Use of Fourier Transform Infrared Spectroscopy in identification of raw milk adulteration

Tatiane B. Coitinho * 2; Laerte D. Cassoli2; Paulo F. Machado2; Pedro H. R. Cerqueira2; Juliana B. Coitinho3; Helen K. Silva2

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* Pádua Dias Ave, 11, CEP:13148-000, Piracicaba, SP, Brazil; 2 Escola Superior de Agricultura “Luiz de Queiroz”, University of São Paulo; 3 Federal University of Espírito Santo, Vitória, ES, Brazil.

* tatianebbc@usp.br

Food adulteration is an ancient practice that has become more sophisticated every day. In recent years, it has been observed several adulteration practices in milk, like the addition of melamine, held in China, and various other compounds such as sodium citrate, sodium chloride, sucrose, bicarbonates, hydrogen peroxide, urea and formaldehyde, occurring in Brazil and other countries, such as India, which creates a major concern throughout the milk production chain. Most of the usual analytical methods have low yield, require the use of reagents and are highly dependent on hand labor, which hampers the monitoring of the milk adulterants as routine. In this context, it is extremely important to develop and validate new methodologies for monitoring the milk on large scale, with quick and low cost analytical methods. Thus, this study aimed to validate a compact equipment (MilkoScan FTI – FOSS ANALYTICAL), which adopts Fourier Transform Infrared Spectroscopy (FTIR) to monitor adulteration in raw milk. To achieve this objective, firstly, the MilkoScan equipment was calibrated with 2497 reference samples. After that, the validation process was done using five adulterants in three different concentrations: starch (500 mg L⁻¹; 750 mg L⁻¹ and 1,000 mg L⁻¹); sodium bicarbonate (600 mg L⁻¹, 1,200 mg L⁻¹ and 1,800 mg L⁻¹); sodium citrate (500 mg L⁻¹; 750 mg L⁻¹ and 1,000 mg L⁻¹); formaldehyde (150 mg L⁻¹, 300 mg L⁻¹ and 450 mg L⁻¹); sucrose (250 mg L⁻¹, 500 mg L⁻¹ and 750 mg L⁻¹), with the addition of two water levels (3% or 6%) or two levels of whey (3% or 6%). Twelve calibrations have been developed for the reference spectrum. To the validation process, different numbers of factors in the statistical model (8, 10, 12, 14, 16 and 18 factors, with unique or double elimination of outliers) were considered. To evaluate the calibrations which shown the best performance in adulterant detection in milk, we applied the Kruskal-Wallis ranking test followed by Dunn's test for multiple comparison of averages (α = 0.05). As results, after the development of calibrations, the equipment showed excellent sensitivity and specificity (84% and 100%, respectively) in the identification of adulterants in raw milk, confirming the possibility of its use directly in fraud detection in the dairy’s milk receiving platform, being possible to identify adulteration before the milk has been sold. Also, since the method utilized is non-specific, it may be possible to identify the fraud independent of which adulterant was used, unlike most of the conventional analytical tests that requires specific tests for each adulterant.

Keywords: adulteration, milk, FTIR, fraud, validation

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