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Comparison of two infrared spectroscopic methods for cheese analysis

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Abstract

Two infrared spectroscopic methods, optothermal near infrared (NIR) spectroscopy and Fourier transform mid-infrared–attenuated total reflection (FTIR–ATR) spectroscopy, were applied to 24 cheese samples in order to obtain protein, fat and moisture contents. Reference values of the protein, fat and moisture contents in weight percent were obtained using standard wet chemistry analysis. Prediction correlation coefficients between 0.93 and 0.96 and standard errors of prediction between 2% and 5% were obtained using optothermal spectroscopy while the corresponding values for FTIR–ATR were 0.81–0.92 and 4–9%. Inhomogeneities in the cheeses, primarily due to the fat droplets, are probably the main reason for the differences in the error sizes. The superior results for optothermal spectroscopy are the more attractive because the instrument is easier to use than the FTIR–ATR instrument, it provides results more quickly with simpler statistical analysis and it is more compact and robust.

Keywords: NIR spectroscopy; Protein; Fat; Moisture; Dairy products; Cheese

1. Introduction

Near infrared spectroscopy has been used more and more in the agricultural and food industries in recent decades. An excellent summary of the field is given by Williams and Norris [1]. In recent years it has become increasingly clear that the application of spectroscopic methods to food analysis can alleviate important problems in the processing and distribution of food and food products [2–4]. More recently the mid-infrared region has attracted considerable interest [5]. It is especially clear that quick, inexpensive, objective methods that do not require special skills on the part of the users are of greatest interest. The ideal case would be that small and medium-sized organizations could handle the spectroscopic instruments in direct connection with their food handling systems, obtaining measurement results in seconds or minutes so that processes could be adjusted and decisions taken quickly. Such smaller production units are not expected to have easy access to highly trained laboratory personnel or to be able to afford expensive and/or sophisticated equipment. Alternatively, quick methods can be used to screen large numbers of samples prior to more detailed analysis of specific samples when and where necessary. The optothermal near infrared (NIR) spectroscopic technique described here can be an appropriate method in some cases while for other applications the repeatability of the measurement results must be improved.

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An example of an industry in which there is a large number of small medium-sized units processing food products is the cheese industry. There are several steps in the cheese-making process that can directly affect the amounts of fat, protein and moisture in the final products, for instance the step or steps in which the curds are separated from the whey. Further, it is known that because of its texture cheese is difficult to analyze spectroscopically using traditional methods. Fourier transform mid-infrared–attenuated total reflection FTIR–ATR and NIR optothermal analysis are two possible methods for determining fat, protein and moisture contents quickly and accurately as well as at reasonable expense to the cheese producer. Here these two methods are evaluated and the results discussed in terms of the morphology of the cheese samples.

It is known that cheeses (and other similar products) are not spatially homogeneous. For instance, the fat content in one part of a large cheese can be of the order of one percent different from that in another part of the same cheese. On another level, the cheese is very inhomogeneous on a submillimeter scale because of the relatively large fat globules. These conditions have to be taken into account in interpreting measurements of fat, protein and moisture contents of cheeses.

Fat globules in milk have diameters of the order of several microns, with wide variations [6]. The casein micelles are much smaller, of the order of 100 nm, and globular proteins are even smaller.

While there is a great deal of detailed information about the morphology of milk fat and other milk components, the situation is quite different concerning cheese, for which vanishingly little published data is available. However, it is felt that during coagulation there is no reason to expect changes in the sizes of fat globules, even if there can be damage to and surface modifications of the fat globule membrane during pasteurization before the cheese-making process. The fat globule membrane can be altered due to the acidity of the cheese starter or due to physical processing such as centrifugation or membrane processing. None of these factors is expected to reduce the sizes of the fat globules significantly. During ripening the activities of the lipases produced by the microorganisms of the starter modify the fat globule structure, increasingly changing the typical round shape of the globules. In some sufficiently aged cheeses the fat globules disappear to be replaced by aggregates of lipolized fat [7, 8]. Thus it appears that the extent of lypolysis is a prime determinant of the morphology of cheeses at the micron level. The morphologies of fresh cheeses are thus expected to be quite similar to that of the milk from which they were produced, while the aged cheeses may be somewhat less inhomogeneous on the micron scale.

2. The cheese samples

Twenty-four cheese samples were studied. Six samples each were obtained from sources in Portugal, UK, Denmark and Finland. They included six fresh cheeses, nine soft cheeses and nine hard cheeses. The widest possible range of cheese types was sought in order that fat, protein and moisture contents show the widest variations possible and so that cheese characteristics affecting the measurement techniques, for instance rheological and mechanical characteristics as well as morphology, be as clearly expressed as possible. However, no separate rheological, mechanical or morphological studies of the cheese samples were carried out.

3. Methods

3.1. Wet chemistry

The wet chemistry analysis of the 24 cheese samples was carried out using standardized methods according to the Nordic Committee on Food Analysis. For each component and each cheese at least two samples were analyzed.

The protein contents were determined using the Kjeldahl method in which "The sample is digested with concentrated sulphuric acid, with the addition of potassium sulphate and copper(II) sulphate. The ammonia formed is distilled off with sodium hydroxide solution and collected in boric acid. The nitrogen content of the sample is then determined by titration with hydrochloric acid".

The fat content was determined according to the Schmid–Bondzynski–Ratslaff method in which "The sample is treated with 8 M hydrochloric acid and after addition of ethanol the liberated fat is extracted with a mixture of diethyl ether and petroleum ether. The solvent is then evaporated and the fat is weighed".