figure 1. Preparation of tail fat samples

## Physicochemical Analysis

Chemical compositions of the samples were determined according to the methods described by AOAC [13], moisture (925.10), crude protein (976.05), ash (923.03) and oil content (920.39). After the samples were diluted by 10%, readings were made using a pH-meter (Starter 2100, Ohaus, USA) and pH values were determined. Water activity was carried out at 25ºC using a measuring device (Aqualab Dew Point Water Activity Meter, USA).

## Cholesterol Analysis

Cholesterol analysis was performed using gas chromatography (Varian, 3400, Varian Inc., Netherlands) [14]. 5-a-Cholestane was used as internal standard solution (IS). Cholesterol (CH) standard (2 mg/mL hexane) was prepared into vials. Cholesterol content was calculated with the help of the following Equation (1):

$$\text{Cholesterol content (mg/100g)} = (\text{mg IS} \times \text{area CH}) / (\text{area IS} \times \text{100g sample}) \quad (1)$$

## Color Analysis

Color analysis was performed using the Colorflex EZ (Hunter Associates Laboratory Inc., USA) measuring device. In the samples, the International Illumination Commission L*, a*, b* values were measured. Prior to undertaking colour readings, the instrument was calibrated using a white standard plate. For each sample, replicate measurements were taken at randomly selected locations across the surface, and the average value was calculated [15].

## Free Fatty Acidity Analysis

Gençcelep [16] method was modified and FFA analysis was performed. After weighing approximately 5 g of the samples taken homogeneously into a 250 mL flask, 50 mL of diethylether:ethanol (1:1,v/v) was added and shaken for 1 minute. 3-4 drops of phenolphthalein indicator were dropped into it and titration was performed with 0.1 N NaOH. The amount of NaOH used was recorded and the % free fatty acidity of the samples was calculated with the following equation (2):

$$\text{FFA (\% in oleic acid)} = (V/m) \times 2.82 \quad (2)$$

where V is the amount of NaOH used (mL), and m is the sample weight (g).

## Peroxide Value Analysis

Approximately 2 g of the samples were weighed into flask and 10 mL of chloroform was added to it and then shaken. 15 mL of acetic acid and 1 mL of saturated potassium iodide were added, respectively, then the lid was closed and shaken for 1 min. 75 mL of distilled water and 1 mL of 1% soluble starch solution were added to the bottles, which were kept in a dark place for about 10 minutes, and titration was carried out with 0.01 N sodium thiosulfate solution. The used amount was recorded, and the result was determined as milliequivalents of $O_2$/kg oil with the help of the following equation (3) [17]:

$$\text{Peroxide value (milliequivalents } O_2\text{/kg oil)} = [\,(a-b) \times N \times 1000] / m \quad (3)$$

where a is the amount of thiosulfate used for the sample in the titration (mL), b is the amount of thiosulfate consumed for the blank in the titration (mL), N is the normality of the thiosulfate, and m is the sample amount (g).

### Thiobarbituric Acid Reactive Substance (TBA) Analysis

Approximately 10 g of sample was weighed for analysis. 25 mL of 20% trichloroacetic acid and 20 mL of distilled water were added and homogenized with Ultra Turrax for about 2 minutes. The homogenized sample was passed through Whatman No: 1 filter paper. 5 mL of the obtained filtrate was taken and transferred to tubes. After adding 5 mL of 0.02 M TBA solution, the lids were closed and shaken. The tubes, which were kept in a boiling water bath at 93ºC for about 30 minutes, were cooled in tap water for 10 minutes after being removed. Then, it was transferred to spectrophotometer (T80+, PG Instruments Limited, UK) cuvettes and the absorbance value was read at 532 nm wavelength. TBA value was determined as mg malonaldehyde/kg sample by multiplying the recorded value with the coefficient of 7.8 [18].

### FTIR Spectroscopy Analysis

The FTIR spectra of the samples were determined with a (Spectrum Two, Perkin Elmer Inc., Netherlands) device at a wavelength of 4000-450 $cm^{-1}$. For the measurement, the fat samples were ground and transferred to the instrument plate in solid form.

### Differential Scanning Calorimetry Analysis

DSC analysis was performed using the DSC (DSC8000, Perkin Elmer Inc., Netherlands) instrument. An empty aluminum DSC container was used as a reference sample. In the study, melting and crystallization processes were carried out at a speed of 10ºC/minute and between 0-100ºC. The melting and crystallization temperatures of the samples were calculated using the instrument software.

### Fatty Acids Composition Analysis

For the analysis of fatty acids composition of fresh and ghee tail fat samples, the samples were derivatized with 1.5 M methanolic HCl and taken into vials and then analyzed in gas chromatography device (Trace 1310, Thermo Scientific, USA). In the analysis, detector temperature and injection temperature are 240°C, detector type is FID (Flame Ionization Detector), helium carrier gas at 1.0 ml/min, oven temperature at 120°C for 0 min, at 210°C for 10 min, 250°C for 1 min parameters were used.

### Statistical Analysis

Trials created within the scope of the study were set up in two repetitions according to a completely random trial plan. The obtained data were subjected to ANOVA analysis via SPSS Statistics program (V21, SPSS Inc., Chicago, USA) and the statistical significance limits of the differences were determined and compared by applying Duncan multiple comparison test ($p>0.05$).

## RESULTS and DISCUSSION

### Compositional Propeties of Fresh and Ghee Tail Fats

The results of moisture, fat, protein, ash and cholesterol values in fresh and heat-treated tail fat (ghee tail fat) are given in Table 1. Composition analysis was not performed in the samples of ghee salted tail fat and ghee additived tail fat obtained by adding salt and antioxidant, because it was predicted that the salt and additive used would not have much effect on the composition.

The moisture content of fresh tail fat is higher than that of ghee tail fat. In the process of obtaining ghee oil as a result of heat treatment, water is separated from the fatty tissue and a tissue with a very high oil content is left. The difference in moisture content between the obtained fats are very important for both technological and storage quality. As a matter of fact, the moisture content of adipose tissues affects the efficiency, and in this case, the yield value of an adipose tissue with a low moisture content is higher than an adipose tissue with a high moisture content. In addition, the hydrolytic degradation potential of water shortens the storage life in adipose tissue with high moisture content [8].

A portion of the protein (3.47%) found in fresh tail fat remained in the cartilage part as a result of melting, and therefore the amount was lower in ghee tail fat (2.98%). There was no statistically significant difference between the amounts of ash and cholesterol in fresh tail fat and ghee tail fat. However, due to the removal of water by the effect of temperature, very small decreases occurred in their amounts. Cholesterol analysis results in fresh and ghee tail fats were determined as 61.10 mg/100 g and 59.02 mg/100 g, respectively.

Table 1. Composition analysis results of fresh and ghee tail fat

| | Moisture (%) | Crude fat (%) | Crude protein (%) | Ash (%) | Cholesterol (mg/100 g) |
|---|---|---|---|---|---|
| Fresh tail fat | 11.2±0.3$^{a}$ | 85.0±0.8$^{b}$ | 3.47±0.3$^{a}$ | 0.07±0.02$^{a}$ | 61.10±0.02$^{a}$ |
| Ghee tail fat | 0.4±0.02$^{b}$ | 96.0±0.6$^{a}$ | 2.98±0.2$^{b}$ | 0.05±0.02$^{a}$ | 59.02±0.02$^{a}$ |

$^{a-b}$Differences between means with different letters in the same column are significant. Results are mean value ± standard deviation.

### Physical and Physico-chemical Properties of Fresh and Ghee Tail Fats

The pH, $a_w$ and color values (L*, a* and b*) results of fresh tail fat, ghee tail fat, salted tail fat and additived tail fat are given in Table 2. When Table 2 was examined, the pH value of the fresh tail fat sample was 5.69 lower than the other samples. It is thought that the increase in pH value of 6.17 in ghee tail fats is caused by the loss of buffering properties of the proteins in the composition with the application of heat treatment. When ghee tail fat, salted tail fat and antioxidant added tail fat were

compared among themselves, no statistical difference was found in pH value (p>0.05). However, with the dissolution of the added salt, some pH change was observed in the salted sample due to the Na and Cl elements, but it did not show a statistical difference. It is known that the pH values of animal fats are generally in the range of 6-7. While the water activity value of the fresh tail fat group samples was determined as 0.988, this value was determined as 0.830 in the ghee tail fats with the application of heat treatment. In the salt and antioxidant added samples, the water activity value was determined as 0.728 and 0.780, respectively. It is thought that the heat treatment application in our research may have reduced the water activity value by affecting the amount of water in the tail fat, and it may have had a lowering effect on the water activity due to the water binding property of the salt. It has been determined that BHA, which is used as an antioxidant in the research, also has a water activity-lowering effect. It is known that low water activity has a limiting effect on deterioration such as enzymatic reactions, oxidation and growth of microorganisms. It is thought that the difference in water activity, which is one of the important parameters in the processing and preservation of foods, may be one of the important parameters affecting the storage stability of tail fat.

Table 2. pH, $a_w$ and color analysis results of tail fats with different application processes before storage

| | pH | $a_w$ | L* | a* | b* |
|---|---|---|---|---|---|
| Fresh tail fat | 5.69±0.1$^{b}$ | 0.988±0.1$^{a}$ | 74.48±0.02$^{a}$ | 1.38±0.02$^{a}$ | 6.15±0.2$^{a}$ |
| Ghee tail fat | 6.17±0.1$^{a}$ | 0.830±0.1$^{b}$ | 74.20±0.03$^{a}$ | 0.20±0.01$^{b}$ | 5.40±0.2$^{a}$ |
| Salted tail fat | 6.26±0.1$^{a}$ | 0.728±0.1$^{c}$ | 75.45±0.02$^{a}$ | 0.05±0.01$^{b}$ | 5.20±0.2$^{a}$ |
| Tail fat with additives | 6.19±0.1$^{a}$ | 0.780±0.1$^{c}$ | 74.55±0.02$^{a}$ | 0.62±0.02$^{b}$ | 5.83±0.2$^{a}$ |

$^{a-c}$Differences between means with different letters in the same column are significant. Results are mean value ± standard deviation. $a_w$: water activity

The L* values of fresh tail fat, ghee tail fat, salted tail fat, antioxidant added tail fat were determined as 74.48, 74.20, 75.45 and 74.55, respectively, and the difference between them was not statistically significant (p>0.05). However, when the L* gloss values of the samples were examined, it was determined that the gloss values of the samples studied with the addition of salt slightly increased with the effect of salt. While the a* value of the fresh tail fat sample was 1.38, the a* value of ghee tail fat was 0.20, the a* value of salted tail fat was 0.05, and the a* value of additived tail fat was 0.62. It is thought that the heat treatment application that we used to obtain ghee tail fat reduces this value by affecting the a* value. Considering the b* values, the highest value was observed in fresh tail fat samples as 6.15, this value was determined as 5.40 in ghee tail fat, 5.20 in salted tail fat and 5.83 in additived tail fat.

## Fatty Acid Composition of Fresh and Ghee Tail Fats

The fatty acid compositions of fresh tail fat and ghee tail fat samples are shown in Table 3. It was determined that the heat treatment application did not have a significant (p>0.05) effect on the fatty acid composition. The difference may be due to the proportional distribution of fatty acids in the ghee tail fat sample with water removal. Three main fatty acids that dominate both samples were determined. Oleic acid, one of the unsaturated fatty acids, was found to be 20.5% and 21.26% in fresh tail fat and ghee tail fat samples, respectively. In saturated fatty acids, palmitic acid was determined as 21.09% in fresh tail fat and 22.02% in ghee tail fat, while stearic acid was determined as 20.61% in fresh tail fat and 20.82% in ghee tail fat. In addition to these, less myristic acid, heptadeconoic acid, linoleic acid, pentadeconoic acid, α-linolenic acid, myristoleic acid were detected. Except for these, all the remaining fatty acids remained below 1%.

When the palmitic acid contents of the samples were evaluated, although there was not statistically significant (p>0.05) difference between them, the palmitic acid content of ghee tail fat (22.02%) was found to be higher than that of fresh tail fat (21.09%). The palmitic acid content of the samples detected in the study is similar to the literature. Mehran and Filsoof [19], in their study, reported that the palmitic acid ratio in the tail fat of Iranian domestic sheep varies between 18.2% and 23.6%. Yılmaz [8] found the palmitic acid content of the tail tissue to be 19.79% on average. Tüfekci et al. [20] in their study on the tail fat of sheep from different breeds bred in the Black Sea Region, they determined the palmitic acid contents of the tail fats of Artlı, Çepni, Karayaka and of sheep as 21.81%, 25.28%, 25.47% and 26.28%, respectively. When the stearic acid contents of the samples were examined, it was found that the stearic acid content of the fresh tail fat sample was 20.61%, and the stearic acid content of the ghee tail fat was 20.82%. These values are partly similar to the literature, partly differing. Mehran and Filsoof [19] reported that the rate of stearic acid in the tail fat of Iranian domestic sheep varies between 7.1 and 22.1%. In the study, the amount of oleic acid in fresh and ghee tail fat samples was determined as 20.50% and 21.26%, respectively. Mehran and Filsoof [19] determined this value as 39.6-53.5% in their study on tail fat of Iranian domestic sheep. Yılmaz [8] determined the amount of oleic acid belonging to sheep tail fat tissues as 35.65%, Ünsal and Aktaş [7] as 28.37%. Total saturated fatty acids were 60.35% in fresh tail fat and 61.97% in ghee tail fat (p>0.05). Total unsaturated fatty acids were 39.65% and 38.03%, respectively (p>0.05). Since the amount of saturated fatty acids is higher, the tail fats are solid at ambient temperature.

Table 3. Fatty acid composition of fresh and ghee tail fat

| Fatty acids | % Fatty acids | |
|---|---|---|
| | Fresh tail fat | Ghee tail fat |
| C14:0 | 6.94±0.23[a] | 7.20±1.04[a] |
| C14:1 | 1.33±0.05[a] | 1.38±0.20[a] |
| C15:0 | 6.77±1.16[a] | 5.54±0.99[a] |
| C16:0 | 21.09±0.61[a] | 22.02±3.15[a] |
| C17:0 | 6.42±0.21[a] | 6.88±0.77[a] |
| C17:1 | 4.68±0.11[a] | 4.81±0.58[a] |
| C18:0 | 20.61±0.65[a] | 20.82±0.04[a] |
| C18:1n9 | 20.50±0.96[a] | 21.26±1.51[a] |
| C18:2n6 | 6.64±0.12[a] | 7.01±0.75[a] |
| C18:3n3 | 2.09±0.07[a] | 2.15±0.28[a] |
| Total Saturated Fatty Acid | 60.35±1.78 | 61.97±5.74 |
| Total Unsaturated Fatty Acid | 39.65±1.78 | 38.03±5.74 |
| Mono Unsaturated Fatty Acids | 26.51±0.00 | 27.45±0.00 |
| Poly Unsaturated Fatty Acids | 8.73±0.00 | 9.16±0.00 |
| PUFA/SFA | 0.15±0.00 | 0.15±0.00 |
| SFA/UFA | 1.52±0.00 | 1.63±0.00 |

[a-e] Differences between means with different letters in the same column are significant. Results are mean value ± standard deviation.

Although the composition of fatty acids obtained in the study of fat samples is similar to the information in the literature, it has been determined that there are partial differences. These differences may be caused by factors such as sheep type, age, nutritional status. As a matter of fact, the fact that tail fat contains essential fatty acids in its structure can be considered among the features that make tail fat important.

## Calorimetric Properties of Fresh and Ghee Tail Fats

DSC is the most widely used thermo-analytical technique to study oils and fats and is a widely accepted method for determining the crystallization and melting properties of fats [21]. DSC thermograms of fresh and ghee tail fat samples are given in Figures 2 and 3. When the DSC thermogram of the fresh tail fat sample was examined (Figure 2), one crystallization peak at 14.77°C and three melting peaks at 10.26°C, 23.34°C and 34.62°C were obtained. One crystallization peak at 19.82°C and three melting peaks at 11.19°C, 26.12°C and 36.88°C were detected in the DSC thermogram of ghee tail fat (Figure 3). Yılmaz and Karakaya [21] conducted thermal analysis of lipids isolated from various tissues of sheep fat and obtained two similar crystallization peaks (31.25-24.69°C and 7.44-3.90°C) and melting peaks (15.36-13.44°C and 45.98-44.60°C) in the DSC thermograms of tallow and intestinal fat. When compared with this information examined in the literature, the results found in the research differ. As a result, although the thermograms of fresh tail fat and ghee tail fat are similar to each other, the differences may be due to small differences in fatty acid composition.

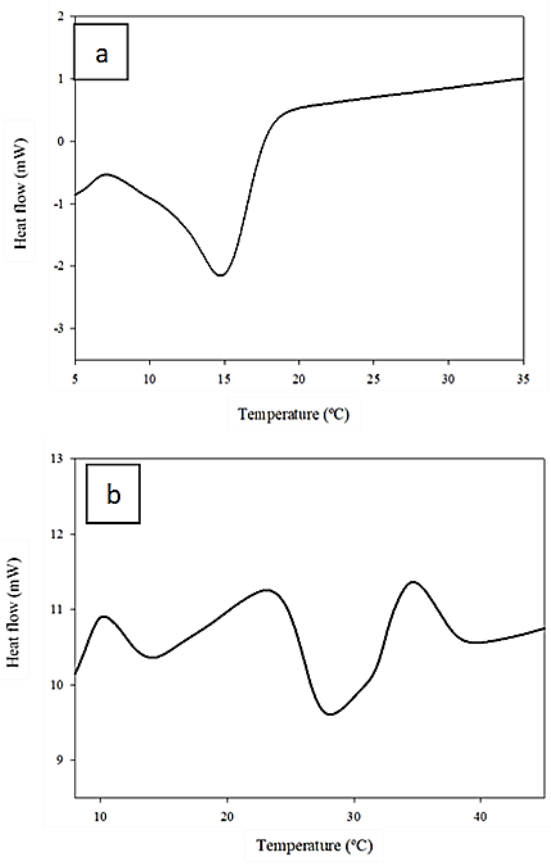


Figure 2. Crystallization (a) and melting (b) DSC thermogram of fresh tail fat sample

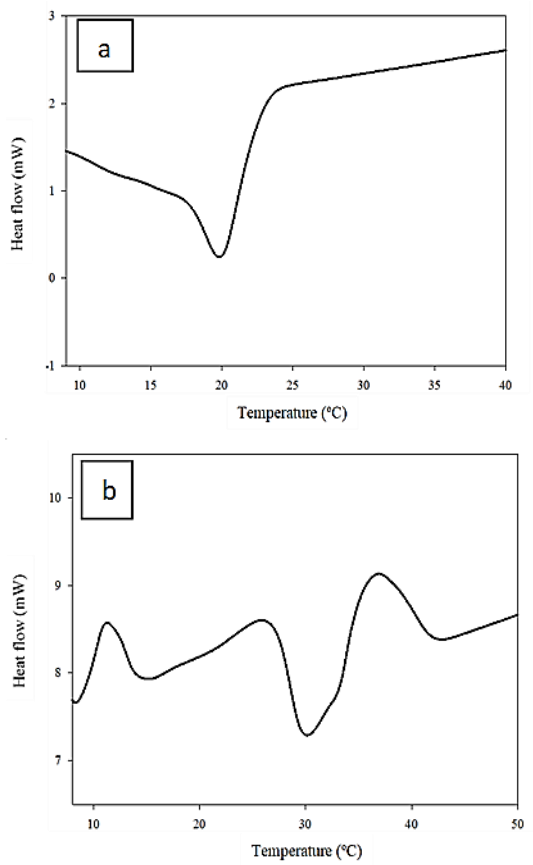


Figure 3. Crystallization (a) and melting (b) DSC thermogram of ghee tail fat sample.

## FTIR Spectral Properties of Fresh and Ghee Tail Fats

FTIR spectra of fresh tail fat and ghee tail fat is given in Figures 4 and 5, respectively. 4000-3050 $cm^{-1}$ absorption frequency determines the O-H bond type and the state of water, alcohol, hydroperoxides [22]. When the spectra of fresh tail fat (Figure 4.) and ghee tail fat (Figure 5.) were examined, a difference was found in the frequency range of 4000-3050 $cm^{-1}$, which is thought to be caused by the decrease in the amount of water in the ghee tail fat samples with the application of heat treatment. It is thought that the absorption frequency of 3025-2850 $cm^{-1}$, which gives information about C-H stretching, methylene and methylene groups at the end of the fatty acid chain [22], is parallel to the fact that the fatty acid compositions of fresh and ghee tail fat samples are similar. 1870-1550 $cm^{-1}$ absorption frequency C=O stretch gives information about the ester bond between fatty acid and glycerol [22]. When the absorption frequency of 1870-1550 $cm^{-1}$ is examined in both spectra, it is seen that the deterioration in the triglyceride structure is more in the fresh tail fat sample. It has been determined that the FTIR spectrum of sheep tail fat obtained in a study is similar to the spectrum obtained in our study [23].

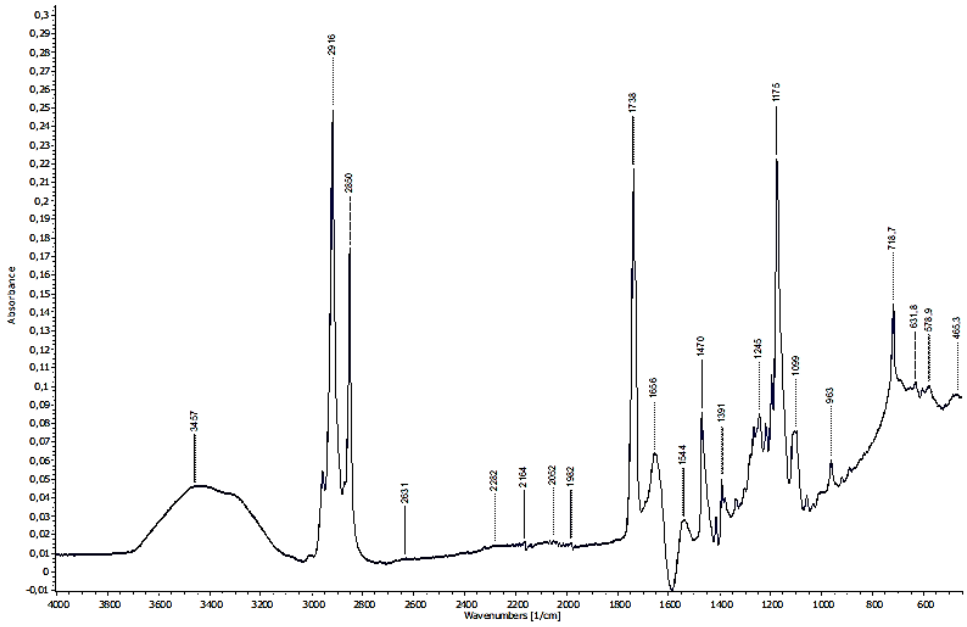


Figure 4. FTIR spectrum of fresh tail fat sample

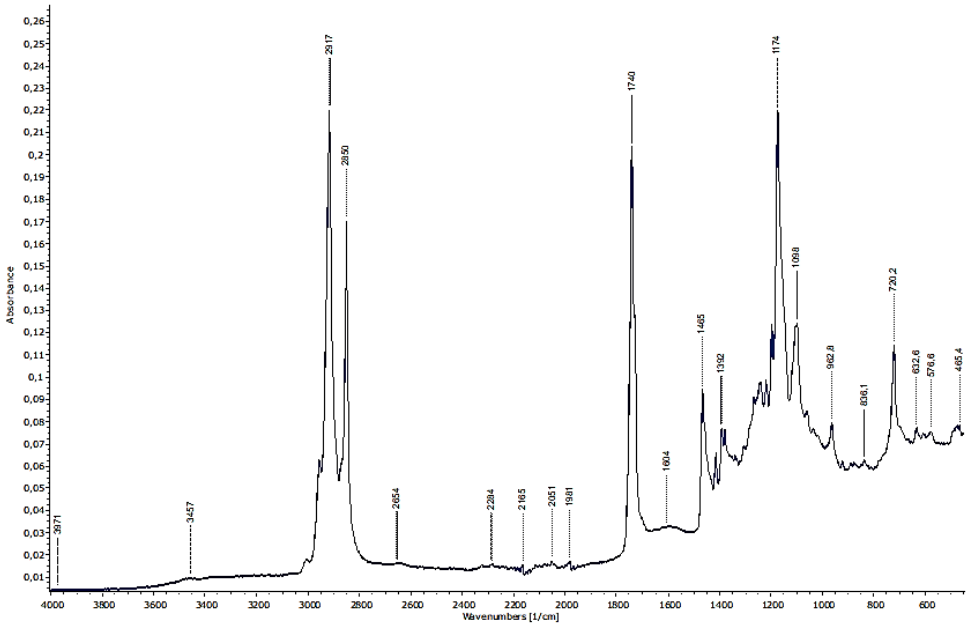


Figure 5. FTIR spectrum of ghee tail fat sample

## Results of Analyses in Tail Fats during Storage

Hydrolysis and oxidation are the most important spoilage factors in storage. In this direction, free fatty acidity (FFA) analysis was performed to determine the extent of hydrolysis. In order to determine the degree of oxidation, the primary products of oxidation were determined by the peroxide number, and TBA analysis was performed to determine the secondary products formed in the advanced stages of oxidation. Samples with different applications (fresh tail fat, ghee tail fat, salted tail fat, additived tail fat); The analyzes were made on the 0th, 15th, 30th, 60th and 90th days stored at room temperature (25ºC), +4ºC and -18ºC. In addition, fresh tail fat stored at room temperature was analyzed on the 7th day. According to the results of the 7th day analysis and due to bad odor and deterioration, fresh tail fat stored at room temperature was excluded from the analysis plan.

Free fatty acids, which are not bound to triglyceride structure in the structure of oils, are an important quality parameter for oils. The change in free fatty acidity during storage gives information about the rancidity of the product, and by detecting the change in free fatty acidity, how far the hydrolysis mechanism has progressed can be determined. The FFA values determined during the storage of tail fats with different application processes are given in Table 4. The highest free fatty acidity value was found as 3.28% in the fresh tail fat sample, which was not processed after being minced, while this value was determined as 0.47%, 0.45% and 0.42% in the ghee tail fat, salted tail fat and additived tail fat, respectively. The difference between fresh and ghee tail fat was significant ($p<0.05$). Free fatty acidity values determined in ghee tail fat, salted tail fat and additived tail fat were between the same values on average and were not statistically different ($p>0.05$). A lower percentage of FFA was determined in ghee tail fats compared to fresh tail fat samples. It may be due to the fact that hydrolytic degradation is largely controlled by removing water with the application of heat treatment. FFA values determined at 25, 4 and -18ºC were determined as 1.43, 1.14 and 0.53%, respectively, and it was observed that different temperature values had a significant ($p<0.05$) effect on free fatty acidity. As the temperature value is lowered, the decrease in % FFA value can be said to slow down the activity of the lipase enzyme by limiting the activity of the enzymes in the low temperature application. The effect of storage time on the formation of free fatty acidity is important. The highest % FFA value (2.28) was found in the analysis results performed on the 7th day for the samples stored at 25ºC. A decrease in FFA value occurred after the 15th day and this is expected, as the free fatty acidity formed until the 15th day was broken down into further degradation products as the storage progressed [8, 12]. After being minced, fresh tail fat stored at 25ºC without any treatment showed a significant increase in free fatty acidity until the 15th day, and it was also determined that it could not be used as food due to the bad smell and spoilage. In addition, it is seen that the heat treatment application has a limiting effect on the formation of free fatty acidity.

Table 4. FFA (%), peroxide (meqg $O_2$/kg) and TBA (mg MDA/kg) values of tail fats stored in different treatments, temperatures and times

| Parameters | N | FFA | Peroxide | TBA |
|---|---|---|---|---|
| *Treatment* | | | | |
| Fresh tail fat | 50 | 3.28±3.59$^{a}$ | 4.75±5.71$^{a}$ | 0.93±0.89$^{a}$ |
| Ghee tail fat | 64 | 0.47±0.07$^{b}$ | 2.67±1.37$^{b}$ | 0.72±0.39$^{b}$ |
| Salted tail fat | 64 | 0.45±0.10$^{b}$ | 2.45±1.23$^{c}$ | 0.81±0.38$^{b}$ |
| Additived tail fat | 64 | 0.42±0.09$^{b}$ | 2.28±1.11$^{d}$ | 0.76±0.36$^{b}$ |
| *Storage temperature ($^{o}$C)* | | | | |
| 4 | 84 | 1.14±1.58$^{b}$ | 3.73±4.46$^{a}$ | 0.94±0.70$^{a}$ |
| 25 | 84 | 1.43±2.96$^{a}$ | 2.97±2.10$^{b}$ | 0.89±0.40$^{a}$ |
| -18 | 84 | 0.53±0.25$^{c}$ | 2.11±1.06$^{c}$ | 0.55±0.32$^{b}$ |
| *Storage (day)* | | | | |
| 0 | 48 | 0.49±0.10$^{d}$ | 1.03±0.12$^{f}$ | 0.31±0.08$^{e}$ |
| 7 | 16 | 2.28±3.24$^{a}$ | 5.45±2.81$^{a}$ | 0.83±0.19$^{cd}$ |
| 15 | 48 | 1.54±3.41$^{b}$ | 2.44±0.87$^{e}$ | 0.90±0.46$^{bc}$ |
| 30 | 48 | 0.88±1.11$^{c}$ | 4.58±5.74$^{b}$ | 0.97±0.60$^{ab}$ |
| 60 | 48 | 0.94±1.36$^{c}$ | 3.14±1.33$^{c}$ | 1.06±0.69$^{a}$ |
| 90 | 48 | 0.92±1.37$^{c}$ | 2.85±1.06$^{d}$ | 0.76±0.31$^{d}$ |

$^{a-f}$ Differences between means with different letters in the same column are significant. Results are mean value ± standard deviation. N: Number of samples, FFA: Free fatty acidity, TBA: Thiobarbituric acid reactive substance

The peroxide value, which is an indicator of the progress in lipid oxidation, is obtained by measuring the peroxide (R-OOH) value, which is one of the main reaction products formed in the first stage of oxidation [12]. Table 4. shows the peroxide values determined during the storage of tail fats with different application processes. Peroxide values of fresh tail fat, ghee tail fat, salted tail fat and additived tail fat samples were determined as 4.75, 2.67, 2.45, 2.28 in meqg $O_2$/kg, respectively, and the differences between them were found to be statistically significant ($p<0.05$). It is thought that the removal of water by heat treatment prevents oxidation, thus causing a lower peroxide value detected in ghee tail fat, and at the same time, peroxides that are not heat-resistant substances may be removed by heat treatment, so the peroxide value is lower in ghee tail fats. Likewise, it can be said that the salt and additive added to the samples reduce the peroxide value in salted and additeved tail fats by inhibiting oxidation. It has been observed that the temperature has a significant effect on the peroxide value in tail fats stored at different temperatures. The peroxide values of tail fats were determined as 2.97 meqg $O_2$/kg at 25ºC, 3.73 meqg $O_2$/kg at 4ºC and 2.11 meqg $O_2$/kg at -18ºC. Lower peroxide values were obtained at -18ºC compared to other temperatures. It is expected that the lowest peroxide value will be detected at -18ºC in the research. An increase was observed in peroxide values until the 30th day of storage, and a decrease was observed in the following periods. It is estimated that this result may be caused by the breakdown of hydroperoxides formed in the initial stage of oxidation to malonaldehydes. It is thought that the changes in peroxide values are due to the fact that the hydroperoxides released in the first stage of oxidation are not stable and can turn into different decomposition products as the degradation progresses [8, 12].

TBA analysis is the most suitable method for the measurement of oxidative rancidity in fat-containing foods, and in this method, malonaldehyde is measured as the secondary oxidation product of polyunsaturated fatty acids [18]. There is an increase in the number of TBA (mg malonaldehyde/kg) in parallel with the accumulation of short carbon chain products that cause rancidity. The TBA values determined during the storage of tail fats with different application processes are presented in Table 4. While the TBA value was found to be 0.93 mg malonaldehyde/kg in the fresh tail fat group samples, it was determined as 0.72 mg malonaldehyde/kg, 0.81 mg malonaldehyde/kg and 0.76 mg malonaldehyde/kg in ghee tail fat, salted tail fat, and additived tail fat, respectively. The difference between fresh and ghee tail fat was found to be statistically significant. The increase in TBA value determined in salted tail fat may be due to the prooxidative effect of salt. TBA values of tail fats stored at 25ºC, 4ºC and -18ºC were determined as 0.89 malonaldehyde/kg, 0.94 malonaldehyde/kg and 0.55 malonaldehyde/kg, respectively. The lowest TBA value was found in the samples stored at -18ºC, and it is thought that freezing at -18ºC delays lipid oxidation in the samples, compared to storage at room temperature and +4ºC, which may be a better method for the preservation of tail fat. There are statistically significant ($p<0.05$) differences between storage times. In parallel with the storage period of tail fat, an increase occurred in TBA values until the 60th day, and then a decrease was observed. It is thought that the decrease in the TBA value observed after the 60th day may be due to the transformation of the released malonaldehydes into further degradation products. Yılmaz (2009) reported that the TBA value in fat samples increased depending on the storage period [8].

## CONCLUSION

Within the scope of the study, the effects of heat treatment, temperature, storage time, salt and antioxidant addition on the storage stability of heat-treated tail fat were investigated and some physical and chemical properties of tail fat were investigated. The high amount of fat in the tail tissue has made it an important source of fat and increased demand [24]. It has been determined that fresh tail fat and ghee tail fat have lower cholesterol amounts compared to many

foodstuffs, and it can be said that they are preferable in terms of nutrition thanks to this feature. The amount of water, which affects technological properties of food, has been reduced by the application of heat treatment. In this context, the water activity value was low in ghee tail fat and this improved the technological and storage conditions of tail fat. Color is one of the most important features of acceptability for consumers, and the absence of a negative effect of heat treatment on color can increase the preferability of ghee tail fats. It was determined that the heat treatment did not have a significant effect on the fatty acid composition, and the fact that the saturated fatty acid content of fresh and ghee tail fat was lower than that of other animal fats enabled tail fat to be considered as an important source of fat in terms of nutrition. The fact that the melting temperatures in DSC thermograms were lower than body temperature in fresh tail fat and ghee tail fat showed that the absorption by the body was high and made tail fat preferable. FFA formation was significantly prevented by inactivating the enzymes in the structure with heat treatment. Removal of water by heat treatment prevented hydrolytic rancidity and peroxide values decreased. Fresh tail fat stored at room temperature deteriorated within 7 days, while samples stored at +4ºC deteriorated within 15 days. However, ghee tail fat could be preserved at these storage temperatures for 60 days without deterioration. As a result, heat treatment of tail fat and keeping it at low temperatures affected the storage stability of tail fat and allowed it to remain intact for a longer period of time.

## AUTHOR CONTRIBUTIONS

Conceptualization, literature review, organization, analysis was done by MB. Review and editing were done by HG. HG had supervised the entire research works. Literature review, critical analysis of data, manuscript review and editing were written by MOY. All authors of this research read the manuscript and agreed to publish it.

## CONFLICT OF INTEREST

The authors declare no conflict of interest.

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**Research Paper / Araştırma Makalesi**

# Validation of an HPLC-UV Method for Simultaneous Analysis of Ascorbic and Oxalic Acids in Beverages

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## ABSTRACT

A reversed-phase high-performance liquid chromatography (RP/HPLC-UV) method was developed for the rapid and simultaneous analysis of ascorbic and oxalic acids in both industrial and freshly squeezed beverages. Due to the rapid degradation of ascorbic acid, its stability was also investigated. The analysis was performed using an Inertsil ODS-3 column with a mobile phase consisting of 0.01 mol $L^{-1}$ $KH_2PO_4$ phosphate buffer at pH 2.3. Detection wavelengths were set at 245 nm for ascorbic acid and 205 nm for oxalic acid. The limits of detection (LOD) were 1.4 mg $L^{-1}$ for ascorbic acid and 1.3 mg $L^{-1}$ for oxalic acid. After 48 hours of opening an industrial fruit juice package, an 18.5% reduction in ascorbic acid content was found. The method demonstrated within-day repeatability (n = 3) of 4.1% for ascorbic acid and 4.6% for oxalic acid. Between-day precision, expressed as relative standard deviation (RSD), was 3.85% for ascorbic acid and 4.33% for oxalic acid, based on 10 replicates (n= 10). The accuracy of the method was validated with an average recovery rate ranging from 93% to 104%.

**Keywords:** Liquid chromatography, Ascorbic acid, Oxalic acid, Beverage, Fruit juice

## İçeceklerde Askorbik ve Oksalik Asitlerin Eşzamanlı Analizi İçin HPLC-UV Yönteminin Validasyonu

## ÖZ

Ters faz yüksek performanslı sıvı kromatografisi (RP/HPLC-UV) yöntemi, endüstriyel ve taze sıkılmış içeceklerde askorbik ve oksalik asitlerin hızlı ve eş zamanlı analizi için geliştirilmiştir. Askorbik asidin hızlı bozulması nedeniyle, stabilitesi de araştırılmıştır. Analiz, pH 2.3'te 0.01 mol $L^{-1}$ $KH_2PO_4$ fosfat tamponundan oluşan hareketli faz ile Inertsil ODS-3 kolon kullanılarak gerçekleştirilmiştir. Tespit dalga boyları askorbik asit için 245 nm ve oksalik asit için 205 nm olarak ayarlanmıştır. Tespit limitleri (LOD), askorbik asit için 1.4 mg $L^{-1}$ ve oksalik asit için 1.3 mg $L^{-1}$ olarak belirlenmiştir. Endüstriyel meyve suyu paketinin açılmasından 48 saat sonra, askorbik asit içeriğinde %18.5'lik bir azalma tespit edilmiştir. Yöntem, gün içi tekrarlanabilirlik (n= 3) açısından askorbik asit için %4.1 ve oksalik asit için %4.6 oranında bir doğruluk göstermiştir. Günler arası hassasiyet, bağıl standart sapma (RSD) olarak ifade edilmiş ve 10 tekrar (n= 10) temelinde askorbik asit için %3.85 ve oksalik asit için %4.33 olarak hesaplanmıştır. Yöntemin doğruluğu, %93 ile %104 arasında değişen ortalama geri kazanım oranı ile doğrulanmıştır.

**Anahtar Kelimeler:** Sıvı kromatografisi, Askorbik asit, Oksalik asit, İçecek, Meyve suyu

## INTRODUCTION

The importance of a healthy and balanced diet is an undisputed fact of life today. Fresh fruit and fruit juice are one of the most important links in the healthy food chain. Organic acids, natural compounds found mainly in fruits, differ in their additive or preservative content between fresh and industrial fruit juices. Fruit juices are an important source of vitamin C and minerals for humans, and their consumption is increasing as ready-made fruit juices become more widely available. Hence, the detection and determination of organic acids in fruit juices are critical for quality and process control. The concentration of organic acids in fruits are important for the taste, color, aroma, stability, nutritional value, and overall quality of fruit juices.

The amount of organic acids in fruits and vegetables depends on factors such as the type of fruit, soil, and stress conditions to which the fruit is exposed. Fruit juices contain oxalic acid, an antioxidant and weak organic acid, and ascorbic acid, commonly known as vitamin C. Vitamin C (L-ascorbic acid) is a water-soluble vitamin. Fruit juices are an important source of vitamin C for humans and since ready-made fruit juices are easy to obtain, their use is becoming more widespread [1-5]. It protects our immune system and increases our resistance. Ascorbic acid and calcium ascorbates are used as antioxidants in pharmaceutical preparations and the food industry. They are essential for growth and development, as well as playing a significant role in cell renewal, protection, and defense against oxidative stress [6]. However, ascorbic acid is highly sensitive to various deteriorative factors during food processing and storage, making it one of the most vulnerable vitamins [7]. It is easily lost during food processing, storage, and preparation. Due to its sensitivity to processing, ascorbic acid loss is used as a criterion to assess the negative effects of many food processing techniques. Additionally, oxalic acid, being a weak acid, has the tendency to form stable complexes with calcium in the body, leading to the formation of calcium oxalate precipitates. This not only prevents the absorption of calcium in the human body but also easily initiates the formation of bladder stones. Excess oxalic acid can affect human health [8]. Therefore, the determination of the amount of oxalic acid in some samples, such as fruits, etc. has very important practical importance [9].

