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Determination of the properties of industrial and local butter sold in the Erzurum market

Erzurum piyasasında satışa sunulan endüstriyel ve yerel tereyağının özelliklerinin geleneksel ve modern analiz yöntemleri ile belirlenmesi

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ABSTRACT

Butter, a highly preferred type of fat in the food industry, is susceptible to adulteration due to its high market price. In this study, the characteristics of industrial butter and local butter sold in the market were determined using traditional and modern methods, and the results were compared. The aim was therefore to detect adulterated fats. The traditional methods used for fat analysis were Saponification Number, Iodine Number, and Reichert-Meissl Number. The modern methods used were GC, DSC, and FTIR analyses. The analyses revealed that modern methods provided more accurate results compared to traditional methods. Additionally, it was found that industrial products had less adulteration compared to local products sold without packaging.

Keywords: Butter, GC, FTIR, DSC, Saponification and Iodine Number, Reichert-Meissl Number.

ÖZ

Gıda sektöründe çok tercih edilen bir yağ çeşidi olan tereyağı, yüksek piyasa değeri sebebiyle tağşişe açık bir üründür. Bu çalışmada piyasada satılan endüstriyel tereyağı ve yerel tereyağının özellikleri geleneksel ve modern yöntemler kullanılarak belirlenmiş ve çıkan sonuçlar birbirleriyle kıyaslanmıştır. Böylece katkılı yağların tespit edilmesi amaçlanmıştır. Yağ analizlerde kullanılan geleneksel yöntemler; Sabunlaşma Sayısı, İyod Sayısı, Reichert-Meissl Sayısıdır. Yağ analizlerinde kullanılan modern yöntemler ise; GC, DSC, FTIR analizleridir. Yapılan analizler sonucunda modern yöntemlerin geleneksel yöntemlere göre daha kesin sonuçlar verdiği görülmüştür. Ayrıca endüstriyel ürünlerin ambalajsız olarak satılan yerel ürünlere göre daha az katkılı olduğu tespit edilmiştir.

Anahtar Kelimeler: Tereyağı, GC, FTIR, DSC, Sabunlaşma ve İyot Sayısı, Reichert-Meissll Sayısı.

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Introduction

Butter, a type of fat rich in milk fat, is a highly nutritious fat that melts at body temperature, is easily digestible, and is also rich in vitamin A and/or β-carotene and essential fatty acids (Metin, 2001; Gosewade et al., 2017; Karagözlü & Yılmaz, 2020). In addition to a high amount of milk fat and water, butter also contains phospholipids, sterols and sterol esters, hydrocarbons, free fatty acids, complex glyceride oils, fat-soluble vitamins, and minerals. Butter has a higher economic value compared to other fats. For this reason, it is highly susceptible to adulteration. The source of adulteration in butter is typically margarine (Çakmakçı et al., 2020).

Margarines contain significantly higher amounts of trans fats compared to butter. While butter contains 3-8% trans fats, margarine contains approximately 10-35% trans fats. Trans fatty acids, which are not naturally occurring, are typically produced through the hydrogenation of vegetable oils and are highly dangerous to human health. For this reason, detecting adulterated fats is crucial both for health and economic reasons. Therefore, alongside traditional methods such as the Saponification Number, Iodine Number, and Reichert-Meissl (RM) number, modern methods like GC, FTIR, and DSC are also used.

There are many studies in the literature aimed at identifying adulteration during the production of butter. In one study, researchers determined the melting and crystallization curves of beef body fat (BBF) and margarine using differential scanning calorimetry (DSC), by gradually cooling from 70 °C to -40 °C. Afterward, they added 5%, 10%, and 20% margarine and BBF to the butter. When BBF or margarine was added to the butter, they observed an increase in the areas of the first and second peaks in the crystallization curves, with the second peak becoming more prominent. Based on these results, the researchers suggested that the DSC technique could be used to detect adulteration in butter (Aktaş & Kaya, 2001).

