J. Dairy Sci. 99:9471–9482
http://dx.doi.org/10.3168/jds.2016-11618
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Effect of homogenizer performance on accuracy and repeatability of mid-infrared predicted values for major milk components[1]

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ABSTRACT

Our objective was to determine the effect of mid-infrared (MIR) homogenizer efficiency on accuracy and repeatability of Fourier transform MIR predicted fat, true protein, and anhydrous lactose determination given by traditional filter and partial least squares (PLS) prediction models. Five homogenizers with different homogenization performance based on laser light-scattering particle size analysis were used. Repeatability and accuracy were determined by conducting 17 sequential readings on milk homogenized externally to the instrument (i.e., control) and unhomogenized milk. Milk component predictions on externally homogenized milks were affected by variation in homogenizer performance, but the magnitude of effect were small (i.e., <0.025%) when milks were pumped through both efficient and inefficient homogenizers within a MIR milk analyzer. Variation in the in-line MIR homogenizer performance on unhomogenized milks had a much larger effect on accuracy of component testing than on repeatability. The increase of particle size distribution [d(0.9)] from 1.35 to 3.03 μm (i.e., fat globule diameter above which 10% of the volume of fat is contained) due to poor homogenization affected fat tests the most; traditional filter based fat B (carbon hydrogen stretch; −0.165%), traditional filter-based fat A (carbonyl stretch; −0.074%), and fat PLS (−0.078%) at a d(0.9) of 3.03 μm. Variation in homogenization efficiency also affected traditional filter-based true protein test (+0.012%), true protein PLS prediction (−0.107%), and traditional filter-based anhydrous lactose test (+0.027%) at a d(0.9) of 3.03 μm. Effects of variation in homogenization on anhydrous lactose PLS predictions were small. The accuracy of both traditional filter models and PLS models were influenced by poor homogenization. The value of 1.7 μm for a d(0.9) used by the USDA Federal Milk Market laboratories as a criterion to make the decision to replace the homogenizer in a MIR milk analyzer appears to be a reasonable limit, given the magnitude of effect on the accuracy of fat tests. In the future, as new PLS models are developed to measure other components in milk, the sensitivity of the accuracy of the predictions of these models to factors such as variation of homogenizer performance should be determined as part of the ruggedness testing during PLS model development.
Key words: mid-infrared, homogenization, testing accuracy

INTRODUCTION

Mid-infrared (**MIR**) milk analysis is based on the principle that each specific chemical bond absorbs MIR energy at a specific wavelength, and the measurement of the intensities of the absorption peaks makes it possible to quantify milk components (Goulden, 1964; Biggs, 1967; Biggs et al., 1987). The MIR energy passing through inefficiently homogenized milk can be distorted by the Christiansen light-scattering effect, which causes a shift in the apparent wavelength of maximum absorption by the carbonyl and carbon-hydrogen groups to a longer wavelength affecting the accuracy of MIR readings (Goulden, 1961). This shift in wavelength or light absorbance may have a negative effect on the accuracy of the determination of the concentration of the major components of milk. As a result, quality-assurance programs for MIR milk analysis often include a test to determine if the homogenizer in a MIR milk analyzer is working properly (Lynch et al., 2006). Normally, a laboratory would need to pump an unhomogenized milk through the homogenizer on their infrared milk analyzer, collect the instrument-homogenized milk,

Received June 15, 2016.
Accepted August 16, 2016.
[1]Use of names, names of ingredients, and identification of specific models of equipment is for scientific clarity and does not constitute any endorsement of product by authors or Cornell University.
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and send the milk to another laboratory for a laser light-scattering particle size analysis to determine if the homogenizer was functioning properly (Lynch et al., 2006). Di Marzo and Barbano (2016) reported that the systematic shift in the MIR absorbance spectra due to the Christiansen effect could be modeled using partial least squares (**PLS**) and enable a prediction of particle size distribution **d**(**0.9**) in real-time operation of the MIR milk analyzer.

