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Original scientific paper

Detection of milk fat in dairy products — an alternative approach

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A B S T R A C T

Milk fat is a highly valuable product, which is why accurate determination of its content in milk and milk products is very important. The use of the GC-FID method in our study proved to be very precise, as in the case of other authors, which signifies the importance of using this method to quantify milk fat. A total of 51 samples of dairy products were analyzed for fatty acid composition with particular attention to butyric acid. Butyric acid contents were in the range from 3.4 ± 0.73 in yogurt to 4.60 ± 0.08 in butter. Milk fat was in the range from 98.5 ± 4.77 in yogurt to 115.0 ± 1.73 in butter. Our results were in accordance with those of many other authors. Development of butyric acid and milk fat analyses in dairy products by GC-FID is essential for laboratories that must conduct analyses for food production, quality control during production, and inspection tasks for the import and export of these food products.

1. Introduction

Milk and milk products have great importance in human nutrition, especially for children and the elderly, due to the products' unique composition of macro and micronutrients. Essential nutrients such as high-value proteins, easily digestible lipids, sugars, minerals and vitamins, especially vitamins A, D, K, vitamins B2, B6, B12 (*Dons et al*., 2023), are all components that make milk and milk products very healthy for consumption. From a health perspective, the importance of regular consumption of milk products is reflected in the improvement of digestion and the health status of the gastrointestinal tract, reduction in the risk of cardiovascular diseases and of the occurrence of cancer, and prevention of type 2 diabetes (*Hasegawa and Bolling*, 2023). The milk product industries have a leading role in the production of functional food, as these products strengthen the immune system, kill pathogenic microorganisms and lower blood pressure (*Korhonen*, 2006). Also, milk and its products that are available on the market do not require additional preparation before consumption, but are ready for use, which adds to the popularity of these products. According to FAO data, world production of milk and milk products has reached 927,800 million tons with a tendency of further growth (*FAO*, 2022).

Milk products include: products of sour-milk fermentation (yogurt, sour milk, acidophilic milk, kefir), milk drinks (chocolate milk, white coffee, milk cocktails with fruit juices etc.), condensed milk (condensed and evaporated), milk powder, butter, cheeses, cream, sour cream, ice cream etc. Milk fat

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plays a very important role in producing the aforementioned foods. In human nutrition, milk fat is the most complex of all fats, having a wide range of lipid types with different molecular configurations, fatty acid chain lengths, and degrees of saturation (*Rico and Razzaghi*, 2023). This chemical composition of milk fat has important roles from an organoleptic point of view in milk products, realizing an adequate texture and enabling an attractive taste for consumers. However, the human population is increasing, which increases the consumption of food and, therefore, the consumption of milk products.

As a consequence, milk and milk product industries strive to improve production methods, in order to increase their capacities, and at the same time maintain their product quality (*Genis et al*., 2021). However, some misusage of new methods is most often reflected in the partial or complete replacement of milk fat with vegetable oils, which are significantly cheaper than milk fat on the market. Mostly, palm oil, soybean oil and sunflower oil are used (*Kim et al*., 2015). The misuse of these products as counterfeit milk and milk products, has expected economic importance, apropos profit, so it also has great ethical significance, because these fraudulent foods are not declared, and they can cause specific health problems for consumers who are subjected to this deception (*Gutiérez et al.*, 2008). According to the literature, milk and milk products are among the leading categories of foods that are counterfeited (*Johnson*, 2014).

The aim of the present study is to analyse butyric acid and milk fat contents in various dairy products and to compare the butyric acid content with experimentally determined milk fat content according to the Roese-Gottlieb method.

2. Materials and methods

Samples — A total of 51 samples of dairy products were analysed for fatty acid composition with particular attention to butyric acid. Dairy samples were „kačkavalj", a type of Serbian cheese, cream, sour cream, butter, cream cheese, yogurt, sour milk, kefir, semi-hard cheese and fresh cheese. The analysed samples are sold on the Serbian market.