It is important to identify the organic acids present in fruits and vegetables. Total acidity, microbial stability, freshness, and other sensory and chemical properties of the matrix are all influenced by the amounts and relative ratios of organic acids. Many methods have been published for the determination of organic acids in foods and beverages such as cheese, tomatoes, green beans, carrots, apples, kiwi, blackberries, currants, fruit juices, grape must, and wine. In particular, methods such as spectroscopy [10,11], electrochemical [12-15], spectrofluorimetry [16,17], high-performance liquid chromatography [18,19] and colorimetry [20] were used to determine ascorbic and oxalic acids.

Among these methods, chromatographic methods are mainly based on GC or HPLC separations and the simultaneous determination of organic acid amounts. There are several methods of analysis of fruit and fruit juices based on GC. Although these methods offer excellent separation and sensitivity, they have disadvantages such as being time-consuming, involving derivatization steps, and the use of toxic derivative markers. Additionally, the high temperature needed for these analyses can lead to sample deterioration. On the other hand, the disadvantage of HPLC methods is based on low separation power and high detection limits. Nevertheless, HPLC separations are effective methods for the separation and quantification of organic acids due to its simplicity and better chromatographic conditions [21].

This study aims to establish a direct RP/HPLC method for the simultaneous analysis of ascorbic and oxalic acids in both industrial and freshly squeezed beverages, such as iced tea and fruit juices, under optimized conditions. Additionally, the levels of ascorbic and oxalic acids present in various beverages are profiled and quantified.

## MATERIALS and METHODS

### Reagents and Samples

HPLC-grade phosphoric acid and sodium dihydrogen phosphate monohydrate were purchased from Merck (Darmstadt, Germany). The standards of ascorbic and oxalic acids were of analytical purity and were purchased from Sigma-Aldrich. Solutions were prepared and diluted using ultra-pure deionized water with a resistance of 18 MΩ·cm that was acquired from an ultra-pure water system (Human Power I plus Water Purification System).

Fruit juices and cold teas were purchased from local markets in Denizli (Türkiye). The samples analyzed were new-production, 100% fruit juices (orange, cherry, pomegranate, apple, apricot, mixed fruit, and cold tea in 1 L TetraPak packages) without any preservatives or added sugars. Natural orange juice samples were obtained from a local market in Denizli, squeezed in the juice apparatus, and analyzed without delay. All samples were run in triplicate.

### Preparation of Standard Solutions

To prepare solutions with concentrations of 1000 mg $L^{-1}$ of ascorbic acid and oxalic acid, 0.05 g of solid ascorbic acid and 0.05 g of solid oxalic acid were weighed and dissolved in an aqueous buffer (pH 2.5). These solutions were prepared in 50 mL volumetric flasks. Working standard solutions were prepared by diluting the appropriate volume of stock solutions with buffer at concentrations between 10-500 mg $L^{-1}$ of ascorbic acid and 15-500 mg $L^{-1}$ of oxalic acid. All standard solutions were stored at 4°C.

### Sample Preparation

The ready-to-drink fruit juices were directly analyzed. For this purpose, three samples of the same type of fruit juice

(3 L) were homogenized simultaneously. Prior to injection, they were centrifugated at 4,000 rpm for 10 min and filtered through a 0.45 µm membrane filter (Millipore Corporation, France). The mobile phase (2.5 pH buffer solution) was degassed in an ultrasonic bath before use. The prepared fruit juice samples were further diluted 5-fold and then injected into the HPLC (20 µL).

The stability of ascorbic acid concentration over time and the effect of dilution with the mobile phase were also evaluated. For this purpose, the same juice sample was analyzed every 15 min for up to 90 min to detect changes in ascorbic acid concentration.

### Instrument and Chromatographic Conditions

A Hettich EBA 20 centrifuge was used for centrifugation of the sample before injection. An ultrasonic bath from Bandelin Sonarex (Berlin, Germany) was used to remove dissolved oxygen from the mobile phase. Chromatographic separations were performed using an HPLC system from Shimadzu (Kyoto, Japan) equipped with the following: LC-20AD pump, SPD-M20A photodiode array detector (DAD), SIL-20A automatic sampler, CTO-20A column oven, and DGU-20A5 degasser. Automatic sampling was performed with an autosampler volume of 20 µL. An Inertsil ODS-3 column (250×4.6 mm; GL Sciences, Japan) was used as the analytical column at 25°C. The mobile phase consisted of a 0.01 mol $L^{-1}$ $NaH_2PO_4.H_2O$ buffer solution (adjusted to pH 2.5 with phosphoric acid) and was eluted isocratically at a flow rate of 0.9 mL $min^{-1}$.

Standard solutions of ascorbic and oxalic acids were injected separately to determine their retention times and elution order. The results obtained and chromatograms were processed via LabSolutions (Shimadzu).

### Analytical Parameters of the Method

The analytical parameters of a method include linear range, precision, accuracy, limit of detection (LOD), and limit of quantification (LOQ) [22]. Once the method was optimized, the determination of ascorbic acid and oxalic acid in real samples was carried out.

### Linearity

Analytes were identified by comparing the UV-visible region spectra of ascorbic and oxalic acids via standard addition. The linearity of the RP/HPLC-UV method was studied by constructing seven-point calibrations for selected organic acids over a wide concentration range. Calibration curves were generated by plotting peak areas against the concentration of the selected organic acids. Correlation coefficients were calculated from the best-fit line. The experimental results were expressed as the mean (X)±standard deviation (SD) of three replicates.

### Repeatability and Accuracy

The repeatability and reproducibility of the method were calculated by taking the percent relative standard deviation of the retention times (RSD, %) for six independent samples containing known amounts of ascorbic and oxalic acid, both on the same day (intraday) and on different days (interday).

For the determination of the organic acids studied, the accuracy of the RP/HPLC-UV system was expressed as the relative error term and calculated by ten repeated injections of the solution with standard addition. The concentrations were calculated from the calibration curve equation for each organic acid.

### Limit of Detection and Limit of Quantification

The theoretical LOD and LOQ for the analyzed ascorbic acid and oxalic acid were represented by 3.3 times and 10 times the ratio of the standard deviation of the lowest concentration value to the slope of the calibration graph. The equations for calculating LOD and LOQ are provided below [23]:

$$\text{LOD (mg } L^{-1}) = (3.3 \times \text{residual standard deviation of y - intersection of the regression line) / slope} \quad (1)$$

$$\text{LOQ (mg } L^{-1}) = (10 \times \text{residual standard deviation of y - intersection of the regression line) / slope} \quad (2)$$

The content of ascorbic and oxalic acids in the samples was determined by interpolating from the calibration curve, taking into account the dilution factors applied during sample preparation.

### Recovery Study

The accuracy of the method was calculated as the recovery efficiency by adding known amounts of the studied organic acids. For this purpose, Ascorbic acid and oxalic acid were spiked into freshly squeezed orange juice at concentrations of 20 mg $L^{-1}$ , 25 mg $L^{-1}$, 50 mg $L^{-1}$, and 100 mg $L^{-1}$ prior to extraction. Analyte addition was repeated three times.

### Stability of Ascorbic Acid

The oxidation of ascorbic acid during sample preparation affects the measurement results. The stability of ascorbic acid was tested in freshly squeezed orange juice obtained immediately after squeezing or, in the case of commercially available orange juice samples, immediately after opening the package. Both samples were diluted 5-fold with the mobile phase and filtered through a 0.45 µm membrane filter. Prior to sample preparation, both orange juices were stored at 5 °C.

## RESULTS and DISCUSSION

### Optimization of the RP/HPLC-UV Method

RP/HPLC coupled with UV detection is a common technique used for the determination of organic acids. Separating and quantifying organic acids using high-performance liquid chromatography is challenging because these acids have similar chemical structures and spectral properties.

In the RP/HPLC-UV method, a UV region was scanned to determine a common wavelength for ascorbic acid and oxalic acid. Since the chemical structures of the organic acids being studied are different, they exhibited maximum absorption at different wavelengths. Ascorbic acid was determined at a maximum absorbance wavelength of 245 nm, while oxalic acid was at 205 nm. However, the acceptable common wavelength for both ascorbic and oxalic acids was determined to be 220 nm.

Generally, when C18 is used as the packing material, it can be operated within the pH range of 2 to 7. When the pH is ≤ 2.00, protonation can occur, which may increase solubility. However, this condition is limited by column efficiency. On the other hand, when the pH is ≥ 7, the siloxane material that makes up the column packing can undergo hydrolysis, causing the column packing to degrade.

Most organic acids have relatively low and similar pKa values. This limits the pH values that can be used for chromatographic separation [24]. An acidic eluent (pH=1.5-2.5) is necessary to keep organic acids protonated. This ensures the best interaction between organic acids and the stationary phase, resulting in optimal separation. The solubility performance of ascorbic and organic acids was tested within a pH range of 2-3. For the simultaneous determination of ascorbic and oxalic acids with optimal separation and peak shape, a mobile phase consisting of a 0.02 mol $L^{-1}$ $NaH_2PO_4.H_2O$ buffer solution was used at a flow rate of 1 mL $min^{-1}$.

In Figure 1, the standard solution of ascorbic acid, ascorbic acid peaks in ready-to-drink orange juice and apple juice are superimposed, and in Figure 2, the standard solution of oxalic acid, oxalic acid peaks in ready-to-drink orange juice and apple juice are superimposed. The peaks are seen overlapped on top of each other.

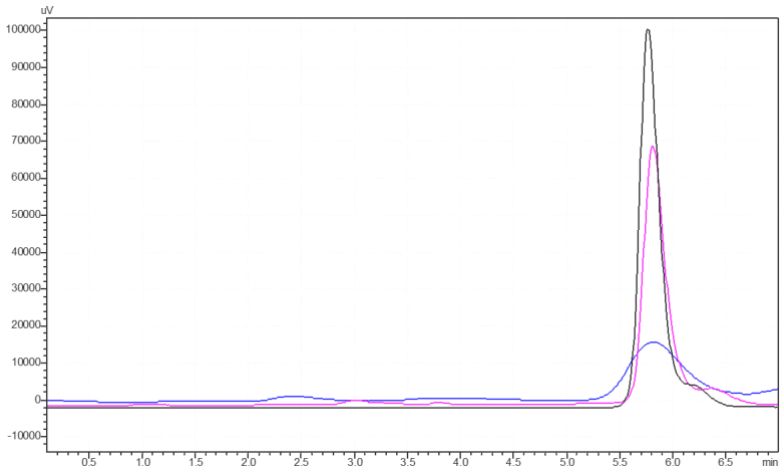


Figure 1. Chromatogram of the standard solution of the ascorbic acid (black), ready-to-drink orange juice (pink), and ready-to-drink apple juice (blue) at 245 nm

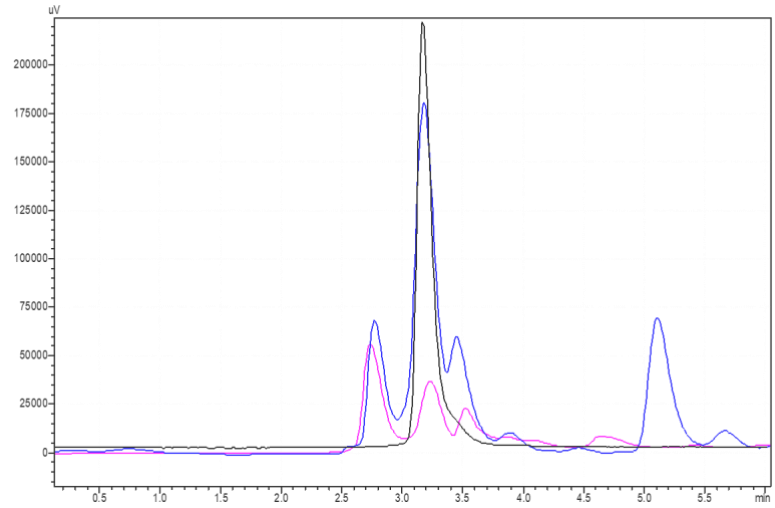


Figure 2. Chromatogram of the standard solution of the oxalic acid (black), ready-to-drink orange juice (pink), and ready-to-drink apple juice (blue) at 205 nm

According to Figures 1 and 2, the analysis time required for the separation of ascorbic and oxalic acids using an ODS column at 1 mL $min^{-1}$ was less than 6.5 min.

Evidently, the method provides good selectivity and resolution for the simultaneous determination of ascorbic acid and oxalic acid.

## Method Validation

### Linearity, LODs, LOQs, Reproducibility and Accuracy

In the RP/HPLC-UV method, the linear range of the calibration curve was established for ascorbic acid within the concentrations of 10, 20, 30, 50, 100, 250, and 500 mg $L^{-1}$, and for oxalic acid within the concentrations of 15, 30, 50, 100, 250, and 500 mg $L^{-1}$. Linearity was evaluated based on the calibration curves. Each standard was injected three times to assess repeatability. The calibration curves for ascorbic and oxalic acids were linear based on a high correlation coefficient ($R^2$). The method exhibits a wide linear range.

The repeatability of the RP/HPLC-UV method for retention times, both intraday and interday, was obtained through repeated injections of standard solutions at 250 mg $L^{-1}$. The calculated LOD and LOQ, retention times ($t_R$), and %RSD of retention times for the method are provided in Table 1. The intraday and interday %RSD for retention times were less than 4.6%. As seen in Table 1, the LOQ for each acid has a good correlation coefficient ($R^2 \geq 0.997$). Coupled with good stability and repeatability values, these data indicate that the applied method can be reliably used for the simultaneous analysis of ascorbic and oxalic acids in fruit juices. Using a test method that conforms to the guidelines stated in internationally accepted guidance publications allowed the procedure to be validated (Eurachem 2014).

Table 1. Linear calibration equation, range, LOD, LOQ, and precision of retention time ($t_R$) of standard organic acids for the RP/HPLC-UV method

| Organic Acids | Analytical Parameters | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Linear Range (mg $L^{-1}$) | Calibration Equation | $R^2$ | LOD (mg $L^{-1}$) | LOQ (mg $L^{-1}$) | $t_R$ (min) | %RSD of $t_R$ | |
| | | | | | | | Intra day | Inter day |
| Ascorbic | 10-500 | y= 65603x-184517 | 0.995 | 1.4 | 4.2 | 6.2 | 4.6 | 4.3 |
| Oxalic | 15-500 | y= 27906x+31434 | 0.997 | 1.3 | 3.9 | 3.1 | 4.1 | 3.8 |

The accuracy of the RP/HPLC-UV method was verified by analyzing standard solutions of ascorbic and oxalic acids with concentrations of three different ppm values within the calibration graph range of 10-500 mg $L^{-1}$. The concentration of the standard solutions was calculated using a calibration curve (Table 2). The relative error was found to be less than %5.

Table 2. Accuracy of the RP/HPLC-UV method for the determination of organic acids

| Organic Acids | Concentration (mg $L^{-1}$) | Calculated Concentration (mg $L^{-1}$) | Relative Error (%) |
|---|---|---|---|
| Ascorbic | 10 | 10.4±1.2 | 4.0 |
| | 30 | 29.5±2.3 | 1.6 |
| | 100 | 97.5±3.4 | 2.5 |
| Oxalic | 20 | 20.9±1.6 | 4.8 |
| | 30 | 29.4±2.5 | 2.0 |
| | 100 | 98.5±3.2 | 1.6 |

### Recovery

The accuracy of the RP/HPLC-UV method for the determination of ascorbic and oxalic acids was also evaluated by calculating the recovery according to the standard addition method to eliminate possible matrix effects. Initially, the amount of ascorbic and oxalic acids in freshly squeezed orange juice was determined. After finding the initial amount, the solution was diluted 5 times, and the addition method was then performed. 20, 25, 50, and 100 mg $L^{-1}$ ascorbic acid were added to the diluted juice. The recovery results obtained are given in Table 3. According to Table 4, recovery values in the range of 81-102% in apple juice and 81-103% in orange juice were obtained for the studied organic acids. The low recovery value might be due to the sample preparation. This step included filtration and solid-phase extraction. These values indicate that the accuracy of the applied analytical method is highly efficient and applicable for the simultaneous determination of ascorbic and oxalic acids in various industrial and freshly squeezed fruit juices.

Table 3. Recovery of ascorbic acid added to fresh orange juice

| Initial Content (mg $L^{-1}$) | Added (mg $L^{-1}$) | Found (mg $L^{-1}$) | Recovery±Standard Deviation (%) |
|---|---|---|---|
| 35.0±1.6 | 25 | 55.5±2.5 | 82±2.2 |
| | 50 | 85.7±3.2 | 101±1.5 |
| | 100 | 127.0±4.1 | 92±1.7 |

Table 4. Recovery of organic acids added to industrial apple and orange juices

| Juice | Organic Acid | Initial Content (mg $L^{-1}$) | Added (mg $L^{-1}$) | Found (mg $L^{-1}$) | Recovery±Standard Deviation (%) |
|---|---|---|---|---|---|
| Apple | Ascorbic | 4.0±0.6 | 20 | 22.0±1.7 | 90.2±2.1 |
| | | | 50 | 53.1±2.7 | 98.3±1.7 |
| | | | 100 | 106.0±3.1 | 102.0±1.3 |
| | Oxalic | 72.0±1.3 | 20 | 88.3±2.4 | 81.7±2.2 |
| | | | 50 | 121.1±2.5 | 98.1±1.4 |
| | | | 100 | 174.5±4.9 | 102.5±1.6 |
| Orange | Ascorbic | 26.7±1.2 | 20 | 44.5±1.5 | 89.2±2.4 |
| | | | 50 | 74.1±1.3 | 94.8±1.7 |
| | | | 100 | 130.1±4.1 | 103.3±1.4 |
| | Oxalic | 12.7±1.4 | 20 | 29.1±1.5 | 81.7±2.3 |
| | | | 50 | 64.1±1.6 | 102.7±1.7 |
| | | | 100 | 113.0±3.9 | 100.3±1.3 |

## Stability Results for Ascorbic Acid

The stability of ascorbic acid was also examined in freshly squeezed and industrial orange juice samples at room temperature after dilution with the mobile phase. The label information on the industrial orange juice recommended consumption within 3 days of opening the package. In the case of the chosen industrial orange juice, the ascorbic acid concentration was intermittently analyzed during the first 48 hours after the package was opened (Figure 2). The ascorbic acid concentration decreased by 1.5% after 4 hours of opening the package, by 9% after 24 hours, and by 18.5% after 48 hours. Thus, after opening the package of industrial orange juice and diluting the sample, it was observed that there was no significant decrease in ascorbic acid concentration 48 hours later (2 days) compared to the initial concentration ($p < 0.05$). Therefore, it is possible to accurately analyze orange juice without significant ascorbic acid loss for at least 48 hours after opening the package. Similarly, fresh-squeezed orange juice was also monitored for a decrease in ascorbic acid concentration for 48 hours after dilution with the mobile phase, and the results were comparable to those obtained with industrial juice (Figure 3).

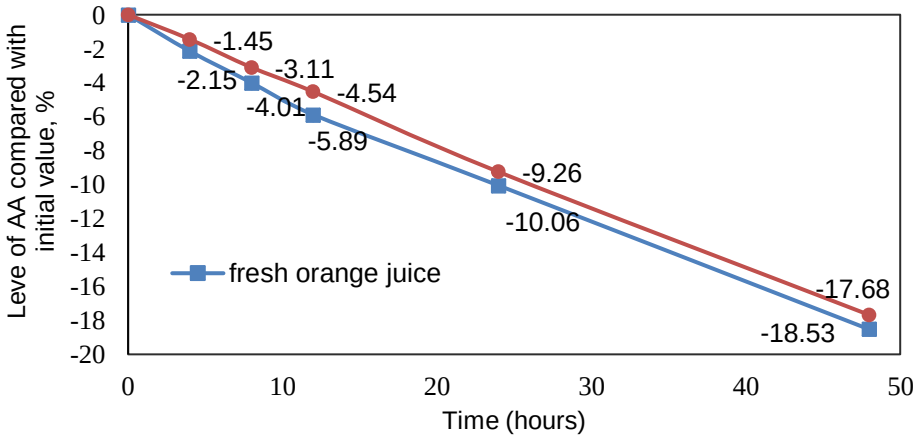


Figure 3. Stability of ascorbic acid in freshly squeezed and industrial orange juice at room temperature

## Real Sample Analysis

The proposed method was applied to determine ascorbic and oxalic acids in various brands of beverages purchased from the local market (Table 5).

According to Table 5, ascorbic acid concentration was highest in orange juice (133.5±5.07 mg $L^{-1}$) and oxalic acid concentration was highest in apple juice (360±4.28 mg $L^{-1}$). Both ascorbic and oxalic acids were found in the fruit juices studied. The recommended daily allowance (RDA) of vitamin C is 100-120 mg $day^{-1}$ [1]. RDA is the recommended daily intake. It is the minimum amount needed daily in nutrients that varies depending on age, gender, and body weight.

When daily vitamin C intake is adequate and regular, the risks of heart disease, cancer, and stroke are reduced. The daily amount of vitamin C that should be taken from the diet varies between 35-100 mg depending on various factors. This amount is estimated to be 35 mg for infants, 60 mg for adults, and 100 mg for nursing mothers [1]. According to the Food and Agriculture Organization, the RDI value of ascorbic acid for adults is recommended as 45 mg [25]. According to some data in the literature, the concentration of ascorbic acid in orange juice was found to be 32±1.2 mg 100 $mL^{-1}$ [26], 344.3 mg $L^{-1}$ [27], 490 mg $L^{-1}$ [28]. Accordingly, the concentration of ascorbic acid in orange juice in our study (133.5±0.07 mg $L^{-1}$) is lower than the literature data. We can think that this may be due

to the origin of the fruit and/or the difference in temperature conditions during storage and transportation after the fruit has become juice. The concentration of ascorbic acid in the studied orange juice does not meet the daily intake of one glass of orange juice (200 mL, 26.8 mg). Therefore, it may be recommended to drink at least two glasses of orange juice per day. Freshly squeezed orange juice had a higher concentration of ascorbic acid (35 mg $L^{-1}$) than industrial orange juice. This means that consuming freshly squeezed orange juice will provide the body with a higher intake of vitamin C than industrial orange juice. So freshly squeezed orange juice should be consumed. In addition to orange juice, ascorbic acid concentration in cold tea was also found to be high (98.5±2.2 mg $L^{-1}$). The concentration of ascorbic acid in other fruit juices studied was about one-tenth of the concentration in orange juice and was low.

Table 5. Concentrations of ascorbic and oxalic acids in various beverages

| Beverage Type | Ascorbic Acid Concentration (X±S.D, mg $L^{-1}$) | Oxalic Acid Concentration (X±S.D, mg $L^{-1}$) |
|---|---|---|
| Orange | 133.5±5.1 | 63.5±2.6 |
| Cherry | 15.0±1.8 | 271.0±5.1 |
| Pomegranate | 13.0±1.2 | 172.0±3.7 |
| Apple | 20.0±1.1 | 360.0±4.3 |
| Apricot | 12.5±1.1 | 220.0±3.5 |
| Mix | 22.5±1.2 | 274.0±4.4 |
| Ice Tea | 98.5±2.2 | 44.0±1.4 |

Table 6 gives a summary of the literature studies with similar analysis methods to our study. Each study has advantages over the other in terms of LOD, LOQ, and recovery values in terms of analysis method. In the developed method, ascorbic acid and oxalic acid were determined simultaneously. This method is of more practical importance, especially for the simultaneous determination of trace levels of oxalic acid with ascorbic acid.

Table 6. Comparison of the described method with other methods in the literature

| Compound | $t_R$[1] | Range (ppm) | Mobile Phase | FR[2] | IV[3] | WL[4] | Column Type | %R | LOQ (ppm) | LOD (ppm) | RsDr (%) | RsDt (%) | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ascorbic Acid | 4.3 | 1-100 | %0.1 Formic acid | 1.0 | 10 | 245 | ODS-3 | | | | | | [29] |
| Ascorbic Acid | 4.2 | 2-100 | Phosphate buffer pH:2.2 | 1.0 | 20 | 245 | ODS-4 | 93 | 2.00 | 0.50 | 0,9 | 4.5 | [27] |
| Oxalic Acid | 2.2 | 5-80 | Phosphate buffer pH:2.2 | 1.0 | 20 | 210 | ODS-4 | 105 | 5.00 | 1.00 | 4.9 | 5.4 | |
| Ascorbic Acid | 4.2 | 6-330 | Phosphate buffer pH:2.6 | 0.5 | 20 | 250 | RP-C18 | | 0.03 | 0.10 | 1.9 | 3.8 | [26] |
| Ascorbic Acid | 5.3 | 1-215 | Phosphate buffer/ACN pH:4.75 | 1.2 | 20 | 205 | S5NH2 | 94 | | 0.18 | 0.9 | 4.0 | [30] |
| Oxalic Acid | 11.9 | 15-150 | Sulfuric acid pH:2.5/MeOH | 0.35 | 20 | 215 | VP-ODS | 85 | | 0.94 | 5.2 | 4.5 | [31] |
| Ascorbic Acid | 14.5 | 10-100 | Sulfuric acid pH:2.5/MeOH | 0.35 | 20 | 215 | VP-ODS | 106 | | 5.17 | 0.3 | 1.6 | |
| Ascorbic Acid | 8.3 | 1-100 | Aqueous sulfuric acid pH:2.2 | 0.6 | 20 | 210 | ODS | | 1.05 | 0.34 | 1.0 | 1.2 | [24] |
| Oxalic Acid | 5.7 | 0,1-100 | Aqueous sulfuric acid pH:2.2 | 0.6 | 20 | 210 | ODS | | 4.08 | 1.34 | 0.2 | 1.4 | |
| Ascorbic Acid | 6.2 | 10-500 | Phosphate buffer pH:2.5 | 1.0 | 20 | 245 | ODS-3 | 93 | 4.20 | 1.40 | 4.1 | 3.9 | This Study |
| Oxalic Acid | 3.1 | 15-500 | Phosphate buffer pH:2.5 | 1.0 | 20 | 205 | ODS-3 | 104 | 3.90 | 1.30 | 4.6 | 4.3 | |

[1]Retention time (min), [2]Flow rate (mL $min^{-1}$), [3]Injection volume (microliter), [4]Wavelength (nm)

## CONCLUSIONS

The present study describes the simultaneous analysis of ascorbic and oxalic acids in various fruit juices using RP/HPLC-UV. The linearity of the method is very high over a wide concentration range ($R^2$ 0.997-1.002). The relative error is less than 5%. The wide linear range obtained allows the determination of ascorbic and oxalic acids in more samples with the current method. The theoretical LOD and LOQ were 1.4 mg $L^{-1}$ and 4.2 mg $L^{-1}$ for ascorbic acid and 1.3 mg $L^{-1}$ and 3.9 mg $L^{-1}$ for oxalic acid, respectively. Extraction recoveries were between 82-103.3% for ascorbic acid and 81.7-102.5% for oxalic acid. The proposed method was applied to a real sample. Ascorbic acid was highest in orange juice and oxalic acid was highest in apple juice. Ascorbic acid was found in higher concentrations in freshly-squeezed orange juice than in industrial orange juice. But even this value (35 mg) is lower than the RDI for ascorbic acid for adults (45 mg), according to the Food and Agriculture Organization. In this case, drinking 2 glasses (400 mL) of freshly-squeezed orange juice can be recommended to get enough vitamin C daily. The developed method provides an easy, simple, and acceptable solution for the accurate, reliable, and precise simultaneous determination of ascorbic and oxalic acids in a variety of fruit juices with a wide linear range and interchangeable LOD and LOQ values. The RP/HPLC-UV method used offers convenience with uncomplicated sample preparation. This method is suitable for high-throughput analysis of multiple samples.

## DISCLOSURE STATEMENT

The authors report no conflict of interest.

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**Research Paper / Araştırma Makalesi**

# Consumer Interest and Its Effect on Purchase Intention for Plant-Based Milk Substitutes

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## ABSTRACT

Interest in plant-based milk substitutes has been increasing steadily. This growing demand can be attributed to various factors, including health concerns, environmental awareness, and ethical values. Among the health-related reasons are lactose intolerance and cow's milk allergy, both of which have become increasingly prevalent worldwide. Additionally, the rising diversity in consumer diets, such as veganism and vegetarianism, has made plant-based milk substitutes a suitable alternative for these groups. Plant-based milk substitutes are suspensions made from various raw materials, such as cereals, pseudo-cereals, legumes, nuts, and seeds, dissolved in water. These products resemble cow's milk in appearance. In many countries, including Türkiye, the term "milk" is used in a broader sense to refer not only to animal-derived milk but also to plant-based milk-like beverages, which have become more prominent in recent years. The purpose of this study was to investigate the relationship between plant-based milk substitutes, consumer interest in these products, and purchase intention. A total of 400 consumers aged 18 years and older participated in the study, with 392 valid questionnaires included in the analysis. Correlation analysis was conducted to evaluate the relationship between consumer interest and purchase intention, while regression analysis was used to determine the effects of interest on purchase intention. Results of collinearity analyses revealed a positive and significant relationship between consumer interest and purchase intention. Furthermore, the regression analysis demonstrated that consumer interest has a significant impact on purchase intention. It was concluded that as consumer interest increases, purchase intention also rises.

**Keywords:** Plant-based milk substitute, Consumer Interest, Purchase Intention

## Tüketici İlgisi ve Bitki Bazlı Süt Alternatiflerine Yönelik Satın Alma Niyeti Üzerindeki Etkisi

## ÖZ

Bitki bazlı süt alternatiflerine olan ilgi giderek artmaktadır. Bu artan talep, sağlık endişeleri, çevresel farkındalık ve etik değerler gibi çeşitli faktörlere dayandırılabilir. Sağlıkla ilgili nedenler arasında laktoz intoleransı ve inek sütü alerjisi yer almakta olup, her ikisi de dünya genelinde giderek daha yaygın hale gelmiştir. Ayrıca, veganlık ve vejetaryenlik gibi tüketici diyetlerindeki çeşitliliğin artması, bitki bazlı süt alternatiflerini bu gruplar için uygun bir seçenek haline getirmiştir. Bitki bazlı süt alternatifleri, tahıllar, psödo-tahıllar, baklagiller, sert kabuklu meyve ve tohumlar gibi çeşitli hammaddelerin suda çözülmesiyle elde edilen süspansiyonlardır. Bu ürünler, görünüm olarak inek sütüne benzemektedir. Türkiye dahil birçok ülkede, "süt" terimi, yalnızca hayvansal kaynaklı sütü değil, aynı zamanda son yıllarda daha belirgin hale gelen bitki bazlı süt benzeri içecekleri de kapsayacak şekilde daha geniş bir anlamda

kullanılmaktadır. Bu çalışmanın amacı, bitki bazlı süt alternatifleri, bu ürünlere olan tüketici ilgisi ve satın alma niyeti arasındaki ilişkiyi araştırmaktır. Çalışmaya 18 yaş ve üzeri toplam 400 tüketici katılmış olup, analizde 392 geçerli anket değerlendirilmiştir. Tüketici ilgisi ile satın alma niyeti arasındaki ilişkiyi değerlendirmek için korelasyon analizi yapılmış, ilginin satın alma niyeti üzerindeki etkilerini belirlemek için ise regresyon analizi uygulanmıştır. Kollineerlik analizlerinin sonuçları, tüketici ilgisi ile satın alma niyeti arasında pozitif ve anlamlı bir ilişki olduğunu ortaya koymuştur. Ayrıca, regresyon analizi, tüketici ilgisinin satın alma niyeti üzerinde anlamlı bir etkisi olduğunu göstermiştir. Sonuç olarak, tüketici ilgisi arttıkça satın alma niyetinin de arttığı sonucuna varılmıştır.

**Anahtar Kelimeler:** Bitki bazlı süt ikamesi, Tüketici ilgilenimi, Satın alma niyeti

## INTRODUCTION

A visit to the dairy section of any grocery store shows a wide range of plant-based milk substitutes based on a wide variety of herbal products. The popularity of plant-based milk substitutes has significantly increased in recent years. Many consumers today limit or avoid milk and dairy consumption for a variety of reasons, including milk protein allergies, lactose intolerance, personal and environmental health concerns, and dietary differences, such as vegetarian diets [1]. In this context, sales of plant-based milk substitutes have been steadily increasing at a high rate over the years. The global plant-based milk substitutes market is estimated to reach a market size of $47.2 billion by 2033, with an annual growth rate of 9.9% between 2023 and 2033 [2].

Plant-based milk substitutes are defined as aqueous extracts from raw plant materials that are a source of protein and calories for human consumption, have a lower percentage of fat compared to cow's milk, and are cholesterol- and lactose-free [3]. In addition to the advantageous effect they provide to consumers due to their nutritional value, plant-based milk substitutes are also economically favorable for the industry, as their production costs can be lower than those of cow's milk. Another advantage is that food industry residues that are rich in proteins and carbohydrates can be used as raw materials. These materials are often discarded or sold cheaply as feed. However, when they are recycled, environmental benefits such as less water is needed to treat wastewater and the impact on climate change and ecotoxicity can be reduced [4].

Plant-based milk substitutes are extracts obtained by dissolving nuts, cereals, pseudocereals, seeds, and legumes in water. Although plant-based milk substitutes do not contain the nutritional content of animal dairy products, they have similar sensory and functional properties. They have been described as a healthy alternative product group owing to their nutrient content, including healthy fatty acids and carbohydrates, as well as B, E, and antioxidants. They are also an important product group for consumers with protein allergies and lactose intolerance [5]. Plant-based milk substitutes are considered a food source for the development of products that can be an alternative to animal milk and for the production of products with nutritional content that can be considered as qualified products. Plant-based milk substitutes contain minerals, vitamins, and many health-beneficial components, and are considered functional products. They are offered as nutritious alternative products, such as special drinks, cheese, and yogurt, for consumers suffering from protein allergies and lactose intolerance as well as for consumers following a vegan diet [6].

Although different raw materials are used in the production of plant-based milk substitutes, their production stages are generally similar. First, the raw material was soaked in water for a few hours prior to processing. The mixing process was then carried out with water. After the mixture was obtained, filtration was performed to separate the residues that did not dissolve in water, and components such as sweeteners, sugar, and stabilizers were added upon request. In the last production step, stability, homogenization, and pasteurization processes were carried out (Figure 1). Liquid extracts were obtained using these processes [7].

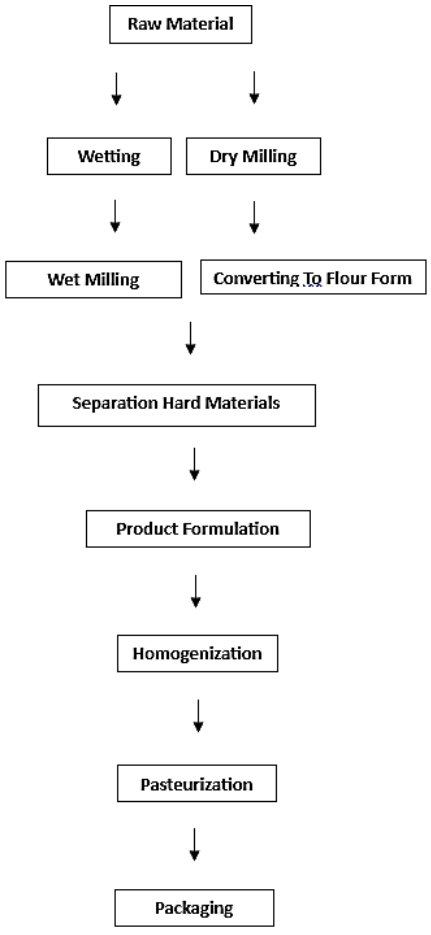


Figure 1. Production scheme of plant-based milk substitutes [8]

To improve the quality and acceptability of plant-based milk substitutes, a number of technological interventions have been applied to improve product stability, eliminate bad tastes, inactivate or remove inhibitors, and improve shelf life [9].

Table 1 shows the processes applied for the production of plant-based milk substitutes and their advantages.

Table 1. Technological interventions applied to the production of plant-based milk substitutes and their objectives [4]

| Ultrasound procedure | Pulsed electric field | High intensity ultrasound irradiation | Ohmic heating | Ultra-high and high pressure homogenization |
|---|---|---|---|---|
| Improving shelf life | Improving shelf life<br>Inactivation of inhibitors | Sedimentation and product stability | Removal of bad taste and aroma | Improving shelf life<br>Sedimentation and product stability |

Today's consumers are becoming more nutritionally conscious and changing their eating habits towards a healthier, sustainable and ethical diet, which is reflected in the demand for plant-based milk substitutes. However, the majority of consumers also have reservations about including such beverages in their daily diet.

Some factors limit consumers' consumption of plant-based milk substitutes. The most important of these factors are taste and appearance. Taste and appearance are the most important purchasing criteria of consumers. The plant material used as raw material in plant-based milk substitutes may have a green, gray, and brownish color due to the natural color of the plant material used as raw material, has a sandy structure due to its insoluble particles, and the feeling it leaves in the mouth, which can negatively affect consumers' desire to buy [10].

Another factor is that consumers lack knowledge of the social, economic, and health impacts of replacing animal-based foods with plant-based foods in their dietary preferences. Economically, it is also noted that in some countries, plant-based milk substitutes are offered at high prices, making it difficult for consumers from all walks of life to access these products [8].

The aim of this research is to measure the relationship between consumers' interest and purchase intention towards plant-based milk, which has entered our lives for various reasons, considering the increasing demand for cow's milk substitutes that have no allergic effects, nutritional, sensory, and technological properties, and to raise awareness.

## METHODS

This study examines the consumer profile of plant-based milk and the relationship between consumer interest and purchase intention. The main mass of research consists of individuals aged 18 years and older living in Türkiye. This study was conducted in January and February 2024. The research sample comprised 400 people, and a face-to-face and online survey method was applied. Eight questionnaires found to be incomplete and inaccurate were excluded, and 392 questionnaires were included in the analysis.

The questionnaire consists of three parts. In the first part, a 7-point semantic differences scale was developed by Zaichkowsk [11]. It consists of 10 items (worthless/ valuable, unattractive/ attractive, ordinary/ magical, means nothing to me/ means something to me, unnecessary/ necessary, boring/ interesting, unimportant/ important, irrelevant/ relevant to me, unexciting/ exciting, and inappropriate/ appropriate) to measure the participants' level of interest in plant-based milk.

In the second part of the study, in order to measure the participants' purchase intention towards plant-based milk, a 4-item, 5-point Likert (1: strongly disagree, 5: strongly agree) purchase intention scale was taken from the research site www.surveymonkey.com [12].

The last part of the questionnaire contained five questions (gender, age, education, occupation, and income) to reveal the demographic characteristics of the participants.

## RESULT and DISCUSSION

The demographic characteristics of the participants are presented in Table 2. Accordingly, it can be said that the gender distribution of the participants is approximately equal. Approximately two-thirds of the participants were aged between the ages of 21-40. It can be said that the marital status of the participants is approximately half and half. Most of the participants (77.5%) were university graduates, and the proportions of workers, civil servants, and self-employed were close to each other. Civil servants provided the highest level of participation by civil servants. Consumer interest in milk and its substitutes is influenced by advertising and promotion [13], product quality and price [14], sociodemographic factors [15], [16], and consumer education [17].

Studies have shown that young people are likely to prefer milk [18]. Similar studies have determined that age affects purchasing behavior, and that the level of education has a significant effect on purchasing behavior [15]. In this respect, when the age range of the participants was taken into consideration and the effect of occupational groups was also evaluated, it can be expected that the results of the analyses would be positively affected. Young people's interest in such product groups is likely to affect their interest and

purchase intentions. Especially in young people under the age of 30, the likelihood of trying and preferring plant-based milk and its products increases because of health concerns, environmental awareness, and veganism, and concerns such as the following trends among young people [19-21].

