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Determination of fat, protein, moisture, and salt content of Cheddar cheese using mid-infrared transmittance spectroscopy[1]

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ABSTRACT

The objective of our work was to develop and evaluate the performance of a rapid method for measuring fat, protein, moisture, and salt content of Cheddar cheese using a combination mid-infrared (MIR) transmittance analysis and an in-line conductivity sensor in an MIR milk analyzer. Cheddar cheese was blended with a dissolving solution containing pentasodium triphosphate and disodium metasilicate to achieve a uniform, particle-free dispersion of cheese, which had a fat and protein content similar to milk and could be analyzed using a MIR transmittance milk analyzer. Annatto-colored Cheddar cheese samples (34) from one cheese factory were analyzed using reference chemistry methods for fat (Mojonnier ether extraction), crude protein (Kjeldahl), moisture (oven-drying total solids), and salt (Volhard silver nitrate titration). The same 34 cheese samples were also dissolved using the cheese dissolver solution, and then run through the MIR and used for calibration. The reference testing for fat and crude protein was done on the cheese after dispersion in the dissolver solution. Validation was done using a total of 36 annatto-colored Cheddar cheese samples from 4 cheese factories. The 36 validation cheese samples were also analyzed using near-infrared spectroscopy for fat, moisture, and the coulometric method for salt in each factory where they were produced. The validation cheeses were also tested using the same chemical reference methods that were used for analysis of the calibration samples. Standard error of prediction (SEP) values for moisture and fat on the near-infrared spectroscopy were 0.30 and 0.45, respectively, whereas the MIR produced SEP values of 0.28 and 0.23 for moisture (mean 36.82%) and fat (mean 34.0%), respectively. The MIR also out-performed the coulometric method for salt determination with SEP values of 0.036 and 0.139 at a mean level of salt of 1.8%, respectively. The MIR had an SEP value of 0.19 for estimation at a mean level of 24.0% crude protein, which suggests that MIR could be an easy and effective way for cheese producers to measure protein to determine protein recovery in cheese making.
Key words: mid-infrared, Cheddar cheese, composition

INTRODUCTION

Measurement of cheese yield, recovery of fat and protein, and evaluation of cheese composition control in large-scale cheese factories is an important aspect of cheese factory management. A system for evaluation and control of these parameters was reported by Margolies et al. (2017). During the testing of a cheese yield evaluation system in large commercial cheese factories, it was observed that the daily mean bias error in measurement was as high as ±0.5% for fat and moisture in commercial cheese factories. It was concluded that the accuracy of rapid secondary testing methods needed improvement (Margolies et al., 2017).

The chemical reference methods for measurement of fat (e.g., Babcock and Mojonnier ether extraction), CP (Kjeldahl), moisture (forced air oven-drying), and salt (Volhard silver nitrate titration) are accurate but are too slow and impractical for rapid analysis of large numbers of cheese samples in a commercial quality assurance (**QC**) laboratory. Therefore, more rapid and cost-effective methods such as near-infrared (**NIR**) reflectance for fat, protein, and moisture (Rodriguez-Otero et al., 1995; McKenna, 2001) and coulometric methods (Varcoe, 2001) for salt determination are currently used in cheese factories.

The NIR methods for measurement of cheese composition use partial least squares (**PLS**) calibration models that are developed locally in each cheese factory and often require a large number of cheese samples (200 to 400) of each cheese type to be tested by reference chemistry and the NIR within each cheese factory (McKenna, 2001; Barbano and Lynch, 2006). Although using NIR for cheese composition analysis is

