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Suitability of the infrared spectroscopy and the rheological method for distinguishing traditional cheese from industrial Turoš cheese

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Abstract

The aim of this paper was to determine suitability of the infrared spectroscopy and the rheological method for distinguishing traditional cheese from industrial Turoš cheese. Turoš cheese belongs to the group of sour, dried, cone shaped cheeses with added salt and red dry pepper. Cheeses were sampled from 15 family farms and from market (industrial cheeses from five different batches). The rheological parameters of analysed cheeses were in accordance with the chemical composition of the same samples. Infrared spectroscopy of cheeses show good relation with the chemical composition and it has been proved to be a fast and effective method when compared to textural and standard chemical analysis for monitoring the standard procedure of production of sour, dried cheeses such as Turoš cheese. The extensive variability of all the parameters was a result of unbalanced production of Turoš cheese among family farms. Industrial production of Turoš cheese demonstrates more uniformity in relation to traditional on-farm cheese production.

Key words: sour dried cheese, chemical composition, texture, infrared spectroscopy, Turoš

Introduction

Turoš cheese belongs to the group of sour, dried, cone shaped cheeses (Fig. 1) with added salt and red dry pepper. It is traditionally produced on family farms in north-western Croatia (Međimurje County) and in Hungary by Pomurje Croats (Zala County) (Kerecsényi, 1983; Andrić et al., 2003). Cheeses of similar properties are produced in nearby Croatian counties: Prgica cheese in the Podravina region, Suhi cheese in the Moslavina and Posavina regions (Andrić et al., 2003), and Kvargl in the Bjelovar and Bilogora region (Kirin, 2004). Moreover, in this group of cheeses the Slovenian Suhi cheese from the Prlekija and Slovenske Gorice regions also could be included (Čuček et al., 2007), as well as Austrian Kugelkäse from the Danube region, and French Boulette d' Avesnes from the Nord-Pas-de-Calais region (Harbutt, 2000).

To achieve the most efficient monitoring of standardised production of Turoš cheese, it is advisable to apply a fast and cost-effective monitoring methods, such as infrared spectroscopy and textural analysis. Since the beginning of the 1960s, texture has started to be used as one of the relevant components in sensor evaluation of food (Costell and Duran, 2002), and the first fundamental rheological researches on four

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Figure 1. Turoš cheese (T2)

kinds of British cheeses were carried out by Davis in 1965 (Holsinger, 1995). Texture also represents a very important indicator of quality since it influences the customer's choice during purchase of cheese (Antoniou et al., 2000).

More and more studies suggest the vibrational spectroscopic analysis as suitable tool for discovering and establishing the characteristics and composition of dairy products (Karoui et al., 2010; Dufour, 2011). The advantages of that kind of analysis are speed, simplicity, accuracy and the possibility of processing more parameters at the same time. The standard physicochemical analyses of cheeses are often expensive and require time, and with the use of spectroscopic analysis accurate results in a short period of time can be obtained (Karoui et al., 2006). Spectroscopic analyses of cheeses are predominantly used for the determination of protein, fat and total solids composition. Moreover, the methodology is frequently used for determination of geographical origin of traditional cheeses, as was demonstrated for Emmentaler (Pillonel et al., 2002; Kocaoglu-Vurma, 2009), Mozzarella (Brescia et al., 2003), and Pecorino cheeses (Lerma-Garcia et al., 2010). However, sour, dried cheeses were not taken into consideration by this method. Therefore, the aim of this paper was to determine suitability of the infrared spectroscopy and the rheological method for distinguishing traditional cheese from industrial Turoš cheese.

Material and methods

Cheese manufacture on family farms

Turoš cheese was manufactured in October and November of 2012. Cheese was produced from raw cow's milk and without adding starter cultures on 15 family farms. Milk was left to sour for two days in a 2.5 to 5 litre jar (traditionally it was produced in a 2-3 litre clay pot) in a warm place (about 27 °C). After milk acidification (<4.6 pH) sour cream from the surface of the jar was removed by spoon. Skimmed sour milk was then poured into a pot placed on a moderate heat source (42 °C) for 3 hours (without stirring), until the whey was removed. The cheese curd was poured into a cheese cloth and left to drain for 24 hours, followed by the addition of salt and red pepper. According to Tišlarić (1992), 20 g of salt and 10 g of red pepper were added on 1000 g of fresh cheese. The cheese mixture was shaped by hand into cones and left to dry at room temperature (between 19 and 22 °C and relative air humidity of 55%) for 7 days.