In another study, researchers tested several modern and traditional methods to identify adulteration in butter. They examined three samples: pure butter, pure margarine, and a 50-50 butter-margarine blend (BM). They analyzed the chemical and physical properties of these samples using traditional methods such as the iodine number, saponification number, and Reichert-Meissell (RM) number, as well as modern methods like DSC, GC, and FTIR. The results showed that both methods could be used to detect adulteration in butter, but modern methods were faster and more reliable (Naktiyok & Doğan, 2021).

In another study, researchers attempted to separate butter from beef fat (BF) by using chemometric techniques combined with Fourier Transform Infrared (FTIR) spectroscopy. Spectral bands associated with butter, BF, and their mixtures were interpreted by correlating them with bands that spectroscopically represent pure butter and BF. The results of the study demonstrated that FTIR spectroscopy, along with multivariate calibration, can be used to detect and quantify BF in butter formulations for authentication purposes (Nurrulhidayah et al., 2013).

In another study, the aim was to determine the β-sitosterol content in butter and cheese samples sourced from the Izmir market. Sterol, fat, and dry matter analyses were performed on 25 butter and 25 cheese samples sold in the İzmir market (Karagözlü & Yılmaz, 2020).

In the present study, the characteristics of industrial butter (IB) and local butter (LB) sold in the Erzurum market were determined using traditional and modern methods, and the results were compared. The aim was to detect adulterated fats.

Methods

Materials

LB and IB used in the study were obtained from a market in Erzurum.

Methods

Traditional Methods

Two important characteristic values used for identifying the type, checking the purity, and classifying fats are the saponification number and the iodine number. The saponification number is the amount of KOH (in mg) required to saponify 1 g of fat. It is commonly used to determine the purity of liquid/solid fats and fatty acids. A saponification number in the range of 210-233 indicates that the butter is pure (Li et al., 1999; Odoom & Edusei, 2015; Xu et al., 2018).

The iodine number indicates the degree of saturation and unsaturation of the fat. It shows the amount of iodine required to saturate the double bonds in 100 g of fat. The number of double bonds in the fat can be determined using the iodine number. The more double bonds the fat contains, the more sensitive it is to oxidation. Pure butter is expected to have an iodine number in the range of 20.4-34.6 (Knothe, 2002; El-Nabawy et al., 2023).

One of the traditional methods, the Reichert-Meissl (RM) number, is an important parameter used to determine whether different oils have been added to butter for adulteration purposes and to check its purity. The Reichert-Meissl (RM) number is a simple, fast, and inexpensive method used to determine the water-insoluble fatty acids that evaporate with steam in 5 grams of fat. After the butter is saponified, small molecular fatty acids are distilled with steam, and the distillate is titrated with an alkaline solution. An RM number in the range of 17-35 indicates that the butter is pure (Naktiyok & Doğan, 2021).

Modern Methods

Gas chromatography (GC) is an analytical method used to determine the chemical composition and quantity of components in a sample consisting of a mixture. Typically, these chemical components are organic molecules or gases. GC-MS is commonly employed for the identification of these components (Mallia et al., 2008; Szabóová et al., 2018; Yaminifar et al., 2021). The image of the GC device used in the analyses is shown in Figure 1 (Agilent-6850-Us104022005).

Figure 1.
GC device used in the analyses.

The Differential Scanning Calorimeter (DSC), used for the thermal analysis of materials, allows for the easy analysis and comparison of the water and fatty acid compositions of butter and margarine. DSC is a precise, fast, and reproducible method that measures the amount of energy absorbed or released by a sample during heating or cooling. By comparing the DSC curves of edible fats in both pure and mixed forms, valuable information can be obtained.