Table 2. Demographic characteristics of participants in the study

| | F | Percentage | | F | Percentage |
|---|---|---|---|---|---|
| Gender | | | Marital status | | |
| Woman | 208 | 53.1 | Single | 207 | 52.8 |
| Male | 184 | 46.9 | Married | 185 | 47.2 |
| Age | | | Profession | | |
| 20 and under | 73 | 18.6 | Not working | 43 | 11.0 |
| 21-30 | 164 | 41.8 | Worker | 97 | 24.7 |
| 31-40 | 96 | 24.5 | Officer | 121 | 30.9 |
| 41-50 | 41 | 10.5 | Self-employment | 96 | 24.5 |
| 51 and over | 18 | 4.6 | Student | 35 | 8.9 |
| Education | | | Revenue | | |
| Primary education | 34 | 8.7 | 15000 TL and under | 98 | 25.0 |
| High school | 54 | 13.8 | 15001-25000 TL | 143 | 36.5 |
| Associate degree | 126 | 32.1 | 25001-35000 TL | 75 | 19.1 |
| Bachelor's degree | 95 | 24.2 | 35001-45000 TL | 46 | 11.7 |
| Postgraduate | 83 | 21.2 | 45001 TL and over | 30 | 7.7 |

The Kaiser-Meyer-Olkin (KMO) value of the scale used in the study was 0.936 and the Bartlett Sphericity Test value was p<0.05 (p:0.000) and it was determined that the scale was suitable for factor analysis. The results of confirmatory and exploratory factor analyses are presented in Table 3. Accordingly, the interest scale consisted of a single dimension and was represented by 10 items. Purchase intention consists of a single dimension, and is represented by three items. The scale items represented 68% of the total explained variance. The reliability values were 0.953 for the interest scale and 0.888 for the purchase intention scale. Thus, the scale is highly reliable.

Table 3. Shopping influencers scale factor analysis results

| Items | Factor Loadings | Eigenvalue | Variance Explained | Cronbach α |
|---|---|---|---|---|
| Factor 1. Interest | | 8.286 | 59.184 | 0.953 |
| Worthless/Valued | 0.838 | | | |
| Not Attractive/Attractive | 0.828 | | | |
| Ordinary/Enchanting | 0.805 | | | |
| Doesn’t/do not mean anything to me | 0.792 | | | |
| Unnecessary/Necessary | 0.774 | | | |
| Boring/Interesting | 0.769 | | | |
| Unimportant/Important | 0.742 | | | |
| Irrelevant to me/Relevant for me | 0.733 | | | |
| Unexciting/Exciting | 0.719 | | | |
| Not suitable / Suitable | 0.708 | | | |
| Factor 2. Purchase Intention | | 1.293 | 9.239 | 0.888 |
| I plan to buy milk-like herbal drinks in 6 months. | 0.889 | | | |
| I plan to buy milk-like herbal drinks in 9 months. | 0.858 | | | |
| I plan to buy milk-like herbal drinks in 3 months. | 0.821 | | | |

Statistical data revealing the interest and purchase intention profiles of the participants towards plant-based milk are presented in Table 4. Accordingly, participants' level of interest in plant-based milk was high ($\bar{x}$=4.11). Although the participants' purchase intention towards plant-based milk was lower than interest ($\bar{x}$=3.12), it can be said that it was above average. These results show that the participants were interested in plant-based milk but were more hesitant to purchase it. Plant-based milk substitutes are generally more expensive than animal milk. This is thought to be because of the costlier processing of the product and the limited number of companies engaged in production. Some studies state that price changes positively affect purchase probabilities [21, 22]. Other situations identified in the literature are that consumers find plant-based milk substitutes and analogue products obtained from them nutritionally inadequate and sensory inappropriate; as a result, they do not want to buy them [19, 21, 23]. However, more processed packaged product groups may also contain negative messages for consumers. Consumers may classify plant-based milk substitutes as more processed products and, therefore, may choose not to prefer them [19].

Table 4. Shopping influencers statistics

| | $\bar{x}$ | S |
|---|---|---|
| Interest | 4.1133 | 1.7388 |
| Purchase Intention | 3.1259 | 1.13te 36 |

The findings of the correlation analysis applied to examine the possible relationship between interest in and purchase intention towards plant-based milk are presented in Table 5. Accordingly, there is a significant positive relationship between interest and purchase intention.

Table 5. Correlation analysis for the relationship between the sub-dimensions of the scales

| | | Purchase Intention |
|---|---|---|
| Interest | Pearson Correlation | 0.642** |
| | Sig. (2-tailed) | 0.000 |
| | N | 392 |

**Significant relationship at 0.01 significance level

According to the results of the correlation analysis, the results of the regression analysis conducted to reveal the strength of the relationship between the variables with a significant positive relationship are presented in Table 6.

Accordingly, consumers' interest has a significant effect on purchase intention. Accordingly, as interest increases, purchase intention also increases. There is a fixed purchase intention of 1.403 units for plant-based milk and each unit increase in interest increases purchase intention by 0.642.

Table 6. Regression analysis for the relationship between the sub-dimensions of the scales

| Independent Variable | Dependent Variable | Fixed* | (β) | St. Error | (t) | p |
|---|---|---|---|---|---|---|
| Interest | Purchase Intention | 1.403 | 0.642 | 0.025 | 16.550 | 0.000 |

*The regression equation: Purchase Intention = 1.403 + 0.642×Interest

The results of research conducted in January and February 2024 across Turkey contain similar findings to studies on consumer interest and purchase intention in the literature. In our country, people are interested in plant-based milk substitutes, but remain somewhat hesitant about purchasing them. This interest is generally influenced by increasing milk allergy and perception of healthy nutrition [4, 8, 24], curiosity and perception of social media [25-27] environmental impacts of milk production facilities, protection of animal rights, and preference for a vegan lifestyle [27-29].

## CONCLISION

The results of the study show a strong relationship between consumer interest in plant-based milk substitutes and purchase intention. Consumers were found to be interested in and intended to purchase plant-based milk substitutes due to factors such as health concerns, environmental impacts, and ethical values. In this study, purchase intention was found to be lower than the level of interest, suggesting some hesitation among consumers to purchase these products. This hesitation could be due to factors such as economic reasons and the taste and texture characteristics of the product. Furthermore, based on the research results, it was observed that consumer interest in plant-based milk substitutes and purchase intentions are associated with consumers' demographic characteristics. For example, young and middle-aged respondents were found to have higher interest and purchase intentions towards plant-based milk substitutes than other age groups. Similarly, the interest and purchase intention of respondents with higher levels of education towards plant-based milk substitutes were more pronounced than for those with lower levels of education. These results provide important guidance for developing marketing strategies and communicating effectively with consumers regarding plant-based milk substitutes. Improving the taste and texture characteristics of the product, reviewing pricing, and providing consumers with more information on the health benefits and environmental impacts of the product could increase purchase intention and expand the market share of plant-based milk substitutes. In conclusion, consumer interest in and purchase intention for plant-based milk substitutes is a complex process shaped by the interaction of health, environmental, and social factors. Future research could conduct further analysis on this topic to gain a deeper understanding of the effects of these factors on consumer behavior and contribute to more effective management of marketing strategies for plant-based milk substitutes.

## CONFLICT OF INTEREST:

The authors declare that they do not have any conflict of interest.

## ETHICAL REVIEW

Necessary ethical documents were obtained and expressed before analysis.

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**Araştırma Makalesi / Research Paper**

# Limonlarda Bulunan Bazı Fungisit Kalıntıları Üzerine Evsel Gıda İşleme Yöntemlerinin Etkisi

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## ÖZ

Bu çalışmanın amacı, limon meyvesi ve ürünlerindeki bazı fungisit kalıntılarına evsel işleme yöntemlerinin etkilerini belirlenmektir. Araştırmada, limon ve işlenmiş ürünlerinde imazalil ve thiophanate-methyl kalıntılarının analizi için QuEChERS (Hızlı, Kolay, Ucuz, Etkili, Sağlam ve Güvenli) yöntemi başarıyla uygulanmıştır. Limonun meyve eti, suyu ve reçel gibi işlenmiş ürünlerinde gerçekleştirilen analizler, pestisit kalıntı seviyelerinin %88 ile %100 arasında önemli ölçüde azaltılabildiğini ortaya koymuştur. Bu durum, pestisitlerin fizikokimyasal özelliklerine (log Po/w değeri, polarite ve çözünürlük gibi) ve limonun biyolojik yapısına bağlanmıştır. Ancak, limon kabuğu ve rendelenmiş dondurulmuş kabuk gibi ürünlerde pestisit kalıntı seviyelerinde artış gözlenmiştir. Bu artış, pestisitlerin kabuk yüzeyinde birikme eğilimiyle ilişkilendirilmiştir. Sonuç olarak, işleme faktörlerinin pestisitlerin fizikokimyasal özelliklerine ve uygulanan işleme yöntemlerine bağlı olarak değiştiği görülmüştür. Meyve eti, limon suyu ve reçel gibi ürünlerde işleme faktörleri 1'den küçük bulunmuş, bu da bu işlemlerin pestisit kalıntılarını azaltmada etkili olduğunu göstermektedir. Öte yandan, kabuklu ürünlerde işleme faktörlerinin 1'den büyük olduğu ve bu işlenmiş ürünlerde kalıntı birikiminin daha fazla olduğu tespit edilmiştir.

**Anahtar Kelimeler:** Limon (*Citrus lamas*), Pestisit kalıntıları, Fungisit, Evsel gıda işlemleri, İşleme faktörü

## Effect of Household Food Processing Methods on Some Fungicide Residues in Lemons

## ABSTRACT

The aim of this study is to determine the effects of household processing methods on certain fungicide residues in lemon fruit and its products. In the research, the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method was successfully applied for the analysis of imazalil and thiophanate-methyl residues in lemons and processed products. Analyses on the fruit pulp, juice, and processed products like jam revealed that pesticide residue levels could be significantly reduced by 88 to 100%. This was attributed to the physicochemical properties of the pesticides (e.g., log Po/w value, polarity, and solubility) and the biological structure of lemon. However, an increase in pesticide residue levels was observed in products such as lemon peel and grated frozen peel. This increase was associated with the tendency of pesticides to accumulate on peel surface. Consequently, it was observed that processing factors varied depending on the physicochemical properties of the pesticides and the processing methods applied. Processing factors were less than 1 in products such as fruit pulp, lemon juice, and jam, indicating that these processes are effective in reducing pesticide residues. On the other hand, processing factors were greater than 1 in peel-containing products, indicating higher residue accumulation in these processed products.

**Keywords:** Lemon (*Citrus lamas*), Pesticide residues, Household food processing, Processing factor

## GİRİŞ

Limon, zengin besin içeriği ve sağlık üzerindeki çeşitli yararlarıyla dikkat çeken bir turunçgil meyvesidir. Yüksek C vitamini içeriği, bağışıklık sistemini desteklemenin yanı sıra hücresel oksidatif stresi azaltarak kronik hastalıklara karşı koruma sağlamaktadır [1]. Öte yandan, limonun besin profili potasyum, kalsiyum ve magnezyum gibi temel minerallerin yanı sıra anti-inflamatuar özelliklere sahip flavonoidleri de içermektedir; bu biyoaktif bileşiklerin kardiyovasküler sağlığı desteklediği ve yaşlanma belirtilerini azaltmada etkili olduğu bilinmektedir [2, 3]. Son yıllarda dünya genelinde ve Türkiye'de limon üretiminde bir artış gözlemlenmektedir. 2023-2024 döneminde küresel narenciye üretiminin yaklaşık 103.7 milyon tona ulaşacağı öngörülmektedir. Türkiye'de ise özellikle Akdeniz ve Ege bölgelerinde yoğunlaşan limon üretimi, 2022-2023 sezonunda yaklaşık 1.4 milyon ton olarak gerçekleşmiştir. Türkiye, küresel limon ticaretinde önemli bir konuma sahip olup üretiminin %50'sinden fazlasını ihraç etmektedir [4]. Dünya çapında İspanya, Arjantin ve Meksika gibi ülkelerle rekabet eden Türkiye, özellikle Avrupa pazarında güçlü bir yere sahiptir. FAO verileri, dünya genelinde limon üretim alanlarının genişlediğini ve artan talebe bağlı olarak üretimin de yükseldiğini göstermektedir [5].

Limon üretimindeki bu artış, aynı zamanda pestisit kalıntılarının kontrol edilmesi gerekliliğini de gündeme getirmektedir. Limonun besin değeri kadar pestisit kalıntılarının gıda güvenliği ve insan sağlığı açısından oluşturduğu riskler de önemlidir [6, 7]. Tarımda yaygın olarak kullanılan pestisitlerin yanlış veya aşırı dozlarda uygulanması, ürünlerde istenmeyen düzeyde kalıntı birikimine yol açarak tüketici sağlığını olumsuz etkileyebilmektedir. Bu doğrultuda, limon gibi ürünlerin tüketici güvenliğini artırmak amacıyla çeşitli gıda işleme yöntemleri uygulanarak pestisit kalıntıları azaltılmaktadır. Bu bağlamda, pestisitlerin insan sağlığı üzerindeki olası etkileri ve işleme yöntemlerinin bu kalıntıları azaltmadaki etkinliğini inceleyen çalışmalar önemli bir yere sahiptir [7-12].

Tarımsal ürünlere zarar yapan ve ürün kayıplarına neden olan pestleri (böcek, mikroorganizma, fungus ve yabancı) kontrol altına almak amacıyla çeşitli pestisitler yaygın olarak kullanılmaktadır [13-15]. Bu bağlamda, sistemik özellikteki imazalil ve thiophanate-methyl, fungisitleri özellikle turunçgil ürünlerinde sıkça uygulanan iki önemli pestisit olarak öne çıkmaktadır [6, 11, 16, 17]. İmazalil, turunçgillerde fungal hastalıkların kontrolünü sağlamak ve ürünün raf ömrünü uzatmak amacıyla öncelikli olarak tercih edilmektedir. Hidrofobik özelliklere sahip olması nedeniyle kabukta daha yüksek birikme eğilimindedir; bu durum, kabuğun işleme tabi tutulmasıyla pestisit kalıntı düzeyinde azalmalara yol açabilmektedir [18, 19]. Öte yandan, thiophanate-methyl, geniş spektrumlu bir fungisit olarak mantar kaynaklı hastalıkların önlenmesinde etkili olup, uygulamadan sonra bitki içinde sistemik bir dağılım göstermektedir. Her iki pestisit de özellikle turunçgillerde hasat sonrası süreçlerde kullanılmakta olup, ürünlerin sağlık ve kalite standartlarını koruma amacıyla tercih edilmektedir [20, 21]. Ancak, hasat sonrasında ürünlerde kalıntı olarak bulunabilen bu pestisitler, tüketicilerin kontrolü dışında olup, insan sağlığına zarar verebilmekte ve dünya genelinde gıda ürünlerinin ticaretine önemli bir engel teşkil etmektedir. Gıdalardaki pestisit kalıntılarının konumu; gıda materyalinin türü ve miktarı, kullanılan pestisit molekülünün kimyasal özellikleri ve çevresel koşullara bağlı olarak değişiklik gösterebilmektedir [22]. Ancak, bu pestisitlerin kalıntıları tüketici sağlığı ve çevre açısından olumsuz etkiler yaratabileceğinden, ürünlerdeki kalıntı seviyelerinin kontrolü ve azaltılmasına yönelik önlemler alınması büyük önem arz etmektedir [18, 23].

Pestisitlerin tarımsal üretim süreçlerinde kullanımı, özellikle hasat öncesi ve sonrası aşamalarda mahsul verimliliğini artırarak üreticilerin ekonomik getirisini yükseltmektedir. Ancak, bu kimyasalların hatalı veya gereğinden fazla kullanımı, gıda ürünlerinde kalıntı birikimine yol açarak insan sağlığı üzerinde potansiyel zararlara ve çevresel kirliliğe neden olabilmektedir. Bu durum, hem gıda güvenliği standartlarını tehdit etmekte hem de sürdürülebilir tarım uygulamaları üzerinde olumsuz etkiler yaratmaktadır [24-26]. Hasat döneminde tüketilen ham tarımsal ürünlerde (Raw Agricultural Commodity-RAC) maksimum kalıntı limitlerinin (MRL) aşılması riski bulunmakta olup, tüketim öncesinde bu ürünlerin büyük çoğunluğu çeşitli işleme aşamalarından geçirilmektedir [27]. Nitekim, gıdalardaki pestisit kalıntı seviyeleri; yıkama, soyma, meyve suyu sıkma, kaynatma, kurutma, fermantasyon veya pişirme gibi temel işleme yöntemleriyle azaltılabilir. Bununla birlikte, pestisitlerin işleme süreçlerinde kimyasal yapı değişikliklerine uğrayarak toksisiteyi artırabileceği veya azaltabileceği de bilinmektedir. Özellikle imazalil ve thiophanate-methyl gibi pestisitlerin moleküler yapılarındaki değişiklikler, tüketici sağlığı açısından potansiyel riskler oluşturabilir [28]. Bu bağlamda meyve ve sebzelerin uygun işleme yöntemleriyle işlenmesi, kalıntı seviyelerini azaltarak tüketici güvenliğini sağlama açısından önem arz etmektedir [16, 29, 30). İşleme faktörü (İf), işlenmiş gıdalardaki pestisit kalıntı seviyesinin, ham tarımsal üründeki kalıntı seviyesine oranı olarak tanımlanır [27]. Bu değer, gıda işleme sırasında pestisitlerin ne ölçüde indirgenip indirgenmediğini belirlemek için kritik bir gösterge olarak kullanılır. İf değeri, pestisitlerin fizikokimyasal özellikleri (suda çözünürlük, log Po/w vb.) ve pestisitlerin uygulama zamanlaması gibi faktörlere bağlı olarak değişiklik gösterebilir [7, 30]. Özellikle işlenmiş ürünlerin güvenilirliği açısından, İf değerlerinin değerlendirilmesi büyük önem taşırmaktadır [31]. Sonuç olarak, pestisitlerin tarımsal üretim süreçlerinde etkin kullanımı ürün verimini artırsa da aşırı veya yanlış kullanımları sağlık risklerini beraberinde getirebilir. Bu sebeple, pestisit kalıntılarının denetimi ve kullanılan gıda işleme tekniklerinin etkinliği, gıda güvenliği ve halk sağlığı açısından hayati önem taşımaktadır. Gıda ürünlerindeki pestisit kalıntılarının insan sağlığı üzerindeki potansiyel etkileri, günümüzde önemli bir tartışma konusudur. İmazalil ve thiophanate-methyl gibi yaygın olarak kullanılan fungisitlerin kalıntılarının yüksek seviyelerde bulunması, kanserojen etkiler, hormonal dengesizlikler ve bağışıklık sisteminin zayıflaması gibi sağlık sorunları

ile ilişkilendirilmektedir [15, 32]. Bu durum, tüketici güvenliği açısından pestisit kalıntılarının daha sıkı kontrol edilmesi gerekliliğini gündeme getirmektedir.

Tüketiciler tarafından sıklıkla tercih edilen ev tipi işleme yöntemleri, pestisit kalıntılarının azaltılmasında önemli bir rol oynamaktadır. Bu çalışmanın amacı, limon meyvesinde bulunan imazalil ve thiophanate-methyl pestisitlerinin kalıntılarını azaltmaya yönelik farklı işleme tekniklerinin etkinliğini değerlendirmektir. Ayrıca, bu kimyasalların fizikokimyasal özellikleri ve halk sağlığı üzerindeki potansiyel etkileri ile ilgili literatür de kapsamlı bir şekilde ele alınacaktır. Pestisitlerin işleme süreçlerinde kimyasal yapılarındaki değişiklikler, toksisiteyi artırabileceği veya azaltabileceği için, bu tekniklerin pestisitlerin yapısal özellikleri üzerindeki etkilerini anlamak büyük önem taşımaktadır. Bu bağlamda, evde uygulanan işleme yöntemlerinin imazalil ve thiophanate-methyl kalıntılarının azaltılmasındaki etkinliğini ortaya koymak amacıyla yapılacak değerlendirme, halk sağlığı açısından kritik bir öneme sahiptir. Sonuç olarak, çalışmamız, limon meyvesindeki bu pestisit kalıntılarının ev tipi işleme teknikleriyle nasıl azaltılabileceğini incelemeyi hedeflemektedir.

## MATERYAL ve METOT

### Materyal

Bursa'daki bir pazardan aynı yıl yaklaşık 30-40 kg ağırlığında limon örnekleri (*Citrus lamas*) temin edilmiştir. Bu örnekler, analitik işlemler öncesinde 5-7°C sıcaklık ve %90-95 bağıl nem koşullarında uygun şartlarda muhafaza edilmiştir. Toplu örneklerde yer alan limonların bireysel kütleleri 156 g ile 185 g arasında değişiklik gösterdiğinden, Avrupa Komisyonu'nun yasal düzenlemeleri [33] doğrultusunda, en az 1 kg ağırlığında ve en az 10 meyve içeren laboratuvar örnekleri hazırlanmıştır. Toplu örneklerden üçü "işlem görmemiş kontrol grubu (K)" olarak ayrılmış, geri kalan laboratuvar örnekleri ise ilerleyen kısımlarda detaylandırılan pestisit uygulama sürecine tabi tutulmuştur.

### Kimyasal ve Çözeltiler

QuEChERS ekstraksiyon kitleri, 6000 mg susuz magnezyum sülfat ($MgSO_4$) ve 1500 mg susuz sodyum asetat (NaOAC) içeriğine sahip olarak tedarik edilmiştir. Ayrıca, 1200 mg $MgSO_4$ ve 400 mg birincil ve ikincil aminler (PSA, partikül boyutu 40 µm) içeren temizleme kitleri Chromabond (Almanya) firmasından sağlanmıştır. Pestisit kalıntı analizlerinde kullanılmak üzere gerekli çözücüler olan asetonitril, buzlu asetik asit, metanol ve formik asit gibi kimyasallar ise Merck (Almanya) firmasından temin edilmiştir. Pestisit kalıntı analizleri için her biri %99 saflıkta olan imazalil ve thiophanate-methyl standartları, Dr. Ehrenstorfer (Almanya) tarafından sağlanmıştır.

Çalışmada pestisit çözeltilerinin hazırlanmasında, %1 asetik asit içeren asetonitril ile 1 mg/mL konsantrasyonunda stok çözeltiler oluşturulmuştur. Bu stok çözeltiler, 20 ila 800 µg/L aralığında olacak şekilde çeşitli seyreltmeler yapılarak çalışma çözeltilerine dönüştürülmüştür. Hedef analitlerin konsantrasyon aralığını kapsayacak şekilde yedi farklı seviye matrisle eşleştirilmiş kalibrasyon standartları, çalışma çözeltilerinden türetilmiştir. Stok çözeltiler, -18 °C'de kahverengi cam şişeler içinde bir yıl boyunca saklanmış; kısa süreli çözeltiler ise 4°C'de en fazla bir hafta boyunca muhafaza edilmiştir. Tüm analizlerde, Merck National Q saflaştırma sisteminden elde edilen deiyonize su kullanılmıştır.

### Cihaz

Pestisit kalıntı analizleri için, Agilent 1260 II model LC-MS-MS-6470A sıvı kromatografisi-tandem kütle spektrometresi (LC-MS/MS) sistemi kullanılarak gerçekleştirilmiştir. Bu sistemde 2.1 mm × 150 mm × 2.7 µm boyutlarında Agilent Poroshell $C_{18}$ analitik kolonu kullanılmıştır. Kütle spektrometresi için çalışma parametreleri, 325°C ısı bloğu sıcaklığı, 400°C kurutma gazı sıcaklığı, püskürtme gazı için 10 L/dakika, kurutma gazı için 11 L/dakika ve nebülizatör gazı için 14 L/dakika azot gazı akış hızları ile ayarlanmıştır. Kılcal voltaj 3000 V olarak belirlenmiştir. Analiz, her bir bileşen için pozitif elektrosprey iyonizasyon (ESI) modunda gerçekleştirilmiştir.

Mobil faz, 0.3 mL/dakika akış hızında, %0.1 formik asit içeren 5 mM amonyum asetat (bileşen A) ve metanol-su karışımından (bileşen B) oluşmaktadır. Gradyan elüsyon programı, ilk olarak 0.5 dakika boyunca %80 A ve %20 B ile başlar, ardından 10 dakika içinde %95 B'ye doğrusal olarak artar ve 3 dakika boyunca bu oran korunur. Toplam koşum süresi (total run time) 13 dakika olup, ardından başlangıç koşullarını kullanan son 3 dakikalık bir çalışma gerçekleştirilmiştir. Çalışma sırasında akış hızı 0.5 mL/dakika ve enjeksiyon hacmi 1 µL olarak belirlenmiştir.

Çalışmada kullanılan diğer ekipmanlar arasında homojenizatör (Recht GM 200, Haan, Almanya), soğutmalı santrifüj (Sigma 2-16P, Osterode, Almanya), üstten yüklemeli hassas terazi (Shimadzu ATX224, Japonya), 5 mL'lik politetrafloroetilen (PTFE) şırıngalar, 0,45 µm gözenek çapına sahip PTFE filtreler, Eppendorf otomatik pipetler (10, 100, 1000 µL) ve 1.5 mL vial şişeleri yer almaktadır.

### Pestisit İşlemi

Bu çalışmada kullanılan limon örneklerinin aktif maddeleri, tarımsal uygulamalarda yaygın kullanım alanlarına ve kalıntı bulunma sıklıklarına dayalı olarak belirlenmiş ve laboratuvar ortamında ticari formülasyon çözeltilerine daldırılarak hazırlanmıştır [7, 16, 23, 34, 35]. Bu yöntemle, laboratuvar örneklerinde hem birimler arası hem de birim içi homojen bir dağılım sağlanmış ve tespit edilebilir düzeyde kalıntı elde edilmiştir [34]. Ticari formülasyonlar olarak, imazalil ve tiyofanat-metil sırasıyla Emtop (60%, ıslanabilir toz) ve Novamite (110 g/L, süspansiyon konsantre) şeklinde seçilerek yerel bir marketten temin edilmiştir.

İşleme faktörlerinin hesaplanmasında temel kriterlerden biri, kalıntıların ham tarımsal ürünlerde (RAC) tespit

edilebilir seviyede olmasıdır. Bu nedenle, bitki koruma ürünlerinin önerilen dozların üzerinde uygulanmasına izin verilir ve RAC'ler hasat döneminden önce toplanabilir [27]. Ön çalışmalar temel alınarak, formülasyonlar önerilen dozun yaklaşık bir ila dört katı olacak şekilde hazırlanmıştır [7, 16, 35]. Hazırlanan formülasyon çözeltileri, yeterli sayıda plastik kap içinde 10 L'lik karışımlar halinde hazırlanmış ve tüm limon örnekleri 30 dakika süreyle bu çözeltilere daldırılmıştır. İşlem sonrası örnekler, polipropilen örtüler üzerinde 3-4 saat boyunca güneş ışığında kurutulmuş, ardından analiz öncesi +4°C'de 1 gün süreyle muhafaza edilmiştir.

## Evsel İşlemler

Uygulanan evsel işlemlerin yöntemi Acoğlu Çelik ve Yolcı Ömeroğlu tarafından [7] detaylı bir şekilde açıklanmıştır. Ticari formülasyonlarla ilaçlanan ve ilaçlanmayan örneklerden, üç laboratuvar örneği sırasıyla ilaçlanmış kontrol numunesi (İK) ve ilaçlanmamış kontrol numunesi (K) olarak ayrılmıştır ve bu örnekler hiçbir ev tipi işleme yöntemine maruz bırakılmamıştır. Bu kontrol numuneleri, işleme adımlarının etkilerini değerlendirebilmek ve uygulanan tedavi yöntemlerinin karşılaştırılması için baz alınan örnekler olarak saklanmıştır. Her bir işlem adımı, farklı üç laboratuvar örneği ile üç kez tekrarlanmıştır. İşlem öncesinde, her bir meyve birimi 2-3 dakika boyunca akan musluk suyu altında yıkanmıştır. *Kabuk Soyma İşlemi (S) ve Meyve Eti (ME)* numunelerini elde etmek için limon örneklerinin kabukları ve beyaz iç kısımları, meyve eti kısmından bıçak yardımıyla dikkatlice ayrılmıştır. Kabuk/meyve ağırlık oranları (%) %26 ile %32 arasında değişmiştir. *Limon Suyu Üretimi (LS)* aşamasında limon örnekleri, mutfak bıçağı kullanılarak iki eşit parçaya ayrılmış ve limon suyu, bir mutfak robotu (Arzum, Türkiye) yardımıyla elde edilmiştir. Elde edilen limon sularının ortalama pH değeri, Mettler Toledo Seven Compact pH/İyon pHmetre (Kanada) kullanılarak 2.2±0.05 olarak belirlenmiştir. *Limon Kabuğu Rendelenmesi ve Dondurulmuş Koşullarda Saklanması (LRD)* aşamasında limon kabukları rendelenmiş ve -20°C'de üç ay boyunca saklanmıştır. Saklama süresi boyunca her ay analitik örnekler alınmıştır. Limon kabukları, meyve etleri, limon suyu ve rendelenmiş kabuklar, sonraki analizlere kadar polipropilen numune kaplarında -20°C'de muhafaza edilmiştir. *Limon Reçeli Üretimi (LR)* için [35] tanımlanan tarif uygulanmıştır. Çalışma kapsamında, meyvenin dış kabuğu hassas bir şekilde rendelenerek çıkarılmış ve ardından posaların 15 dakika süreyle üç defa su içerisinde kaynatılmıştır. Dış kabuğun acı tadını gidermek amacıyla her kaynatma işlemi sonrası su taze su ile değiştirilmiştir. Üretimin diğer aşamaları ise geleneksel reçel üretim yöntemlerine paralel olarak, 95°C'de 30 dakika süreyle pişirme işleminden oluşmaktadır. Elde edilen reçellerin ortalama pH değeri 3,45±0,06, suda çözünür kuru madde miktarı (Brix) ise 72.65±0.64 g/100 g olarak belirlenmiştir (RA-500 Model Kyoto Electronics Manufacturing Co. Ltd., Japonya). Limon reçeli, sonraki analizlere kadar oda sıcaklığında saklanmıştır.

## Pestisit Kalıntı Analizi

Her bir laboratuvar örneği, pestisit kalıntı analizi sürecinde ayrı ayrı işlenmiştir. Laboratuvar numunelerinin homojenizasyonu için, K, İK, S, ME kodlu örneklerde bulunan her bir limon dörde bölünmüş ve çapraz karşılıklı iki kısım alınarak, 2-3 mm partikül boyutuna ulaşana kadar homojenize edilmiştir (RechtGM 200, Haan, Almanya). LR kodlu laboratuvar örnekleri tamamen homojenize edilirken, LRD ve LS kodlu örnekler bu işleme tabi tutulmamıştır. Laboratuvar örneklerinden alınan analitik örnekler, ekstraksiyon ve clean-up (temizleme işlemi) adımlarına kadar -20°C'de PTFE örnek kaplarında muhafaza edilmiştir. Pestisit kalıntı analizinde kullanılan ekstraksiyon, clean up ve sıvı kromatografisi-tandem kütle spektrometresi (LC-MS/MS) prosedürleri, QuEChERS [36] olarak bilinen ve geçerliliği kanıtlanmış standart çoklu kalıntı analiz yöntemi temel alınarak uygulanmıştır. Bu metodoloji, çeşitli pestisitlerin analizi için hızlı, kolay, ucuz, etkili, sağlam ve güvenli ekstraksiyon yöntemi kullanılmıştır. Bu yöntem, aynı anda birden fazla kalıntı analizi yapabilme yeteneği ile tanınmakta olup, yüksek verimlilik sağlamaktadır. Yöntemin ayrıntıları, LC-MS/MS tanımlamaları ve ekipman parametreleri Tablo 1'de sunulmuştur. Bu standartlaştırılmış yöntem, farklı örnek tiplerinde pestisit kalıntı analizlerinde yüksek doğruluk ve tutarlılık sağlar.

Metodun uygulanmasından önce laboratuvarımızda yapılan yöntem doğrulama (method verification) çalışması ile analizlerin güvenilirliği sağlanmış ve her analiz serisi sırasında kalite kontrol çalışmaları, Avrupa Birliği SANTE/11312/2021 Rehber Belgesi [37] ve EURACHEM yönergeleri [38, 39] doğrultusunda gerçekleştirilmiştir. Bu kalite güvence prosedürleri, analizlerin doğru ve tutarlı sonuçlar üretmesini sağlamak amacıyla büyük bir titizlikle uygulanmıştır.

## İşleme Faktörü

İşlem faktörü (İf), ham tarımsal üründeki pestisit miktarının, işlenmiş ürüne oranıdır [27, 30]. 1'den küçük veya daha yüksek bir faktör, sırasıyla azalma veya artmayı göstermektedir. İşleme faktörünün denklemi eşitlik 1'de verilmiştir.

$$\text{İf} = \frac{\text{B}}{\text{A}} \quad (1)$$

Burada B, işlenmiş limon numunelerindeki (S, ME, LS, LRD ve LR) kalıntı seviyesini ifade eder. A ise ham tarımsal ürünlerdeki (İK) kalıntı seviyesini ifade eder.

Tablo 1. Pestisitlerin fizikokimyasal özellikleri [40] ve LC-MS/MS koşulları
*Table 1. Physicocehmical properties of pesticide [40] and LC-MS/MS conditions*

| Aktif Bileşenler | | Imazalil | Thiophanate-methyl |
|---|---|---|---|
| Pestisit Özellikleri | Molekül Formülü | $C_{14}H_{14}Cl_2N_2O$ | $C_{12}H_{14}N_4O_4S_2$ |
| | Etli Şekli | Sistemik | Sistemik |
| | Molekül Ağırlığı (g/mol) | 297.179 | 342.388 |
| | Suda çözünürlük (mg/L) | 180 (20°C) | 26.6 (20°C) |
| | Log Po/w[1] | 3.82 | 1.50 |
| | Kaynama Noktası (°C) | 347 | 342 |
| LC-MS/MS Koşulları | Alıkonma Zamanı ($t_R$) | 8.685 | 7.953 |
| | MRL (mg/kg) | 5 | 6 |
| | LOQ (mg/kg) | 0.01 | 0.01 |
| | Kone Voltajı | 120 | 120 |
| | Öncül İyon | 297.1 | 343.1 |
| | Parçalanma İyonu | 41.2 | 151 |
| | Çarpışma Enerjisi | -21 | -3 |

[1]*Log Po/w "Pestisitlerin oktanol/su bölme katsayısı (LogPo/w), bir bileşiğin oktanol içindeki çözünürlüğünün (polar olmayan bir çözücü) sudaki çözünürlüğüne (polar bir çözücü) oranını temsil eder.*
[1]*The octanol/water partition coefficient (Log Po/w) represents the ratio of a compound's solubility in octanol (a nonpolar solvent) to its solubility in water (a polar solvent).*

## İstatistiksel Analiz

Tüm evsel işlemler üç tekrarlı olarak yürütülmüş; her laboratuvar örneği için pestisit kalıntı analizleri iki tekrarlı şekilde gerçekleştirilmiş ve her bir analitik örnek için LC-MS/MS enjeksiyonları iki kez yapılmıştır. Elde edilen veriler, ortalama ± standart sapma olarak raporlanmıştır. Farklı işlem yöntemlerinin pestisit kalıntı seviyeleri üzerindeki etkilerini ve işlem faktörlerinin indirgeme oranlarındaki anlamlı farklılıklarını belirlemek amacıyla Varyans Analizi (ANOVA) kullanılmış, anlamlı farklılıkların tespiti için ise Tukey Post Hoc testi uygulanmıştır. İstatistiksel analizler SPSS yazılımı (sürüm 28.0; SPSS Inc., Chicago, IL, ABD) kullanılarak yapılmış olup, $p < 0,05$ düzeyi istatistiksel olarak anlamlı kabul edilmiştir.

## BULGULAR ve TARTIŞMA

Pestisit kalıntı analizlerinde uygulanan analitik yöntemin doğrulanması laboratuvarımızda başarıyla doğrulanmış olup, portakal [35] ve limon [7] matrislerinde yapılan çalışmalarda rapor edilmiştir. Elde edilen sonuçlara göre, yöntemin ortalama geri kazanım oranı (doğruluk ölçütü olarak %70-120 aralığında), kesinlik (tekrarlanabilirlik ve ara kesinlik için göreli standart sapma, RSDr ve RSDwR <%20) ve LOQ değeri (0.01 mg/kg < MRL), Avrupa SANTE/11312/2021 Kılavuz Belgesi [37] tarafından belirlenen kriterlere uygun bulunmuştur. Bunun yanı sıra, ölçüm belirsizliği de maksimum %50 göreli genişletilmiş belirsizlik sınırına uygun olacak şekilde hesaplanmıştır. Analitik yöntemin doğrusallığını değerlendirmek ve her numunede mevcut kalıntı seviyelerini ölçmek amacıyla, her analiz partisinde yedi noktalı matris uyumlu bir kalibrasyon eğrisi oluşturulmuştur. Bu eğri, 10 µg/kg ile 1500 µg/kg aralığındaki konsantrasyonları kapsamaktadır. Kalibrasyon eğrisi, yöntemin hesaplama sınırının (LOQ) altında kalıntı seviyelerine sahip kontrol limon örneklerinin ekstraktlarından hazırlanmıştır. Ağırlıklı doğrusal regresyon analizi sonucunda, belirleme katsayısı ($R^2$) 0.9990'dan büyük olan bir kalibrasyon fonksiyonu elde edilmiştir; böylece konsantrasyonların µg/kg cinsinden hesaplanması mümkün hale getirilmiştir [41]. Ayrıca, her bir analitik parti süresince konsantrasyon sapmaları ve geri kazanım oranları içeren kalite kontrol süreçleri uygulanmıştır. Kalibrasyon fonksiyonu kullanılarak standartların gerçek konsantrasyonları ile hesaplanan değerler arasındaki farklılıklar sistematik olarak analiz edilmiştir. Bu sapmalar, her partiye özel kalite kontrol süreci kapsamında değerlendirilmiş ve -%3.0 ile %19.0 arasında değişmiştir. Bu oranlar sırasıyla, SANTE/11312/2021 Kılavuz Belgesi'nde [37] belirtilen ±%20 ve kabul sınırları ile uyumludur. Ayrıca, ölçüm sınırı (LOQ) seviyesinde kör limon örneklerine analit eklenerek yapılan analizlerde, her partideki bireysel geri kazanım oranlarının %83 ile %117 arasında değiştiği saptanmış ve bu oranlar kılavuzda belirtilen %60-140 aralığına uygun bulunmuştur [37].

### Fungisit Kalıntılarının Evsel Gıda İşlemleri Sırasında Değişiminin İncelenmesi

Ekonomik Kalkınma ve İşbirliği Örgütü (OECD) tarafından 2008 yılında yayımlanan rehberde, işleme faktörlerinin hesaplanabilmesi için, işlenmemiş tarımsal ürünlerdeki kalıntı seviyelerinin tayin limitinin (LOQ) üzerinde olması gerektiği belirtilmiştir [27]. Laboratuvar koşullarında, daldırma yöntemi ile ilaçlanan ancak işlem görmemiş limonlarda tespit edilen pestisit kalıntı seviyeleri 0.014 mg/kg ile 4.173 mg/kg arasında değişmiştir (Tablo 2). Bu seviyeler, analiz metodunun LOQ seviyesi olan 0.010 mg/kg'dan yüksek olduğundan, OECD kriterleriyle uyumlu olduğu sonucuna varılmıştır. Tablo 2'de sunulan istatistiksel analiz, farklı evsel işleme yöntemlerinin, İK örneklerinde gözlemlenen kalıntı seviyeleri ile karşılaştırıldığında, evsel gıda işleme yöntemlerinin kalıntı seviyelerini önemli ölçüde değiştirdiğini ($p< 0.05$) göstermektedir.