Samples

Step 1

Cheeses were sampled from 15 family farms, and one sample of cheese per family farm was acquired for standard physical and chemical analysis.

All farms were situated in north-western Croatia, in Međimurje County.

Step 2

From 15 family farms included in the test, five representative family farms were chosen: Three family farms whose cheeses were, according to the chemical composition, the closest to the mean values (T1, T2 and T3) and two family farms with extreme values (T4 and T5). Five Turoš cheeses from the same batch were sampled from each of those five family farms (25 cheeses in total). Two were used for standard physical and chemical analysis, as well as for spectroscopy analysis, and three for textural testing.

In order to distinguish industrially from traditionally produced Turoš cheeses, 25 industrially produced cheeses were purchased from market at the same time that cheese was manufactured on family farms; five different batches (5 different dates of production), each with five samples.

Physicochemical analyses

Physicochemical analysis included determination of the fat content according to the Van Gulik method (ISO, 2009), the protein content according to the Kjeldahl method (ISO, 2003), the total solids according to the reference method (ISO, 2008) and the salt content according to Mohr (AOAC, 935.43). The pH was measured by a pH-meter (Mettler Toledo, Seven Multi, according to manufacturer's instructions). The measurements were performed by the Reference laboratory of Dairy Science Department at the Faculty of Agriculture of the University of Zagreb. Two samples per batch were analysed. All measurements were done in duplicate.

Textural analysis

To determine the texture parameters of cheeses, a texture analyser (model TA Plus, Lloyd Instruments, UK) equipped with a 500 N load cell (model XLC -0500-A1) and cylindrical probe (model FG/CY3) was used. Instrument was connected to a personal computer. The software used was Nexygen Plus 3. Samples were taken in the middle of the cone and cut using a sampler sharpened at the top and squared at the cross section. The samples were cut into 28 x 28 x 28 mm sized cubes and wrapped in a plastic film. Prior to penetration test, samples were left for about 2 hours in order to reach room temperature (22±3 °C) and the cylinder probe was lubricated with mineral oil. During the test, the cylinder probe was penetrated into the cheese samples at a crosshead speed of 5 mm/min until the moment when sample fractured and then was withdrawn in zero position. Three replicates were analysed for each sample. Four rheological parameters were evaluated: 1) Maximum load produced during the penetration test; 2) Breaking load - load at which sample broke; 3) Brittleness, calculated as being the distance travelled to the point of fracture; 4) The total work done during the penetration test. The measurements were performed by the Department of Plant Sciences, Institute for Adriatic Crops and Karst Reclamation, Split.

Spectroscopic analysis

Infrared spectra were recorded with a resolution of 4 cm^{-1} on an ABB Bomem (Quebec City, Canada) MB102 single-beam spectrometer, equipped with CsI optics and a DTGS detector. The multiple-reflection (MR) ATR-IR spectra were recorded on a Thermo Scientific Spectra-Tech Foundation Multi-Bounce HATR using a horizontal multiple-reflection ATR zinc selenide (ZnSe) prism with 45° angle of incidence and a number of reflections of 10. Each spectrum was recorded as the ratio of the sample spectrum to the spectrum of the empty ATR plate. For each cheese sample the measurements were taken in triplicate at room temperature.

All spectra were smoothed and transformed to second derivative form by the Savitzky-Golay algorithm using a polynomial of degree two and a window size of 15 points in total, followed by normalization by multiplicative signal correction (MSC) (Ilari et al., 1988). Pre-processed spectra were used to evaluate similarities between samples by using the principal component analysis (PCA). The measurements were performed by the Department of Organic Chemistry and Biochemistry, Ruđer Bošković Institute, Zagreb.

