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Quality parameter assessment of grated Parmigiano–Reggiano cheese by waveguide spectroscopy

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ABSTRACT

Parmigiano–Reggiano (P–R) cheese is still one of the most valuable Protected Designation of Origin (PDO) cheeses of Italy. The rind percentage (from 12% to 50%), the ripening (from 13 to 31 months), the moisture content and the differences between true P–R and competitors were determined by means waveguide spectroscopy. Preliminary tests were carried out in the 2–20 GHz frequency range (with a span of 1 GHz) to investigate which 1 GHz frequency range contains most information on the rind percentage and on the months of ripening.

Partial Least Squares (PLS) regression was used to predict rind percentage, months of ripening and moisture in the previously selected frequency ranges (2–3, 5–6 and 17–18 GHz for the rind percentage; 2–3, 5–6 and 16–17 GHz for the months of ripening). Moreover, Soft Independent Modelling of Class Analogy (SIMCA) analysis was used to discriminate the samples according to the rind percentage. Principal component analysis (PCA) was used to discriminate true P–R cheese from competitors. PLS models (*test set validation*) showed R^2 values up to 0.944 (root mean square error of prediction in test set validation, $RMSE_p$ = 3.4%), 0.966 ($RMSE_p$ = 1.2 months) and 0.786 ($RMSE_p$ = 0.99%) for the prediction of rind percentage, months of ripening and moisture percentage, respectively. As a result only a part of the prediction of rind percentage and ripening can easily be attributed to the moisture.

For each considered frequency range, all samples belonging to the classes characterized by 12% and 50% of rind were correctly classified. Competitors were clearly separated from P–R cheese by the PCA analysis of the spectral signals and their moisture was predicted with a R^2 values up to 0.942 (*test set validation*). In this case, the discrimination power can be mainly attributed to moisture content.

1. Introduction

Parmigiano–Reggiano (P–R) cheese is one of the oldest traditional cheeses produced in Europe, and it is still one of the most valuable Protected Designation of Origin (PDO) cheeses of Italy (Zannoni, 2010). The cheese is produced from bovine milk in a limited geographic area in northern Italy, exclusively from raw and unheated bovine milk. The Parmigiano–Reggiano cheese Consortium (CFPR) is the institutional system that encompasses all the cheese dairy factories, and regulates the cheese production in terms of cow feeding, cheese manufacture and ripening processes. These rules are based on secular experience closely related to regional habits and practices and also on technological innovation that has left the uniqueness of the product unaltered (EC, 510/2006).

The denomination of origin is extended to grated P–R cheese made exclusively with whole cheese. The grating operation must occur in the defined geographical area of cheese production, and packaging must take place immediately, without any processing or addition of substances to modify the conservation and the original organoleptic characteristics. The grated cheese must be characterized by the absence of additives, a moisture content not less than 25% and not more than 35%, and rind not more than 18%, a non-crumbly aspect and homogeneous particles with diameter less than 0.5 mm not exceeding 25% (D.P.C.M., 4/11/1991).

Therefore, to ensure the final quality of the product and guarantee the authenticity of grated P–R, it is important to monitor the preparation of the grated product in the diary factories, because the marks embossed on the rind identifying the P–R are lost in the grated product. Since 1998, the department of P–R quality control (DQC PR) has been the official authority commissioned by the Italian ministry of agriculture, to ascertain the compliance to the P–R DOP standards in each phase of the production chain. In particular, the DQC PR carries out random analyses on the grated product each year to assess the absence of additives, determination of free amino acids and moisture content, sensory analysis, and evaluation of the particle size.

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It is well known that the cheese making technology and the dimension of the wheel (22–24 cm high, 40–45 cm diameter) of this long-ripened cheese (minimum 12 months of ripening) lead to differential development of bacterial growth and to evolution of the chemical/physical parameters between the inner and the outer zones during the ripening (Malacarne et al., 2009; Tosi et al., 2008; De Dea Lindner et al., 2008; Panari et al., 2003; Careri et al., 1996; Pecorari et al., 1995). To determine the rind percentage and the months of ripening of the grated P–R by referring only to the moisture value of the product, is difficult because the packs are filled with grated cheese from different areas of the wheel.

It therefore becomes important to find a method to determine the rind percentage and ripening of the grated P–R cheese based not only on the moisture variation.

The possibility of detecting the addition of extra rind to grated cheese (Grana Padano) and the ripening time of the P–R cheese has been studied by means of destructive chemical methods (Cattaneo et al., 2008; Consonni and Cagliani, 2008; Shintu and Caldarelli, 2005).

Dielectric spectroscopy provides a window into the material and allows internal properties, composition and structure of the material to be assessed (Everard et al., 2006). The dielectric properties change with composition, temperature and frequency at which dielectric measurement is taken (Ryynaänen, 1995).