### Kabuk Soyma İşlemi ve Meyve Eti

Soyma işlemi (S), çeşitli meyve ve sebzelerin işlenmesinde kritik bir ilk aşama olarak kabul edilmektedir. Ürünlerin dış katmanlarını veya kabuklarını çıkarmak amacıyla uygulanan bu işlem, pestisit kalıntılarının seviyesini azaltmada etkili bir yöntem sunar. Evsel işlemlerde yaygın olarak kullanılan mekanik soyma, meyve etindeki (ME) pestisit kalıntılarını azaltmada başarılı olurken, endüstriyel süreçlerde sıklıkla tercih edilen kimyasal soyma ise bu etkiyi daha da artırabilmektedir [42]. Araştırmalar, mekanik ve kimyasal soyma yöntemlerinin yanı sıra buharla soyma ve dondurma gibi çeşitli tekniklerin de pestisit kalıntılarını etkin bir şekilde azaltabileceğini göstermektedir. Ancak, bu yöntemlerin etkinliği; pestisitlerin kimyasal yapısı, sistemik yayılım durumu ve çevresel koşullara bağlı olarak değişiklik gösterebilmektedir [43, 44]. Pestisitlerin büyük bir kısmı doğrudan ürünlerin yüzeyine uygulandığından, kabuk soyma işlemi, kütikül tabakasına nüfuz etmiş pestisit kalıntılarını azaltmada en etkili yöntemlerden biri olarak kabul edilmektedir [7, 44]. Limon kabuklarına uygulanan kabuk soyma işleminin sonucunda, kontrol örnekleriyle (İK) karşılaştırıldığında kalıntı konsantrasyonlarında istatistiksel olarak anlamlı bir artış meydana gelmiştir ($p<0.05$). Konsantrasyondaki artış 1.8 ila 3.2 kat arasında değişmiştir. Limon kabuklarının meyve posasından ayrılması, pestisit kalıntılarında dikkate değer bir azalmaya neden olmuştur. Özellikle soyma işlemi, posadaki imazalil kalıntısını %97 oranında, thiophanate methyl konsantrasyonunu ise %94 oranında azaltmıştır. Bu bulgu, kabukların uzaklaştırılmasının pestisit maruziyetini önemli ölçüde düşürebileceğini göstermektedir. Özellikle kabuk soyma işlemi, yüzeyde birikmiş olan pestisitlerin uzaklaştırılmasında etkili olmuş, meyve etinde kalan pestisit seviyelerinde belirgin bir azalma sağlamıştır. Bu sonuç, pestisitlerin büyük ölçüde kabuk kısmında yoğunlaştığını ve soyma işlemiyle önemli ölçüde bertaraf edilebileceğini ortaya koymaktadır. Limon ve benzeri turunçgil ürünlerinde tüketici sağlığını koruma açısından kabuğun uzaklaştırılmasının etkili bir yöntem olduğu sonucuna varılabilir. Bulgularımızla uyumlu olarak [45] araştırmasında limon örneklerinde fenhexamid kalıntısının %43'ünün kabuktan albedoya, %18'inin ise albedodan meyve etine geçtiği belirlenmiştir. Portakal örneklerinde, imazalil kalıntısının %58'inin kabuktan albedoya, yalnızca %6'sının ise meyve etine geçtiği saptanmıştır. Mandalina örneklerinde ise imazalilin yalnızca %1.6 oranında meyve etine geçiş yaptığı tespit edilmiştir. Fungisitlerin meyve etine geçiş oranının nispeten düşük olmasının, analiz edilen bileşiklerin ve meyve kabuklarının fizikokimyasal özelliklerinden kaynaklandığı rapor edilmiştir. Ayrıca, kabuklardaki pestisit seviyelerinin, bütün meyvelere kıyasla belirgin şekilde daha yüksek olduğu ortaya konmuştur. Ürünlerin menşei hakkında yeterli bilgiye sahip olunmaması da göz önünde bulundurulduğunda, narenciye kabuklarının yemek veya içecek hazırlamada kullanılmasının önerilmediği vurgulanmıştır. Portakal kabuklarında imazalil konsantrasyonunun 3.5 ila 3.6 kat arasında arttığı tespit edilmiştir [46]. Turunçgil meyvelerinde spirodiclofen kalıntısının kabuk ve meyve eti içindeki dağılımını değerlendirmiştir. Kabuklarda, tüm turunçgillerle karşılaştırıldığında sırasıyla 5.1, 2.9 ve 1.9 kat oranında spirodiclofen konsantrasyonunda artış tespit edilmiştir. Ancak meyve eti örneklerinde kalıntı seviyeleri hesaplama limit değerinin altında kaldığı saptanmıştır [47].

Bir başka çalışmada, limon, portakal, greyfurt, mandalina, pomelo ve misket limonu gibi altı farklı turunçgil türünde pestisit kalıntıları analiz edilmiştir. Çalışmada, en sık tespit edilen fungisit kalıntısı imazalil olup, bu kalıntıya test edilen örneklerin %88'inde rastlanmıştır. Bunu, örneklerin %57'sinde tespit edilen bir diğer fungisit olan pyrimethanil takip etmiştir. Kalıntıların dağılımını değerlendirmek ve tüketicilerin diyet kaynaklı maruziyet riskini tahmin edebilmek amacıyla, kalıntılar kabuk ve meyve eti ayrı ayrı analiz edilmiştir. Turunçgil türüne ve çeşidine bağlı olarak, kabuk ile meyve eti arasındaki ağırlık oranı %15 (misket limonu) ile %42 (portakal) arasında değişiklik göstermiştir. Araştırmada, analiz edilen pestisitlerin çoğu için meyve etinde, kabuğa kıyasla belirgin şekilde daha düşük kalıntı seviyeleri tespit edilmiştir. Sistemik ve temas pestisitleri karşılaştırıldığında, temas pestisitlerinin büyük çoğunluğunun kabukta yoğunlaştığı belirlenmiştir. Öte yandan, sistemik pestisitlerde transfer oranı önemli ölçüde daha yüksek olup, %30 (prochloraz) ile %70 (spirotetramat) arasında değişmiştir. Bu durum, sistemik pestisitlerin püskürtüldüğünde bitki yapraklarına nüfuz etmesi ve bitki dokuları aracılığıyla meyveye geçerek posada, kabuktakine göre daha fazla yoğunlaşabilmesi ile açıklanabileceğini belirtmişlerdir [48]. Benzer şekilde, literatürde yer alan diğer araştırmalar da kabuk soyma işleminin meyve ve sebzelerdeki pestisit kalıntılarını kayda değer oranda azalttığını göstermiştir [7, 20, 35, 47, 49, 50]. Pestisit kalıntılarının büyük bir kısmı, genellikle meyve kabuğunun soyulmasıyla birlikte uzaklaştırılmaktadır. Ancak, pestisit kalıntılarının sistemik olarak meyve dokusuna yayılması durumunda, soyma işlemi her zaman pestisit kalıntılarında belirgin bir azalmaya yol açmayabilir [42]. Elma kabuklarının soyulması, pestisit seviyelerinde %24 (carbendazim) ile %100 (triflumuron, thiodicarb, tebuconazole) arasında değişen oranlarda bir azalma sağlamıştır [11].

Portakallar üzerinde yapılan bir çalışmada, kabuklardaki pestisit içeriğinin, meyve etine kıyasla belirgin şekilde daha yüksek olduğu (%7.5-17.9) tespit edilmiştir. Fakat prochloraz kalıntısı için durum farklı olup, meyve etindeki seviyesi %65.4 olarak belirlenmiştir. Bu nedenle turunçgil meyvelerinin kabukları genellikle doğrudan tüketilmez; ancak limon kabuğu, baharat değeri taşıması nedeniyle bir istisna oluşturur [51]. Bunun yanı sıra, turunçgil kabukları, şekerleme ürünlerine katkı maddesi olarak işlenebilmekte veya esansiyel yağ üretimi için hammadde olarak kullanılabilmektedir [11]. Bu bağlamda, pestisitlerin seviyelerinin izlenmesi ve uluslararası gıda güvenliği standartlarına uygunluğunun sağlanması, tüketicinin sağlığı açısından kritik öneme sahiptir [30].

Benzer bulgular, daha önce yapılan çalışmalarla da uyum göstermektedir. Örneğin, [52] tarafından

gerçekleştirilen bir araştırmada, soyma işleminin domatesteki thiophanate-methyl kalıntısında %84.2 ve carbendazim kalıntısında %87.3 oranında kayıplara neden olduğu bildirilmiştir. Bunun yanı sıra, [53] tarafından yapılan bir başka çalışmada, domateslerin kabuklarının soyulmasının chlorothalonil, oxadixyl ve thiophanate-methyl kalıntılarında sırasıyla %96, %60 ve %94 oranında azalma sağladığı tespit edilmiştir. Bu bulgular, farklı pestisit türlerinde soyma işleminin etkili bir azaltma yöntemi olabileceğini göstermektedir.

Bu araştırma kapsamında elde edilen bulgular, pestisitlerin kabuk tabakasında kaldığını ve meyve etine doğru difüzyonlarının düşük düzeyde gerçekleştiğini göstermektedir. Pestisit kalıntılarındaki artış, pestisitlerin etki mekanizmalarının yanı sıra, özellikle oktanol-su katsayısı (log Po/w) ve suda çözünürlük gibi fiziksel ve kimyasal özellikleri ile ilişkilendirilebilir. İmazalil ve thiophanate-methyl her iki pestisit de sistemik etki göstermektedir; ancak imazalil, thiophanate-methyl'e kıyasla daha yüksek suda çözünürlük ve log Po/w (Tablo 1) değerlerine sahip olduğundan, daha fazla azalma göstermektedir. Bu durum, bu pestisitlerin meyve etine difüze olmak yerine, meyve kabuğundaki kütiküler mumlara veya daha derin katmanlara tutunma eğiliminde olduğunu göstermektedir [43, 54]. Turunçgillerin dış yüzeyinde yer alan kütin ve mum tabakalarının pestisit kalıntılarının fiziksel korunmasında kritik bir rol oynadığını rapor etmiştir [55]. Ayrıca, Liu ve ark. [56] tarafından gerçekleştirilen bir çalışmada, turunçgil kabuklarındaki spirotetramat kalıntılarının başlangıç konsantrasyonlarına göre arttığı belirlenmiştir. Bu durum, kütiküler balmumunun pestisit kalıntılarının meyve etine doğru difüzyonunu engelleyerek bir bariyer görevi gördüğü şeklinde yorumlanmıştır. Bu bulgu, pestisit kalıntılarının meyve kabuğunda birikim eğilimini açıklamakta ve fizikokimyasal özelliklerin kalıntı dağılımındaki önemini vurgulamaktadır. Portakal [35] ve limon [7] üzerinde gerçekleştirdikleri çalışmalarda benzer pestisit kalıntısı bulgularına ulaşmışlardır. Her iki çalışmada da, turunçgil ve limon kabuklarındaki kütiküler mum tabakasının, pestisit kalıntılarının meyve etine difüzyonunu engelleyerek etkin bir taşıma bariyeri görevi gördüğü sonucuna varılmıştır. Bu bulgular, farklı pestisit-matriks kombinasyonlarını ele alan çeşitli çalışmalarda da benzer şekilde gözlemlenmiştir. Özellikle kabukların soyulmasının, meyve etindeki pestisit kalıntılarını önemli ölçüde azalttığı sıkça vurgulanmıştır; bu durumun, pestisitlerin fizikokimyasal özelliklerinden etkilendiği bildirilmektedir. Başka bir çalışmada, pestisitlerin kütiküler mum tabakasına tutunmasının, meyve etine doğru difüzyonunu fiziksel olarak engellediği belirtilmiştir [57].

Benzer şekilde, Liu ve ark. [58] tarafından yapılan araştırmada, üzüm meyvelerinin soyulması işlemi sonrasında dinotefuran, imidacloprid, acetamiprid, triadimefon, triadimenol, tebuconazole, azoxystrobin, pyraclostrobin, fluxapyroxad, pydiflumetofen ve difenoconazole gibi pestisit kalıntıları incelenmiştir. Araştırma bulguları, soyma işleminin hedef pestisitlerin kalıntı seviyelerinde belirgin bir azalma sağladığını göstermiş; uzaklaştırma oranlarının %13 ila %91 arasında değiştiği rapor edilmiştir. Bu durum, kabuk üzerinde bulunan mumsu tabakanın soyulmasıyla, pestisit kalıntılarının etkili bir şekilde azaltılabileceğini ortaya koymaktadır.

Bu sonuçlar, önceki çalışmalarla da tutarlıdır [43, 53, 54, 55, 59]. Söz konusu araştırmalar, balmumu tabakasının pestisit kalıntılarının meyve etine geçişini kısıtlayarak koruyucu bir bariyer oluşturduğunu göstermiştir. Dolayısıyla, meyve yüzeyindeki pestisitlerin kütiküler tabaka tarafından engellendiği ve bu bariyerin ortadan kaldırılmasıyla birlikte kalıntı miktarında azalma gözlendiği sonucuna varılabilir. Bu durum, soyma işleminin fungisit kalıntılarının azaltılmasında önemli bir rol oynadığını ortaya koymaktadır.

## Limon Suyu Üretimi

Meyve suyu eldesi, bitki dokularından sıvının çıkarılması işlemidir ve hızlı bir şekilde büyük miktarlarda meyve suyu tüketmek, genellikle tercih edilen bir yöntemdir [60]. Bu çalışmada, limon meyvelerinin tamamı kullanılarak evsel yöntemlerle limon suyu (LS) elde edilmiş ve bu işlem sırasında pestisit kalıntı seviyelerinin işlenmiş numunelerde önemli ölçüde değiştiği tespit edilmiştir.

Pestisit kalıntılarının meyvelerden meyve suyuna geçişi, meyve kabuğu ve posasında kalan miktarlara ek olarak, pestisitlerin fizikokimyasal özelliklerine de bağlı olarak değişiklik göstermektedir. Meyve suyu eldesi sırasında uygulanan santrifüjleme veya filtrasyon gibi berraklaştırma işlemleri, pestisit kalıntı seviyelerinde belirgin bir azalma sağlamaktadır. Bu berraklaştırma süreçlerinin, pestisitlerin sudaki çözünürlüğü ve fiziksel ayrılabilirliği üzerinde etkili olduğu düşünülmektedir, dolayısıyla meyve suyu ürünlerinde kalıntı miktarlarını azaltma potansiyeline sahiptir [43, 61]. Bu çalışmada, limon suyunda imazalil kalıntısının %92 oranında azalırken, thiophanate-methyl kalıntısı %88 oranında azaldığı tespit edilmiştir. Limon meyve suyuna işlenirken yüksek oktanol-su bölme katsayısı (log Po/w) ile ilişkili olduğu düşünülerek imazalil kalıntısı daha fazla azalmıştır (Tablo 1).

Ev yapımı meyve suyu üretim sürecinde herhangi bir ısıtma işlemi (sterilizasyon/pastörizasyon) uygulanmadığından, meyve suyundaki pestisit kalıntılarındaki azalmanın, dış yüzeydeki balmumu ve kütiküler tabakaya pestisitlerin yapışmasına bağlanabileceği ve bunun da yüksek oktanol-su bölme katsayısı (log Po/w) ile ilişkili olduğu düşünülmektedir [34]. Domateslerden meyve suyu elde etme işlemi, her iki pestisitin uzaklaştırılmasında en etkili yöntem olarak tespit edilmiştir. Domates suyunda metalaxyl kalıntılarında %66 oranında, chlorpyrifos kalıntılarında ise %98 oranında kayda değer bir azalma gözlemlenmiştir. Araştırmacılar, bu yüksek azalma oranlarını, pestisitlerin fiziko-kimyasal özellikleri ve özellikle log Po/w (oktanol-su dağılım katsayısı) değerleri ile ilişkilendirmiştir: Yüksek log Po/w değerlerine sahip olan pestisitler, hidrofobik doğaları nedeniyle meyve suyu içerisinde daha az çözünmekte ve büyük ölçüde kabuk veya posa kısmında tutulmaktadır [34]. Carbendazim, thiamethoxam,

imidacloprid, acetamiprid, prochloraz ve difenoconazole kalıntılarının, berrak ve bulanık elma suyu üretim süreçleri ile hızlandırılmış depolama koşullarındaki davranışları kapsamlı bir şekilde değerlendirilmiştir. Berrak elma suyu üretiminde uygulanan enzimleme işlemi, pestisit kalıntılarında %1.9 ile %31.6 arasında düşük düzeyde bir azalmaya neden olmuştur. Klarifikasyon ve saflaştırma adımlarını takiben yapılan filtrasyon aşaması, kalıntı seviyelerinde %14.0 ile %87.5 arasında önemli bir azalma sağlamıştır. Öte yandan, bulanık elma suyu üretiminde santrifüj işlemi, pestisit kalıntılarında %6.3 ile %88.9 arasında değişen oranlarda azalmasına yol açmıştır. Elde edilen bulgular, farklı işleme adımlarının pestisit kalıntılarının azaltılmasında önemli bir rol oynadığını göstermekte ve meyve suyu üretim süreçlerinin kalıntı yönetimi üzerindeki etkisini vurgulamaktadır [62].

Başka bir çalışmada, üzümden meyve suyu üretimi sırasında dinotefuran, imidacloprid, acetamiprid, triadimefon, triadimenol, tebuconazole, azoxystrobin, pyraclostrobin, fluxapyroxad, pydiflumetofen ve difenoconazole kalıntıları incelenmiştir. Çalışmada, hedef pestisitlerin kalıntı seviyelerinde belirgin azalmalar gözlemlenmiş ve uzaklaştırma oranlarının %6 ile %62 arasında değiştiği rapor edilmiştir. Posada ise nispeten daha yüksek düzeyde pestisit kalıntılarının tespit edilmesi, posanın düşük su içeriğine bağlı olarak pestisitlerin konsantrasyonunun artması ile ilişkilendirilmiştir [58]. Başka bir çalışmada, turunçgil meyve suyu üretimi sırasında prochloraz kalıntılarının başlangıçtaki seviyelere kıyasla %94.3 ve %94.5 oranında azaldığı tespit edilmiştir. Benzer şekilde, kasugamisin kalıntılarında %96'nın üzerinde bir azalma kaydedilmiş, oxine-bakır kalıntılarında ise bu oranlar sırasıyla %95.6 ve %94.6 olarak belirlenmiştir. Fenaminstrobin kalıntılarında %94.2 ve %95.2 oranında azalma gözlemlenmiştir. Bu sonuçlar, turunçgil meyve suyu üretim süreçlerinin dört farklı fungisit kalıntısını etkili bir şekilde azaltabildiğini göstermektedir. Özellikle, yağda çözünebilen pestisitlerin büyük bir kısmının kabuk ve posada tutulduğu, bu nedenle meyve suyuna geçişinin sınırlı olduğu ifade edilmiştir. Bu durum, presleme ve posanın ayrılması gibi işleme adımlarının pestisit kalıntılarının gideriminde önemli bir yöntem olduğunu ortaya koymaktadır [63]. Benzer şekilde, tatlı portakal meyvesi ve yan ürünlerinde, endüstriyel meyve suyu üretim süreci boyunca spiropidion ve beş ana metabolitinin kalıntı seviyeleri incelenmiştir. Portakal suyu üretiminde uygulanan sıkma, filtrasyon, sterilizasyon ve konsantrasyon gibi işlem basamaklarının, pestisit kalıntılarını %34.2 ile %70.8 oranında azalttığı belirlenmiştir. Bu bulgular, meyve suyu işleme süreçlerinin pestisit kalıntılarının azaltılmasında etkili bir rol oynadığını vurgulamakta ve bu tür işlemlerin gıda güvenliği üzerindeki olumlu etkilerini ortaya koymaktadır [64].

Bir çalışmada, armut suyu üretimi sırasında chlorpyrifos kalıntılarının tamamen (%100) giderildiği tespit edilmiştir. Benzer şekilde, elma suyu üretiminde mancozeb kalıntılarının %100 oranında ortadan kalktığı rapor edilmiştir [65]. Ayrıca, literatürde yer alan diğer çalışmalar, meyve suyu üretim süreçlerinde pestisitlerin davranışları ile bu pestisitlerin fizikokimyasal özellikleri arasındaki ilişkiyi desteklemektedir [34, 58, 63, 64, 66].

## Limon Kabuğu Rendelenmesi ve Dondurulmuş Koşullarda Depolanması

Dondurma, gıda kalitesini etkileyen kimyasal reaksiyonları engellemek için etkili bir yöntem olup, bu sayede gıdanın tadı, dokusu ve besin değeri alternatif koruma yöntemlerine kıyasla daha iyi korunmaktadır [43]. Limon kabukları, hem evsel hem de ticari dondurma yöntemleriyle korunabilir; bu sayede atıkların geri dönüştürülmesi sağlanır ve kabukların fizikokimyasal bütünlüğü bozulmadan raf ömrü uzatılır. Tablo 1'de, limon kabuğu üretimi sırasında ve -20°C'de saklama süresi boyunca pestisit kalıntılarındaki değişiklikler sunulmuştur. Limon kabuğuna işlendikten sonra, imazalil ve thiophanate-methyl kalıntılarının konsantrasyonlarının, kontrol limon örneklerine (İK) göre istatistiksel olarak anlamlı şekilde daha yüksek olduğu gözlemlenmiştir ($p<0.05$). Üç aylık depolama süreleri sonucunda pestisit kalıntılarının anlamlı derecede azaldığı görülmüştür ($p<0.05$). Bu nedenle depolama süresinin kalıntı konsantrasyonlarını etkilediği sonucuna varılmıştır. Bu gözlem, pestisitlerin limon meyvesinin dış yüzeyinde birikmesinden kaynaklanmaktadır. Çalışma kapsamında analiz edilen pestisitlerin yüksek yağ çözünürlükleri, bu pestisitlerin kalıntılarının limonun mumsu dış yüzeyine adsorplanmasına yol açmıştır. Bulgularımızla korelasyon gösteren çalışmalar literatürde yer almaktadır [7, 35].

Yapılan bir araştırmada, haşlama işlemleri, dondurma ve donmuş depolamanın (-20°C) pestisit kalıntıları üzerindeki etkisini incelemek ve on aylık bir süre boyunca ıspanakta bulunan chlorantraniliprole, lambda-cyhalothrin ve fluopicolide kalıntı miktarları incelenmiştir. Bu kapsamda, dondurulmuş depolama süresi (0-300 gün) boyunca, farklı sürelerle haşlanmış (2, 6 ve 10 dakika) ve ardından -20°C'de dondurulmuş ıspanak örneklerinde önemli değişiklikler gözlemlenmiştir. Chlorantraniliprole kalıntı düzeyleri, haşlama süresine bağlı olarak 2, 6 ve 10 dakika haşlanan örneklerde depolama süresinin sonunda (300. gün) sırasıyla 5.83, 12.15 ve 11.94 mg $kg^{-1}$ seviyelerine ulaşarak anlamlı bir artış göstermiştir ($p<0.05$). Benzer şekilde, lambda-cyhalothrin kalıntılarında depolama süresince artış eğilimi tespit edilmiş; bu artış özellikle 105. günden itibaren belirginleşerek, 300. günde sırasıyla 4.17, 6.38 ve 8.63 mg $kg^{-1}$ seviyelerine ulaşmıştır. Diğer yandan, fluopicolide içeriği, 2 dakika haşlanan örneklerde depolama süresince sabit kalmış; ancak 6 ve 10 dakika haşlanan örneklerde depolama süresinin sonunda anlamlı bir artış kaydedilmiştir [67]. Başka bir çalışmasında, 14 yerel taze balık örneğinde (tilapia, kefal ve yayın balığı) yedi farklı pestisitin (endosulfan, heptaklor, malathion, chlorpyrifos, bifenthrin, deltamethrin ve fenoxycarb) kalıntı seviyeleri incelenmiştir. Kontamine balıkların -70°C'de bir ay süreyle dondurularak muhafaza edilmesi, pestisit kalıntılarında yalnızca %6-30 oranında bir azalma sağlamıştır. Ancak bu süreç sonucunda elde edilen kalıntı seviyeleri, önerilen maksimum kalıntı limit değerlerinin altına düşmemiştir [68]. Elde edilen bu

bulgular, dondurma işleminin pestisit kalıntılarının giderilmesi üzerinde sınırlı bir etkiye sahip olduğunu ortaya koymaktadır.

Elde ettiğimiz sonuçlar, literatürde sunulan sınırlı verilerle genellikle korelasyon göstermektedir. Kabak örneklerinde −30°C'de 15 ve 30 günlük depolama koşullarında diethofencarb, pyriproxyfen, trifloxystrobin, imidacloprid ve myclobutanil kalıntıları incelenmiştir. Dondurma işlemi sonrasında ve depolama süresince, kabak örneklerinde trifloxystrobin ve myclobutanil kalıntı seviyelerinin büyük ölçüde korunduğu ve bu pestisitlerdeki kayıpların %1'in altında kaldığı belirlenmiştir. Buna karşılık, imidacloprid ve diethofencarb kalıntılarında sırasıyla %31.7 ve %9.8 oranında daha yüksek düzeyde kayıplar tespit edilmiştir. Ancak, depolama süresinin (15 ve 30 gün) bu pestisitlerin konsantrasyonları üzerinde anlamlı bir değişikliğe yol açmadığı gözlemlenmiştir. Buna karşın, düşük depolama sıcaklıklarının pestisit kalıntılarına etkisi üzerine yapılan bazı çalışmalarda azalmalar rapor edilmiştir [69]. Örneğin, mango örneklerinde −20 °C'de difenoconazole kalıntılarında [70], taze, çiğ uskumru filetolarında −20 °C'de piretroit pestisitlerde [71] ve elmalarda −25 °C'de fungisit kalıntılarında [72] belirgin azalmalar gözlemlenmiştir. Bu çalışmalar, düşük depolama sıcaklıklarının bazı pestisit türlerinde kalıntı düzeylerini önemli ölçüde azaltabileceğini ortaya koymaktadır. Düşük sıcaklıkların, organik bileşiklerin kimyasal reaktivitesini ve bozunumunu azalttığı geniş bir şekilde kabul edilen bir durumdur. Pestisitlerle kontamine olmuş gıdaların çok düşük sıcaklıklarda depolanmasının, genellikle pestisit seviyeleri üzerinde anlamlı bir etkisi olmadığı bildirilmektedir [68]. Bununla birlikte, gıda örneklerinin matrisinde bulunan enzimler ve mikroorganizmalar, pestisit kalıntılarının bozunmasına yol açabilmektedir. Düşük sıcaklıklar, enzimatik ve mikrobiyal aktiviteleri sınırlayarak bu bozunmayı engelleyebilir, ancak aynı zamanda pestisit kalıntılarının stabilitesini de etkileyebilir [67]. Özellikle, dondurma işleminin rendelenmiş limon kabukları gibi gıda örneklerinde buz kristallerinin oluşması, bitki dokularında önemli hasarlara yol açabilmektedir. Bu hasarlar, su kaybı, hücre yapılarının bölünmesi, konsantrasyon değişiklikleri veya bileşiklerin bozunmasını etkileyebilir [73]. Bu mekanizmalar, donmuş gıdalarda pestisit kalıntılarının seviyelerini etkileyebilir.

Ayrıca, pestisit moleküllerinin fizikokimyasal özellikleri de bu süreçleri etkileyen önemli bir faktördür. Pestisitlerin moleküler yapısı, taşınım davranışları ve çevresel koşullar, kalıntıların bozulma hızını ve miktarını belirleyen kritik etmenlerdir [67]. Dondurma ve depolama koşullarının pestisitlerin davranışına olan etkilerini daha iyi anlayabilmek için bu faktörlerin her birinin detaylı bir şekilde incelenmesi gerekmektedir.

Tablo 2. Limon numunelerinde farklı işleme tekniklerinin pestisit kalıntı miktarlarına etkisi (n=3)
*Table 2. Effect of different processing techniques on pesticide residue levels in lemon samples (n=3)*

| Evsel İşlemler (Kod) | İmazalil | Thiophanate-methyl |
|---|---|---|
| İşlemsiz (İK) | 1.730±0.242$^{c}$ | 0.253±0.028$^{c}$ |
| Kabuk Soyma (S) | 3.030±0.526$^{b}$ | 0.813±0.115$^{b}$ |
| Meyve Eti (ME) | 0.056±0.003$^{d}$ | 0.014±0.002$^{d}$ |
| Limon Suyu (LS) | 0.146±0.023$^{d}$ | 0.031±0.05$^{d}$ |
| Dondurulmuş Limon Kabuğu Rendesi (Depolamanın 1. ayı) ($LRD_1$) | 4.173±0.077$^{a}$ | 0.716±0.005$^{b}$ |
| Dondurulmuş Limon Kabuğu Rendesi (Depolamanın 2. ayı) ($LRD_2$) | 4.163±0.213$^{a}$ | 1.230±0.173$^{a}$ |
| Dondurulmuş Limon Kabuğu Rendesi (Depolamanın 3. ayı) ($LRD_3$) | 3.116±0.083$^{b}$ | 0.563±0.142$^{b}$ |
| Limon Reçeli (LR) | 0.116±0.024$^{d}$ | <LOQ |

*Sütunlardaki farklı harfler istatistiksel olarak önemli farklılıkları temsil etmektedir (p<0.05). LOQ: belirleme sınırı, 0.01 mg/kg*
*Different letters in the columns represent statistically significant differences (p<0.05). LOQ: limit of detection, 0.01 mg/kg*

## Limon Reçeli Üretimi

Reçel, şekerin meyve veya sebze posası ile diğer malzemeler (şeker, asit vb.) karıştırılarak, uygun kıvama gelene kadar pişirilmesiyle hazırlanan yarı katı bir gıda ürünüdür [74, 75]. Reçel yapım sürecinde, pestisitlerin davranışı, sudaki çözünürlükleri, kaynama noktaları ve diğer fizikokimyasal parametrelerle doğrudan ilişkilidir. Isıl işlem sırasında, pestisit kalıntıları yüksek sıcaklık etkisiyle bozunarak azalabilir. Bu süreçte, uygulanan yüksek sıcaklık, pestisitlerin buharlaşma, hidroliz veya diğer bozunma yolları aracılığıyla giderilmesine katkı sağlamaktadır [60]. Bu çalışmada, limon reçeli üretimi sırasında thiophanate-methyl kalıntısı tespit edilemezken, imazalil kalıntısında %93 oranında bir azalma gözlemlenmiştir. Reçel üretim sürecinde, kaynatma işlemi ön işlem olarak uygulanmış ve bu esnada su, üç kez taze su ile değiştirilmiştir. Son aşamada ise meyvelere taze su ve şeker eklenerek pişirme işlemi gerçekleştirilmiştir. Reçeldeki pestisit kalıntı seviyelerindeki azalmanın, kaynama esnasında suda çözünebilen pestisitlerin uzaklaştırılması [43, 60], buharlaşma, ayrışma, ısıl bozunma, işlem süresi ve pişirme sürecinde uygulanan sıcaklık gibi faktörlerle ilişkili olduğu değerlendirilmektedir. Reçel yapım sürecinde pestisitlerin davranışları, suda çözünürlüğü ve kaynama noktası gibi fizikokimyasal özelliklerine bağlı olarak değişiklik gösterebilmektedir. Isıl işlem sırasında pestisit kalıntıları, yüksek sıcaklık etkisiyle bozunarak azalabilmektedir. Bu nedenle, reçel üretimi sırasında uygulanan işlemler, pestisit kalıntılarının etkin bir şekilde azaltılmasına katkı sağlayabilmektedir [60].

Bu çalışmada, piyasadan temin edilen çilek örneklerine 2 mg $kg^{-1}$ konsantrasyonunda karıştırılan piretroid grubu pestisitler (bifenthrin, carbofuran, chlorfenpayer, chlorpyrifos, cypermethrin, deltamethrin, esfenvalerate, ethion, fenvalerate, lambda-cyhalothrin, permethrin ve thiamethoxam) üzerine odaklanılmıştır. Çalışma kapsamında, bu pestisitlerin çileklerin reçel üretim sürecindeki davranışları ve kalıntı seviyelerinde meydana gelen değişimler sistematik olarak incelenmiştir. Ev tipi işleme yöntemleri ile hazırlanan çilek reçeli örneklerinde, chlorpyrifos hariç (%51.8 azalma), tespit edilen tüm pestisit kalıntılarının konsantrasyonlarında %96'nın üzerinde bir azalma gözlemlenmiştir. Araştırma, pestisit kalıntılarının azaltılmasına yönelik çeşitli basit yöntemlerin etkinliğini değerlendirmiştir. Bu bağlamda, reçel üretimi sırasında uygulanan yıkama, ısıtma ve kurutma işlemleri, pestisit kalıntılarının uzaklaştırılmasında etkili temel yöntemler arasında yer aldığı belirlenmiştir [76]. Başka bir çalışmada, muz kabuğu unu ilavesi yapılan reçel örneklerinde pestisit kalıntıları incelenmiştir. Analiz edilen bileşiklerin (azoxystrobin, bifenthrin, difenoconazole ve simazine) azalma oranlarının %28 ile %60 arasında değiştiği belirlenmiştir. Bu azalmanın, yüksek sıcaklıkların etkisiyle analitlerin termal kararsızlığından kaynaklandığı rapor edilmiştir. Elde edilen bulgular, reçel üretim sürecinde uygulanan ısıl işlemlerin pestisit kalıntılarının azaltılmasında etkili olduğunu ancak tamamen ortadan kaldırılmasını sağlayamadığını göstermektedir [75].

Yapılan bir çalışmada, Brezilya ve İspanya'dan temin edilen sekiz farklı markaya ait toplam 51 adet kayısı, üzüm, şeftali, ananas ve çilek reçeli örneği analiz edilmiştir. İncelenen örneklerin %80'inde en az bir pestisit kalıntısı tespit edilmiş ve toplamda 42 farklı pestisit belirlenmiştir. En yüksek kalıntı düzeyleri, Brezilya kaynaklı çilek reçellerinde gözlemlenmiş; bu reçellerde difenoconazole, procymidon ve thiophanate-methyl yüksek konsantrasyonlarda tespit edilmiştir. İspanya kaynaklı çilek reçellerinde ise penconazole ve spinosyn A en sık rastlanan pestisitler arasında yer almıştır. Üzüm reçellerinde pyrimethanil, ananas reçellerinde ise carbendazim öne çıkan pestisitler olmuştur. Bu bulgular, pestisit kalıntılarının nihai üründe kalabildiğini ve bu durumun insan sağlığı açısından potansiyel bir risk teşkil edebileceğini göstermektedir. Bu nedenle, reçel ürünlerinin pestisit kalıntıları açısından düzenli olarak denetlenmesi büyük önem taşımaktadır [77].

Benzer şekilde, siyah frenk üzümü reçeli üretimi sırasında thiophanate-methyl kalıntısının %82 oranında, difenoconazole kalıntısının ise %29 oranında azaldığını rapor etmiştir. Bu durum, reçel üretiminde kullanılan pişirme tekniği ve üründeki pestisit kalıntısının fizikokimyasal özelliklerine bağlı olarak azalma oranlarının değişebileceğini ortaya koymaktadır. Özellikle, kaynatma ve pişirme işlemlerinin, su çözünürlüğü ve kaynama noktası gibi pestisitlerin fizikokimyasal parametrelerine göre kalıntı seviyelerini önemli ölçüde etkileyebileceği anlaşılmaktadır. Dolayısıyla, reçel yapım sürecinde kullanılan ısıl işlemlerin pestisit kalıntılarının azaltılmasında etkili olabileceği, ancak bu etkinliğin pestisitin türüne ve üründeki davranışına göre değişiklik gösterebileceği sonucuna varılmıştır [60]. Bu bulgularla uyumlu olarak, turunçgil marmelat üretimi sırasında spirotetramatın metabolitlerinden B-keto'nun başlangıç konsantrasyonuna göre %68 oranında azaldığını; diğer metabolitlerin (B-enol, B-glu ve B-mono) ise tamamen yok olduğunu rapor etmiştir [56]. Yapılan başka bir çalışmada ise, elma reçelinde mancozeb kalıntısının %100 oranında azaldığını göstermiştir [65].

Başka bir çalışmada ise portakal reçeli ve guava meyvesi reçellerinde perfekthione, vydate ve teldor kalıntıları incelenmiş; portakal reçelinde sırasıyla %87.5, %100 ve %100 oranında azalma tespit edilirken, guava meyvesi reçelinde ise bu oranlar sırasıyla %72.5, %100 ve %100 olarak belirlenmiştir. Bu sonuçlar, yüksek sıcaklıkların etkisiyle ısıl işlem sırasında bu pestisitlerin sudaki hidrolizine bağlı olarak kalıntıların azalabileceğini göstermektedir [78]. Bu bulgular, yukarıda açıklandığı üzere reçel üretim süreçlerinde gerçekleştirilen aşamalardan kaynaklanmaktadır. Ayrıca, pestisit kalıntılarındaki azalmanın yalnızca suda çözünürlüklerine değil, aynı zamanda ısıl işlem sırasında gerçekleşen kimyasal ve termal bozunmalara da bağlı olduğu söylenebilir [7, 60, 65, 75, 78].

## İşleme Faktörü

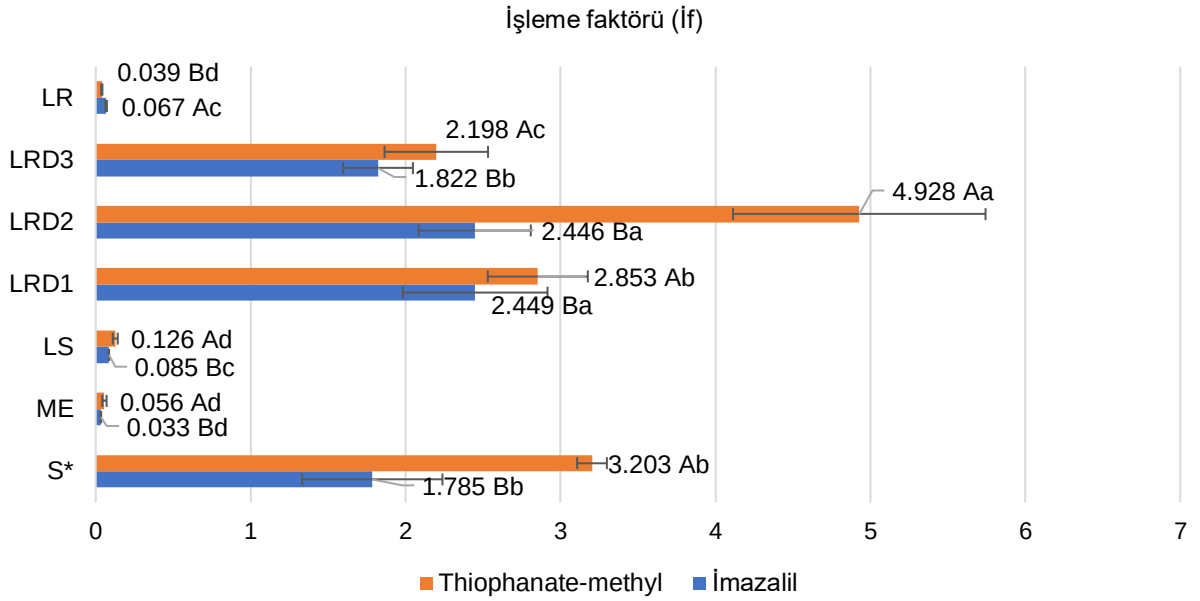
Gıda işleme süreçleri, genellikle birden fazla aşamadan oluşur ve bu aşamalar pestisit kalıntı seviyelerinde azalma ya da artışa yol açabilir [69]. Şekil 1, evsel işleme yöntemlerinin farklı aşamalarında pestisit kalıntı seviyelerinde meydana gelen değişikliklere ilişkin bulguları sunmaktadır. Araştırmada, İf, işlenmiş gıdada tespit edilen kalıntı miktarının, ham tarımsal üründeki kalıntı miktarına oranı olarak tanımlanmış ve hesaplanmıştır [27].

İK numunelerinde tespit edilen pestisit kalıntı konsantrasyonları 0.014 mg/kg ile 4.173 mg/kg arasında değişiklik göstermiştir (Tablo 2). Bu değerlerin analitik yöntemin kantitatif belirleme limiti (LOQ) üzerinde olması, işleme faktörlerinin hesaplanmasında OECD kılavuzunda belirtilen kriterlerin karşılandığını göstermektedir [27]. İşleme faktörün etkinliği, test edilen bileşiklerin fizikokimyasal özelliklerine, etki mekanizmalarına (Tablo 1), seçilen meyve veya sebze türünün biyolojik özelliklerine ve uygulanan teknolojik işlemlerin koşullarına bağlıdır [79]. Yapılan analizlerde, en düşük işleme faktörleri meyve eti (ME), limon suyu (LS) ve limon reçeli (LR) için saptanmıştır. Bu işlemler sonucunda incelenen pestisitler için işleme faktörleri (İf) 1'den düşük bulunmuş, bu da taze ürüne kıyasla tüketiciler için daha düşük bir risk düzeyi olduğunu işaret etmektedir.