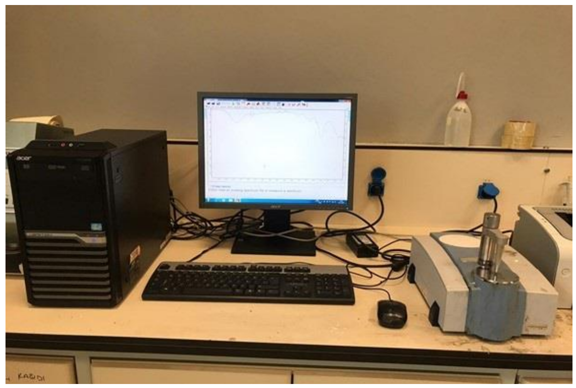
It is well known that fats do not have a specific melting point but rather a melting temperature range. This method, which identifies the correct melting range among multiple endothermic peaks, is widely used in the literature (Aktaş & Kaya, 2001; Tomaszewska-Gras et al., 2013; Dahimi et al., 2014; Dolatowska-Zebrowska et al., 2019). Figure 2 shows the DSC sample preparation apparatus and the DSC device used in the analyses (Perkin Elmer-Diamond DSC-536NG101703).

Figure 2.
a) *DSC sample preparation apparatus* ***b)*** *DSC device used in the analyses.*

(a) (b)

FTIR spectroscopy is used for the qualitative analysis and characterization of fats, proteins, and carbohydrates in various food samples. Particularly, FTIR analysis, along with certain techniques, is commonly applied to distinguish butter from other edible fats and to determine the acidity, fat content, and moisture of butter for quality control. There are numerous examples of this in the literature (Didar, 2022; Nilchian et al., 2022; Akram et al., 2023; Ariza-Ortega et al., 2023). The FTIR device used in the analyses is shown in Figure 3 (Bruker-Alpha-103011).

Figure 3.
FTIR device used in the analyses.



Results and Discussion

Gas Chromatography (GC) Analyses

The results of GC-FID analyses for, IB and LB are presented in Table 1.

Table 1.

Fatty acid compositions(%) for IB and LB as determined by GC-FID analysis.

Fatty Acids	IB (%)	LB (%)
C4:0 (Butyric)	2.12	1.70
C6:0 (Caproic)	1.54	1.09
C8:0 (Caprylic)	1.00	0.60
C12:0 (Lauric)	3.06	1.78
C14:0 (Myristic)	11.23	9.19
C15:0 (Pentadecanoic)	1.31	2.19
C16:0 (Palmitic)	33.31	29.39
C16:1 (Palmitoleic)	1.56	1.51
C18:0 (Stearic)	11.03	12.64
C18:1 11t (Vaccenic)	1.75	1.94
C18:1 9c (Oleic+Elaidic)	24.42	28.60
C18:2 9c12c (Linoelaidic)	4.20	3.79

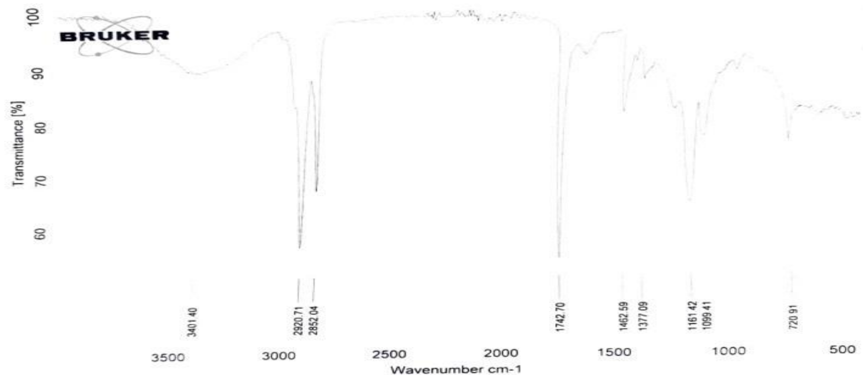
IB: Industrial Butter, LB: Local Butter

The results show that IB contains 85% milk fat based on calculations from butyric acid, while LB contains 68% milk fat. The remaining composition is believed to be due to adulteration. The level of adulteration in LB was found to be higher. It was determined that the adulteration is due to the addition of a fat containing C18:1 oleic acid.