Results and discussion

Physicochemical composition of Turoš cheese

The mean values of physical and chemical characteristics of Turoš cheese produced on 15 family

Table 1. The mean value (range) and standard deviation of the physicochemical composition of Turoš cheese made on family farms (n=15)

	Physicochemical composition						
	Fat (g/100 g)	Protein (g/100 g)	Total solids (g/100 g)	Salt (%)	pH	Moisture in total solids-nonfat (g/100 g)	Solids non fat (g/100 g)
mean (range)	14.90 (22.00)	28.80 (17.63)	57.28 (32.12)	3.84 (2.32)	4.45 (0.45)	57.57 (24.62)	25.56 (31.26)
standard deviation	±5.32	±4.86	±8.48	±0.83	±0.11	±7.27	±6.83

range - between farms

farms are presented in Table 1. Based on the obtained results, Turoš cheese belongs to semi-hard cheese, with the moisture content total solids-nonfat range from 54 % to 69 %. In relation to the milk fat content in total solids of cheese, this cheese can be categorised into the group of fat cheeses, with fat content in total solids between ≥25 % and <45 % (Official Gazette No. 20/09.).

The chemical composition of the cheeses showed a great deviation from the mean value, as expressed by range values in Table 1. The extensive variability of all the parameters was a result of unbalanced production of Turoš cheese among 15 family farms. To improve quality of Turoš cheese it is necessary to standardise the production.

Traditionally produced Turoš cheese (from the five representative family farms) contained lower milk fat, in comparison to the industrial Turoš cheeses (Table 2). such findings might be explained by the time of milk acidification. During two days of milk acidification considerable part of fat was spontaneously separated on milk surface and removed from

Table 2. The mean value (range) of the physicochemical composition of Turoš cheese made on family farms and in the dairy industry (five family farms and five batches of the industrial Turoš cheeses)

Turoš cheese	Physicochemical composition						
Batch	Fat (g/100 g)	Protein (g/100 g)	Total solids (g/100 g)	Salt (%)	pH	Moisture in total solids-non fat (g/100 g)	Solids non fat (g/100 g)
T1	13.50 (2.00)	27.05 (0.37)	56.30 (0.32)	4.42 (0.07)	4.47 (0.03)	57.20 (1.68)	23.97 (3.41)
T2	18.50 (2.00)	25.37 (0.87)	56.04 (0.87)	3.81 (0.29)	4.61 (0.01)	62.46 (1.08)	33.00 (3.02)
T3	19.50 (5.00)	30.06 (0.76)	59.34 (0.80)	3.48 (0.09)	4.43 (0.07)	60.16 (4.20)	32.83(7.99)
T4	5.50 (0.00)	34.70 (0.18)	56.74 (0.90)	4.19 (0.06)	4.45 (0.03)	48.76 (0.90)	9.69 (0.15)
T5	11.75 (5.50)	26.46 (2.21)	49.95 (7.22)	3.03 (0.04)	4.61 (0.17)	61.80 (2.72)	23.32 (5.64)
I1	28.00 (1.00)	30.05 (0.35)	64.91 (1.38)	2.67 (0.01)	4.49 (0.01)	63.09 (2.38)	43.15 (2.46)
I2	25.00 (2.00)	35.06 (0.73)	65.23 (0.50)	2.29 (0.12)	4.45 (0.01)	59.77 (1.50)	38.32 (2.78)
I3	27.25 (0.50)	32.28 (1.75)	64.29 (0.38)	2.44 (0.22)	4.44 (0.08)	62.96 (0.12)	42.39 (0.53)
I4	26.25 (1.50)	32.11 (1.31)	63.91 (0.63)	2.24 (0.13)	4.39 (0.14)	62.35 (2.13)	41.08 (2.75)
I5	37.75 (0.50)	31.14 (1.18)	72.27 (0.44)	1.85 (0.00)	4.57 (0.01)	65.48 (0.06)	52.23 (0.37)

range - within the batch
T - Turoš cheese made on family farms
I - Turoš cheese made in the dairy industry

Table 3. The mean value (range) of the textural properties of Turoš cheese made on family farms and in dairy industry (n=10)