2.1. Fatty acid analysis by gas chromatography

The total fat content was determined according to Roese-Gottlieb method (AOAC,1995). Fatty acids methyl esters (FAMEs) were analysed with a gas-liquid chromatograph (GLC, Shimadzu 2010, Japan) combined with flame ionization detector and capillary HP-88 column (length 100 m, i.d. 0.25 mm, film thickness 0.20 µm). Injector and detector temperatures were maintained at 250°C and 280°C, respectively. Nitrogen was used as the carrier gas at flow rate of 1.87 mL min^{-1}. The injector split ratio was set at 1:50. Injector syringe volume was 10 µL and injection volume was 1 µL. The oven with column was programmed: temperature starting at 50°C and ending at 230°C was applied. Total analysis time was 63.12 min. The chromatographic peaks in the samples were identified by comparing FAME peaks with peaks in Supelco 37 Component FAME mix standard (Supelco, Bellefonte, PA).

2.2. Statistical analysis

All values are expressed as mean ± standard deviation. The statistical analysis was performed with software XLSTAT2023 (trial version for Microsoft Excel, Addinsoft, NY, USA). Prior to statistical analyses, all data sets were grouped according to similar production methods.

3. Results and discussion

After the tests carried out on the samples grouped in Table 1, the butyric acid contents ranged from 3.94 ± 0.73 to 4.60 ± 0.08 respectively. The results show that the percentage C4:0 in the milk products was satisfactory in all samples, which was confirmed by the obtained calculated results of milk fat, which ranged from 98.5 ± 4.77 to 115.0 ± 1.73. The importance of using this method, which determines the percentage of butyric acid in milk products, through which we calculated the percentage of milk fat, is becoming increasingly important.

This study showed the capability of GC-FID to analyse butyric acid and milk fat in dairy products with reliable results. According to the Regulation (EC) No 900/2008 (2008), milk fat content is calculated based on butyric acid content.

Comparison of the butyric acid content against Roese-Gottlieb fat for the industrially-produced milk products are presented in Figure 1.

In Figure 1, it can be seen that analysed dairy products were grouped according to their milk fat content vs. butyric acid content in corresponding milk fat. Other authors stated that analytical errors of ± 10% should be allowed for when calculating butyric acid (C4:0) content (*Molkentin and Precht*, 2000). Our results for milk fat content and butyr-

Table 1. Butyric acid content (% of total fat), milk fat (calculated based on butyric acid) and milk fat measured by the Roese-Gottlieb method

	Butyric acid (C4:0) (% of fat)	Milk fat (calculated based on butyric acid)	Milk fat by Roese-Gottlieb method (%)
Kačkavalj (n=6)	4.38 ± 0.81	109.5 ± 4.16	26.08 ± 0.14
Cream (n=3)	4.47 ± 0.19	111.7 ± 4.61	45.50 ± 0.41
Sour cream (n=3)	4.21 ± 0.35	105.2 ± 4.72	23.15 ± 0.17
Butter (n=3)	4.60 ± 0.08	115.0 ± 1.73	82.50 ± 0.18
Cream cheese (n=3)	4.29 ± 0.50	107.2 ± 4.31	15.75 ± 0.16
Yogurt, sour milk, kefir (n=8)	3.94 ± 0.73	98.5 ± 4.77	2.80 ± 0.33
Cheese — hard, semi-hard, fresh (n=25)	4.47 ± 0.33	111.7 ± 6.09	26.36 ± 2.95