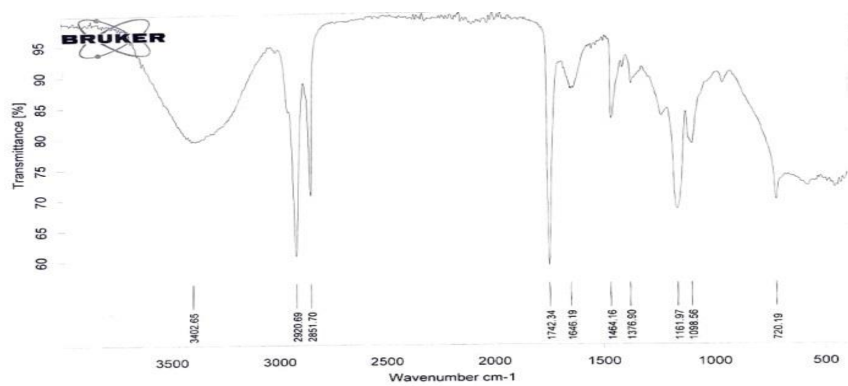
Fourier Transform Infrared (FTIR) Spectroscopy Analysis

Figure 4.

*FTIR spectra of the samples **(a)** FTIR spectrum of IB **(b)** FTIR spectrum of LB.*



(a)



(b)

The structural properties of the samples (IB and LB) were analyzed using FTIR spectroscopy. The FTIR spectra of the samples are shown in Figure 4, and the wavenumbers of the characteristic groups are presented in Table 2. The FTIR analysis revealed the presence of O-H, (-CH(CH_2)), C=O, C=N, -C-H, C-O ester, and -HC=CH- bands in both IB and LB. According to the results in the table, the FTIR analysis showed that both IB and LB exhibited nearly identical peak bands, although the protein content in LB was found to be lower.

Table 2.

Wavenumbers of characteristic groups in the samples.

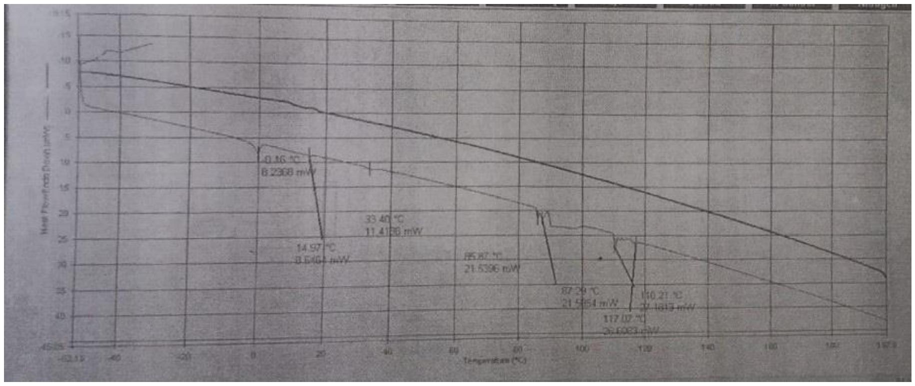
Wavenumber (cm^{-1})	Assignment	IB	LB
3600-3000	O-H water streching	3401	3402
2920	-CH(CH_2) Asymmetric streching (fat)	2920	2920
2850	-CH(CH_2) Symmetric streching (fat)	2852	2851
1740	-C=O(ester), COO-, COOH streching (fat)	1742	1742
1690-1640	C=N streching (protein)	1690-1650	1646
1465	-C-H(CH_2 and CH_3) fat	1462	1464
1377	-C-H(CH_3) symmetric streching	1377	1376
1237	Streching vibration of C-O ester groups (fat)	1230	1230
1170	Streching vibration of -C-O ester groups (fat)	1161	1161
1097	-C-O streching (fat)	1099	1098
967	-HC=CH- streching (trans groups, fat)	995	993
724	-HC=CH- (cis) streching (fat)	720	720