Turoš cheese	Rheological parameters			
Batch	Maximum Load (N)	Load at Break (N)	Brittleness (mm)	Total work done during the test (Nmm)
T1	31.65 (2.84)	28.85 (2.48)	2.50 (0.27)	52.54 (6.21)
T2	28.42 (3.49)	26.20 (1.77)	2.73 (0.17)	48.52 (4.76)
T3	30.63 (6.19)	28.21 (6.78)	2.47 (0.09)	49.17 (13.07)
T4	51.94 (21.55)	47.60 (19.42)	2.92 (0.65)	111.82 (55.22)
T5	13.01 (4.41)	11.86 (3.97)	3.45 (0.37)	29.16 (5.37)
I1	22.35 (5.79)	20.47 (4.59)	3.12 (0.98)	42.87 (19.33)
I2	31.69 (4.53)	28.72 (4.08)	2.17 (0.58)	43.49 (11.48)
I3	30.47 (9.97)	27.42 (8.08)	2.54 (0.57)	48.92 (21.75)
I4	26.17 (2.86)	24.68 (2.23)	2.59 (0.18)	41.65 (4.89)
I5	24.31 (1.34)	22.15 (0.81)	2.66 (0.30)	42.49 (4.41)

sour milk in traditional production of Turoš cheese. In the industrial production, pasteurised milk is used in which 1/10 dosage of rennet and full dosage of starter cultures are added. The rennet and starter cultures contribute to a faster acidification (seventeen hours) and larger recovery of fat compared to Turoš cheese produce on family farms (Kršev et al., 1992).

Higher salt content was determined in cheeses produced on family farms in relation to the ones produced in industrial conditions. Cheeses T1, T2, and T3 made on family farms showed more standardized composition in relations to cheeses T4 and T5. The cheeses from family farm T4 had lower content of milk fat, moisture and higher content of proteins. The cheeses from family farm T5 showed lower content of total solids, and the highest variation among batches. Therefore, it is obvious that farms such as T4 and T5 should be excluded from the standardization process for branding of Turoš cheese. Chemical composition confirmed standardised procedure of production among the batches of industrial cheeses. The only exception was sample I5 which was considerably different from all the other industrial samples with high content of milk fat and total solids, and in lower content of salt. All analysed cheeses showed uniformity in pH (Table 2).

Textural analysis

Textural properties of food imply a group of physical characteristics which follow their structural elements. They can be perceived by pressing to deformation, disintegration and structure of food under the effect of force (Bourne, 2002).

All four parameters analysed by rheological measurements (Table 3) showed uniformity for eight cheeses: three from family farms (T1, T2 and T3), and all from industry (I1, I2, I3, I4 and I5). Cheeses T4 and T5 were different from all the other cheeses, which was expected regarding the large deviation in their chemical composition when compared to all the other analysed cheeses. The cheese structure depends on casein matrix in which water and milk fat are incorporated (Hort and Grys, 2001). The hardness and elasticity of cheeses highly depend on the quantity of protein in the cheese (Prentice, 1993; Puđa, 2009). The water in protein matrix has a "plastic" effect on texture and it makes it less elastic, or that it shows the tendency to crack during the compression. Effect of milk fat on texture depends on its physical characteristics. At lower temperatures, milk fat is mainly in a solid condition and contributes to the elasticity of the cheese body. Milk fat becomes liquid and viscous with the increase of the temperature, and plays like a lubricant on the touching surfaces alongside casein. Because of that, the point of crack of the protein matrix can be achieved with the lower force (Fox et al., 2000).