Commercial Homogenization of Milk

The recommended milk temperature for commercial milk homogenization is between 60 to 75°C, which achieves breakage of milk fat into smaller fat globules and reduce the tendency of fat globules to aggregate and rise to the top of container of fluid milk (Trout, 1950; Walstra et al., 2005). If the temperature of homogenization of milk is below the melting point of milk fat (i.e., <40°C), fat will be in the solid state, resulting in incomplete fat dispersion and ineffective homogenization (Trout, 1950; Bylund, 1995). In a typical commercial homogenizer, a high-pressure positive displacement pump forces heated milk through a narrow gap in the homogenizer valve (Mulder and Walstra, 1974; Phipps, 1985). As the milk is forced through the gap at high pressure, the linear velocity of the milk and shear forces increase. Often, the high-velocity milk is projected against a surface to create high turbulence and more shear, leading to a reduction in fat globule size (Mulder and Walstra, 1974; Walstra et al., 2005). The high-pressure positive displacement pump can be equipped with a single piston or with multiple pistons (3, 5, or 7). In a single-piston homogenizer, the valves open and close with every stroke of the piston and the flow pressure goes from zero to the set pressure for that stage and back to zero as the valve opens and closes. The multiple pistons are operated intentionally out of phase to achieve a constant applied pressure, continuous flow of milk, and uniform homogenization. The higher the number of pistons, the more the pressure fluctuations are minimized (Phipps, 1985) and the homogenizer valves are running open continuously with relatively constant pressure decrease across the gap. In this way, the valve wear is minimized and consistent particle size is achieved (Walstra, 1975).

Typical homogenization of pasteurized fluid milk is done with a 2-stage homogenizer with a first-stage pressure of 20 MPa and second-stage pressure of 5 MPa (Walstra et al., 2005). The second valve should always operate at lower pressure (i.e., 20% of the total pressure; Walstra, 1975). The function of the high-pressure first stage is to break fat globules to smaller sizes. The newly formed small fat globules are no longer exclusively covered with the original milk fat globule membrane. Instead, they are also covered with protein adsorbed from the milk plasma (Walstra et al., 2005). In the turbulent environment created by velocity of the milk and shear forces in the milk exiting the first stage, the small fat globules may start to collide before they are completely covered with protein, leading to fat globule coalescence (Mulder and Walstra, 1974). The first stage creates up to a 10-fold increase of the milk fat-plasma interfacial surface area. If the surface of the newly formed fat globules lacks protein, the small fat globules may easily come together to share protein at their interface, forming clusters (Mulder and Walstra, 1974). The function of the low-pressure second stage is to break up the fat globule clusters (Walstra, 1975; Phipps, 1985; Walstra et al., 2005). At the second stage the pressure is low so that the new surface created is insignificant and new clusters are not formed (Mulder and Walstra, 1974). Enough time needs to be given for the newly formed fat globules to cluster after milk passes through the first-stage valve so that the second-stage valve will be able to fulfill its role in breaking clusters (Walstra et al., 2005). The typical d(0.9) in a commercially homogenized milk is about 1.2 to 1.8 μm (Caplan and Barbano, 2013).

Homogenization in a MIR Milk Analyzer

The MIR homogenizer designs are slightly different than a commercial homogenizer. All homogenizers within MIR milk analyzers are single-piston homogenizers; thus, the pressure across the homogenizer stages is going from zero to full pressure and back to zero with every pump stroke during the pumping of a single milk sample. Some MIR homogenizer designs have the springs in the milk flow, and this is different than commercial homogenizers. A 2-stage homogenizer with the springs in the milk flow is shown in Figure 1. This design of homogenizer has been used by Multispec (no longer in business), Bentley Instruments (Chaska, MN), and Delta Instruments (Drachten, the Netherlands). This type of homogenizer includes the 2 stages connected in series within a single homogenizer housing, which is mounted in the MIR as shown in Figure 1A. In Figure 1B, the internal parts of the homogenizer (#1) are shown. The strength of the first- (#8) and second-stage (#13) springs are different (Figure 1B). Heated milk (about 40°C) is pumped through the homogenizer and reaches the first-stage seat (#4) and the ball (#7). Higher milk temperatures are not used in MIR milk analyzers because of the negative effect of high milk temperatures on the cuvette. The high milk pressure (about 15 MPa) operating against the spring (#8) forces the ball off the seat and opens a narrow

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