IB: Industrial Butter, LB: Local Butter

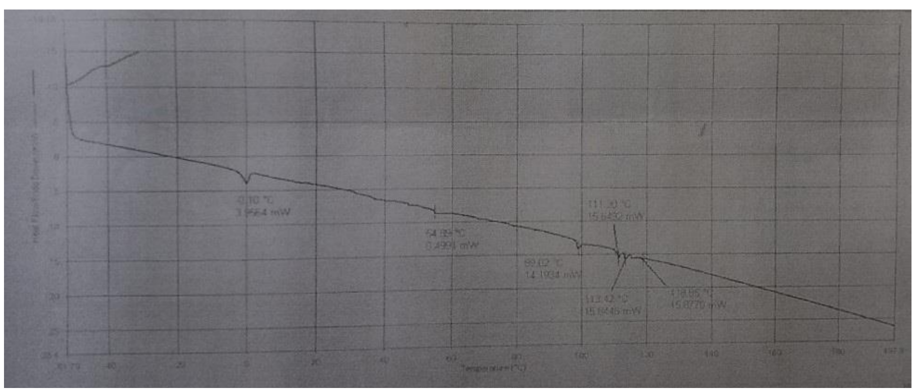
Differential Scanning Calorimetry (DSC) Analysis

Figure 5.

*DSC melting/crystallization cycles **(a)** DSC cycles of IB **(b)** DSC cycles of LB.*



(a)



(b)

The DSC device used for measurement operates within a temperature range of -65 °C to 200 °C, with nitrogen gas used as the protective atmosphere. The crystallization and melting points of the fat samples were determined. The DSC melting/crystallization cycles obtained for İB and LB are shown in Figure 5.

The DSC analysis revealed melting peaks of fatty acids in IB at temperatures of -0.16 °C, 14.97 °C, 33.40 °C, 85.87 °C, 87.29 °C, 110.21 °C, and 117.07 °C. The melting peaks of fatty acids in LB were observed at -0.10 °C, 54.89 °C, 99.02 °C, 111.30 °C, 113.42 °C, and 118.85 °C. When comparing the DSC cycles, it was observed that the areas of the first and second peaks in the DSC cycle of LB (Figure b) were larger. This suggests the possibility of adulteration in the LB.

Results of Analyses Using Traditional Methods

The analyses of IB and LB were completed using traditional fat analysis methods. The results are presented in Table 3.

Table 3.

Saponification number, iodine number, and free fatty acid analysis results of IB and LB.

Traditional Fat Analysis	IB	LB

Saponification Number	213	215
Iodine Number (g)	28.76	25.38
Free Fatty Acid (%Oleic acid)	1.196	1.7

IB: Industrial Butter, LB: Local Butter

As seen in Table 3, the free acidity of both IB and LB was found to be suitable for edible butter. Similarly, the saponification and iodine number values were also found to be within the range suitable for butter.

The results in the table show that the saponification number in LB was higher than that in IB, indicating a slightly higher amount of foreign substances that have undergone saponification. The lower iodine number suggests a lower degree of unsaturation. The high free fatty acid content in LB suggests the possibility of adulteration.

Conclusions

When comparing modern and traditional methods, it was found that modern methods provided more detailed, clearer, and faster results than traditional methods. Furthermore, due to the reduced occurrence of human errors, more accurate results were obtained. Based on the GC analysis data, it was determined that IB contains 85% milk fat based on butyric acid calculations, while LB contains 68% milk fat. IB was found to contain a higher percentage of milk fat compared to handmade LB. This indicates that IB, produced using machines in large facilities and sold in packaged form, is more reliable than handmade LB sold without packaging, with less adulteration found in packaged products.

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