Table 4. The mean value (range) and standard deviation of the physicochemical composition of Turoš cheese made on family farms (T1, T2 and T3)

	Physicochemical composition (T1,T2 and T3)						
n = 3	Fat (g/100 g)	Protein (g/100 g)	Total solids (g/100 g)	Salt (%)	pH	Moisture in total solids-nonfat (g/100 g)	Solids non fat (g/100 g)
mean (range)	17.17 (6.00)	27.49 (4.69)	57.23 (3.30)	3.90 (2.94)	4.50 (0.18)	59.94 (5.26)	29.93 (9.03)
standard deviation	± 3.22	± 2.38	± 1.83	± 0.48	± 0.09	± 2.64	± 5.17

range - between T1, T2 and T3

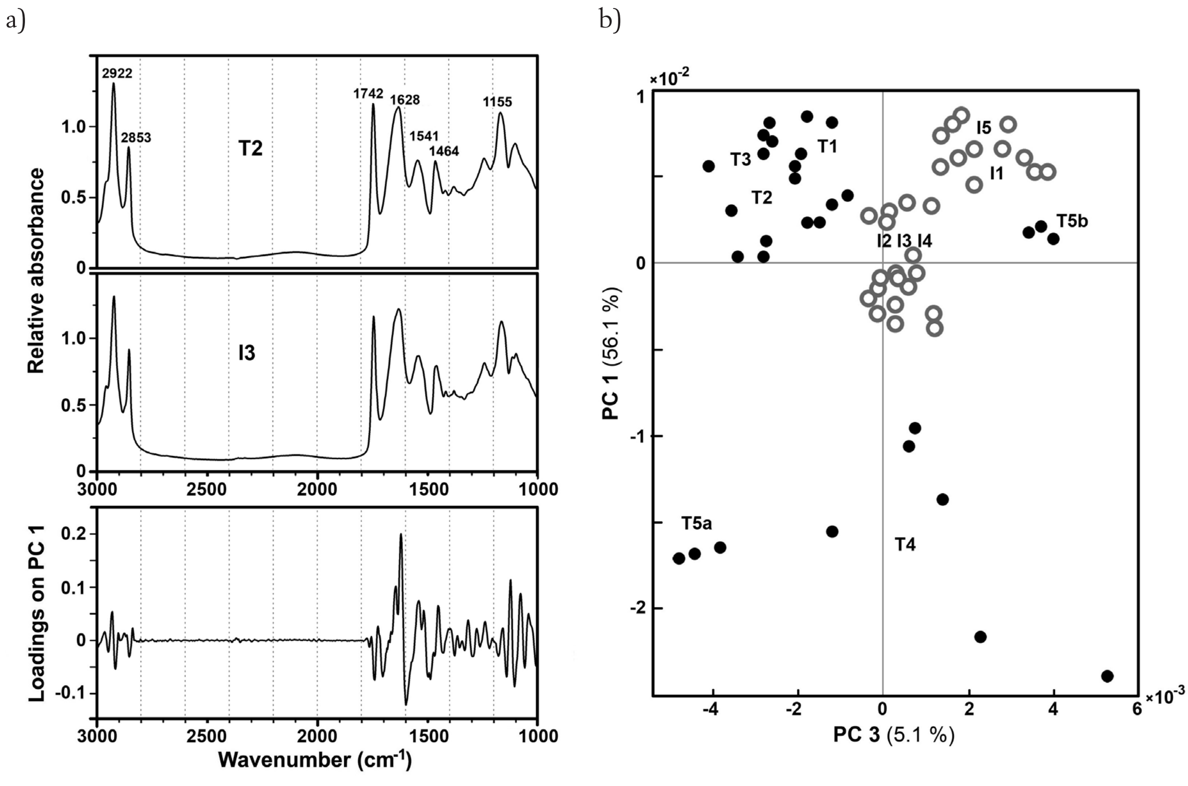

Figure 2. a) MSC corrected reflectance IR spectra of T2 sample of Turoš cheese made on family farm (up) and of I3 sample of industrial Turoš cheese (middle), and loadings plot on the first principal component of the PCA (down). b) PCA plot of IR spectral data (second derivative and MSC corrected spectra): (T) 10 samples from family farms (5 farms, each with 2 samples), (I) 10 samples from dairy industry (5 batches, each with 2 samples); three spectra per sample were measured. The spectral region of 1000 cm^{-1} to 3000 cm^{-1} was selected for data analysis. The percent variances for the first five PCs are 56.08, 28.52, 5.12, 2.75, and 1.96

The rheological properties of cheeses T4 (with low fat content, low moisture in total solids-nonfat content, and greater content of proteins) and T5 (with low total solids) confirmed previous findings.