A large number of studies have been conducted to investigate the dielectric properties of food products such as cereals (Tabelsi and Nelson, 2003), fruit and vegetable (Nelson et al., 1994; Wang et al., 2003;), bread dough (Zuercher et al., 1990), and meat and egg (Zhang et al., 2004; Sipahioglu et al., 2003; Ragni et al., 2006). On the contrary, dielectric measurement techniques have not been used extensively in the analysis of cheese properties. By using different probes and frequency ranges, research showed that dielectric properties depend on the chemical composition of the cheese, such as the moisture and inorganic salt (Everard et al., 2006; Fagan et al., 2005) and fat contents (Herve et al., 1998; Green, 1997). In particular, using a coaxial line probe between 300 MHz and 3 GHz and chemometric models, Fagan et al., 2005) reported correlation values of R^2 = 0.98 and R^2 = 0.91 (full cross validation), respectively for the prediction of moisture and inorganic salt.

The present research intends to non-destructively predict the rind percentage, ripening and moisture content of the grated P–R by means of waveguide spectroscopy, a technique already set up and used in previous studies on eggs (Ragni et al., 2010) and kiwifruits (Ragni et al., 2012). In addition differences between true P–R and competitors were determined. The explored range of frequency was from 2 to 20 GHz (with steps of 1 GHz). Predictive (PLS) and classification (SIMCA) models of rind percentage and ripening were built in the best frequency ranges previously selected from preliminary trails.

2. Materials and methods

2.1. Instrumental chain

The instrumental chain used in this study consisted of a rectangular waveguide, a sinewave sweep oscillator (HP 8350B + 8352B), with a frequency range from 0.01 to 20 GHz, and a spectrum analyzer (HP 8566B), with a range from 100 Hz to 22 GHz connected to a PC (Fig. 1). The spectral information was collected by LabVIEW 8.2 (National Instruments Corporation, Austin, Texas).

The waveguide is made of aluminum and is characterized by the following internal dimensions, in mm: 244 (length); 96 (width), 46 (height). The thickness of aluminum is 2 mm. The guide is terminated to minimize the reflections with a 234 mm long termination section. The absorbing material of the pyramidal element is graphite mixed with sand.

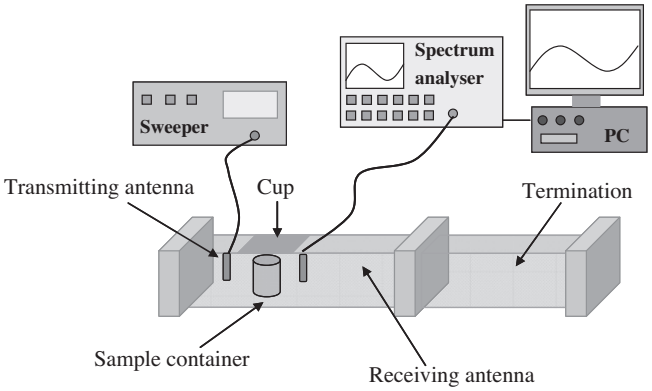

Fig. 1. Layout of the instrumentation used.

A transmitting antenna is connected by a coaxial cable to the sweeper and the transmitted/reflected signal, detected by the receiving antenna, is captured by a spectrum analyser. Antenna location and configuration were chosen for the propagation dominant mode TE10 (Mahmoud, 1991; Marcuvitz, 2005), with a cut-off frequency of 1.561 GHz. The main body of the antenna is made by a 8 mm (diameter) × 20 mm (length) brass cylinder. The cylinder is welded on the terminal of a N type connector. The antennas were mounted on the longer side of the guide section, placed along the longitudinal axis and oriented along the transversal axis of the guide. The distance between the center of the two antennas is 125 mm, while the distance from the wall to the transmitting antenna is 62 mm.

To insert the sample, a square window (70 × 70 mm^2), with aluminum cap, is located in the middle of the upper side of the guide. The sample (15 ± 0.1 g) was placed inside a glass cylinder with the following dimensions: 34 mm (diameter), 44 mm (height), 1.4 mm (thickness). The cylinder was placed at the center of the two antennas and aligned with them along the longitudinal axis of the guide. The distance between the cap and the cylinder was of 2 mm. The grated cheese was poured inside the cylinder and it was brought to volume through a light compression provided by a slight shaking. The small changes occurring in the mass, volume and in the way in which the grated cheese is added to the cylinder (intensity of the shaking) did not affect the resulting signal. This assertion was confirmed by the fact that the resulting signals of various replicas of the same samples were very close.

2.2. Samples

All samples of grated P–R (170 samples) and non-P–R (15 samples) cheese were provided by the CFPR from local grating companies, and the particle size ranged between 0.5 and 1.5 mm.

The P–R cheese samples were manufactured (grated and packaged) in the area of origin. All the grated P–R samples derived from several certified whole cheese wheels, and purchased by the grating companies at various dairy farms in such area.

The grating companies provided samples with different months of ripening and rind percentage in order to randomize the source of the samples belonging to a particular class. This randomization rules out a possible relationship between the particle size and the sample classes.

The rind percentage ranges were chosen by the CFPR based on checks drawn from grated cheeses found on the market and with reference to the standard (D.P.C.M. 4/11/1991).