Buna karşılık, kabuk kısmında (S) ve limon kabuklarının rendelenip dondurulması (LRD) işleminde pestisit konsantrasyonlarında artış gözlenmiş ve bu işlemler için İf değerleri 1'den büyük olarak belirlenmiştir. Bu durum, pestisitlerin fizikokimyasal özelliklerinden biri olan log Po/w değerleriyle ilişkilendirilmiştir. Kalıntı seviyelerinin azaltılmasında önemli etkiye sahip temel parametreler

arasında çözünürlük, polarite ve aktif maddenin bitki dokusuna nüfuz etme yöntemi bulunmaktadır. Yüksek polariteye sahip pestisitler (örneğin, imazalil, logPo/w = 3.82), suda yüksek çözünürlük gösterenler (imazalil, 20 °C'de 180 mg/L) ve sistemik etkisiyle çalışanlar (imazalil ve thiophanate-methyl) bitki dokularına daha derinlemesine nüfuz eden ve asimilasyon akışıyla hareket eden sistemik bileşiklerin işleme sırasında azaltılması kabuklarda ve kabukların rendelenip dondurulması sırasında daha zor olmuştur.

Literatürde yapılan araştırmalarda da benzer şekilde işleme faktörlerinin hesaplandığı, bu faktörlerin aktif maddenin türüne, fizikokimyasal özelliklerine ve formülasyonda kullanılan taşıyıcıların türüne bağlı olarak değişiklik gösterdiği rapor edilmiştir [7, 79, 80-85]. Bu sonuçlar, mevcut çalışmadan elde edilen bulgularla uyumlu olduğunu göstermektedir.




Şekil 1. Farklı evsel tekniklerin işleme faktörleri (İf) (Sütunlardaki farklı harfler istatistiksel olarak önemli farklılıkları temsil etmektedir ($p<0.05$). *S (Soyma), ME (Meyve eti), LS (Limon suyu), $LRD_1$ (Dondurulmuş Limon Kabuğu Rendesi (Depolamanın 1. ayı)), $LRD_2$ (Dondurulmuş Limon Kabuğu Rendesi (Depolamanın 2. ayı)), $LRD_3$ (Dondurulmuş Limon Kabuğu Rendesi (Depolamanın 3. ayı))

*Figure 1. Processing factors (Pf) of different household techniques (Different letters in the columns represent statistically significant differences ($p<0.05$). *S (peel), ME (pulp), LS (lemon juice), LRD1 (frozen grated lemon peel (1st month of storage)), LRD2 (frozen grated lemon peel (2nd month of storage)), LRD3 (frozen grated lemon peel (3rd month of storage))*

## SONUÇ

Bu çalışmada, limonların evsel işleme yöntemleriyle (kabuk soyma ve meyve eti, limon suyu üretimi, reçel üretimi, kabukların rendelenerek dondurulması gibi) işlenmesi sırasında pestisit kalıntı seviyelerinde meydana gelen değişimler incelenmiştir. Araştırma sonuçları, pestisitlerin fizikokimyasal özelliklerinin (log Po/w değeri, çözünürlük ve polarite), etki mekanizmalarının yanı sıra işlenen ürünün biyolojik özelliklerinin, kalıntı seviyelerindeki değişimlerde belirleyici bir rol oynadığını ortaya koymaktadır.

Yüksek polariteye sahip (örneğin log Po/w değeri düşük olan) ve suda yüksek çözünürlük gösteren pestisitler, daha etkili olduklarından, işleme sırasında daha kolay uzaklaştırılmıştır. Özellikle, meyve eti, limon suyu üretimi ve reçel yapımı gibi işlemlerde İf değeri 1'den düşük olarak tespit edilmiş, bu da pestisit kalıntılarının önemli ölçüde azaldığını ortaya koymuştur. Buna karşın, kabuk soyma ve dondurma süreçlerinde, pestisitlerin konsantrasyonlarının artış göstermesi nedeniyle İf değerleri 1'den büyük olarak bulunmuştur. Bu durum, sistemik pestisitlerin bitki dokularına derinlemesine nüfuz etmesi ve taşınabilir özellikleri nedeniyle işlemle tamamen uzaklaştırılamadığını göstermektedir.

Sonuç olarak, bu çalışma, evsel işleme yöntemlerinin pestisit kalıntılarını azaltmada etkili olduğunu, ancak bu etkinliğin pestisit türüne ve fizikokimyasal özelliklerine bağlı olarak değişiklik gösterebileceğini ortaya koymuştur. Ayrıca, elde edilen bulgular, tüketicilerin güvenli gıda tüketimini sağlamak adına evsel işleme yöntemlerini bilinçli bir şekilde kullanmasının önemini vurgulamaktadır. Bununla birlikte, laboratuvar koşullarında ürünlerin pestisitlerin ticari formülasyonlarına daldırılması, gerçek işleme süreçlerinin etkilerini tam olarak yansıtmamaktadır. Bu nedenle, daha doğru ve güvenilir işleme faktörlerinin belirlenmesi için gelecekteki çalışmalarda, pestisit uygulamalarının tarlada gerçekleştirilmelidir. Tarlada işlenmiş bitkilerde, pestisitler hasat öncesi sürece bağlı olarak bitkinin farklı dokularına nüfuz edebilir. Pestisitlerin bitkilerden emilimi ve taşınması, işleme sürecinde pestisit kalıntılarının kaderini doğrudan etkileyebilmektedir. Bu bağlamda, mevcut çalışma bir vaka çalışması olarak değerlendirilmeli ve tarlada işlenmiş bitkiler kullanılarak farklı gıda türleri ve işleme

yöntemlerini içeren daha kapsamlı araştırmalar yürütülmelidir.

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**Research Paper / Araştırma Makalesi**

# Sustainability of Hospital Catering Services: Water and Carbon Footprint

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## ABSTRACT

It is important that mass catering services in hospitals adhere to health quality standards and are carried out sustainably and reliably, aligning with the planning of patients' nutritional treatments in clinical services. This study aims to analyze the carbon and water footprints of menus used in hospital catering services. The research was conducted across all four seasons, with 31-day months included to standardize the number of days in the selection of seasonal menus. Among the menus, one of the most frequently prepared meals using traditional production methods was selected separately for lunch and dinner. In the study, carbon and water footprint calculations were performed for pre-selected meals used in hospital catering services. During spring, summer, autumn, and winter, the carbon footprint levels of the first meal group were significantly higher than those of the second and third meal groups ($p<0.001$). Similarly, in all seasons, the water footprint levels of the first meal group were significantly higher than those of the second and third meal groups ($p<0.001$). Hospital catering services, which primarily serve patients, staff, and visitors within mass nutrition systems, play a vital role in protecting health, supporting medical nutrition treatments, and contributing to the sustainable nutrition chain. In this context, to ensure sustainability in hospital catering services, it is essential to develop guidelines tailored to public catering systems and consider the carbon and water footprints of these menus.

**Keywords**: Sustainability, Carbon footprint, Water footprint, Catering, Hospital

## Hastane Yemek Hizmetlerinin Sürdürülebilirliği: Su ve Karbon Ayak İzi

## ÖZ

Hastanelerdeki toplu yemek hizmetlerinin sağlık kalite standartlarına uygun, sürdürülebilir ve güvenilir bir şekilde gerçekleştirilmesi, klinik hizmetlerdeki hastaların beslenme tedavilerinin planlamasıyla uyumlu olması önemlidir. Bu çalışma, hastane yemek hizmetlerinde kullanılan menülerin karbon ve su ayak izlerini analiz etmeyi amaçlamaktadır. Araştırma, dört mevsim boyunca gerçekleştirilmiş ve mevsimsel menü seçiminde gün sayısını standartlaştırmak için 31 günlük aylar dahil edilmiştir. Menüler arasında, geleneksel üretim yöntemleriyle en sık hazırlanan yemeklerden biri, öğle ve akşam yemekleri için ayrı ayrı seçilmiştir. Çalışmada, hastane yemek hizmetlerinde kullanılan önceden seçilmiş yemekler için karbon ve su ayak izi hesaplamaları yapılmıştır. İlk yemek grubunun karbon ayak izi seviyeleri, ilkbahar, yaz, sonbahar ve kış mevsimlerinde, ikinci ve üçüncü yemek gruplarına göre anlamlı derecede daha yüksek bulunmuştur ($p<0.001$). Benzer şekilde, tüm mevsimlerde, ilk yemek grubunun su ayak izi seviyeleri, ikinci ve üçüncü yemek gruplarına göre anlamlı derecede daha yüksek bulunmuştur ($p<0.001$). Hastane yemek hizmetleri, toplu beslenme sistemleri içinde öncelikli olarak hastalara, personele ve ziyaretçilere hizmet vererek sağlığın korunmasında, tıbbi beslenme tedavilerinin desteklenmesinde ve sürdürülebilir beslenme zincirine katkıda bulunmada hayati bir rol oynamaktadır. Bu bağlamda, hastane yemek hizmetlerinde sürdürülebilirliği sağlamak için kamu yemek sistemlerine özel yönergeler geliştirilmesi ve bu menülerin karbon ve su ayak izlerinin dikkate alınması gereklidir.

**Anahtar Kelimeler**: Sürdürülebilirlik, Karbon ayak izi, Su ayak izi, Yemek hizmetleri, Hastane

## INTRODUCTION

It is of great importance that mass nutrition services carried out within the scope of quality standards in health in hospitals are carried out sustainably and reliably following the planning of the patient's nutritional treatment in clinical services. In hospital menus, both healthy nutrition-specific and disease-specific therapeutic diet meals should be prepared and standardized in quality, taste, and variety that will increase the consumption of patients and should be offered to the consumption of patients [1]. In addition, taking measures to ensure a sustainable service in all processes in hospital catering services is one of the issues that have gained importance in recent years [2].

The term sustainability was first used in the late 1980s by the Brundland Commission in its report "Our Common Future". Sustainability is defined as "the frugal, regenerative use of renewable resources" and in another definition, it is defined as "the conservation of assets needed by economic, social and ecological systems, at least at the level needed" [3]. In the "Brundtland Report" published by the World Commission on Environment and Development (WCED) in 1987, the concept of sustainable development was defined as "a development that focuses on meeting the needs of the present without compromising the ability of future generations to meet their own needs" [4].

Sustainable development is not only multidimensional but also dynamic. Wealth, development, and success, which are generally measured by the level of gross domestic product, need to be renewed to include social and environmental indicators. In this respect, ecological footprint and carbon footprint appear as indicators that measure environmental sustainability. Ecological footprint is defined as the area of fertile land and water used for the reproduction of consumed resources and the disposal of wastes. The concept of carbon footprint is called a subset of the "ecological footprint" proposed by Wackernagel in the early 1990s [5]. Climate change, which manifests itself as an important problem of the world, refers to "the changes in the global climate system and consequent changes in ecosystems due to the excessive increase in the accumulation of greenhouse gases in the atmosphere due to anthropogenic effects" [6]. The rapid rise in global temperature is due to the increasing greenhouse effect due to the release of anthropogenic greenhouse gases into the atmosphere. The carbon footprint of a product or service refers to its total greenhouse gas (GHG) emissions at each stage of its lifecycle: production, use/consumption, and disposal. Global Warming Potential (GWP) is calculated mathematically and its unit is carbon dioxide equivalent ($CO_2$-e) [7]. Current dietary trends indicate a 95.0 percent increase in global demand for meat and animal food products, which will increase food-related GHG to 80.0 percent by 2050 [8]. An indicator of sustainability in societies is the measurement of the amount of water used within the country and on a global scale. The concept of water footprint was first introduced by Hoekstra in 2002 to identify water use along the supply chains of products and services. The concept of water footprint is an expression of both direct water use and indirect water use in the production process in all processes from raw material processing, direct operations and consumer use of the product. Methodologies for calculating the water footprint of products have been developed by Hoekstra et al. In the case of agricultural products, the water footprint is usually expressed in $m^3$/ton or litres/kg. Other ways to express the water footprint of a product are water volume/kcal (for food products in the context of diets) or water volume/joule (for electricity or fuels) [9,10]. The water footprint of a product is also defined as the volume of freshwater used to produce the product, measured along the full supply chain. The water footprint shows not only the volume of water used but also the type of water used, where, and when it is used. The blue, green, and grey water footprints are the three components of the water footprint that represent water use and quality [11]. Blue water footprint refers to the consumption of blue water resources (surface and groundwater) along the supply chain of a product. Green water footprint refers to the consumption of green water resources (rainwater stored in the soil as soil moisture). Grey water footprint refers to pollution and is defined as the volume of freshwater required to assimilate a load of pollutants according to current ambient water quality standards [12]. Approximately one-third of the total water footprint of agriculture in the world is generated by the production of animal products. It is reported that the water footprint of any animal product is more than the water footprint of plant products with equivalent nutritional value [13].

## MATERIALS and METHODS

### Place and Time of the Study

This study was planned and conducted in a Research and Application Hospital in Isparta to examine the carbon and water footprints of the menus used in hospital catering services between August 2020 and March 2021. Approval for the research was obtained from Gazi University Ethics Commission with the research code 2020-168 on 21.02.2020. The legal permission for the study to be conducted in hospital kitchen menus was obtained on 26.06.2020 with the decision numbered 26515734-605.01-E. The entire budget of the study belongs to the researcher.

### General Plan of Study

In the study, carbon and water footprint calculations of pre-selected meals applied in hospital catering services were made by the researcher in person. The study was conducted in 4 seasons. To standardize the number of application days in the selection of seasonal menus, 31-day months were included in the study. August menu for the summer season, October menu for the autumn season, January menu for the winter season, and March menu for the spring season were taken into consideration.

In the hospital, 3 food groups are applied to 3 meal alternating menus without a set selection. Only the selected meals at lunch and dinner were included in the

study. Due to the frequent repetition of some of the meals in the menus and the presence of meals with similar workflow and raw material content in the food groups, one of the most frequently produced meals using traditional production methods was selected separately for lunch and dinner. If the selected meal was repeated in that month, it was not re-evaluated. The standard recipe of the selected meal and the amounts of nutrients in a portion were obtained from the institutional authority through the executive dietician.

In the study, the first group of meal types consisted of large pieces of meat meals, small pieces of meat meals, meatballs, chicken meals, fish meals, meaty vegetable meals, meaty legume meals, egg meals, and liver meals, the second group of meal types consisted of pasta, rice, soups, vegetable meals with olive oil, legume meals with olive oil, Turkish ravioli, pastries, and flatbreads, and the third group consisted of salads, milk desserts, pastry desserts, compotes, and pleasantries, fruits, and others (yogurt, pickles, etc.).

## Carbon and Water Footprint Calculation

The average carbon and water footprint factors published in the extant literature were utilised in the carbon and water footprint calculations of the selected meals included in the study. As Turkey-specific data on the carbon and water footprint factors of foods is lacking, the carbon and water footprint factors obtained from meta-analyses [14, 15] and studies [16, 17] in the existing literature were used. These sources provided robust and widely recognized estimates that were deemed appropriate for the scope of this investigation. The carbon footprint factors for each food item were quantified in units of kilograms per product (kg-product), while the water footprint factors were measured in cubic meters per ton ($m^3$/ton). This differentiation in units reflects the distinct nature of carbon and water usage associated with food production and consumption. To ensure consistency and accuracy in the footprint calculations, each of these food-specific factors was meticulously converted into grams per product (gram-product). This conversion was essential for facilitating precise calculations at the portion level. The carbon and water footprint factors for each food are expressed in kilograms per product and cubic metres per ton, respectively.In the study, each of the food-specific factors was converted into a gram per product to calculate the carbon and water footprints of one portion of the meals included in the standard meal recipes applied in the institution. Subsequently, these standardized footprint factors were applied to determine the carbon and water footprints of individual meal portions. The meals analyzed were based on standard meal recipes that are routinely implemented within the institution under study. By adopting this methodological approach, the research was able to provide detailed insights into the environmental impacts of each meal component, thereby enabling a comprehensive assessment of the overall sustainability of the institution's meal offerings.

From the ingredients in the recipes, pepper paste, black cumin, noodles, hazelnut, semolina, baking powder, kadayif, cocoa, black pepper, kemal pasha, cornichon pickles, curry sauce, lemon salt, pasta, Turkish ravioli, parsley, puff pastry, corn starch, leek, proline, saffron, tomato paste, grape vinegar, soda, sumac, granulated sugar, vermicelli, cinnamon powder, salt, vanilla, allspice, phyllo dough, and turmeric are not included in the calculation since they do not have food-specific carbon footprint factors. Also, among the ingredients in the recipes, trout, pumpkin, black cumin, coconut, baking powder, kadayif, cocoa, kashar cheese, kemal pasha, red cabbage, cumin, cornichon pickles, curry sauce, cream, dried thyme, lemon salt, curd cheese, Turkish ravioli, puff pastry, proline, saffron, grape vinegar, soda, sumac, tahini, vermicelli, salt, allspice, yogurt, phyllo dough, turmeric are not included in the calculation since they do not have food-specific water footprint factors.

## Statistical Evaluation of Data

The data were analyzed with IBM SPSS V23. Compliance with normal distribution was analyzed by the Shapiro-Wilk test. The Independent two-sample t-test was used to compare normally distributed data according to binary groups and the Mann-Whitney U test was used to compare non-normally distributed data. One-way analysis of variance was used to compare normally distributed data according to groups of three or more and multiple comparisons were analysed by Duncan test. Kruskal Wallis test was used for the comparison of non-normally distributed data according to groups of three or more. The significance level was taken as $p<0.050$.

## Limitations of the Study

Due to the insufficiency of the literature data, when evaluating the water footprint of the meals, grey, green, and blue water footprints were evaluated as total water footprints, not separately. In addition, the lack of water and the carbon footprint of all foods constitutes another limitation.

## RESULTS

The data obtained in the study conducted to examine the carbon and water footprints of the menus used in hospital catering services are given below under the relevant headings.

Table 1 shows the carbon footprint values of 1 portion of the meals included in the study according to the groups in terms of $CO_2$ equivalent/kg. The meal with the highest carbon footprint value of one portion is elbasan tava (4.098) for the first group and the meal with the lowest carbon footprint value is chickpea with chicken (0.077). For the second group, the highest meal was flatbread with minced meat (2.348) and the lowest meal was sawdust pastry with cheese (0.036). For the third group, the highest meal was cacik (1.183) and the lowest meal was quince compote (0.028).

Table 1. Carbon footprint values for each portion of meals according to groups ($CO_2$ equivalent/kg)

| Group 1 | | Group 2 | | Group 3 | |
|---|---|---|---|---|---|
| Meal Name | $CO_2$ equivalent/kg | Meal Name | $CO_2$ equivalent/kg | Meal Name | $CO_2$ equivalent/kg |
| Elbasan Tava | 4.098 | Flatbread with Minced | 2.348 | Cacik (Tzatziki) | 1.183 |
| Stick Kebab | 4.085 | Wedding Rice | 1.455 | Kalburabasti | 0.436 |
| Hungarian Goulash (Puree) | 4.071 | Trotter Soup | 0.676 | Yogurt | 0.404 |
| Tas Kebab | 4.062 | Fresh Beans with Olive Oil | 0.605 | Cocoa Pudding | 0.380 |
| Forest Kebab | 4.051 | Imambayildi | 0.353 | Keshkul | 0.326 |
| Shepherd Roast with Vegetables | 3.984 | Bulgur Rice with Chicken | 0.347 | Orange | 0.320 |
| Boiled Meat | 3.948 | Manti | 0.332 | Milk Halva | 0.317 |
| Beef Emense Over Rice | 3.541 | Pasta with Yoghurt | 0.305 | Semolina Halva | 0.275 |
| Minced Meat Sauteed Over Rice | 3.418 | Oven Pasta | 0.302 | Oven Rice Pudding | 0.250 |
| Sauteed Meat Over Rice | 3.411 | Puff Pastry with Chicken | 0.286 | Kazandibi | 0.249 |
| Albanian Liver | 3.199 | Tomato Soup | 0.252 | Şekerpare | 0.244 |
| Abant Kebab | 2.978 | Lentil Pastry | 0.243 | Peanut Dream | 0.205 |
| Izmir Meatballs | 2.869 | Yayla Soup | 0.214 | Mixed Salad | 0.145 |
| Dalyan Meatballs (Puree) | 2.811 | Leek with Olive Oil | 0.208 | Tulumba | 0.143 |
| Izmir Meatballs | 2.752 | Cheese Pastry | 0.204 | Seasonal Salad | 0.140 |
| Farm Meatballs | 2.742 | Celery with Olive Oil | 0.198 | Shepherd Salad | 0.137 |
| Grilled Meatballs | 2.702 | Ayran Soup | 0.185 | Spoon Salad | 0.135 |
| Terbiyeli Meatballs | 2.633 | Bulgur Rice | 0.170 | Yoghurt Dessert with Sesame | 0.100 |
| Celery with Meat | 1.748 | Pasta with Cheese | 0.158 | Yogurt Dessert | 0.100 |
| Karnıyarık | 1.629 | Rice | 0.147 | Pumpkin Dessert | 0.098 |
| Patlıcan Oturtma | 1.585 | Gemici Soup | 0.132 | Ashoura | 0.092 |
| Minced Spinach with Yoghurt | 1.568 | Vermicelli Soup | 0.117 | Peach | 0.086 |
| Egg with Minced Meat | 1.561 | Peas with Olive Oil | 0.115 | Carrot Cabbage Salad | 0.083 |
| Fresh Beans with Meat | 1.511 | Cauliflower with Olive Oil | 0.114 | Watermelon | 0.064 |
| Peas with Meat | 1.435 | Rice Soup | 0.113 | Pear | 0.058 |
| Cauliflower with Mince | 1.425 | Bulgur Rice with Vermicelli | 0.110 | Kadayif | 0.053 |
| Okra with Meat | 1.406 | Wire Vermicelli Soup | 0.104 | Wire Kadayif | 0.053 |
| Sour Pumpkin with Meat | 1.400 | Cheese and Walnut Noodles | 0.102 | Kemalpasha | 0.048 |
| Potato with Meat | 1.397 | Meyhane Rice | 0.097 | Mixed Compote | 0.039 |
| Kabak Kalye | 1.396 | Noodles with Cheese | 0.096 | Quince Compote | 0.028 |
| Chickpeas with Meat | 1.386 | Bulgur Rice with Lentils | 0.096 | | |
| Dried Beans with Meat | 1.367 | Kidney Beans with Olive Oil | 0.092 | | |
| Meat Fajita (Saffron Rice Garnish) | 0.988 | Pasta with Tomato | 0.090 | | |
| Chicken Nuget (with Vegetable Garnish) | 0.953 | Ezogelin Soup | 0.087 | | |
| Chicken Fajita | 0.874 | Lentil Soup | 0.081 | | |
| Chicken Over Rice | 0.870 | Bulgur Rice with Chickpeas | 0.081 | | |
| Chicken Casserole | 0.832 | Dried Beans with Olive Oil | 0.070 | | |
| Boiled Chicken | 0.827 | Pasta with Sauce | 0.064 | | |
| Sauced Chicken | 0.822 | Tutmaç Soup | 0.058 | | |
| Creamy Chicken (with Pasta Garnish) | 0.809 | Flatbread with Cheese | 0.052 | | |
| Tavuk Dünyası | 0.809 | Flatbread with Spinach | 0.041 | | |
| Trout (with Potato Garnish) | 0.710 | Sawdust Pastry with Cheese | 0.036 | | |
| Chicken Galantine (Puree) | 0.622 | | | | |
| Chicken Meatloaf | 0.622 | | | | |
| Chicken Bowl with Spinach | 0.607 | | | | |
| Chicken Cauliflower with Bechamel Sauce | 0.452 | | | | |
| Potato with Chicken | 0.327 | | | | |
| Roman Style Spinach | 0.319 | | | | |
| Chickpeas with Chicken | 0.077 | | | | |

Table 2 shows the mean ($\bar{X}$), standard deviation (SD), and upper and lower values of the carbon footprint of 1 portion of the meals included in the study according to the groups in terms of $CO_2$ equivalent/kg. For the first group, the highest average carbon footprint of a portion of the carbon footprint was for large meat meals (4.047±0.067) and the lowest was for fish meals (0.683±0.027). For the second group, the highest meal type was flatbreads (0.814±1.329) and the lowest meal type was legume meals with olive oil (0.078±0.015). For the third group, the highest meal type was yogurt, pickles, and other meals (0.434±0.316), while the lowest meal type was compote and pleasantries (0.034±0.008).

Table 3 shows the mean ($\bar{X}$), standard deviation (SD), median, and lower and upper values of the carbon footprint ($CO_2$ equivalent/kg) of one portion of the meals included in the study according to the groups and seasons. In the spring season, the highest average carbon footprint of one portion for the first group was large meat meals (4.072), while the lowest was egg meals (0.320). For the second group, the highest carbon footprint was flatbreads (2.349) and the lowest was legume meals with olive oil (0.056). For the third group, the highest meal type was yogurt, pickles, etc. ($\bar{X}$±SD=0.314±0.128) and the lowest meal type was fruits ($\bar{X}$±SD=0.029±0.041). In the summer season, the highest average carbon footprint of a portion of the meal for the first group was large pieces of meat meals (4.098), while the lowest was fish meals (0.656). For the second group, the highest meal type was Turkish ravioli (0.332) and the lowest meal type was flatbreads (0.041). For the third group, the highest meal type was yogurt, pickles, etc. ($\bar{X}$±SD=0.794±0.551), while the lowest meal type was compote and pleasantries (0.040). In the autumn season, the highest average carbon footprint of one portion for the first group was large pieces of meat meals ($\bar{X}$±SD=4.010±0.087), while the lowest was fish meals (0.684). For the second group, the highest meal type was Turkish ravioli (0.332) and the lowest meal type was

pasta ($\bar{X}$±SD=0.080±0.032). For the third group, the highest meal type was yogurt, pickles, etc. ($\bar{X}$±SD=0.314±0.127) and the lowest meal type was fruits ($\bar{X}$±SD=0.072±0.020). For the first group, the highest mean or median of the carbon footprint of a portion of meal in the winter season is small pieces of meat meals (median=3.988), while the lowest is legume meals with meat (median=0.643). For the second group, the highest meal type was Turkish ravioli (0.332) and the lowest meal type was flatbreads (0.052). For the third group, the highest meal type was yogurt, pickles, etc. ($\bar{X}$±SD=0.314±0.127), while the lowest meal types were fruits ($\bar{X}$±SD=0.029±0.041), compote, and pleasantries (0.029). There was no statistically significant difference between the seasons according to the means and medians of the carbon footprint of one portion of all meal types in the first group, second group, and third group (p>0.05).

Table 2. Mean ($\bar{X}$), standard deviation (SD), and lower upper values distribution of carbon footprint ($CO_2$ equivalent/kg) for each portion of meals according to groups

| Carbon Footprint for Each Portion ($CO_2$ equivalent/kg) | | | | |
|---|---|---|---|---|
| Meal Group | Meal Types | S | $\bar{X}$±SD | Lower-Upper |
| Group 1 | Large Piece Meat Meals | 4 | 4.047±0.067 | 3.949-4.098 |
| | Small Piece Meat Meals | 12 | 3.839±0.344 | 2.978-4.086 |
| | Meatballs | 13 | 2.855±0.256 | 2.634-3.418 |
| | Chicken Meals | 23 | 0.739±0.187 | 0.327-0.988 |
| | Fish Meals | 3 | 0.683±0.027 | 0.656-0.711 |
| | Vegetable Meals with Meat | 22 | 1.485±0.106 | 1.334-1.749 |
| | Legume Meals with Meat | 7 | 1.140±0.475 | 0.078-1.386 |
| | Egg Meals | 2 | 0.941±0.878 | 0.319-1.562 |
| | Liver Meals | 3 | 3.199±0.000 | 3.199-3.199 |
| Group 2 | Pastas | 15 | 0.131±0.083 | 0.058-0.305 |
| | Rice | 17 | 0.272±0.419 | 0.072-1.455 |
| | Soups | 24 | 0.155±0.167 | 0.039-0.677 |
| | Vegetable Meals with Olive Oil | 6 | 0.266±0.188 | 0.115-0.605 |
| | Legume Meals with Olive Oil | 7 | 0.078±0.015 | 0.056-0.092 |
| | Manti | 4 | 0.332±0.000 | 0.332-0.332 |
| | Pastries | 5 | 0.179±0.100 | 0.036-0.287 |
| | Flatbreads | 3 | 0.814±1.329 | 0.041-2.349 |
| Group 3 | Salads | 12 | 0.132±0.023 | 0.083-0.145 |
| | Milk Desserts | 12 | 0.209±0.109 | 0.092-0.381 |
| | Pastry Desserts | 16 | 0.158±0.118 | 0.048-0.436 |
| | Compote and Pleasantries | 2 | 0.034±0.008 | 0.029-0.039 |
| | Fruits | 9 | 0.052±0.032 | 0.058-0.320 |
| | Others (Yoghurt, Pickles, etc.) | 8 | 0.434±0.316 | 0.223-1.183 |

In Table 4, it is examined whether there is a difference between the meal groups according to the carbon footprint ($CO_2$ equivalent/kg) values of the meal types included in the study within each season. In the spring season, the carbon footprint level (median) of one portion of the meal types in the first meal group is 1,489, while the level in the second meal group is 0,159 and in the third meal group is 0.145. In the summer season, the carbon footprint level (median) of one portion of the meal types in the first meal group is 1.418, in the second meal group it is 0.114, and in the third meal group, it is 0.118. In the autumn season, the carbon footprint level (median) of one portion of the meal types in the first meal group was 1.426, while the level was 0.097 in the second meal group and 0.140 in the third meal group. In the winter season, the carbon footprint level (median) of one portion of the meal types served in the first meal group is 1.407, in the second meal group it is 0.092, and in the third meal group, it is 0.142. In the spring, summer, autumn, and winter seasons, the carbon footprint levels of the first group portion were significantly higher than the carbon footprint levels of the second group and third group portion (p<0.001).

Table 5 shows the water footprint values of 1 portion of the meals included in the study according to the groups in $m^3$/ton. The meal with the highest water footprint value of one portion is stick kebab for the first group (2500.804) and the meal with the lowest water footprint value is chickpea with chicken (119.323). For the second group, the highest meal was wedding rice (985.745) and the lowest meal was Turkish ravioli (56.431). For the third group, the highest meal was mixed compote (618.683) and the lowest meal was cacik (52.175).

Table 6 shows the mean ($\bar{X}$), standard deviation (SD), and lower and upper values of the water footprint of 1 portion of the meals included in the study according to the groups in $m^3$/ton. For the first group, the highest water footprint average of a portion of water footprint was for large meat meals (2447.275±29.554) and the lowest was for fish meals (230.435±0.000). For the second group, the highest meal type was legume meals with olive oil (425.194±10.051) and the lowest meal type was Turkish ravioli (56.431±0.000). For the third group, the highest meal type was compotes and pleasantries (361.958±363.065) and the lowest meal type was yogurt, pickles, and other meals (52.175±0.000).

Table 3. Mean ($\bar{X}$), standard deviation (SD), median and lower upper values of carbon footprint ($CO_2$ equivalent/kg) for each portion of meals according to groups and seasons.

| Mea Group | Meal Types | Spring S | Spring $\bar{X}$±SD Median (Lower-Upper) | Summer S | Summer $\bar{X}$±SD Median (Lower-Upper) | Autumn S | Autumn $\bar{X}$±SD Median (Lower-Upper) | Winter S | Winter $\bar{X}$±SD Median (Lower-Upper) | p |
|---|---|---|---|---|---|---|---|---|---|---|
| Group 1 | Large Piece Meat Meals | 1 | 4.072 | 1 | 4.098 | 2 | 4.010±0.087 | - | - | - |
| | Small Piece Meat Meals | 4 | 3.762 (2.980-3.990) | 2 | 4.057 (4.050-4.060) | 4 | 3.989 (3.410-4.090) | 2 | 3.988 (3.980-3.990) | 0.225** |
| | Meatballs | 2 | 3.080 (2.740-3.420) | 3 | 2.789 (2.750-2.810) | 2 | 2.818 (2.770-2.870) | 6 | 2.722 (2.630-3.420) | 0.313** |
| | Chicken Meals | 6 | 0.818 (0.450-0.990) | 5 | 0.827 (0.360-0.870) | 4 | 0.829 (0.710-0.950) | 8 | 0.716 (0.330-0.870) | 0.665** |
| | Fish Meals | 1 | 0.711 | 1 | 0.656 | 1 | 0.684 | - | - | - |
| | Vegetable Meals with Meat | 3 | 1.533±0.039 | 7 | 1.471±0.083 | 7 | 1.476±0.111 | 5 | 1.490±0.164 | 0.869* |
| | Legume Meals with Meat | 2 | 1.365 (1.360-1.370) | 1 | 1.386 (1.386-1.386) | 2 | 1.288 (1.210-1.370) | 2 | 0.643 (0.080-1.210) | 0.228** |
| | Egg Meals | 1 | 0.320 | 1 | 1.562 | - | - | - | - | - |
| | Liver Meals | 1 | 3.199 | 1 | 3.199 | 1 | 3.199 | - | - | - |
| Group 2 | Pastas | 5 | 0.186±0.113 | 4 | 0.110±0.058 | 2 | 0.080±0.032 | 4 | 0.108±0.058 | 0.350* |
| | Rice | 4 | 0.133 (0.070-0.180) | 4 | 0.244 (0.070-1.450) | 5 | 0.084 (0.070-0.140) | 4 | 0.123 (0.070-1.280) | 0.619** |
| | Soups | 5 | 0.117 (0.040-0.250) | 8 | 0.114 (0.060-0.680) | 7 | 0.104 (0.060-0.670) | 4 | 0.080 (0.060-0.100) | 0.481** |
| | Vegetable Meals with Olive Oil | 2 | 0.407 (0.210-0.600) | - | - | 1 | 0.116 | 3 | 0.198 (0.110-0.350) | 0.248*** |
| | Legume Meals with Olive Oil | 1 | 0.056 | 2 | 0.081 (0.070-0.090) | 2 | 0.082 (0.070-0.090) | 2 | 0.082 (0.070-0.090) | 0.773** |
| | Manti | 1 | 0.332 | 1 | 0.332 | 1 | 0.332 | 1 | 0.332 | - |
| | Pastries | 2 | 0.184 (0.120-0.240) | 1 | 0.205 (0.205-0.205) | 2 | 0.161 (0.040-0.290) | - | - | 1.000*** |
| | Flatbreads | 1 | 2.349 | 1 | 0.041 | - | - | 1 | 0.052 | - |
| Group 3 | Salads | 3 | 0.123±0.035 | 4 | 0.140±0.004 | 3 | 0.122±0.034 | 2 | 0.143±0.004 | 0.653* |
| | Milk Desserts | 2 | 0.237±0.204 | 3 | 0.223±0.119 | 2 | 0.172±0.112 | 5 | 0.204±0.104 | 0.954* |
| | Pastry Desserts | 6 | 0.202±0.104 | 3 | 0.068±0.029 | 3 | 0.211±0.200 | 4 | 0.122±0.092 | 0.342* |
| | Compote and Pleasantries | - | - | 1 | 0.040 | - | - | 1 | 0.029 | - |
| | Fruits | 2 | 0.029±0.041 | 3 | 0.069±0.015 | 2 | 0.072±0.020 | 2 | 0.029±0.041 | 0.318* |
| | Others (Yoghurt, Pickles, etc.) | 2 | 0.314±0.128 | 2 | 0.794±0.551 | 2 | 0.314±0.127 | 2 | 0.314±0.127 | 0.388* |

*One-way analysis of variance, **Kruskal Wallis test, ***Mann-Whitney U test.

Table 0. Comparison of the median values of carbon footprint ($CO_2$ equivalent/kg) for each portion of the first, second, and third groups according to the seasons.

| Season | | Group 1 S | Group 1 Median (Lower-Upper) | Group 2 S | Group 2 Median (Lower-Upper) | Group 3 S | Group 3 Median (Lower-Upper) | p |
|---|---|---|---|---|---|---|---|---|
| $CO_2$ Footprint | Spring | 21 | $1.489^a$ (0.319-4.072) | 21 | $0.159^b$ (0.039-2.349) | 15 | $0.145^b$ (0.000-0.404) | <0.001* |
| | Summer | 22 | $1.418^a$ (0.108-4.098) | 21 | $0.114^b$ (0.041-1.455) | 16 | $0.118^b$ (0.039-1.183) | <0.001* |
| | Autumn | 23 | $1.426^a$ (0.245-4.086) | 20 | $0.097^b$ (0.036-0.668) | 12 | $0.140^b$ (0.054-0.436) | <0.001* |
| | Winter | 23 | $1.407^a$ (0.078-3.994) | 19 | $0.092^b$ (0.052-1.277) | 16 | $0.142^b$ (0.000-0.404) | <0.001* |

*Kruskal-Wallis test, a-b: There is no difference between groups with the same letter

Table 7 shows the mean ($\bar{X}$), standard deviation (SD), median, and lower upper values of the water footprint ($m^3$/ton) of one portion of the meals included in the study according to the groups and seasons. In the spring season, the highest average water footprint of one portion for the first group was large meat meals (2459.529) and the lowest was fish meals (230.435). For the second group, the highest meal type was flatbreads (792.428) and the lowest meal type was Turkish ravioli (56.431). For the third group, the highest meal type was milk desserts ($\bar{X}$±SD=326.149±18.736), while the lowest meal type was salads ($\bar{X}$±SD=131.303±6.288). In the summer season, the highest mean or median of the water footprint of one portion for the first group was small pieces of meat meals (median=2469.135) and the lowest was fish meals (230.435). For the second group, the highest meal type was rice ($\bar{X}$±SD=439.631±378.735) and the lowest meal type was Turkish ravioli (56.431). For the third group, the highest meal type was compotes and pleasantries (618.684), while the lowest meal type was yogurt, pickles, etc. (52.176). In the autumn season, the highest mean or median of the water footprint of a portion of water footprint for the first group was small pieces of meat meals (median=2470.705) and the lowest was fish meals (230.435). For the second group, the highest meal type was vegetable meals with olive oil (483.026), while the lowest meal type was Turkish ravioli (56.431). For the third group, the highest meal type was milk desserts ($\bar{X}$±SD=300.801±54.583), while the lowest meal type was yogurt, pickles, etc. (52.175). In the winter season, the highest mean or median of the water footprint of one portion of the first group was small pieces of meat meal (median=2469.135) and the lowest was legume meal with meat (median=583.598). For the second group, the highest meal type was legume meals with olive oil (median=423.850) and the lowest meal type was Turkish ravioli (56.431). For the third group, the highest meal type was milk desserts ($\bar{X}$±SD=303.633±33.485) and the lowest meal type was compotes and pleasantries (105.232). There was no statistically significant difference

between the seasons according to the means and medians of the water footprint of one portion of all meal types in the first group, second group, and third group ($p>0.05$).