ic acid content were in accordance with the studies by *Molkentin and Precht* (2000) (3.43–3.59%) and *Molkentin and Precht* (1998) (3.06–3.42%). The butyric acid and milk fat contents for butter were in accordance with published results obtained by *Danudol and Judprasong* (2022). They reported butyric acid 3.6% and milk fat 100.02% in butter. The butyric acid content in bovine milk was consistent with the data of *Mänsson* (2008) (4.4%), *Salamon et al.* (2006) (2.8–3.6%) and *Adamska et al.* (2014) (3.79–4.2%), but which can depend on the season. In a report by *Sacchi et al.* (2018), the GC/FID method was compared with ^{13}C NMR spectroscopy for analysis of butyric acid to evaluate the content of milk fat. They concluded that both methods can be used successfully, even though they stated that the ^{13}C NMR method has a lower detection level. Therefore, those authors concluded that correlation between the two independent methods was excellent. They reported butyric acid in the range from 1.91 to 8.44% for cow dairy products from the literature and milk fat contents ranging between about 22 and 26%. High variability in the composition of milk fat even from cows fed the same diet has been previously reported (*Bobe et al.*, 2003).

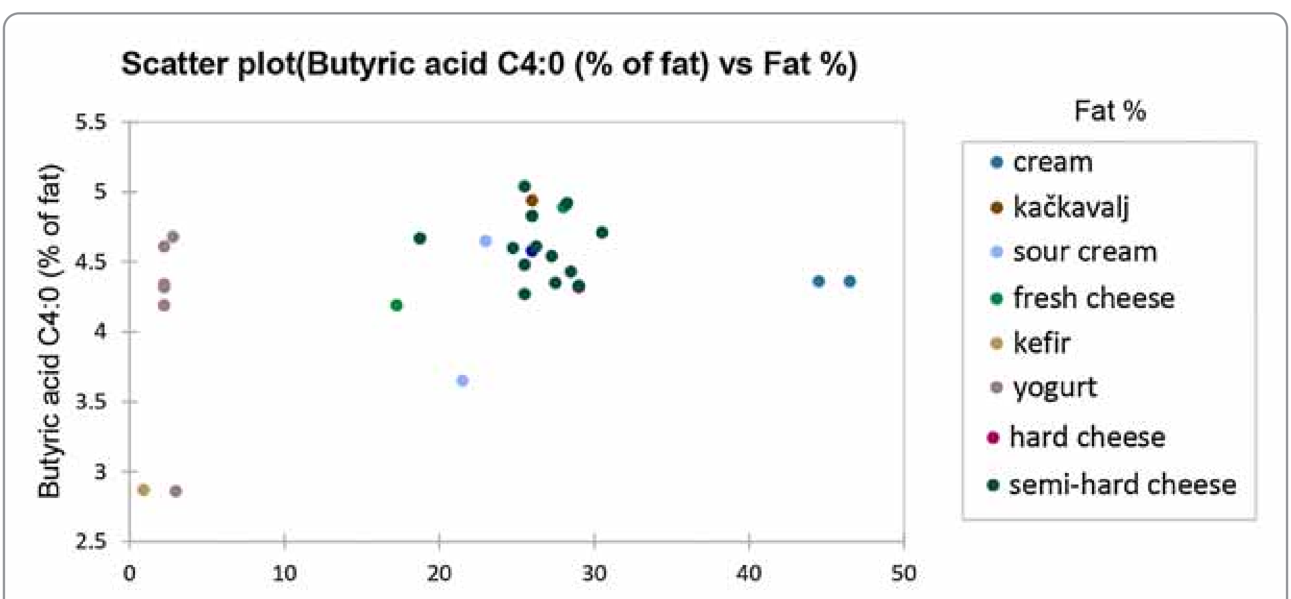

Figure 1. Butyric acid content in different dairy products (g/100 g fat) on the Serbian market

4. Conclusion

This study revealed that determination of the milk fat based on butyric acid content in extracted fat from dairy products gave significant information on quality, and indirect, on authenticity of product. Milk fat is a highly valuable nutrient, so the exact determination of its content in milk and milk products is very important. Development of butyric acid and milk fat analysis in dairy products by GC-FID is essential for laboratories that analysed dairy, and in general, food products, considering quality control during production, and inspection tasks for the import and export of these food products.

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