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appropriate in terms of the ease of sample preparation and speed of analysis, the work required to develop calibrations for each cheese type within each cheese factory is not practical or cost effective. Holroyd (2011) indicated that there has been an evolution of equipment manufacturer-specific NIR calibration approaches using multiple linear regression and PLS over the years. If a factory has more than one NIR analyzer within their laboratory, separate calibration development may be required for each instrument. The accuracy of each prediction model for each cheese type is dependent on the accuracy of the reference chemistry performed in the cheese factory and the concentration range and distribution of fat, protein, and moisture in the specific calibration samples used for PLS model development (McKenna, 2001; Barbano and Lynch, 2006). Consistency of sample presentation (particle size distribution) is often a source of analytical variation in NIR cheese analysis (Holroyd, 2011). In a review of NIR analysis of cheese, Holroyd (2011) gave the Fonterra Research Centre (Palmerston North, New Zealand) perspective on the evolution of NIR analysis of cheese for a large group of cheese factories in New Zealand over the last 30 yr, and on the evolution of NIR analytical technology. Holroyd (2011) reported that the following factors are important in NIR cheese analysis: (1) careful sample preparation using several cheese cores from each block, (2) representative sampling of the block, (3) a knowledge of the sample matrix, and (4) sample preparation that provides a uniform presentation of the sample to the NIR. The accuracy of analytical performance of an indirect measurement method's prediction of reference chemistry is called the standard error of prediction (**SEP**). Holroyd (2011) reported the SEP metrics from many studies of NIR cheese analysis performance ranging from 0.2 to 0.5% for fat, protein, and moisture during the period from 1993 to 2003. In 2003, Fourier transform NIR instruments became more common and some individual companies developed global PLS models for cheese analysis that reduced the amount of reference chemistry, time, and cost required at the local level for calibration, while maintaining SEP values more consistently in the range of 0.2 to 0.35% for fat and moisture prediction (Holroyd, 2011).

Mid-infrared (**MIR**) milk analyzers are commonly used in cheese factories for analysis of milk, cream, and liquid whey products. The understanding of measurement of fat and protein using MIR is well developed (Kaylegian et al., 2006b, 2009) and accepted in official methods for milk (AOAC, 2016, method number 972.16). While various instrument manufacturers have developed applications notes for analysis of cheese samples using MIR liquid analyzers, utilization of this approach for cheese analysis is not common. Unlike NIR, MIR methods use well documented wavelengths for measurement of fat, protein, and lactose (Kaylegian et al., 2009) and this approach could be applied to cheese analysis. The challenge is sample preparation to convert the solid cheese into a particle-free liquid that could be pumped through the MIR milk analyzer flow system. A method for dissolving cheese for MIR analysis was described by Sjaunja (1999) using a solution of disodium metasilicate and pentasodium triphosphate. The potential advantage of this approach would be that liquid calibration samples might be prepared centrally and used on multiple instruments in multiple factories as is currently done for milk (Kaylegian et al., 2006a, 2006b) and whey analysis by MIR. This would reduce the need for reference chemistry analysis of cheese in cheese factories and provide an analytical alternative to NIR analysis of cheese using a piece of equipment that is normally available in cheese factories. The objective of our work was to develop and evaluate the performance of a rapid method for measuring fat, protein, moisture, and salt content of Cheddar cheese using a combination MIR transmittance analysis and an in-line conductivity sensor within the MIR milk analyzer.

MATERIALS AND METHODS

Experimental Design

Calibration. Cheese samples were diluted about 10:1 (wt/wt) with a dispersing liquid, blended, and pumped through a MIR milk analyzer (LactoScope FTIR Advanced, Delta Instruments, Drachten, Netherlands). Cheese samples were between 5 and 10 d of age at the time of analysis. Calibration of the MIR milk analyzer was done using 34 individual annatto-colored full-fat Cheddar cheese samples that represented different individual batches of cheese from one large commercial cheese factory. Reference chemistry for fat, protein, salt, and TS was determined for each cheese sample and a linear slope and intercept adjustment was made for estimation of fat (separately for fat A, B, and A+B), protein, salt, and TS. Fat A is a fat measurement based on the carbonyl stretch and fat B is based on carbon hydrogen stretch within neutral and polar lipids (Kaylegian et al., 2009). Moisture was calculated as 100 minus TS.

Validation. Whereas the calibration was based on full-fat annatto-colored Cheddar cheese from one factory, validation was done with 36 full-fat annatto-colored Cheddar cheeses from 4 different factories (6 from each of 2 factories and 12 from each of the other 2 factories). One of the 4 factories (6 cheese samples) was the