Table 5. Water footprint values for each portion of meals according to groups ($m^3$/ton)

| Group 1 | | Group 2 | | Group 3 | |
|---|---|---|---|---|---|
| Meal Name | $m^3$/ton | Meal Name | $m^3$/ton | Meal Name | $m^3$/ton |
| Stick Kebab | 2500.804 | Wedding Rice | 985.745 | Mixed Compote | 618.683 |
| Tas Kebab | 2493.257 | Imambayildi | 887.710 | Melon | 481.000 |
| Elbasan Tava | 2466.800 | Flatbread with Minced | 792.428 | Ashoura | 339.397 |
| Hungarian Goulash (Puree) | 2459.529 | Peas with Olive Oil | 483.026 | Keshkul | 333.670 |
| Shepherd Roast with Vegetables | 2448.152 | Kidney Beans with Olive Oil | 433.253 | Tulumba | 329.385 |
| Forest Kebab | 2445.012 | Dried Beans with Olive Oil | 414.450 | Kalburabasti | 320.640 |
| Boiled Meat | 2403.243 | Bulgur Rice with Chicken | 401.945 | Cocoa Pudding | 312.901 |
| Albanian Liver | 2305.617 | Ayran Soup | 393.304 | Pumpkin Dessert | 303.500 |
| Beef Emense Over Rice | 2238.116 | Trotter Soup | 391.559 | Milk Halva | 298.865 |
| Minced Meat Sauteed Over Rice | 2137.400 | Rice | 343.525 | Peanut Dream | 287.199 |
| Sauteed Meat Over Rice | 2137.105 | Cheese and Walnut Noodles | 335.088 | Kazandibi | 279.395 |
| Izmir Meatballs | 2055.794 | Oven Pasta | 327.707 | Şekerpare | 265.483 |
| Abant Kebab | 1830.356 | Leek with Olive Oil | 324.556 | Oven Rice Pudding | 262.205 |
| Farm Meatballs | 1793.819 | Fresh Beans with Olive Oil | 292.267 | Apple | 192.400 |
| Dalyan Meatballs (Puree) | 1748.165 | Noodles with Cheese | 288.688 | Watermelon | 192.400 |
| Izmir Meatballs | 1706.668 | Lentil Pastry | 284.555 | Mandarin | 192.400 |
| Terbiyeli Meatballs | 1694.146 | Celery with Olive Oil | 271.810 | Banana | 192.400 |
| Grilled Meatballs | 1672.565 | Puff Pastry with Chicken | 266.449 | Orange | 192.400 |
| Dried Beans with Meat | 1095.127 | Cheese Pastry | 247.329 | Peach | 192.400 |
| Celery with Meat | 1056.837 | Pasta with Cheese | 244.503 | Fresh Grape | 192.400 |
| Chickpeas with Meat | 1048.453 | Cauliflower with Olive Oil | 216.370 | Pear | 184.400 |
| Karnıyarık | 1034.306 | Bulgur Rice with Lentils | 207.163 | Yoghurt Dessert with Sesame | 166.219 |
| Egg with Minced Meat | 1029.048 | Lentil Soup | 204.131 | Yogurt Dessert | 166.219 |
| Patlıcan Oturtma | 1012.270 | Bulgur Rice with Chickpeas | 200.637 | Semolina Halva | 158.064 |
| Fresh Beans with Meat | 956.686 | Ezogelin Soup | 190.834 | Mixed Salad | 136.910 |
| Chicken Nuget (with Vegetable Garnish) | 945.871 | Tutmaç Soup | 181.788 | Shepherd Salad | 135.981 |
| Meat Fajita (Saffron Rice Garnish) | 935.935 | Bulgur Rice with Vermicelli | 178.760 | Kadayif | 135.500 |
| Sour Pumpkin with Meat | 927.030 | Flatbread with Cheese | 170.650 | Wire Kadayif | 135.500 |
| Okra with Meat | 916.512 | Pasta with Tomato | 162.070 | Carrot Cabbage Salad | 132.495 |
| Peas with Meat | 910.454 | Rice Soup | 162.043 | Spoon Salad | 132.445 |
| Potato with Meat | 906.581 | Yayla Soup | 158.956 | Seasonal Salad | 124.505 |
| Kabak Kalye | 906.145 | Bulgur Rice | 158.867 | Quince Compote | 105.232 |
| Cauliflower with Mince | 896.414 | Pasta with Sauce | 158.082 | Kemalpasha | 89.100 |
| Minced Spinach with Yoghurt | 888.695 | Meyhane Rice | 157.770 | Cacik (Tzatziki) | 52.175 |
| Sauced Chicken | 778.883 | Pasta with Yoghurt | 150.537 | | |
| Creamy Chicken (with Pasta Garnish) | 774.901 | Vermicelli Soup | 126.484 | | |
| Tavuk Dünyası | 774.901 | Wire Vermicelli Soup | 126.484 | | |
| Chicken Fajita | 770.010 | Tomato Soup | 123.877 | | |
| Boiled Chicken | 738.787 | Flatbread with Spinach | 122.986 | | |
| Chicken Casserole | 733.493 | Gemici Soup | 107.052 | | |
| Chicken Over Rice | 716.460 | Sawdust Pastry with Cheese | 73.633 | | |
| Chicken Meatloaf | 590.065 | Manti | 56.431 | | |
| Chicken Galantine (Puree) | 589.034 | | | | |
| Chicken Bowl with Spinach | 512.328 | | | | |
| Chicken Cauliflower with Bechamel Sauce | 409.067 | | | | |
| Potato with Chicken | 352.081 | | | | |
| Zucchini with Bechamel Sauce | 346.915 | | | | |
| Roman Style Spinach | 335.360 | | | | |
| Trout (with Potato Garnish) | 230.435 | | | | |
| Chickpeas with Chicken | 119.323 | | | | |

In Table 8, it is examined whether there is a difference between the meal groups according to the water footprint ($m^3$/ton) values of the meal types included in the study within each season. In the spring season, the water footprint level (median) of one portion of the meal types in the first meal group was 1012.270, in the second meal group the level was 247.329, and in the third meal group, the level was 192.400. In the summer season, the water footprint level (median) of one portion of the meal types in the first meal group was 941.859, in the second meal group it was 181.788 and in the third meal group, it was 184.400. In the autumn season, the water footprint level (median) of one portion of the meal types in the first meal group was 1034.306, in the second meal group it was 202.384 and in the third meal group, it was 192.400. In the winter season, the water footprint level (median) of one portion of the meal groups in the first meal group was 916.512, in the second meal group the level was 216.370 and in the third meal group, the level was 192.400. In the spring, summer, autumn, and winter seasons, the water footprint levels of the first group one portion were significantly higher than the water footprint levels of the second group and third group one portions ($p<0.001$).

Table 6. Distribution of mean ($\bar{X}$), standard deviation (SD), and lower and upper values of water footprint (m$^3$/ton) for each portion of meals according to groups.

| | | Water footprint of 1 Portion (m$^3$/ton) | | |
|---|---|---|---|---|
| Meal Group | Meal Types | S | $\bar{X}$±SD | Min-Max |
| Group 1 | Large Piece Meat Meals | 4 | 2447.275±29.554 | 2403.243-2466.800 |
| | Small Piece Meat Meals | 12 | 2371.883±205.310 | 1830.356-2500.804 |
| | Meatballs | 13 | 1825.884±167.693 | 1672.565-2137.400 |
| | Chicken Meals | 23 | 675.423±167.101 | 346.915-945.871 |
| | Fish Meals | 3 | 230.435±0.000 | 230.435-230.435 |
| | Vegetable Meals with Meat | 22 | 906.030±178.840 | 140.402-1056.837 |
| | Legume Meals with Meat | 7 | 928.807±357.639 | 119.323-1095.127 |
| | Egg Meals | 2 | 682.204±490.512 | 335.36-1029.048 |
| | Liver Meals | 3 | 2305.617±0.000 | 2305.617-2305.617 |
| Group 2 | Pastas | 15 | 232.867±76.951 | 150.537-335.088 |
| | Rice | 17 | 364.040±306.855 | 157.770-985.745 |
| | Soups | 24 | 208.842±101.212 | 107.052-393.304 |
| | Vegetable Meals with Olive Oil | 6 | 412.623±249.499 | 216.370-887.710 |
| | Legume Meals with Olive Oil | 7 | 425.194±10.051 | 414.448-433.254 |
| | Manti | 4 | 56.431±0.000 | 56.431-56.431 |
| | Pastries | 5 | 223.859±85.392 | 73.633-284.555 |
| | Flatbreads | 3 | 362.021±373.504 | 122.986-792.428 |
| Group 3 | Salads | 12 | 132.546±5.168 | 124.505-136.910 |
| | Milk Desserts | 12 | 310.378±31.721 | 262.205-339.398 |
| | Pastry Desserts | 16 | 210.144±92.336 | 89.100-329.385 |
| | Compote and Pleasantries | 2 | 361.958±363.065 | 105.232-618.684 |
| | Fruits | 9 | 221.095±91.649 | 184.400-481.000 |
| | Others (Yoghurt, Pickles, etc.) | 8 | 52.175±0.000 | 52.175-52.176 |

Table 7. Mean ($\bar{X}$), standard deviation (SD), median and lower upper values of water footprint (m$^3$/tonne) of 1 portion of meals according to groups and seasons.

| | | Spring | | Summer | | Autumn | | Winter | | p |
|---|---|---|---|---|---|---|---|---|---|---|
| Meal Group | Meal Types | S | $\bar{X}$±SD Median (Lower-Upper) | S | $\bar{X}$±SD Median (Lower-Upper) | S | $\bar{X}$±SD Median (Lower-Upper) | S | $\bar{X}$±SD Median (Lower-Upper) | |
| Group 1 | Large Piece Meat Meals | 1 | 2459.529 | 1 | 2466.800 | 2 | 2431.386 (2431.386-2431.386) | - | - | - |
| | Small Piece Meat Meals | 4 | 2341.564(1830.356-493.257) | 2 | 2469.135 (2445.012-2493.257) | 4 | 2470.705 (2137.105-2500.804) | 2 | 2469.135 (2445.012-2493.257) | 0.631** |
| | Meatballs | 2 | 1965.610 (1793.819-2137.400) | 3 | 1748.165 (1706.668-1793.819) | 2 | 1901.932 (1748.069-2055.794) | 6 | 1727.417 (1672.565-2137.400) | 0.433** |
| | Chicken Meals | 6 | 727.624 (409.067-935.935) | 5 | 733.493 (346.915-778.883) | 4 | 756.844 (733.493-945.871) | 8 | 653.263 (352.081-774.901) | 0.348** |
| | Fish Meals | 1 | 230.435 | 1 | 230.435 | 1 | 230.435 | - | - | - |
| | Vegetable Meals with Meat | 3 | 957.556 (888.695-1012.270) | 7 | 910.454 (888.695-1012.270) | 7 | 906.581 (140.402-1034.306) | 5 | 916.512 (888.695-1056.837) | 0.890** |
| | Legume Meals with Meat | 2 | 1071.500(1047.873-1095.127) | 1 | 1048.453 | 2 | 1071.5 (1047.873-1095.127) | 2 | 583.598 (119.323-1047.873) | 0.325** |
| | Egg Meals | 1 | 335.360 | 1 | 1029.048 | - | - | - | - | - |
| | Liver Meals | 1 | 2305.617 | 1 | 2305.617 | 1 | 2305.617 | - | - | - |
| Group 2 | Pastas | 5 | 233.903±78.441 | 4 | 234.137±87.004 | 2 | 247.290±124.166 | 4 | 223.091±80.318 | 0.990* |
| | Rice | 4 | 423.825±382.687 | 4 | 439.631±378.735 | 5 | 217.790±72.821 | 4 | 411.477±392.675 | 0.695* |
| | Soups | 5 | 181.788 (123.877-393.304) | 8 | 160.500 (107.052-391.559) | 7 | 190.834 (123.877-393.304) | 4 | 197.483 (126.484-393.304) | 0.572** |
| | Vegetable Meals with Olive Oil | 2 | 308.412 (292.267-324.556) | - | - | 1 | 483.026 | 3 | 271.810 (216.370-887.710) | 0.800*** |
| | Legume Meals with Olive Oil | 1 | 433.253 | 2 | 423.851 (414.447-433.253) | 2 | 423.852 (414.450-433.253) | 2 | 423.850 (414.450-433.250) | 0.893** |
| | Manti | 1 | 56.431 | 1 | 56.431 | 1 | 56.431 | 1 | 56.431 | - |
| | Pastries | 2 | 265.942 (247.329-284.555) | 1 | 247.330 | 2 | 170.041 (73.633-266.449) | - | - | 0.667*** |
| | Flatbreads | 1 | 792.428 | 1 | 122.986 | - | - | 1 | 170.650 | - |
| Group 3 | Salads | 3 | 131.303±6.288 | 4 | 132.461±5.642 | 3 | 135.129±2.328 | 2 | 130.708±8.772 | 0.811* |
| | Milk Desserts | 2 | 326.149±18.736 | 3 | 317.488±33.113 | 2 | 300.801±54.583 | 5 | 303.633±33.485 | 0.837* |
| | Pastry Desserts | 6 | 227.748±82.912 | 3 | 130.273±38.825 | 3 | 261.842±109.502 | 4 | 204.867±111.643 | 0.358* |
| | Compote and Pleasantries | - | - | 1 | 618.684 | - | - | 1 | 105.232 | - |
| | Fruits | 4 | 192.400 (184.400-192.400) | 5 | 192.400 (184.400-481.000) | 6 | 192.400 (184.400-481.000) | 4 | 192.400 (184.400-192.400) | 0.844** |
| | Others (Yoghurt, Pickles, etc.) | - | - | 1 | 52.176 | 1 | 52.175 | - | - | - |

*One-way analysis of variance, **Kruskal Wallis test, ***Mann-Whitney U test

Table 8. Comparison of median values of water footprint (m$^3$/ton) for each portion of the first, second, and third groups according to seasons

| Season | | Group 1 | | Group 2 | | Group 3 | | p |
|---|---|---|---|---|---|---|---|---|
| | | S | Median (Lower-Upper) | S | Median (Lower-Upper) | S | Median (Lower-Upper) | |
| Water Footprint | Spring | 21 | 1012.270$^a$ (230.435-2493.257) | 21 | 247.329$^b$ (56.431-985.745) | 15 | 192.400$^b$ (124.505-339.397) | <0.001* |
| | Summer | 22 | 941.859$^a$ (230.435-2493.257) | 21 | 181.788$^b$ (56.431-985.745) | 17 | 184.400$^b$ (52.176-618.684) | <0.001* |
| | Autumn | 23 | 1034.306$^a$ (140.402-2500.804) | 20 | 202.384$^b$ (56.431-483.026) | 15 | 192.400$^b$ (52.175-481.000) | <0.001* |
| | Winter | 23 | 916.512$^a$ (119.323-2493.257) | 19 | 216.370$^b$ (56.431-985.745) | 16 | 192.400$^b$ (89.100-339.397) | <0.001* |

*Kruskal-Wallis test, a-b: There is no difference between groups with the same letter.

## DISCUSSION

In catering services, various activities cause environmental impacts at all stages from raw material procurement, acceptance, storage, food production, distribution, and service. Environmental sustainability in hospital catering services, which are usually carried out as service procurement, is increasingly recognized and researched [18-21]. This study was planned and conducted to examine the carbon and water footprints of menus used in hospital catering services. To the best of our knowledge, this is the first study conducted to determine the carbon and water footprints of menus used in hospital catering services. As a result of the study, the carbon footprint assessment of catering services and the meals on the menu were discussed.

With the increasing awareness of climate change, it is seen that the number of researchers and companies who want to calculate the carbon footprint of food products,

determine their impact on global warming, and increase awareness by sharing this data with consumers is increasing and this trend is becoming popular [22]. The high proportion of discarded food waste produces negative environmental impacts that are fuelling climate change. The breakdown of food in landfills produces methane, a powerful GHG with a GWP 104 times higher than carbon dioxide [23]. In the study conducted by Madalı et al. (2021) vegetable dishes with meat, fish, and turkey, and legumes with meat and chicken dishes were found among the food types with high SG emission values. Accordingly, it was determined that the GHG emission of the food types containing animal products was higher than the other food types and the first group meals had higher GHG emission values than the other groups [24]. In a study conducted in the USA, GHG emissions associated with food waste were analyzed using the Life Cycle Assessments approach. The total emissions from the production, processing, packaging, distribution, retail sale, and disposal of food were found to be 112.9 million metric tons (MMT) $CO_2$ equivalent. Beef was identified as the largest source of loss-related emissions, accounting for 16.0 percent of loss-related emissions, despite accounting for 22.0 percent of food losses per kilogram [25]. In another study conducted in the Netherlands, the current Dutch dietary pattern and the GHG emissions of 4 different dietary patterns were evaluated. Consumption patterns consist of healthy dietary patterns with and without meat, and diets containing nutrients with lower environmental impacts. At the end of the study, it was revealed that eliminating meat products from the diet and/or consuming only foods with low GHG emissions would reduce the average GHG emission by 28.0-46.0%. However, it was also emphasized in the study that consumption patterns in which only foods with low GHG emissions are consumed may cause deficiency in terms of some nutrients [26]. Adequate and balanced menus in hospital catering services ensure reduced GHG emissions [27]. In the study, in parallel with the results of other studies, the first three food types with the highest carbon footprint were determined as large piece meat dishes, small piece meat dishes, and liver dishes. This is an expected result considering that the minimum amount of red meat used in these products is 50 g and the maximum amount is 150 g according to the standard recipes of the institution. There was no statistically significant difference between the seasons according to the means and medians of the carbon footprint of all meal types in the first group, second group, and third group ($p>0.05$). One of the reasons for this result is that the standard recipes of these meals applied in the institution do not show seasonal differences, although the products that are abundant and cheap in season are used in the recipes. Similar to other studies, the carbon footprint levels of the first group were significantly higher than the carbon footprint levels of the second group and the third group in the spring, summer, autumn, and winter seasons ($p<0.001$). The use of meat or the addition of eggs in almost all of the first group meals was considered as one of the factors increasing the carbon footprint of the first group meals.

The commercial sector, which includes health care, public institutions, and restaurants, reportedly consumes 900 million gallons of water per day, ranging from 1.5 gallons per meal for school lunches to 2.0 gallons per meal for all-day restaurants or cafeterias [28]. In addition to the total water used in the production stages of catering services, the total water footprint of the meals varies depending on the type of raw material used in the meals. When calculating the total water footprint, attention is paid to indirect water use in addition to the water used in production and consumption. In other words, both the direct water use and the indirect water use of a product along the production line should also be calculated [29]. In this study, while evaluating the water footprint of meals, grey, green, and blue water footprints were evaluated as total water footprints, not separately. In a study conducted in India, 5 different diets were evaluated in terms of water footprint. The diets were categorized as rice with less variety; rice and fruit; wheat and legumes; wheat, rice and oils; and rice and meat. While the green water footprint of rice-based diets was higher, the blue water footprint of the wheat-based diet was found to be higher. In addition, it was determined that the environmental impact of the rice and meat diet model was higher than the other models [30]. Mekonnen and Hoekstra (2012) reported that the average water footprint per calorie for beef was 20 times higher than for cereals and starchy crops; the water footprint per gram of protein for milk, eggs, and chicken meat was 1.5 times higher than for legumes [15]. In a study conducted in Turkey, it was calculated that the food group that increased the water footprint the most was small piece meat dishes, similar to GHG emissions. Vegetable dishes with meat (11.9%) were shown to increase the water footprint level significantly in the summer season, while large meat dishes (11.4%) and meatballs (11.3%) were shown to affect water footprint levels at similar rates, although the frequency of serving them was lower compared to vegetable dishes with meat [31]. In a study conducted in thirteen cities in Mediterranean countries where the water source is from outside the city, the water footprint of the current diet was determined to be between 3277 L/g and 5789 L/g per capita. These values were shown to be about thirty times higher than local water use. In addition, in this study, 3 different diet types were created, and in the calculations; it was determined that the Mediterranean diet could reduce the water footprint by 19.0% to 43.0%, the pesco vegetarian diet by 28.0% to 52.0% and the vegetarian diet by 30.0% to 53.0% [32]. Uçar and Çapar emphasized that Turkey is not a water-rich country and that it is a good practice to export products that do not provide added value to the country but have a high share in water consumption [33]. In the study, the first three meal types with the highest total water footprint in parallel with the carbon footprint were calculated as large piece and small piece meat dishes and liver dishes; the first group water footprint levels were found to be significantly higher than the second group and third group water footprint levels in all seasons ($p<0.001$). It is known that the water footprint increases as well as the carbon footprint due to the increase in the amount of meat used in meat-containing dishes. Considering that the largest part of the water footprint of consumption in Turkey is caused by agriculture with 89.0%, this is an expected result and consistent with the previously mentioned studies.

## CONCLUSIONS

This study was carried out on 62 meals in selected months representing each season and 248 meals in total, produced in the kitchen of a research and application hospital in Isparta. The carbon and water footprint of the meals were calculated and evaluated. In the spring, summer, autumn, and winter seasons, the carbon footprint levels of the first group were found to be significantly higher than the carbon footprint levels of the second and third pots ($p<0.001$). Again, in all seasons, the first-group water footprint levels were significantly higher than the second and third-group water footprint levels ($p<0.001$).

Hospital catering services, which serve primarily patients, staff, and patient visitors within the collective nutrition systems, have an important place both for the protection of health and support for medical nutrition treatment and their effects on the sustainable nutrition chain. Hospital catering services, which primarily serve patients, staff, and patient visitors within collective nutrition systems, have an important place both in terms of health protection and support for medical nutrition therapy and in terms of their effects on the sustainable nutrition chain. Health professionals play a key role in the implementation of sustainable food systems. For this reason, it is important to plan training activities to increase the awareness and knowledge levels of dietitians, physicians, nurses, and other auxiliary health personnel, especially food services nutrition dietitians.

### AUTHOR CONTRIBUTIONS

HB, SB contributed to idea conception, design, data collection, data analysis, data interpretation. HB, SB contributed to manuscript drafting and manuscript revision. HB, SB supervised the whole study and revised the final manuscript. All authors approved the final manuscript.

### DATA AVAILABILITY

The dataset generated and analyzed during the current study is available from the corresponding author on reasonable request.

### ETHICAL APPROVALS

This study was performed in accordance with the Declaration of Helsinki. Approval for the research was obtained from Gazi University Ethics Commission with the research code 2020-168 on 21.02.2020.

### CONFLICT of INTERESTS

All authors declare that they have no competing interests.

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**Araştırma Makalesi / Research Paper**

# Termosonikasyon ve Mikrodalga Ön İşlemlerinin Kırmızı Pancar (*Beta vulgaris L.*) Pestilinin İnce Tabaka Kuruma Kinetiği Üzerine Etkileri

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## ÖZ

Meyve ve sebzelerin zengin biyoaktif bileşenler içermesi, pestillerin fonksiyonel bir atıştırmalık olarak tüketilmesini cazip kılmaktadır. Koyu kırmızı bir kök sebzesi olan pancar (*Beta vulgaris* L.), sağlık üzerindeki olumlu etkileri ve zengin besin içeriği ile son yıllarda dikkat çekmiştir. Bu çalışmanın amacı, termosonikasyon ve mikrodalga ön işlemlerinin fonksiyonel bir atıştırmalık olarak geliştirilen kırmızı pancar pestilinin ince tabaka kuruma kinetiği üzerindeki etkilerini araştırmaktır. Ayrıca ön işlem süresini ve etkisini azaltmak için pestil herlesinin suda çözünür kuru madde oranını geleneksel yöntemlerde uygulanan 40°Brix (Bx) değerinden 15-20°Bx'e düşürme olanağının irdelenmesi hedeflenmiştir. Yapılan çalışma sırasında pestil herlesine uygulanan ön işlemlere bağlı olarak kuruma süresi 75-120 dakika arasında değişmiştir. Geleneksel haşlama yöntemine alternatif olarak uygulanan termosonikasyon ön işlemi, kuruma süresini %4-10 aralığında azaltmıştır. Kuruma süresi, termosonikasyon ve mikrodalga ön işleme yöntemlerinin birlikte uygulanmasıyla %12-22 aralığında azalmıştır. Dolayısıyla farklı ön işlem uygulamaları, kırmızı pancar pestillerinin kuruma süresi ve buna bağlı olarak kuruma hızını etkilemiştir. Kırmızı pancar pestillerinin kuruma davranışına en uygun ince tabaka matematiksel modellerinin belirlendiği çalışmada, ön işlem koşulları değiştikçe kurutma kinetiğini açıklayan en uygun model de değişmiştir. Pestillerin efektif nem difüzyon katsayısı ($m^2/s$) $8.91\times10^{-8}$-$1.14\times10^{-7}$ arasında değişmiştir.

**Anahtar Kelimeler:** Kırmızı pancar, Pestil, Mikrodalga, Termosonikasyon, İnce tabaka kurutma modelleri

## Effects of Thermosonication and Microwave Pre-treatments on Thin Layer Drying Kinetics of Red Beetroot (*Beta vulgaris L.)* Pestils

## ABSTRACT

The rich bioactive components found in fruits and vegetables make pestils an attractive option as a functional snack. Beetroot (*Beta vulgaris* L.), a dark red root vegetable, has received a great attention in recent years due to its positive health effects and rich nutritional content. The aim of this study is to investigate the effects of thermosonication and microwave pre-treatments on the thin-layer drying kinetics of red beetroot pestil developed as a functional snack. Additionally, it aims to explore the possibility of reducing the water-soluble dry matter content of the pestil pulp from the traditionally applied 40°Brix (Bx) to 15-20°Bx to minimize pre-treatment time and influence. During the study, the drying time of the pestil pulp varied between 75 and 120 min depending on the pretreatments applied. Thermosonication pre-treatment, used as an alternative to the conventional blanching method, reduced the drying time by 4-10%. When thermosonication and microwave pretreatment methods were applied together, the drying time decreased by 12-22%. Therefore, different pretreatment applications influenced the drying time and, consequently, the drying rate of red beetroot pestils. In the study, which identified the most suitable thin-layer mathematical models for the drying behavior of red beetroot pestils, the optimal model explaining the drying kinetics changed as the pre-treatment conditions varied. The effective moisture diffusion coefficient ($m^2/s$) of the pestils ranged between $8.91\times10^{-8}$ and $1.14\times10^{-7}$.

**Keywords:** Red beet, Pestil, microwave, Thermosonication, Thin layer drying models

## GİRİŞ

Günümüz endüstrisi; artan dünya nüfusu ile birlikte tüketime hazır gıda çeşitliliğinin artırılmasına odaklanmıştır. Bununla birlikte, sağlıklı beslenmeye olan taleplerin karşılanması için ise çevre dostu yaklaşımlar ile enerji ve zaman tasarrufu sağlayacak ve aynı zamanda kaliteli ürünler geliştirecek çalışmaları da ele almaya başlamıştır. Global fonksiyonel ürün pazarının 2030 yılında 285.3 milyon Dolar hacmine ulaşması beklendiğinden [1] bu yükselişe geçen pazara yenilikçi ürün alternatifinin eklenmesi, bilimsel olarak ürün ve proses detaylarının incelenmesi çok önemlidir. Ürünlerin fonksiyonel özelliklerinin ortaya koyulması, geliştirilmesi ve üretim metotlarının optimize edilmesi gibi hususlar ele alınmalıdır ve tüketim oranlarının artırılması için farkındalık yaratma çalışmalarına devam edilmelidir [2].

Chenopodiaceae familyasının koyu kırmızı bir kök sebzesi olan pancar (*Beta vulgaris L*.), sağlık üzerine olumlu etkileri ve zengin besin içeriği ile son yıllarda bilim dünyasının ilgisini çekmektedir. Vitaminler, mineraller, fenoller, karotenoidler, nitrat, askorbik asit ve betalainler gibi temel bileşenleri içeren, bitki dünyasının en zengin gıdalarından biridir [3, 4, 5]. Pancar yaygın olarak sebze formunda tüketilse de çeşitli mutfak ve beslenme amaçları için meyve suyu, turşu, toz ve gıda takviyeleri formuna da işlenebilmektedir [5-10]. Buna rağmen kırmızı pancarın zengin sağlıklı atıştırmalık formülasyonlarında daha fazla yer alması gerektiği düşünülmektedir. Diğer taraftan kırmızı pancar ürüne işlendiği zaman terapötik etkilerinin korunması için buna uygun optimum koşulların seçilmesi önem arz etmektedir [11].

Pestil, meyve pulpuna şeker ve nişasta gibi katkı maddelerinin eklenerek koyulaştırılmasından sonra kurutulmasıyla üretilen geleneksel üründür. Pestillerin uzun raf ömrüne sahip olması, kurutma sonrası gelişen hacim küçülmesi, ağırlığının azalması ve paketleme aşamasının kolay olması gibi özellikleri tercih edilmesini artırmıştır. Üretildiği meyve ve sebzelerin, zengin biyoaktif bileşenleri içermesi, pestillerin fonksiyonel atıştırmalık olarak kullanımını cazip kılmaktadır [12-14]. Literatürde çeşitli meyve ve sebzelerden üretilmiş sağlıklı atıştırmalık kategorisinde sıralanabilecek pestiller üzerine çalışmalar mevcuttur [12, 14-19]. Ancak, literatürde yapılan çalışmalarda ve marketlerde kırmızı pancarından elde edilen fonksiyonel pestil gibi yenilikçi alternatif bir geleneksel ürün bulunmamaktadır.

Kurutma, gıda ürünlerini korumanın geleneksel yollarından biridir. Kurutmanın temel amacı bünyesindeki suyun gıda matrisinden kısmen uzaklaştırılması, sonuç olarak raf ömrünün uzatılması ve gıda bozulmasının önlenmesidir [20]. Kurutma sayesinde, gıdanın hacim ve ağırlığında azalma, buna bağlı olarak ambalaj boyutunda küçülme, nakliyede ve depolamada daha düşük maliyet ve kolaylık gibi avantajlar sağlanmaktadır [21]. Öte yandan, kurutma işlemi, gıdadaki fiziksel ve biyokimyasal değişikliklere bağlı olarak nihai ürünün kalitesini önemli ölçüde etkilemektedir. Gıda matrisindeki bu değişiklikler, kuruma süresi, sıcaklık ve ürünün su aktivitesi gibi faktörlerden etkilenmektedir [22]. Kurutmayı hızlandırmak, enzimlerin etkin bir şekilde inaktivasyonunu sağlamak ve oksidasyona engel olmak amacıyla kurutmadaki bazı dezavantajları azaltmak ve gidermek adına kurutma öncesi ön-muamele teknikleri üzerine çalışmalar yapılmaktadır [23]. Konvansiyonel ön işlemler genellikle sıcak su ile haşlama, buharda haşlama, hiperozmotik solüsyon, alkali solüsyonlar, sülfatlama ve asit ile muameleleri içerir. Bu ön işlemlerin, kuruma süresini kısaltarak ve kaliteyi artırarak kurutma işlemi üzerinde olumlu etkileri olmasına rağmen, özellikle uzun süreli kurutma sırasında kimyasal absorpsiyon, kalite bozulması, yetersiz rehidrasyon, yapısal çökme, besin kayıpları ve yüksek enerji tüketimi gibi olası sorunları da ortaya çıkarabilmektedir. Dolayısıyla, kurutma prosesini daha da iyileştirmek ve geliştirmek için yeni ısıl ve ısıl olmayan ön işlemler de araştırılmaktadır. Yeni ön işlem tekniklerinin geliştirilmesi ve uygulanması, kuruma süresini kısaltmakla birlikte kuruma hızını artırmaktadır, nem dağılımını iyileştirmektedir ve enerji tüketimini azaltmaktadır. Aynı zamanda kurutulmuş ürünlerin fonksiyonel ve besinsel kalite özelliklerini gelenekselden daha iyi hale getirebilmektedir [24]. Bu tip ihtiyaçlar doğrultusunda, soğuk plazma (CP), darbeli elektrik alanı (PEF), yenilebilir film kaplama, termal olmayan ultrasonikasyon, yüksek nemli sıcak havayla haşlama (HHAIB), kızılötesi haşlama (IRB) ve mikrodalga (MW) gibi alternatif termal teknikler araştırılmıştır [24]. Ultrases, 20 kHz veya daha yüksek sonik dalgalarla üretilen bir enerji şeklidir [24]. Meyve sebzelerin kurutulma işlemlerinden önce US ile ön muamelesinin, gıda kalitesini geliştirici etkilere sahip olduğu tespit edilmiştir. Böylece kurutma süresinde azalma, enerji tüketiminde tasarruf ve kurutulmuş ürün kalitesinin korunması sağlanır [24-27]. Mikrodalgalar, 300 MHz ila 300 GHz arasında değişen frekansa sahip elektromanyetik dalgalardır. Gıda endüstrisindeki uygulamaları, işleme süresinin azaltılması, ısıtma verimliliği, güvenli kullanım ve kolaylığı, kalitede iyileşme ve suda çözünür besin kayıplarını azaltma gibi avantajlar nedeniyle büyük popülerlik kazanmıştır [28]. Bu nedenle mikrodalga, yüzey neminin hızla giderilmesi için bir ön işlem tekniği olarak tavsiye edilmektedir [24]. Literatürde, farklı işleme tekniklerinin kırmızı pancarın kuruma kinetiği üzerine etkisinin araştırıldığı çalışmalar mevcuttur. Yapılan bir çalışmada, kırmızı pancar dilimlerine farklı sıcaklık ve sürelerde (65°C ve 85°C'de 10-15 dakika) sıcak su ile haşlama ve ultrasonikasyon (5-10 dakika 40 kHz) ön işlemleri uygulanmış, ardından dondurarak kurutma tekniği ile kurutma gerçekleştirilmiştir. Bu ön işlemler sonrasında haşlama uygulanan örneklerde büzülme oranının azaldığı, aynı zamanda gözenekliliğin arttığı gözlemlenmiştir [29]. Başka bir çalışmada mikrodalga ön işleminin kırmızı pancarın kuruma süresi, kimyasal bileşenler ve besin içeriği üzerindeki etkileri incelenmiştir. Kırmızı pancarın suda haşlama ve mikrodalga (600 W) ön işlemine tabi tutulduktan sonra kurutulması karşılaştırılmış ve mikrodalga ön işleminin, besin bileşenlerinin korunmasına yardımcı olduğu belirtilmiştir [30]. Ayrıca, farklı mikrodalga güçlerinin kırmızı pancar püresinin kuruma karakteristiği üzerine etkileri incelenmiş ve mikrodalga işleminin kuruma süresini önemli ölçüde azalttığı sonucuna varılmıştır [31]. Bu tür ön işlemler, kırmızı pancar ürünlerinin kalitesini iyileştirebilecek potansiyel yöntemler olarak öne çıkmaktadır.

İnce tabaka kurutma modelleri, gıda ve tarım ürünlerinin kurutma kinetiğinin analiz edilmesine yardımcı olarak, ürün kalitesinin korunmasına, hasat ve işleme sırasında ortaya çıkabilecek kayıpların azaltılmasına destek olabilecek yaklaşımlardır [32]. İnce tabaka kurutma denklemleri teorik, yarı teorik ve ampirik modeller olmak üzere üç kategoriye ayrılmaktadırlar [32, 33]. İnce tabaka kurutma modelleri gıdaların ve pestillerin kurutulması sırasında başarıyla uygulanmıştır. Bu çalışmalarda hint inciri pestillerinin kuruma kinetiğini açıklayan en uygun modelin Page modeli olduğu [34], muşmula, dut, havuçlu kırmızıbiber biberli pestilinin kurutulmasının incelendiği çalışmada Page ve Modifiye Page modelleri [13, 35, 36], kivi, muşmula ve mango pestillerinde Midilli modelinin uygun olduğu [14, 37] sonucuna varılmıştır.

Bu çalışmanın amacı elma (doğal tatlandırıcı) ve tapyoka nişastasını (kıvam artırıcı) kullanarak kırmızı pancar pestil formülasyonunu optimize etmek ve geleneksel haşlama ön işlemine alternatif olarak mikrodalga ve termosonikasyon kullanımının ince tabaka kuruma kinetiğine olan etkisini irdelenmektir.

## MATERYAL ve METOT

### Materyal

Çalışma için kullanılan kırmızı pancarlar (*Beta vulgaris var. conditiva*) ve elma (*Starkrimson delicious)* Bursa ilinde yerel bir marketten temin edilmiştir. Çalışma kapsamında kullanılan kırmızı pancarlar ve elmalar, pestil üretim sürecine kadar, 4.0±0.5°C sıcaklıkta buzdolabında 1 ay süreyle saklanmıştır. Diğer hammaddelerden olan sitrik asit Bursa Uludağ Üniversitesi Gıda Mühendisliği Pilot Tesisi'nden temin edilmiştir. Tapyoka nişastası (TMS Organik Gıda San. Tic. Ltd. Şti) ise glutensiz bir ürün olup Talya Foods isimli internet alışveriş marketten temin edilmiştir.

### Pestil Üretimi

Bu çalışma kapsamında yapılan ön denemeler aşamasında, pestil formülasyonunun optimize edilmesi için farklı oranlarda elma, topyaka nişastası ve kırmızı pancar kullanılmıştır. Üretim aşmasında ise uygulanan haşlama süresi ve yöntemi (açık kazan, termosonikasyon ve mikrodalga), koyulaştırma süresi, hedef briks değerleri (40, 20 ve 15°Bx) ve kurutma sıcaklığı gibi parametreler kapsamlı bir şekilde değerlendirilmiştir. Ön denemeler aşamasında bu parametrelerin pestilin hedonik açıdan genel kabul edilebilirliği (9: çok fazla beğendim; 1: hiç beğenmedim) üzerine olan etkisi 12 adet eğitimli panelist tarafından analiz edilmiştir [19]. Genel beğeninin 7 puan üzerinde elde edildiği nihai formülasyon aşağıda açıklanmıştır ve çalışma kapsamında irdelenecek ön işlemlere ait koşullar Tablo 1'de sunulduğu gibi oluşturulmuştur.

Pestillerin üretimi sırasında, kırmızı pancarların ve elmaların yaprakları ve sapları ayrılmıştır. Meyve ve sebzelerin dış yüzeyi, toprak ve diğer kirliliklerden arındırılmak için etkili bir şekilde yıkanmıştır. Yıkama sonrası kabukları soyulmuştur ve elmaların çekirdekleri çıkarılmıştır. Kırmızı pancarlar ve elmalar, bir sonraki ön işlemler aşamasında kullanılmak üzere 2×2×2 cm ebatlarında küpler halinde kesilerek hazırlanmıştır. Küp kesilmiş kırmızı pancar (reçetenin ağırlıkça %47'i) ve elma (reçetenin ağırlıkça %47'i) ağırlıkça "1:1" oranında tartılmıştır ve üzerine ağırlıkça "1:1" oranında su ilave edilmiştir. Sonra, açık kazan, termosonikasyon, termosonikasyon ve mikrodalganın hibrit uygulanmasını içeren ön haşlama işlemine tabi tutulmuşlardır.

Tablo 1. Ön işlem koşulları
*Table 1. Pre-treatment conditions*

| Koşul | Kurutma Öncesi Uygulanan Ön İşlemler | | | | |
|---|---|---|---|---|---|
| | Haşlama | | | Koyulaştırma[6] | |
| | 1. Ön İşlem | 2. Ön İşlem | Püre Eldesi | Süre (min) | Hedef Son Brix (°) |
| Kontrol | Açık kazan-15 dk[1] | - | √[4] | 105 | 40 |
| 1 | Termosonikasyon[2]- 30 dk | - | √[4] | 78 | 40 |
| 2 | Termosonikasyon[2]- 45 dk | - | √[4] | 58 | 40 |
| 3 | Termosonikasyon[2]- 30 dk | Mikrodalga[3]-10 dk | √[4] | 60 | 40 |
| 4 | Termosonikasyon[2]- 45 dk | Mikrodalga[3]-10 dk | √[4] | 30 | 40 |
| 5 | Açık kazan-15 dk[1] | - | √[4] | 63 | 20 |
| 6 | Termosonikasyon[2]- 30 dk | - | √[4] | 27 | 20 |
| 7 | Termosonikasyon[2]- 45 dk | - | √[4] | 6 | 20 |
| 8 | Termosonikasyon[2]- 30 dk | Mikrodalga[3]-10 dk | √[4] | 6 | 20 |
| 9 | Termosonikasyon[2]- 45 dk | Mikrodalga[3]-10 dk | √[4] | 4 | 20 |
| 10 | Termosonikasyon[2]- 45 dk | - | √[5] | - | 15 |
| 11 | Termosonikasyon[2]- 45 dk | Mikrodalga[3]-10 dk | √[5] | - | 15 |

[1]Atmosferik basınçta açık kazanda 95ºC'de ısıl işlem, [2]35 kHz frekansta ve 80°C sıcaklıkta termosonikasyon,[3]Mikrodalga gücü: 360 W, [4]1. Ön işlem sırasında kullanılan su ile birlikte püre haline getirilir, [5]1. Ön işlem sırasında kullanılan su süzüldükten sonra kalan meyve ve sebze püre haline getirilir, [6]Atmosferik basınçta açık kazanda 100-103ºC'de ısıl işlem,
*[1]Heat treatment at 95ºC in an open boiler at atmospheric pressure,[2]Thermosonication at a frequency of 35 kHz and a temperature of 80°C,[3]Microwave power: 360 W, [4]1. Pureed together with the water used during pre-treatment, [5]1. After draining the water used during pre-treatment, the remaining fruit and vegetables are pureed.[6]Heat treatment at 100-103ºC in an open boiler at atmospheric pressure*

Haşlama amaçlı yapılan birinci aşama ön işlemlerin sonunda yumuşayan kırmızı pancar ve elma, haşlama suyu ile birlikte bir parçalayıcıda (Arzum, İstanbul, Türkiye) püre haline getirilmiştir. Bu işlem kontrol üretimi dahil olmak üzere 1 ve 9. Koşulları içeren tüm üretimlerde uygulanmıştır. 10 ve 11. koşullar uygulanarak gerçekleştirilen üretimde, haşlama suyunun süzülmesinin ardından yumuşayan meyve ve sebzeler mekanik bir parçalayıcı kullanılarak püre haline getirilmiştir. Tüm üretimler sonucu elde edilen pürenin %25'si ayrılarak tapyoka nişastası (reçetenin ağırlıkça %5.8'i) ve sitrik asit (reçetenin ağırlıkça %0.2'i) ile karıştılmak üzere ayrılmıştır. Geri kalan püre atmosferik basınçta açık kazanda koyulaştırma (100-103°C) işlemine tabi tutulmuştur. Hedef son çözünür kuru madde oranına ulaşmaya yakın bir zamanda, nişasta içeren püre koyulaştırma işlemi sırasında yavaş yavaş eklenmiştir. Bu aşamadan sonra belirlenen kurutma öncesi hedef suda çözünür kuru madde oranına ulaşıldığında koyulaştırma işlemi sonlandırılmıştır. Ancak 10. ve 11. koşullarda koyulaştırma işlemi uygulanmadığından haşlama işlemi sonrasında elde edilen püreye katkı maddeleri eklenip homojen bir şekilde karıştırılarak kurutma işlemine geçilmiştir. Tüm ön işlem koşullarda gerçekleştirilen üretim sonucu elde edilen koyulaştırılmış püreler (pestil herlesi), kurutma işlemi gerçekleştirilinceye kadar, +4°C'de 1-2 gün süreyle düşük yoğunluklu polietilen film ile paketlenmiş ve analiz edilene kadar depolanmıştır.