The rheological parameters of analysed cheeses were in accordance with the chemical composition of the same samples, as stated in Tables 2 and 3. The one exception were samples of industrial cheese denoted as I5 which did not differ in rheological parameters from other industrial samples, as it was observed from measurement of chemical composition. Since chemical and rheological analyses were not obtained from the same samples, these results could indicate an unbalanced industrial production within one batch I5.

Infrared spectroscopy of Turoš cheese

The typical IR spectra of family farm Turoš cheese (sample T2) and industrial Turoš cheese (sample I3) are presented in Fig. 2. The spectra are dominated by the bands associated with fats (2922, 2853, 1742, 1464 and 1155 cm^{-1}) and proteins (1628 and 1541 cm^{-1}). The difference between the industrial and family farms samples (Fig. 2) was evident in the ratio of bands associated with proteins (1628 and 1541 cm^{-1}) and fats (1742, 1464 cm^{-1}). The PCA of the spectral data reveals good separation of industrial and family farms samples. Moreover, the separation of chemically outlying samples T4 and T5 is also present in the spectral data. The two samples of batch T5 (T5a, T5b), that show great variability in chemical composition, showed significant spectral difference as well. From the above mentioned facts it can be concluded that the IR spectra of cheeses show good relation with the chemical composition. Compared to the standard chemical and textural analysis, IR spectroscopy has proved to be low-cost and a rapid method, which provides a great number of information by only one measurement. IR spectroscopy might serve as a simple and economical method for monitoring Turoš cheese production on family farms, particularly in the case of Turoš cheese branding.

Based on chemical, textural and spectroscopy analyses it is obvious that the cheeses from the family farms T4 and T5 were not representative. Therefore, cheeses T4 and T5 were excluded from the final statistical analysis of Turoš cheese properties. The average values of the physiochemical composition for Turoš cheese, based on family farms T1, T2 and T3, was shown in Table 4.

Values from table 4. are in accordance with the mean composition of Turoš cheese obtained by analysis of samples from the initial 15 family farms (Table 1).

Conclusion

According to chemical composition, there were clear differences among cheeses produced on family farms, especially compared to industrially produced Turoš cheeses. The difference between cheeses produced on family farms and those industrially produced was determined for content of fat, protein, total solids and salt. The analysis of cheese texture and analysis of IR spectrum of cheeses were in good agreement with the chemical analysis. IR spectroscopy of cheeses is clearly a faster and a cheaper method compared to textural and standard chemical analysis. Therefore, it might be used as a single method for monitoring the standard production procedure of sour, dried cheeses such as Turoš cheese.

Prikladnost infracrvene spektroskopije i reološke metode u razlikovanju tradicijskog od industrijskog sira turoša

Sažetak

Cilj ovog rada bio je utvrditi prikladnost infracrvene spektroskopije i reološke metode u razlikovanju tradicijskog od industrijskog sira turoša. Sir turoš pripada skupini kiselinskih, sušenih sireva u obliku stošca s dodatkom soli i mljevene crvene paprike. Sirevi su uzorkovani na 15 obiteljskih poljoprivrednih gospodarstava i u trgovinama (pet različitih šarži industrijskih sireva). Reološki parametri su u skladu s kemijskim sastavom analiziranih sireva. Infracrvena spektroskopija sireva pokazuje dobru povezanost s kemijskim sastavom, a pokazalo se da je ona učinkovit način za praćenje standardnog postupaka proizvodnje kiselinskih, sušenih sireva kao što je sir turoš u odnosu na standardne teksturalne i kemijske analize. Velika varijabilnost svih parametara je posljedica neuravnotežene proizvodnje sira turoša među obiteljskim poljoprivrednim gospodarstvima. Industrijska proizvodnja sira turoša pokazuje veću ujednačenost u odnosu na tradicijsku proizvodnju sireva na obiteljskim poljoprivrednim gospodarstvima.

Ključne riječi: kiselinski sušeni sirevi, kemijski sastav, tekstura, infracrvena spektroskopija, turoš

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