Ön işlemlerin tamamlanmasının ardından, 25.0±0.5 g koyulaştırılmış püre (herle), 8×8×0.5 cm ebatlarında bir kalıp kullanılarak yağlı kağıt üzerine bir spatül yardımıyla serilmiştir. Daha sonra, kurutma işlemi için %20 nispi nemde ve 70°C'de, 2 m/s hızında konveksiyonel kabin tipi kurutucu (Yücebaş Makine Tic. Ltd. Şti., İzmir) kullanılarak kurutma işlemi gerçekleştirilmiştir. Kurutma işlemi, pestillerin nem içeriğinin 0.90 ile 0.10 g su/g kuru madde (KM) oranına ulaşınca sonlandırılmıştır. Kurutma işlemi boyunca, başlangıçta 15 dakika arayla, daha sonra ise sona yaklaşırken 20 dakika aralıklarla, 0.01 g hassasiyetinde dijital bir terazi (Mettler Toledo, MS3002S) ile tartımlar yapılmış ve nem içeriğinin kuruma boyunca değişimi izlenmiştir. Tartımlar, kısa bir sürede (10 s) gerçekleştirilmiştir. Kurutma işlemi tamamlanan pestiller soğutularak 4°C'de depolanmıştır.

## Kurutma Kinetiği

Pestil örneklerinin kurutulması sırasında, nem içeriği değerleri Eşitlik 1 kullanılarak hesaplanmıştır.

$$M_t = \frac{(M-KM)}{KM} \quad (1)$$

Burada; $M_t$ kuruma sürecinin "t" zamanındaki pestillerin nem içeriğini (g su/ g KM), M pestillerin "t" anındaki ağırlığını (g), KM ise pestillerin kuru madde miktarını (g) ifade etmektedir.

Pestil örneklerinin kuruma hızı (KH) Eşitlik 2 kullanılarak hesaplanmıştır.

$$KH = \frac{(M_{t+dt}-M_t)}{dt} \quad (2)$$

Bu eşitlikte KH; kuruma hızı (g su/g KM. dakika), $M_{t+dt}$; t+dt zamanındaki nem içeriği (g su/kg KM), $d_t$ kuruma zamanıdır (dakika).

Nem oranının (MR, birimsiz) hesaplanmasında Eşitlik 3 kullanılmıştır.

$$MR = \frac{(M_t-M_e)}{(M_0-M_e)} \quad (3)$$

Bu eşitlikte, $M_e$; denge anındaki nem içeriği (g su/g KM), $M_o$; başlangıçtaki nem içeriğidir (g su/g KM).

MR değerleri hesaplanırken, $M_e$ değerinin her ürünün son nem içeriği olduğu öngörülmüştür [32, 38].

## İnce Tabaka Matematiksel Modellerle Uyum

Kırmızı pancar pestillerinin kuruma davranışını analiz etmek için farklı ince tabaka kurutma modellerinin kullanılması, ürünlerin nem içeriği ve kuruma hızı gibi önemli parametrelerinin belirlenmesine yardımcı olmaktadır. Bu çalışma kapsamında, yaygın olarak kullanılan yedi farklı ince tabaka kurutma modeli, literatürde tanımlanmış ve önerilmiş olan modeller arasından seçilmiştir. Bu modeller (Tablo 2), kırmızı pancar pestilinin kuruma sürecini analiz etmek ve kuruma kinetiğini anlamak için kullanılmıştır.

Kırmızı pancar pestillerinin deneysel çalışması sırasında elde edilen MR değerlerini açıklayan en uygun ince tabaka kurutma modelleri istatiksel parametreler kullanılarak seçilmiştir. Bu kapsamda korelasyon katsayısını ($R^2$)'nın 1'e yakın olacak şekilde en yüksek değerini sağlayan, tahmini standart hata (RMSE) ve ki-kare ($\chi^2$) değerlerinin de 0'a yakın olacak şekilde en düşük değerlerini sağlayan modeller belirlenmiştir. Bu parametreler Eşitlik 4, 5 ve 6 kullanılarak hesaplama yapılmıştır [32].

$$RMSE = \left[\frac{1}{N}\sum_{t=1}^{N}\left(MR_{exp,i} - MR_{pre,i}\right)^2\right]^{\frac{1}{2}} \quad (4)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{exp,i} - MR_{pre,i})^2}{N-n} \quad (5)$$

$$R^2 = 1 - \left[\frac{(MR_{exp,i}-MR_{pre,i})^2}{(MR_{exp,i}-MR_{ort})^2}\right]^2 \quad (6)$$

Burada, "MR" nem oranını, alt indis olarak kullanılan "exp,i " i. deney sırasında elde edilen değerini, "pre,i" i. tahmin edilen değerini, "ort" ortalama değerini, "N" gözlemlenen deneysel veri adedini," "n" modelde yer alan bağımsız değişken sayısını ifade etmektedir.

Tablo 2. İnce tabaka kurutma modelleri
*Table 2. Thin layer drying models*

| Model No | Model | Denklem* | Kaynaklar |
|---|---|---|---|
| 1 | Lewis (Newton) | MR= exp(-kt) | [39] |
| 2 | Page | MR = exp(-kt$^n$ ) | [40] |
| 3 | Modifiye Page | MR = exp[-(kt)$^n$ ] | [41] |
| 4 | Henderson & Pabis | MR = a exp(-kt) | [42] |
| 5 | Logaritmik | MR = a exp(-kt) + c | [43] |
| 6 | Two Term | MR = a exp(-kt) + (1-a) exp(-kat) | [44] |
| 7 | Wang ve Singh | MR = 1 + at + bt$^2$ | [45] |

**MR: nem oranı; a, b, c: kurutma katsayıları; k: kurutma sabiti; t: kurutma süresi*
**MR: moisture ratio; a, b, c: drying coefficients; k: drying constant; t: drying time*

**Efektif Nem Difüzyon Katsayısının Hesaplanması**

Kırmızı pancar pestil örneklerinin kuruma davranışlarını incelemek için Fick'in İkinci Yasası kullanılmıştır. Bu yasa, kurutma işlemi sırasında nem transferinin difüzyon ile gerçekleştiğini varsaymaktadır. Özellikle plaka şeklindeki malzemeler için geçerli olan bu varsayım, nemin homojen bir şekilde dağıldığı, üründe deformasyon olmadığı, yüzey direncinin ihmal edilebilir düzeyde olduğu ve ortam koşullarının değişiminin önemsiz olduğu durumları kapsamaktadır [46]. Bu bağlamda, sonsuz levhalar için efektif diffüzyon katsayısı literatürde yer alan varsayımlar ve sadeleştirmelerle kırmızı pancar pestillerinin nem difüzyon katsayısı ($D_{eff}$) kuruma süresine karşılık çizilen ln MR grafiğinin eğiminden elde edilmiştir (Eşitlik 7) [47]. Burada sıcaklığın her noktada eşit olduğu öngörülmüştür [48].

$$D_{eff} = \frac{4kL^2}{\pi^2} \quad (7)$$

Burada k, çizilen grafiğin eğimidir. $D_{eff}$ nem difüzyon katsayısı ($m^2/s$), L levhanın yarı kalınlığını ifade etmektedir.

## BULGULAR ve TARTIŞMA

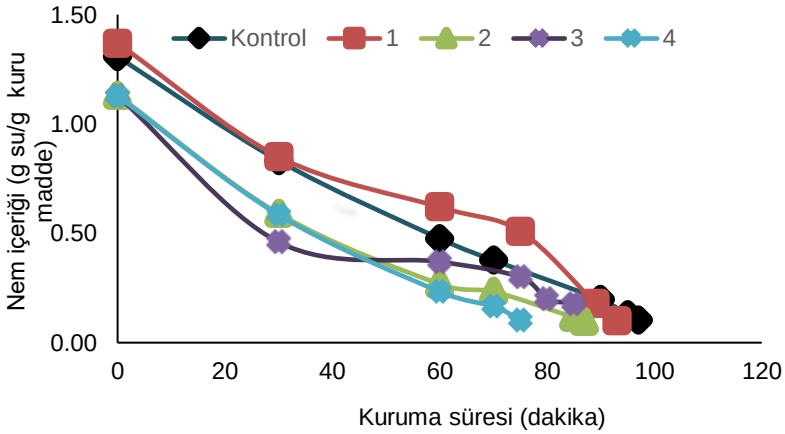
### Kırmızı Pancar Pestilinin Kurutma Kinetiği: Nem Değişimi

Farklı ön işlemlerle (Tablo 1) elde edilen koyulaştırılmış kırmızı pestil püresi (herle) 70°C'de sıcak hava ile kurutulmuştur. Kurutma işlemi sırasında nem içeriklerinin zamanla değişimi Şekil 1-3'de sunulmuştur.

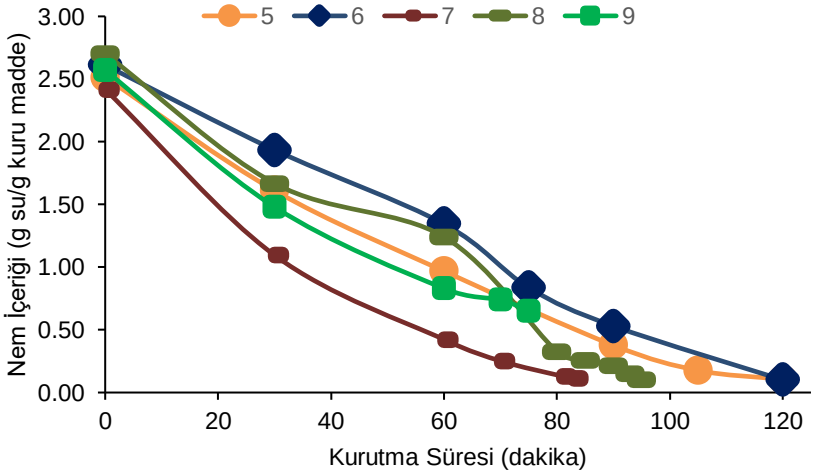
Kurumadde içeriği 40°Bx'e kadar koyulaştırılan ve başlangıç nem içeriği 1.13-1.37 g su/g KM olan kırmızı pancar pestil herleleri, 0.10 g su/g KM'ye kadar kurutulmuştur (Şekil 1). Kurutma süreci sonunda, istenen nem oranına ulaşabilmek için 75-95 dakika arasında bir kurutma süresi gerekmiştir. Benzer şekilde, 20°Bx'e kadar koyulaştırılan ve başlangıç nem içeriği 2.42-2.72 g su/g KM olan kırmızı pancar herlelerinden 75-120 dakika arasında değişen bir kurutma süresi sonrası pestil üretilmiştir (Şekil 2). Çalışma kapsamında, 45 dakika termosonikasyon (10. Koşul) ve 10 dakika mikrodalga (11. Koşul) kombinasyonuyla gerçekleştirilen haşlama ön işlemlerinin ardından, koyulaştırma işlemi uygulanmadan herle elde edilmiştir ve başlangıç nem içeriği 2.84-3.92 g su/g KM olan bu herleler, hedef nem oranı olan 0.10 g su/g KM'ye kadar kurutulmuştur. Gerçekleştirilen kurutma süresi, Şekil 3'te görüldüğü üzere 75-120 dakika aralığında değişmiştir.

Herlenin kurutma öncesi başlangıç suda çözünür kuru madde oranından bağımsız olarak, en fazla kurutma süresine (120 dakika) 5., 6. ve 11. koşullarında ulaşılmıştır. En az kurutma süresi (75 dakika) ise, haşlama işleminin "45 dakika termosonikasyon ve 10 dakika mikrodalga" kombinasyonu ile desteklendiği ön işlem koşullarında (4., 9. ve 11. Koşul) elde edilmiştir. Açık kazanda gerçekleştirilen ön haşlama işlemine alternatif olarak uygulanan termosonikasyon işleminde, kurutma süresinin, artan termosonikasyon süresi ile %4-10 oranında azaldığı gözlemlenmiştir. Termosonikasyon işlemi sonrası haşlama işlemine eklenen 10 dakikalık mikrodalga işlemi ise kurutma süresini %12-22 oranında azaltmıştır. Sonuç olarak, farklı ön işlem uygulamalarının ardından, aynı sıcaklıkta ve koşullarda konveksiyonel yöntemlerle kurutulan kırmızı pancar pestillerinin kuruma sürelerine ön işlemlerin etkisi olduğu gözlemlenmiştir.

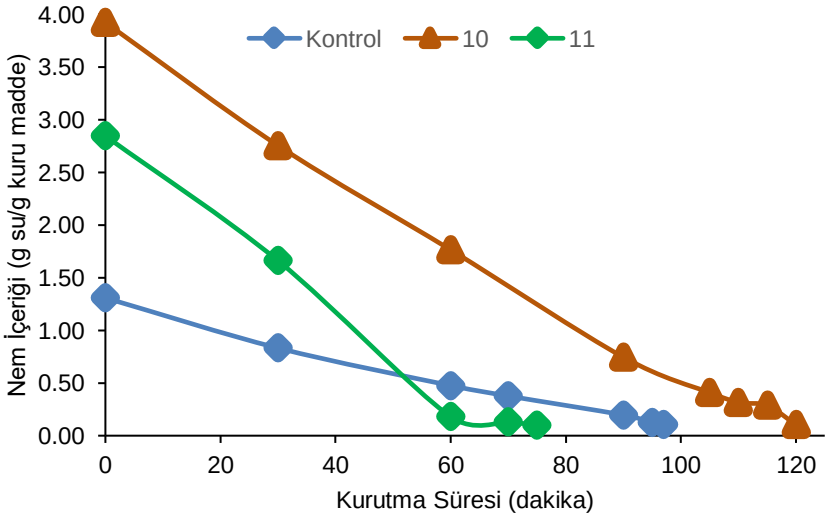
Literatürde yapılan bir çalışmada, havuçlu kırmızıbiber pestil karışımı açık kazanda 40°Bx'e kadar koyulaştırılmış ve akabinde 60 ve 70°C'de sıcak hava kurutma işlemiyle 0.07 g su/g KM'e kadar kurutulmuştur. Kurutma işlemi sırasıyla 175 ve 110 dakikada tamamlanmıştır [13]. Benzer koşullarda koyulaştırma işlemi geçiren muşmula (42° Bx) ve dut pestilinin (45°Bx) 70°C'de sıcak hava kabin kurutucuda kurutulması için kurutma süresi sırasıyla 115 dakika ve 100 dakika olarak raporlanmıştır [35, 36]. Bu çalışma kapsamında "Kontrol" (Tablo 1) koşulunda üretilen kırmızı pancar pestili kurutma süresinin, literatürde raporlanan bu sonuçlarla uyumlu olduğu gözlenmiştir.




Şekil 1. Kurutmalar sırasında pestillerin nem oranı değişimi (Kontrol, 1-4. Koşul).
*Figure 1. Moisture content change of pestils during drying (Control, Condition 1-4).*




Şekil 2. Kurutmalar sırasında pestillerin nem oranı değişimi (5-9. Koşul)
*Figure 2. Moisture content change of pestils during drying (Condition 5-9)*



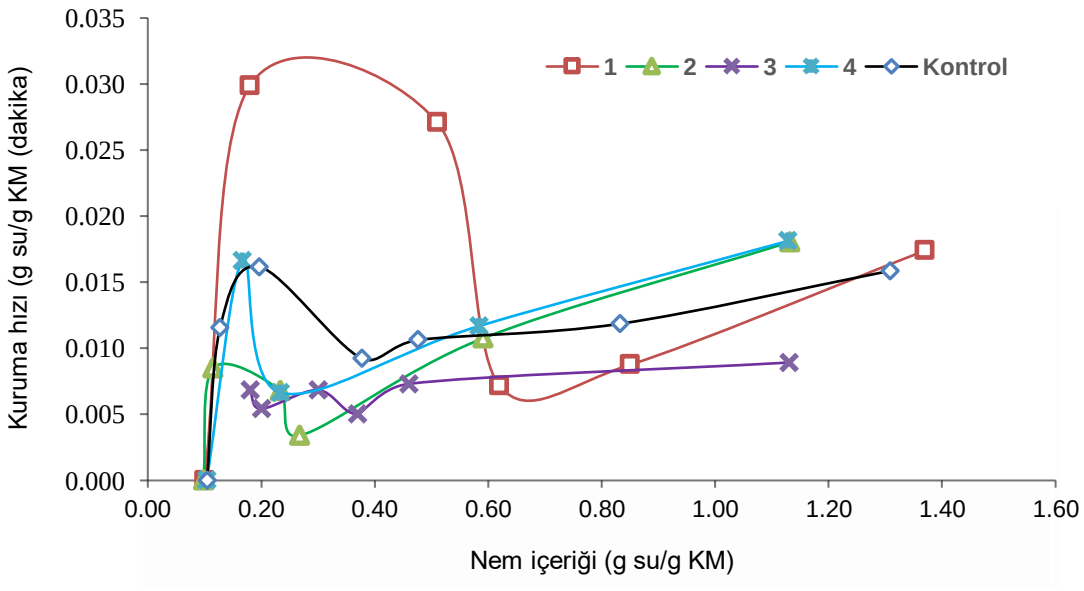

Şekil 3. Kurutmalar sırasında pestillerin nem oranı değişimi (10-11. Koşul).
*Figure 3. Moisture content change of pestils during drying (Condition 10-11).*

Literatürde yapılan çalışmalarda pestil üretiminde kurutma öncesi ön işlem olarak haşlama işlemi yaygın olarak kullanılmaktadır [13, 49]. Ancak, haşlama işlemine alternatif olarak veya destekleyen nitelikte ultrasonikasyon/termosonikasyon ve mikrodalga uygulamalarına ait çalışmalara rastlanmamıştır. Diğer taraftan, bu tekniklerin ön işlem olarak meyve ve sebzelerin kurutulması sırasında kurutma süresini azaltmak ve nihai ürünün kalitesini iyileştirmek amacıyla kullanıldığı raporlanmıştır. Yapılan bir çalışmada, ultrason banyosunda 10, 20 ve 30 dakika süreyle 35 kHz ultrason gücü ile ön işlem görmüş elma küpleri konveksiyon yöntemi kullanılarak 70°C'de ve 1.5 m/s hava hızında kurutulmuştur ve çalışmanın sonucunda 30 dk ultrason banyo uygulamasının, işlem görmemiş örneklere kıyasla kuruma süresini %31 oranında azalmasına neden olduğu raporlanmıştır [50]. Kavun dilimlerinin kurutulması için yapılan bir çalışmada, 25 kHz frekansda 10, 20 ve 30 dakika sürelerinde uygulanan ultrason ön işlemi sonrasında kurutma işlemi 50, 60 ve 70°C sıcaklıklarda gerçekleştirilmiştir. Çalışmanın sonucunda, 20 ve 30 dakika boyunca uygulanan ultrason ön işleminin kuruma süresini sırasıyla %25 (50°C) ve %40 (70°C) oranında azalttığı belirtilmiştir [51]. Benzer şekilde, yer elmasının kurutulması için yapılan bir çalışmada, kurutma öncesi 5-15 dakika arasında uygulanan ultrasonikasyon ön işlemlerinin kurutma süresini azalttığı raporlanmıştır [52].

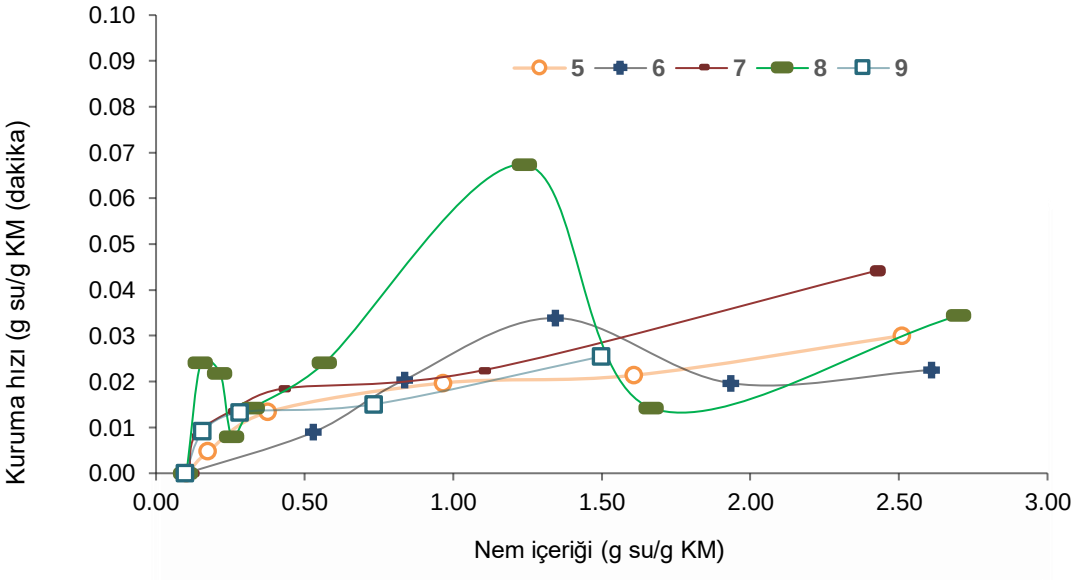
Tatlı patatesin 80°C sıcak hava ile kurutulması öncesi uygulanan mikrodalga ön işleminin, geleneksel buharda ve geleneksel sıcak suda haşlama ön işlemlerine göre kurutma süresini azalttığı raporlanmıştır [53]. Ahududuların üzerine yapılan bir çalışmada, ahududunun mikrodalga enerjisi ve ultrason destekli hibrit bir sistemi (100-200 W, 55°C) kullanılarak kurutulmasında, kurutma süresinin mikrodalga kullanımıyla %79, mikrodalga kullanılmadığı koşullara göre ise konveksiyonel kurutmaya göre %59 oranında azaldığı rapor edilmiştir [54]. Benzer şekilde Abbaspour-Gilandeh ve ark. [55] tarafından alıç meyvesinin kurutulması üzerine yapılan bir çalışmada, ultrasonik ön işleminin mikrodalga destekli sıcak hava kurutma işlemininin diğer farklı kurutma tekniklerine (sıcak hava, mikrodalga-sıcak hava, kızılötesi-sıcak hava, dondurarak kurutma, ultrasonik + sıcak hava, ultrasonik + mikrodalga-sıcak hava, ultrasonik + kızılötesi-sıcak hava) göre kurutma süresini kısalttığı raporlanmıştır. Rocha armutunun kurutulması için yapılan bir çalışmada, mikrodalga (4 dakika boyunca 539 W) ve ultrasonikasyon (35 kHz- 160 W gücü ile 30°C sıcaklıkta 10 dakikalık uygulama) ön işlemleri karşılaştırılmış ve mikrodalga ön işlem koşulunun daha kısa kuruma süresi sağladığı tespit edilmiştir [27]. Kurutma süresinde belirgin değişiklik yaratmayan ultrasonik ön işlem örneklerinde ise kalite özelliklerinin daha iyi korunduğu raporlanmıştır. Literatürde yer alan bu sonuçlarla çalışmamız kapsamında elde edilen sonuçların uyumlu olduğu sonucuna varılmıştır.

### Kırmızı Pancar Pestilinin Kurutma Kinetiği: Kuruma Hızı Değişimi

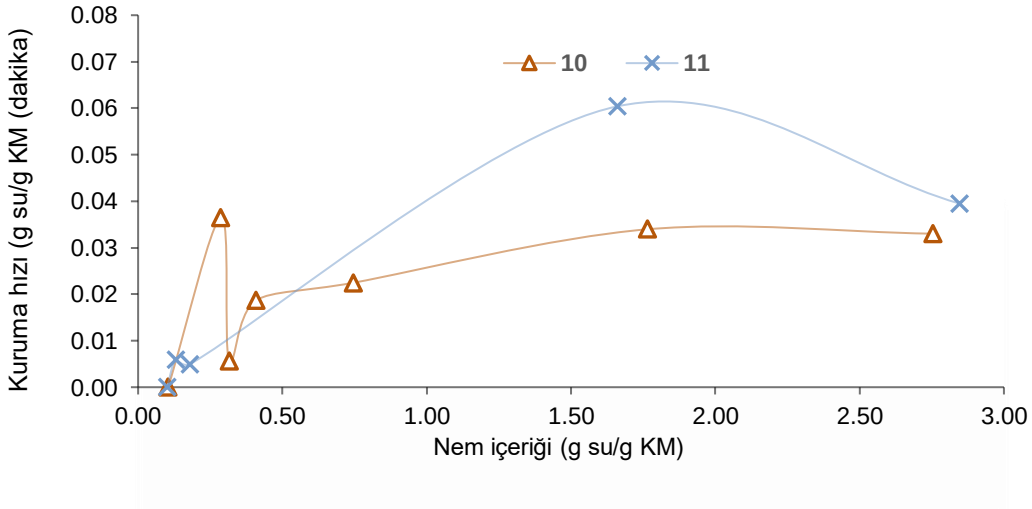
Farklı ön işlem koşullarıyla hazırlanmış herlenin kurutulmasıyla elde edilmiş kırmızı pancar pestil örneklerinin kuruma hızlarını hesaplamak için Eşitlik 2 kullanılmıştır. Kırmızı pancar pestillerinin değişen nem içeriklerine karşı kuruma hızı değişimleri Şekil 4-6'de gösterilmiştir.




Şekil 4. Kurutmalar sırasında pestillerin kuruma hızı değişimi (Kontrol, 1-4.Koşul)
*Figure 4. Drying rate variation of pestils during drying (Control, Conditions 1-4)*




Şekil 5. Kurutmalar sırasında pestillerin kuruma hızı değişimi (5-9.Koşul)
*Figure 5. Drying rate variation of pestils during drying (Conditions 5-9)*




Şekil 6. Kurutmalar sırasında pestillerin kuruma hızı değişimi (10.-11.Koşul)
*Figure 6. Drying rate variation of pestils during drying (10th -11th conditions)*

Şekil 4, 5 ve 6'da görüldüğü üzere gerçekleştirilen termosonikasyon ve termosonikasyon ile kombine edilmiş mikrodalga ön işlem uygulamalarının kuruma hızını önemli ölçüde etkilediği görülmektedir. Başlangıç nem içeriğinin (g su/g KM) diğer koşullara göre yüksek olduğu durumlarda (5.-11. koşullarda) kuruma hızının da başlangıç periyodunda yüksek olduğu görülmektedir. Bu çalışma kapsamında kuruma evresinde genellikle azalan bir kuruma hız periyodu gözlemlenmiştir, yani birim zamanda buharlaşan nem miktarı, bir önceki zamana kıyasla azalmıştır. Kuruma hızının azalmaya başladığı, difüzyonun etkili olduğu dönemlerde oluşan bu hız düşüşleri, yüzeyde oluşan kuru tabakanın altındaki nemli tabakalara büzüşerek baskı yapması ve alt tabakalarda nemin bulunması nedeniyle baskıya direnç göstermesinden kaynaklanabildiği belirtilmektedir. Bu durum, kuruma sonucunda üst tabakada büzüşmenin gerçekleşemediği ve sert kabuk oluşumunun meydana geldiği durumlarda kuruma hızını ani bir şekilde düşürebilmektedir. Kabuk bağlanması, çözünür kuru madde göçüne bağlı olarak da meydana gelebilmektedir [56].

Diğer taraftan bu çalışma kapsamında, sabit oranlı bir kuruma hız periyodunun da belirgin olarak ortaya çıkmadığı da görülmüştür (Şekil 4-6). Isı iletimi ve kütle iletimi hızının eşit olduğu bu periyodun görülmemesi, pestil ürünlerinin üst tabakasında uzun bir süre boyunca sabit su tabakasının bulunmamasından veya nem ölçümlerinin uzun zaman aralıklarında gerçekleştirilmemesinden kaynaklanabildiği düşünülmektedir [57].

Bazı üretim koşullarında (1, 8, 9 ve 10) azalan kuruma hız periyodu sırasında kuruma hızında ani kısa artışlar da gözlenmiştir. En yüksek kuruma hızı, haşlama ön işleminin 45 dakika termosonikasyon ile gerçekleştirildiği ve koyulaştırmanın 20°Bx'e kadar sürdürüldüğü üretimde (8. Koşul), dakikada 0.0067 g su/g kuru madde olarak elde edilmiştir. Literatürde raporlanan ultrason destekli

kurutma çalışmalarında, kuruma hızının genellikle kısa bir süre arttığı belirtilmiştir. Bu dönemin, çoğunlukla ortam ile ürün yüzeyi nemindeki farka bağlı olduğu ve aynı zamanda ön ısıtma dönemi olarak adlandırıldığı belirtilmektedir. Bu ani artış periyodunun ardından daha uzun süre devam eden ve asıl kuruma işleminin gerçekleştiği düşen hız periyodunun gözlemlendiği raporlanmıştır [58-60].

Bozkır ve ark. [61] tarafından Trabzon hurmasının kurutma ve kalite özelliklerine ultrason ve ozmotik dehidrasyon ön işlemlerinin etkisinin araştırıldığı çalışmada, ultrason destekli ozmotik dehidrasyon uygulaması ile kurutulan numunelerin kuruma süresinin önemli ölçüde azaldığı ve kuruma hızının arttığı raporlanmıştır. Yapılan bir araştırmada, sarı manyok örneklerine kurutma işlemi (sıcak havayla konveksiyonel kurutma) öncesi uygulanan ultrasonikasyon işleminin (20 kHz ve 600 W güçte 10 dakika), kuruma süresini kontrol örneklerine göre %35 oranında azalttığı, kuruma hızını da %63'lük arttırdığı belirtilmiştir [58]. Mikrodalga ve ultrason destekli konvektif kurutmanın çilek örneklerinde, ısı ve kütle transferini önemli ölçüde iyileştiren etkileri olduğunu raporlamıştır [62]. Mikrodalganın ısıtma etkisi ve ultrasonun titreşim etkisi ile birlikte hareket eden bu enerji kaynaklarının sinerjistik etkisi, kuruma hızında artışa yol açmıştır.

## İnce Tabaka Matematiksel Modelleri ile Kurutma Davranışının Açıklanması

Farklı ön işlemler sonucu elde edilen kırmızı pancar pestil herlelerinin kuruma davranışına uygun ince tabaka matematiksel modelleri irdelenmiştir. Kurutma modellerine uygun parametreler Tablo 3 ve 4'te sunulmuştur.

Tablo 3. Kuruma modellerine ait sabitler ve modellerin istatistiksel uygunluk parametreleri (Kontrol, Koşul 1-4).
*Table 3. Constants of the drying models and statistical fit parameters of the models (Control, Conditions 1-4).*

| Model No[1] | | | Ön İşlem Koşulları[2] | | | | |
|---|---|---|---|---|---|---|---|
| | | | Kontrol | 1 | 2 | 3 | 4 |
| 1 | Model sabitleri | *k* | 0.02140 | 0.02060 | 0.02490 | 0.01830 | 0.02840 |
| | İstatiksel parametreler | $R^2$ | 0.98360 | 0.93210 | 0.99210 | 0.98340 | 0.99260 |
| | | *RMSE* | 0.01056 | 0.02848 | 0.00917 | 0.17831 | 0.01452 |
| | | $X^2$ | 0.00223 | 0.00982 | 0.00077 | 0.32191 | 0.00172 |
| 2 | Model sabitleri | *n* | 1.43470 | 1.41960 | 1.16690 | 1.34160 | 1.33440 |
| | | *k* | 0.00303 | 0.00310 | 0.01200 | 0.00410 | 0.00690 |
| | İstatiksel parametreler | $R^2$ | 0.94860 | 0.82970 | 0.97330 | 0.97450 | 0.98730 |
| | | *RMSE* | 0.00542 | 0.02017 | 0.00594 | 0.00821 | 0.00440 |
| | | $X^2$ | 0.00063 | 0.00547 | 0.00038 | 0.00078 | 0.00019 |
| 3 | Model sabitleri | *n* | 1.43470 | 1.41960 | 1.16690 | 1.34160 | 1.33440 |
| | | *k* | 0.01755 | 0.01690 | 0.02258 | 0.01650 | 0.02400 |
| | İstatiksel parametreler | $R^2$ | 0.94860 | 0.82970 | 0.97330 | 0.97450 | 0.98730 |
| | | *RMSE* | 0.00542 | 0.02017 | 0.00594 | 0.00821 | 0.00440 |
| | | $X^2$ | 0.00063 | 0.00547 | 0.00038 | 0.00078 | 0.00019 |
| 4 | Model sabitleri | *k* | 0.02140 | 0.02090 | 0.02490 | 0.01830 | 0.02840 |
| | | *a* | 2.14470 | 1.47060 | 1.10010 | 1.52590 | 1.12513 |
| | İstatiksel parametreler | $R^2$ | 0.98360 | 0.91540 | 0.99210 | 0.98340 | 0.99260 |
| | | *RMSE* | 0.07266 | 0.05295 | 0.01419 | 0.07287 | 0.02001 |
| | | $X^2$ | 0.11733 | 0.03770 | 0.00215 | 0.06144 | 0.00392 |
| 5 | Model sabitleri | *k* | 0.03070 | 0.02690 | 0.03460 | 0.02910 | 0.03920 |
| | | *a* | 1.85726 | 1.54130 | 1.11728 | 1.23146 | 1.08112 |
| | | *c* | 0.07972 | 0.07001 | 0.08670 | 0.14173 | 0.09198 |
| | İstatiksel parametreler | $R^2$ | 0.93920 | 0.88750 | 0.95340 | 0.92390 | 0.97060 |
| | | *RMSE* | 0.04899 | 0.06089 | 0.02758 | 0.04433 | 0.02981 |
| | | $X^2$ | 0.05647 | 0.04091 | 0.00974 | 0.02653 | 0.01088 |
| 6 | Model sabitleri | *k* | 0.01356 | 0.01303 | 0.01440 | 0.01057 | 0.01618 |
| | | *a* | 0.57781 | 0.58105 | 0.52380 | 0.73106 | 0.75494 |
| | İstatiksel parametreler | $R^2$ | 0.98360 | 0.93210 | 0.99210 | 0.98340 | 0.99260 |
| | | *RMSE* | 0.04739 | 0.05774 | 0.07691 | 0.06219 | 0.06876 |
| | | $X^2$ | 0.04492 | 0.04483 | 0.06309 | 0.04475 | 0.04633 |
| 7 | Model sabitleri | *b* | 0.00010 | 0.00001 | 0.00010 | 0.00003 | 0.00009 |
| | | *a* | -0.01300 | 0.98738 | -0.01870 | -0.01220 | -0.01880 |
| | İstatiksel parametreler | $R^2$ | 0.93490 | 0.87800 | 0.97390 | 0.88830 | 0.99030 |
| | | RMSE | 0.17720 | 0.06086 | 0.00786 | 0.00838 | 0.00467 |
| | | $X^2$ | 0.69780 | 0.04980 | 0.00066 | 0.00081 | 0.00021 |

[1]Tablo1'de İnce tabaka matematiksel modellerin detayları sunulmuştur. [2]Tablo 2'de ön işlem koşullarının detayları sunulmuştur.
*[1]Details of the thin layer mathematical models are presented in Table 1.[2]Details of the pretreatment conditions are presented in Table 2.*

Tablo 4. Kuruma modellerine ait sabitler ve modellerin istatistiksel uygunluk parametreleri (Koşul 5-11)
*Table 4. Constants of the drying models and statistical fit parameters of the models (Control, Conditions 5-11)*

| Model No[1] | | | Ön İşlem Koşulları[2] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
| 1 | Model sabitleri | *k* | 0.02370 | 0.02030 | 0.03400 | 0.02780 | 0.03200 | 0.02300 | 0.06510 |
| | İstatiksel parametreler | $R^2$ | 0.97770 | 0.96830 | 0.99410 | 0.96180 | 0.99020 | 0.96010 | 0.97900 |
| | | *RMSE* | 0.03524 | 0.02724 | 0.01084 | 0.03696 | 0.01701 | 0.03646 | 0.08232 |
| | | $X^2$ | 0.00894 | 0.00972 | 0.00131 | 0.01383 | 0.00236 | 0.01215 | 0.04879 |
| 2 | Model sabitleri | *n* | 1.43730 | 1.71760 | 1.32260 | 1.67180 | 1.33190 | 1.59820 | 2.04740 |
| | | *k* | 0.00310 | 0.00070 | 0.00840 | 0.00137 | 0.00610 | 0.00138 | 0.00054 |
| | İstatiksel parametreler | $R^2$ | 0.98490 | 0.96950 | 0.98730 | 0.90000 | 0.98430 | 0.97310 | 0.98260 |
| | | *RMSE* | 0.01065 | 0.00913 | 0.00357 | 0.02150 | 0.01782 | 0.01028 | 0.00655 |
| | | $X^2$ | 0.00102 | 0.00120 | 0.00016 | 0.00535 | 0.00209 | 0.00113 | 0.00039 |
| 3 | Model sabitleri | *n* | 1.43730 | 1.71760 | 1.32260 | 1.67180 | 1.33190 | 1.59820 | 2.04740 |
| | | *k* | 0.01790 | 0.01500 | 0.02710 | 0.01939 | 0.02560 | 0.01620 | 0.02520 |
| | İstatiksel parametreler | $R^2$ | 0.98490 | 0.96950 | 0.98730 | 0.90000 | 0.98430 | 0.97310 | 0.98260 |
| | | *RMSE* | 0.01065 | 0.00913 | 0.00357 | 0.02150 | 0.00509 | 0.01028 | 0.00655 |
| | | $X^2$ | 0.00102 | 0.00120 | 0.00016 | 0.00535 | 0.00025 | 0.00113 | 0.00039 |
| 4 | Model sabitleri | *k* | 0.02370 | 0.02190 | 0.03400 | 0.02780 | 0.03200 | 0.02300 | 0.04150 |
| | | *a* | 1.99210 | 1.64560 | 1.21680 | 1.52166 | 1.17304 | 1.47110 | 1.50863 |
| | İstatiksel parametreler | $R^2$ | 0.97770 | 0.96540 | 0.99410 | 0.96180 | 0.99020 | 0.96010 | 0.96870 |
| | | *RMSE* | 0.17710 | 0.05654 | 0.02236 | 0.06286 | 0.02656 | 0.06086 | 0.08948 |
| | | $X^2$ | 0.28229 | 0.04604 | 0.00625 | 0.04572 | 0.00691 | 0.03951 | 0.07205 |
| 5 | Model sabitleri | *k* | 0.02720 | 0.02780 | 0.04620 | 0.33320 | 0.04220 | 0.02530 | 0.05090 |
| | | *a* | 1.29540 | 2.08760 | 1.46961 | 1.66500 | 1.19650 | 1.48914 | 1.62418 |
| | | *c* | 0.04165 | 0.03861 | 0.04712 | 0.03792 | 0.06682 | 0.02652 | 0.03525 |
| | İstatiksel parametreler | $R^2$ | 0.96700 | 0.92540 | 0.95940 | 0.95330 | 0.96860 | 0.97150 | 0.93620 |
| | | *RMSE* | 0.05092 | 0.09636 | 0.05600 | 0.08215 | 0.04084 | 0.06666 | 0.11575 |
| | | $X^2$ | 0.03177 | 0.14857 | 0.04481 | 0.09111 | 0.02044 | 0.05687 | 0.16078 |
| 6 | Model sabitleri | *k* | 0.01510 | 0.01350 | 0.02195 | 0.01734 | 0.02078 | 0.01442 | 0.02592 |
| | | *a* | 0.56970 | 0.62201 | 0.54889 | 0.60344 | 0.53982 | 0.59532 | 0.60138 |
| | İstatiksel parametreler | $R^2$ | 0.97770 | 0.96540 | 0.99410 | 0.96180 | 0.99020 | 0.96010 | 0.96870 |
| | | *RMSE* | 0.06276 | 0.04683 | 0.06017 | 0.05636 | 0.07470 | 0.05448 | 0.06040 |
| | | $X^2$ | 0.03545 | 0.03158 | 0.04525 | 0.03675 | 0.05468 | 0.03166 | 0.03284 |
| 7 | Model sabitleri | *b* | 0.00004 | 0.00008 | 0.00010 | 0.00010 | 0.00010 | 0.00002 | 0.00020 |
| | | *a* | -0.01310 | -0.00900 | -0.02170 | -0.01310 | -0.02000 | -0.01050 | -0.01620 |
| | İstatiksel parametreler | $R^2$ | 0.98070 | 0.90380 | 0.98770 | 0.88790 | 0.99080 | 0.94950 | 0.89320 |
| | | *RMSE* | 0.00786 | 0.22820 | 0.04011 | 0.25862 | 0.00583 | 0.00449 | 0.31303 |
| | | *X2* | 0.00056 | 0.74989 | 0.02011 | 0.77394 | 0.00033 | 0.00021 | 0.88188 |

[1]Tablo1'de İnce tabaka matematiksel modellerin detayları sunulmuştur. [2]Tablo 2'de ön işlem koşullarının detayları sunulmuştur.
*[1]Details of the thin layer mathematical models are presented in Table 1.[2]Details of the pretreatment conditions are presented in Table 2.*

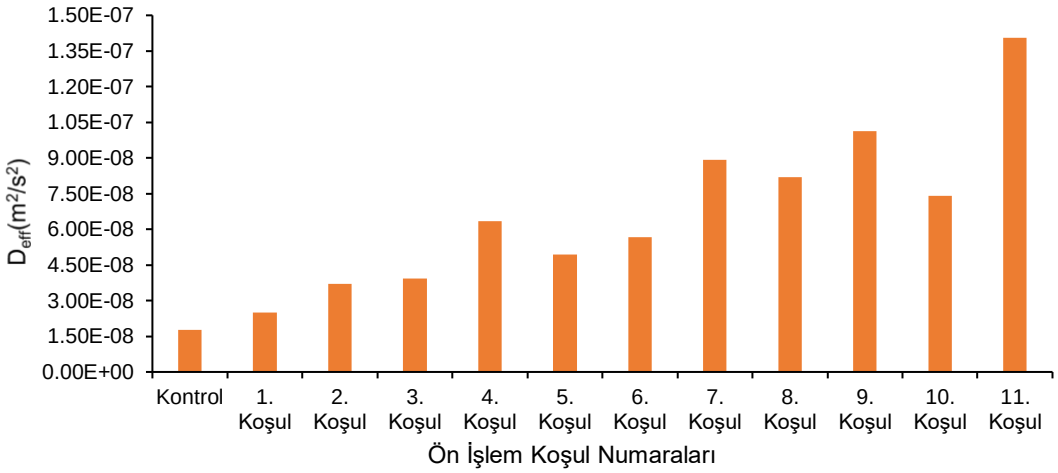
Farklı ön işlemlerden geçirilen yaklaşık 40°Bx'e sahip kırmızı pancar pestillerinin (Kontrol, 1.2, 4. Koşullar) kuruma davranışlarını en iyi açıklayan modeller için en yüksek $R^2$ (0.93210-0.99260) değerini ve en düşük $\chi^2$ (0.00077-0.00982) ve RMSE (0.00917-0.02848) değerlerini veren Lewis, yaklaşık 20°Bx'e sahip kırmızı pancar pestillerinin (5, 6, 10 ve 11) için ise en yüksek $R^2$ (0.9650-0.98490) değerini ve en düşük $\chi^2$ (0.000127 – 0.001108) ve RMSE (0.000655– 0.1065) değerlerini veren Page ve Modifiye Page modelleri olarak belirlenmiştir. Bunların dışında kalan 3. ve 8. Koşulları için en yüksek $R^2$ değerini (0.96180-0.9910) ve en düşük $\chi^2$ (0.03675-0.06309) ve RMSE (0.05636-0.07691) değerlerini Two Term modeli, 7. ve 8. koşulları için en yüksek $R^2$ değerini (0.99020-0.99410) ve en düşük $\chi^2$ (0.00691-0.00625) ve RMSE (0.02236-0.026561) değerlerini Henderson & Pabis modeli sağlamıştır. Elde edilen bu sonuçlar, kırmızı pancar pestili kuruma davranışını açıklayan tek bir matematiksel model olmadığı ve uygulanan ön işlemlere de bağlı olarak farklı modeller ile açıklanabileceği sonucuna varılmıştır.
Mikrodalga ve konvansiyonel kurutma tekniklerini de içeren farklı tekniklerle kurutulan dut ve muşmula pestilinin kurutma kinetiği Page ve Modified Page modeli ile açıklanmıştır [34, 35]. Son yıllarda yapılan güncel bir çalışmada da ısı pompası ile kurutulan kızılcık-kapya pestilinin kuruma davranışını en iyi bu iki modelin açıkladığı raporlanmıştır [63].

Termosonikasyon ön işlemine tabi tutulan pestilin kurutulmasıyla ilgili ince tabaka modellerine uyum literatürde sıklıkla çalışılmasa da, sebze ve meyvelerin kurutulması öncesinde ultrasonikasyon uygulanmasıyla ilgili çalışmalar mevcuttur ve bu alandaki çalışmaların sayısı artmaktadır. Ananas dilimlerinin 40 kHz'de 20 ve 30 dakika ultrasonik banyo ön işlem sonrası 70°C'de sıcak hava ile kurutulmasında, Logaritmik model en yüksek $R^2$ (0.99602 ve 0.99692) en düşük RMSE (0.0156 ve 0.0138) değerleri ile uyum gösteren model olarak belirlenmiştir [64]. Patatesin ultrasonik (20 kHz) ön muamelesi sonrası 60°C'de sıcak hava ile kurutulmasında Modifiye Page ve Henderson & Pabis modelinin en iyi uyumu gösterdiği raporlanmıştır [65]. Tatlı muz kabuğu dilimlerinin kuruma hızlarını ve kalite parametrelerini arttırmak için ultrasonikasyon ve karbonasyon-ultrasonikasyon destekli konvektif kurutma tekniklerinin değerlendirilmesi üzerine yapılan bir araştırmada, Wang and Singh modeli deneysel verilere en iyi şekilde uyan model olarak tanımlanmıştır [66].

Ultrason ön işleminin (0, 20 ve 40 dakika) ve mikrodalga gücünün (120, 150 ve 180 W) olduğu 60°C'de sıcak hava ile birleştirilmiş mikrodalga uygulaması ile kurutulan domates dilimleri çalışmasında Page modeli ($R^2$>0.99) deneysel verilere uyum gösteren en iyi model olarak belirlenmiştir [67].

## Efektif Nem Difüzyon Katsayısı ($D_{eff}$) Değerleri

Değerlendirilen $D_{eff}$ değerleri, Şekil 7'de grafiksel olarak sunulmuştur. Bu değerler, Eşitlik 7 kullanılarak hesaplanmış olup, $8.91\times10^{-8}$ – $1.01\times10^{-7}$ ($m^2/s$) aralığında yer almaktadır.




Şekil 7. Farklı ön işlemler uygulanarak elde edilen kırmızı pancar pestil ürünlerinin $D_{eff}$ değerleri
*Figure 7. $D_{eff}$ values of red beet pestil products obtained by applying different pretreatments*

Kurutma öncesi kırmızı pancar pestili herlesinin sahip olduğu başlangıç suda çözünür kuru madde miktarından bağımsız olarak, herlenin elde edilmesinde uygulanan 30 dk termosonikasyon ön işlemi (1. ve 6. Koşul), sadece açık kazanda haşlama ön işleminin uygulandığı Kontrol ve 5. Koşullara göre $D_{eff}$ değerini $1.77\times10^{-8}$ $m^2/s$'den $2.49\times10^{-8}$ $m^2/s$'e ve $4.94\times10^{-8}$ $m^2/s$'den $5.67\times10^{-8}$ $m^2/s$'ye artırmıştır. Aynı etki 45 dakika olarak uygulanan termosonikasyon (3. ve 7. Koşul) işleminde de görülmüştür. Termosonikasyon işlemi ile hibrit bir şekilde uygulanan mikrodalga işleminin de $D_{eff}$ değerini artırdığı gözlenmiştir. Termosonikasyon uygulaması, kırmızı pancar pestil örneklerinde, mekanik ve termal uyarımlarla oluşan mikroskobik kanalların artmasını sağlamıştır ve bunlarda hücre yapısının zayıflamasına yol açmıştır. Bu durum, sıcak hava ile kurutma sırasında nem transferine karşı iç direncin azalmasına ve yüzeyde oluşan sıcaklık artışının etkisiyle kütle transferinin hızlanmasına neden olmuştur. Mikrodalga ön işlemi de kütle transfer hızını artırarak kuruma süresinde zaman tasarrufu sağlamış ve tüm mikrodalgasız ön işlemlere göre daha etkili bir performans sergilediği gözlenmiştir. Sonuç olarak, Şekil 7'de belirgin bir şekilde görüldüğü üzere, termosonikasyon ve mikrodalga ön işlemleri kırmızı pancar pestil ürünlerinde kütle transferi karekterize eden efektif nem difüzyon katsayısında artışa yol açmıştır.

Bu çalışma kapsamında elde edilen efektif nem difüzyon katsayı aralığı, gıda ürünleri için önerilen sınırlar içindedir. Gözenekli ve katı gıdalarda nem difüzyonu genellikle $3.6\times10^{-10}$ $m^2/s$ ile $3.6\times10^{-5}$ $m^2/s$ arasında değişmiştir [68, 69]. Kırmızı pancar pestilinin kurutulması sırasında uygulanan kurutma koşullarında (70°C'da konvektif sıcak hava ile kurutma) dut pestilinin kurutulmasını inceleyen [35], $D_{eff}$ değerini $8.73\times10^{-8}$ $m^2/s$ olarak, [13] kabak pestilinde $9.61\times10^{-9}$ $m^2/s$, olarak raporlamıştır. Diğer taraftan bu çalışma kapsamında elde edilen sonuçlarla uyumlu olarak literatürde, acı su kabağının sıcak hava ile kurutulması öncesi uygulanan mikrodalga ön işleminin kuruma süresinin azalmasına ve nem yayılımının artmasına yol açtığı [23] ve siyah zeytin kurutulmasında farklı güçlerde uygulanan ultrasonikasyon işleminin $D_{eff}$ değerlerini artırdığı [70] raporlanmıştır.

$D_{eff}$ değeri üzerine kuruma sırasındaki havanın sıcaklığı, gıdanın yapısal özellikleri ve içerdiği nem etkili olduğu literatürde belirtilmiştir. $D_{eff}$ değeri hesaplanması için kullanılan Eşitlik 7, ince tabaka matematiksel modelleri için kullanılan öngörülerle ilişkili olduğu belirtilmektedir ve bu değerin tüm kurutma süresini kapsayacak şekilde hesaplanarak ortaya konulması farklı gıdaların kurutma davranışları ile karşılaştırma yapılmasına olanak sağlamaktadır [20, 71].

## SONUÇLAR

Bu çalışma kapsamında, elma ve tapyoka nişastası gibi bileşenlerin kullanımı ile kırmızı pancar pestilinin formülasyonu oluşturulmuş ve mikrodalga ve termosonikasyon ön işlemlerinin geleneksel haşlama işlemine alternatif olarak kullanım olanakları araştırılmıştır. Farklı ön işlemlerin, kırmızı pancar pestilinin ince tabaka kurutma davranışı üzerine olana etkisi irdelenmiş ve en uygun matematiksel model ortaya konulmuştur. Elde edilen bulgular, termosonikasyon, mikrodalga ve geleneksel haşlama yöntemlerinin kombinasyonlarının pestil üretiminde olumlu etkiler sağladığını ortaya koymuştur. Önerilen alternatif ön işlemlerin kurutma süresini belirgin ölçüde kısalttığı, kuruma hızını artırdığı ve effektif difüzyon katsayısı üzerine etksi üstünde olumlu etkisinin olduğu ortaya

konulmuştur. Özellikle termosonikasyonun hücre yapısını zayıflatarak nem transferini hızlandırdığı ve mikrodalga ön işlemi sıcak hava ile kurutma sürecinde kütle transferini artırarak kuruma süresini önemli ölçüde azaltarak enerji verimliliğini artırmış ve pestilin besin değerini koruma konusunda etkili olmuştur.

Artan tüketici talebi, bu tür inovatif pestil ürünlerinin fonksiyonel gıda pazarında yüksek bir potansiyele sahip olduğunu ortaya koymaktadır. Gelecek çalışmalarda, formülasyonu ve ön işlem koşulları belirlenen kırmızı pancar pestilinin duyusal olarak hedonik tüketici testlerinin yapılması, bu ön işlemlerin ürünün nihai mineral, fenolik kompozisyon, hidrosimetil furfural içeriği gibi özellikleri üzerine olan ektisi irdelenmelidir. Ayrıca pestillerin besin değeri ve duyusal özelliklerini daha da iyileştirmek amacıyla diğer doğal tatlandırıcılar ve kıvam artırıcıların araştırılması önerilmektedir.

## TEŞEKKÜR

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**1.** Çalışmalar A4 boyutunda hazırlanmalı, üstten 2.45 cm, alttan 2.45 cm, sağ ve soldan 1.75 cm boşluk bırakılmalı ve tek kolon olarak hazırlanmalıdır. Metin çift satır aralıklı yazılmalı, paragraflar arasında tek satır boşluk bırakılmalıdır. Metinde bütün satırlar (sürekli) numaralandırılmalıdır.

**2.** Çalışma başlığı 14 punto Arial, koyu, küçük harflerle ve ortalanmış olarak yazılmalıdır. Başlıktan sonra bir satır boşluk bırakılmalı (11 punto); yazar isimleri (yalnızca ilk harfler büyük) 10 punto Arial ve ortalanmış olarak verilmelidir. Yazarların adresleri, telefon ve faks bilgileri ile yazışmalardan sorumlu yazarın e-posta adresi hemen alt satırda 9 punto Arial, ilk harfler büyük olacak şekilde ve ortalanmış olarak yazılmalıdır. Yazarların çalıştıkları kuruluşlar (ve/veya adresler) farklı ise her bir yazar isminin sonuna rakamlarla üst indis konulmalıdır.

**3.** Metin içindeki kısımların başlıkları (ÖZ, ABSTRACT, GİRİŞ vb.) 10 punto Arial ve koyu olarak büyük harflerle yazılmalı, başlıktan sonra bir satır boşluk bırakılarak metine geçilmelidir. Alt başlıklarda ilk harfler büyük, 10 punto Arial ve koyu yazı karakteri kullanılmalıdır. ÖZ'ün altına bir satır boşluk bırakıldıktan sonra en fazla 5 adet Anahtar Kelime konmalıdır. Anahtar Kelimelerden sonra bir satır boşluk bırakılarak İngilizce başlık ve altına ABSTRACT ve Keywords yazılmalıdır. Bir satır boşluk bırakılarak ana metine geçilmelidir.

**4.** Ana metin 9.5 punto Arial olarak hazırlanmalıdır.

**5.** Çalışma başlıca şu kısımlardan oluşmalıdır: Başlık, Yazar İsimleri, Adresleri, İletişim Bilgileri, Yazışmalardan Sorumlu Yazarın E-posta adresi, Öz, Abstract, Ana Metin (Giriş, Materyal ve Metot, Bulgular ve Tartışma, Sonuç), Teşekkür (gerekiyorsa), Kısaltmalar (gerekiyorsa), Kaynaklar.

**6.** Öz ve Abstract 250 kelimeyi geçmemeli, çalışmanın amacını, metodunu ve önemli sonuçlarını içermelidir. Öz tek paragraf olarak yazılmalı ve öz içinde kaynaklara atıf yapılmamalıdır.

**7.** Çalışma içerisinde geçen mikroorganizma isimleri ile Latince ifade ve isimler italik olarak yazılmalı ve kısaltmalarda uluslararası yazım kuralları göz önünde bulundurulmalıdır.

**8.** Tablo başlıkları tablonun üstüne, şekil başlıkları ise şeklin altına yazılmalı ve numaralandırılmalıdır. Kullanılan tablo ve şekillere metin içinde mutlaka atıf yapılmalıdır. Metin içinde geçen veriler tablo ve şekillerin tekrarı olmamalıdır. Tablo ve şekillerin başlıkları içerikleriyle uyumlu ve anlaşılabilir olmalıdır. Şekiller ve resimlerin yüksek çözünürlükte olmasına dikkat edilmelidir. Resimler (ve gerekiyorsa Şekiller) *.jpg formatında metin içerisinde yer almalıdır.

**9.** Metin içerisinde atıflar köşeli parantez içerisinde rakamlarla yapılmalı [1] ve Kaynaklar bölümünde bu numara sırasıyla detayları yazılmalıdır. Kaynakların numaralandırılması MS Word Numaralandırma Kitaplığı kullanılarak yapılmalıdır.

**10.** Kullanılan matematiksel denklemler numaralandırılmalı ve metin içerisinde bu denklemlere atıf yapılmalıdır.

**11.** Kaynaklar kısmı APA yazım stili kullanılarak hazırlanmalıdır. Kaynakların yazımında aşağıdaki örnek yazım biçimleri kullanılmalı ve makalelerin yayınlandığı dergi isimleri kısaltma kullanılmadan ve italik olarak yazılmalıdır. Web adreslerine atıf yapılacağında (mümkün olduğunca Resmi web sayfalarına atıf yapılmalıdır) mutlaka ilgili web adresine erişim tarihi verilmelidir.

**Makale**

[1] Bozkurt, H., İçier, F. (2009). İnegöl köfte üretiminde ohmik pişirmenin uygulanabilirliğinin incelenmesi. *Akademik Gıda,* 9(1), 6-12.

**Kitap**

[2] Kılıç, S. (2001). Süt Endüstrisinde Laktik Asit Bakterileri. Ege Üniversitesi Ziraat Fakültesi Yayınları, Ege Üniversitesi Matbaası, Bornova, İzmir.

**Kitap Bölümü**

[3] Gibson, G.R., Saavedra, J.M., MacFarlane, S., MacFarlane, G.T. (1997). Probiotics and Intestinal Infections. In Probiotics 2: Applications and Practical Aspects, Edited by R. Fuller, Chapman & Hall, 2-6 Boundary Row, London SE1 8HN, England, 212p.

**Kongre-Sempozyum Bildirisi**

[4] Gürsoy, O., Akdemir, O., Hepbaşlı, A., Kınık, Ö. (2004). Recent situation of energy consumption in Turkey dairy industry. *International Dairy Symposium: Recent Developments in Dairy Science and Technology*, May 24-28, 2004, Isparta, Turkey, Book of Proceedings, 10-16p.

**12.** Hakem görüşleri doğrultusunda düzeltilmek üzere yazar(lar)a gönderilen çalışmaların gerekli düzeltmeleri yapılarak yayın ofisine ulaştırılması gereklidir. Editörler tarafından belirtilen süre zarfında gönderilmeyen çalışmalar "ilk defa gönderilmiş çalışma" olarak değerlendirilecektir.

**13.** Yukarıdaki kurallara uygun olarak hazırlanmamış çalışmalar değerlendirmeye alınmaz.

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# Guidelines to Authors

Akademik Gıda® (Academic Food Journal) is a peer reviewed journal where original research and review articles are published in the field of food science and technology. Research notes, mini-reviews, opinions and letters to the editor are also considered for publication. The journal is published trimonthly and each volume is composed of 4 issues per year. Journal articles are published either in Turkish or English. Manuscripts in either good American or British English usage are accepted, but not a mixture of these.

Manuscripts for the Akademik Gıda® (Academic Food Journal) must be sent via the electronic article submission system, which can be located in the official website of the journal, www.academicfoodjournal.com. Manuscripts sent by e-mail are not considered for evaluation. For questions related to the electronic article submission system, contact the editor via e-mail at ogursoy@yahoo.com.

- Authors must specify the type of the manuscript (research articles, review articles, research briefs, mini-review articles, comments and letters to the editor).
- Authors should provide at least 3 potential referees and their contact information (mailing address, e-mail address and phone number).
- Manuscripts to be submitted should be free from any spelling or grammatical error.
- Authors must guarantee that the submitted manuscript is not published anywhere previously and will not be submitted to anywhere before the editorial board makes a final decision on the manuscript.
- The editorial board is authorized to make necessary changes in manuscripts accepted for publication.

**Peer review policy**

Manuscripts pass through initial screening in the editorial office followed by internal review by Editors. After the first evaluation, manuscripts are double-blind-reviewed by a peer review system involving at least two independent reviewers to ensure high quality of manuscripts accepted for publication. The Editors have the right to decline formal review of a manuscript if it is (i) on a topic outside the scope of the Journal, (ii) lacking technical merit, (iii) fragmentary and providing marginally incremental results or (iv) poorly written.

**Publication fee**

There is a 1000 TL submission fee for Turkish manuscripts submitted after February 1, 2024. This fee may not be waived. 750 TL fee is charged for articles submitted to the journal in English.

**Ethics Statement**

Detailed information about journal publication policy, evaluation of manuscripts and ethical issues can be found in the Ethics Statement section.

**Preparation of a manuscript**

**1.** Manuscripts should be prepared in A4 size, and the text must be prepared in a single column format. The text must be double-spaced, and a single space should be left between paragraphs. All lines and pages must be continuously numbered.

**2.** The title must be 14pt Arial, bold, small letters and centered. A blank line should be left after the title, and the names of authors should be given in 10pt Arial and centered. In addition to each author's contact address, the phone and fax numbers and e-mail address of the corresponding author should be provided. If the institutions of the authors are different, superscript numbers should be used to indicate their addresses.

**3.** The headings (e.g. Abstract, Introduction, Materials and Methods etc.) must be 10pt Arial, and should be typed in bold capital letters. Each heading should appear on its own separate line. A blank line should be left after each heading. A list of keywords, a maximum of 5, should be provided below the abstract section of the manuscript.

**4.** The main text should be prepared in 9.5pt Arial.

**5.** Typical articles mainly consist of the following divisions: Title, Author Names, Addresses, Contact Information, Corresponding author's e-mail address, Abstract, Main text (Introduction, Materials and Methods, Results and Discussion, Conclusions), Acknowledgements (if necessary), Abbreviations (if necessary) and References.

**6.** The abstract should not exceed 250 words, and the main purpose and method and the most significant result and conclusion should be presented in the abstract. The abstract should be prepared as a single paragraph, and should not include any citation.

**7.** Latin names in the text should be in italics, and names and abbreviations should follow international rules. If abbreviations that are not standard are unavoidable, they must be defined at their first mention in the text. Consistency of abbreviations throughout the article must be ensured. Internationally accepted rules and conventions must be followed, and the international

system of units (SI) must be used. If other units are mentioned, their equivalents in SI must be provided.

**8.** Table headings should be on the top of each table and figure captions below each figure. Each table or figure must be numbered consecutively in accordance with their appearance in the text. All figures and tables should be cited in the text. The data presented in the tables and figures should not be repeated in the text. Table headings and figure captions should be self-explanatory. Figures and pictures must be provided in high resolution, and pictures (and, if necessary figures) should be included in the text as *. jpg format.

**9.** References in the text should be cited in numbers in square brackets [1] and details of the citations must be provided in the Literature or References section with their respective numbers.

**10.** Mathematical equations should be numbered and cited in the text.

**11.** References should be given according to the APA manual of style. The following formats should be used for the details of cited references, and the journal names must be typed in italics. References to the Web addresses (if necessary, the official web pages should be preferred) must include full web address and the date of access.

**Article**

[1] Güzeler, N., Kaçar, A., Say, D. (2011). Effect of milk powder, maltodextrin and polydextrose use on physical and sensory properties of low calorie ice cream during storage. *Akademik Gıda,* 9(2), 6-12.

**Book**

[2] Kilic, S. (2001). Lactic Acid Bacteria in Dairy Industry. Ege University Faculty of Agriculture Publications, Ege University Press, Bornova, Izmir, Turkey.

**Book Chapter**

[3] Gibson, G.R., Saavedra, J.M., MacFarlane, S., MacFarlane, G.T. (1997). Probiotics and Intestinal Infections. In Probiotics 2: Applications and Practical Aspects, Edited by R. Fuller, Chapman & Hall, 2-6 Boundary Row, London, England, 212p.

**Proceedings of the Congress-Symposium**

[4] Gursoy, O., Akdemir, O., Hepbasli, A., Kinik, O. (2004). Recent situation of energy consumption in dairy industry in Turkey. *International Dairy Symposium: Recent Developments in Dairy Science and Technology*, May 24-28, 2004, Isparta, Turkey, Book of Proceedings, 10-16p.

**12.** A list of the corrections requested by the referees must be provided by the authors, and it must be sent to the editorial office.

**13.** Studies that are not prepared in accordance with the rules above will not be considered for evaluation.

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# Etik Beyanı

Akademik GIDA®, gıda bilimi ve teknolojisi alanında orijinal araştırma ve derleme makalelerinin yayınlandığı hakemli bir dergidir. Dergi üç ayda bir Sidas Medya Ltd. Şti. (Çankaya, İzmir, Türkiye) tarafından yayınlanmaktadır. Derginin genel bilimsel kalitesini iyileştirmek için yayıncı tarafından aşağıdaki yönergeler belirlenmiştir.

## Yayın Politikası

Akademik Gıda dergisine gönderilen tüm makaleler Dergi Editörleri için Davranış Kuralları ve En İyi Uygulama Kılavuzları ve Dergi Yayıncıları için Davranış Kurallarında (Code of Conduct and Best Practice Guidelines for Journal Editors and Code of Conduct for Journal Publishers) belirtilen Genel Kılavuzlara uygun olarak değerlendirilmektedir. Bilimsel yazılar dergiye gönderilmeden önce derginin Yazım Kurallarının okunmasını önemle tavsiye ederiz. Yazarlar aynı zamanda Avrupa Bilim Editörleri Birliği'nin (EASE) (European Association of Science Editors) İngilizce olarak basılacak makaleler için "Bilimsel Makalelerin Yazarları ve Çevirmenleri İçin Rehber"e uymalıdır. Yazarlar, insan veya hayvan verilerini içeren araştırmaları için Uluslararası Tıp Dergisi Editörleri Komitesinin (International Committee of Medical Journal Editors) önerilerini takip etmelidir.

## Makalelerin Değerlendirilmesi

Dergiye gönderilen tüm makaleler, bilimsel içeriklerinin özgünlüğü ve kalitesi ölçütlerine göre değerlendirilir.

- Dergiye gönderilen tüm yazılar, ilk olarak yayın ofisindeki (teknik ve genel kalite değerlendirilmesi açısından) eleme işleminden geçer ve ardından teknik ve bilimsel editörler tarafından değerlendirilir.
- İlk değerlendirmeden sonra, editörler (i) dergi kapsamı dışında kalan bir konu hakkında hazırlanmış makaleleri (ii) teknik olarak eksik/yetersiz makaleleri, (iii) kısmi ve marjinal artan sonuçları içeren makaleleri veya ( iv) kötü yazılmış makaleleri reddetme hakkına sahiptir.
- İlk inceleme sonucunda makalenin ileri değerlendirme için uygun olduğuna karar verilirse, dergide yayımlanmak üzere kaliteli makalelerin seçimini yapmak amacıyla, makaleler çift-körlü (hakemin ve yazar/yazarların birbirlerini görmedikleri) değerlendirme sistemi ile en az iki bağımsız hakemden oluşan bir değerlendirme sürecinde bilimsel incelemeye alınır.
- Hakemler tarafından talep edilirse, makalenin hakem görüşleri doğrultusunda yazarlar tarafından revize edilmiş versiyonu orijinal hakemler tarafından tekrar değerlendirilir. Değerlendirmelerin ardından editörler hakem önerileri doğrultusunda makale hakkındaki nihai kararlarını verirler. Gerekirse editörler, hakemlerin istedikleri tüm şartların yerine getirilmesi için yazarlardan ilave revizyon isteyebilir.
- Kabul edilen makalelerin son versiyonu, yayın öncesi taslağın (galley proof) hazırlanması için teknik editörlere gönderilir. Yazarlardan, makalelerinin dizgisi hazırlanmış taslaklarını son kontrol için yayın öncesinde incelemeleri istenir.
- Tüm makaleler, nihai formlarında DOI numarası almış ve çevrimiçi olarak pdf dosyaları halinde yayımlanır. İlgili veritabanlarında bu şekilde indekslenir.

## Yayın Ücreti

Akademik Gıda dergisinde makalelerin yayınlanması için herhangi bir yayın ücreti talep edilmemektedir.

## Gizlilik

Editörler, Akademik Gıda'ya gönderilen tüm makaleleri tam bir gizlilikle ele alır. Editörler, hakemler haricinde, COPE tavsiyelerine uyulmadığı takdirde, üçüncü şahıslara makale ile ilgili hiçbir bilgi vermezler. Yayınlanmak üzere dergiye gönderilen makaleler hakemler için de gizlidir ve bilimsel değerlendirme için aldıkları makalelerin herhangi bir bölümünü üçüncü şahıslarla paylaşmalarına veya dağıtmalarına izin verilmez. Suiistimal şüphesi olduğunda, hakemlerin derhal gizli bir şekilde yayın ofisine başvurmaları önerilir. Hakemler ayrıca, Dergi Editörleri İçin Davranış Kuralları ve En İyi Uygulama Kuralları ile Dergi Yayıncıları için Davranış Kuralları'nı (Code of Conduct and Best Practice Guidelines for Journal Editors and Code of Conduct for Journal Publishers) takip ederek editöre gizli yorumlarında belirli bir eylem önerebilirler.

Akademik Gıda, çift-kör bir hakem inceleme süreci yürütür, yani çalışmanın eleştirel değerlendirmesini sağlamak için hakemlerin isimleri gizlidir. Hakemlerden, raporlarında adlarını veya irtibat bilgilerini açıklamamaları istenir. Hakem raporları yazarlara gönderilemeden önce bu açıdan kontrol edilir.

## Yazarlık

Bir yazar, bir araştırmanın fikrine veya tasarımına, verilerin elde edilmesine, verilerin analizine veya yorumlanmasına büyük ölçüde katkıda bulunan, makalenin hazırlanmasında, yazılmasında veya gözden geçirilmesinde entelektüel içeriğe eleştirel katkı yapan bireydir. Katkıda bulunanlar diğer kişiler makalenin Teşekkür bölümünde belirtilmelidir ve çalışmanın yazarı olarak kabul edilemez. Tüm yazarların doğru ve tam isimleri ile ORCID kimlikleri dergiye gönderilen

makalenin başlık sayfasında yer almalıdır. Yazarların isimlerinin yanında çalıştıkları kurumlar ve yazışmalardan sorumlu yazarın geçerli bir adresi verilmelidir. Yazışmalardan sorumlu yazarın telefon ve faks numaraları ile e-posta adresi makalenin ilk sayfasında belirtilmelidir. Tüm yazarlar, gönderilen makalenin daha önce herhangi bir yerde yayınlanmadığını ve makale hakkında Akademik Gıda dergisi nihai bir karar vermeden önce makaleyi başka bir dergiye göndermeyeceklerini garanti etmelidir.

**Destekleyen/Finans Sağlayan Kuruluşlar**

Araştırmanın tüm finans kaynaklarına ilişkin detaylar, Teşekkür bölümünde belirtilmelidir. Yazarlar, resmi finansman kurum/larının tam isimlerini ve proje/hibe numaralarını belirtmelidir.

**Yazarlarda Değişiklik**

Makalenin Akademik Gıda'ya sunulmasından sonra yazar isimlerinde değişiklik ancak revizyon sırasında gerekli olan ek çalışmalar durumunda olabilir. Makalenin yayına kabul edilmesinden sonra herhangi bir değişikliğe izin verilmez. Yazarlıktaki değişiklik, hakem görüşlerine verilen cevaplar sırasında yazışmalarda belirtilmeli ve tüm yazarlar tarafından kabul edilmelidir. Yazışmalardan sorumlu yazar, yazarların sırası da dahil olmak üzere makalenin revize edilmiş versiyonundaki değişikliklerden sorumludur.

**Çalışma Verilerinde Düzeltme**

Yayınlanan verilerin doğruluğundan tüm yazarlar sorumlu olmalıdır. Verilerin düzeltilmesi için, yazışmalardan sorumlu yazardan yayın öncesi taslağı (galley proof) incelemesi ve makalenin yayınlanmasından 4 gün önce dikkatlice düzeltmesi istenir.

**Makalenin Geri Çekilmesi**

Bir makalenin geri çekilmesi, gönderim veya yayın hatalarını düzeltmek için kullanılır. Yazarlar makaleyi geri çekebilir ve bu durumda Yayın Etiği Komitesi (COPE) Geri Çekme Kurallarına [(COPE) retraction guidelines] uymalıdır. Tekrarlanan veya benzerlik oranı yüksek bir yayın, verilerin hileli kullanımı, intihal veya etik dışı araştırma yapılması durumunda, makale editör tarafından geri çekilecek ve geri çekilen makale linklerine bağlantı korunacak ancak elektronik veri tabanına (makale sayfasına) bir geri çekme bildirimi eklenecektir.

**Etik Hususlar**

***Çıkar çatışması:***

- Yazar/lar başvuru sırasında herhangi bir çıkar çatışması varsa beyan etmelidir. Yazar/ların başvuru sırasında bilimsel değerlendirme için en az üç potansiyel hakem önermeleri istenir. Önerilen hakemler çalışma arkadaşları, ortak çalıştıkları kişiler veya çalıştıkları kurumların üyeleri olamazlar.
- Hakemler makaleyi değerlendirmelerini önleyen herhangi bir çıkar çatışması olması durumunda Editörleri bilgilendirmesi ve bu konuda COPE kurallarına uyması tavsiye edilmektedir.
- Editörler Kurulu üyeleri veya kurul üyelerinin ortak çalıştıkları kişiler tarafından dergiye gönderilen makaleler için, değerlendirme sırasındaki önyargıları en aza indirgemek amacıyla, değerlendirme süreci ilgili kurul üyelerini dışarıda tutacak şekilde değiştirilerek uygulanır.
- Düzeltmeler (revizyonlar) sırasında, editörler Dergi Editörleri İçin Davranış Kuralları ile En İyi Uygulama Kılavuzu ve Dergi Yayıncıları İçin Davranış Kurallarını (Code of Conduct and Best Practice Guidelines for Journal Editors and Code of Conduct for Journal Publishers) takip ederler.

***İnsan denekleri, hayvan veya bitki içeren araştırmalar***

- Araştırmanın insan denekleri veya hayvanları içermesi durumunda, yazarların Uluslararası Tıp Dergisi Editörleri Komitesinin (the International Committee of Medical Journal Editors) yönergelerini izlemeleri önerilir.
- İnsan denekleri içeren çalışmalarda, deneklerin çalışmaya katılmak için imzaladıkları onamlar yazarlar tarafından sağlanmalıdır. 18 yaşın altındaki deneklerin çalışmaya katılmaları için ebeveyn veya velileri tarafından izin verilmelidir.
- Test edilen tüm denekler için, makalenin, ilgili kurallara ve/veya uygun izinlere veya lisanslara uyumunu gösteren belgelerin sunulması gerekir.
- Hayvanlar üzerinde yapılacak her türlü araştırma kurumsal, ulusal veya uluslararası kurallara uygun olmalı ve etik kurul tarafından onaylanmalıdır.
- Bitki materyallerinin toplanması dahil, bitkiler üzerinde yapılan deneysel araştırmalar, kurumsal, ulusal veya uluslararası kurallara uygun olmalıdır.
- Saha çalışmaları yerel mevzuata uygun olarak yapılmalı ve uygun izinleri ve/veya lisansları belirten bir açıklama makalede yer almalıdır.

***Yayın suistimali***

- Akademik Gıda dergisi, Dergi Editörleri İçin Davranış Kuralları ile En İyi Uygulama Kılavuzları ve Dergi Yayıncıları İçin Davranış Kurallarını (Code of Conduct and Best Practice Guidelines for Journal Editors and Code of Conduct for Journal Publishers) takip eder.
- Makalenin aynı anda birden fazla dergiye gönderilmesi, intihal, yayınlanmış makalenin yeniden yayınlanması, etik kuralların ihlali vb. şüpheli bir suiistimal durumunda, araştırmacılar, hakemler veya okuyucular Yayın Ofisi (ogursoy@yahoo.com) ile iletişime geçmeye teşvik edilir.
- Makaledeki benzerlik oranı tek bir kaynaktan %10'dan fazla olmamak üzere en fazla %25 ile sınırlandırılmıştır. Bu koşula uymayan makaleler reddedilir. Bu şartların ihlal edilmesi durumunda, COPE (COPE recommendations) tavsiyeleri izlenecek ve ilgili tüm taraflara bildirilecektir.

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# Ethics and Publication Malpractice Statement

Akademik GIDA® is a peer-reviewed journal where original research and review articles are published quarterly by Sidas Media Agency Advertisement Consultation Ltd. (Cankaya, Izmir, Turkey) in the field of food science and technology. In order to improve the overall scientific quality of the journal, following guidelines have been established by the publisher.

## Editorial Policy

General Guidelines stated in the Code of Conduct and Best Practice Guidelines for Journal Editors and Code of Conduct for Journal Publishers are followed by all papers submitted to Academic GIDA. Prior to submission, authors are highly recommended to read the Journal's Instructions to Authors. Authors should also follow the European Association of Science Editors (EASE) Guidelines for Authors and Translators of Scientific Articles to be Published in English. For any research involving human or animal data, the recommendations of the International Committee of Medical Journal Editors should be followed by the authors of the manuscripts.

## Peer Review

All contributions are evaluated according to the criteria of originality and quality of their scientific content.

- All manuscripts pass through an initial screening process (technical and overall quality evaluation) in the editorial office followed by an internal review by the technical and scientific editors.
- After the first evaluation, editors have the right to decline formal review of a manuscript if it is (i) on a topic outside the scope of the Journal, (ii) lacking technical merit, (iii) fragmentary and providing marginally incremental results or (iv) poorly written.
- If the manuscript is considered suitable for further evaluation, manuscripts are double-blind-reviewed by a peer review system involving at least two independent reviewers to ensure high quality of manuscripts accepted for publication.
- If requested, the revised version is evaluated by the reviewers, and editors make a decision about final acceptance based on their suggestions. If necessary, further revision can be asked for to fulfil all the requirements of the reviewers.
- The final version is then sent to the technical editor in order to produce a galley proof, and the authors receive this proof for final check before publishing.
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