



Matching portable NIRS instruments for *in situ* monitoring indicators of milk composition



Begoña de la Roza-Delgado ^{a,*}, Ana Garrido-Varo ^b, Ana Soldado ^a,
Amelia González Arrojo ^{a,1}, María Cuevas Valdés ^a, Francisco Maroto ^b,
Dolores Pérez-Marín ^b

^a Nutrition Research Programme, Regional Institute for Research and Agro-Food Development (SERIDA), PO Box 13, 33300 Villaviciosa, Spain

^b Faculty of Agriculture and Forestry Engineering, Department of Animal Production, University of Cordoba, Campus Rabanales, Ctra. Nacional IV – Km 396, 14071 Cordoba, Spain

ARTICLE INFO

Article history:

Received 18 October 2016

Received in revised form

9 January 2017

Accepted 10 January 2017

Available online 11 January 2017

Keywords:

MEMS-NIR

Raw milk

In-situ NIRS analysis

Standardization

Calibration transfer

ABSTRACT

The real time knowledge of dairy milk composition can be used as a tool to guarantee milk quality and safety, offering additional information for dairy producers and consumers. To carry out these *in situ* analyses, methodologies based on Near Infrared (NIR) portable sensors have a great potential as an advisory tool. The main goals of the present work have been to develop a methodology using a hand-held portable NIR spectrophotometer to collect raw milk spectra, including the development of calibration models for the analysis of protein, fat and solids-non-fat (SNF) of raw milk and further to transfer the developed models to another portable unit. A total of 542 fresh milk samples were scanned over the NIR spectral range (1600–2400 nm) using a hand-held MicroPhazir™ (MP) NIR spectrometer and different instrumental configurations. The best results for repeatability and reproducibility calculated as root mean squared (RMS) were obtained using a 17 mm cuvette thickness. The displayed predictive ability of calibration models measured as Standard error of prediction/Standard error of cross validation were 0.96; 0.72 and 0.83 for fat, protein and SNF contents, respectively. For cloning purposes an additional MP unit (satellite) has been used. A standardization set of 10 samples enabled standardization of both instruments. After applying standardization matrix, Standard error of differences between master and satellite reached great reduction, 68% for fat, 66% for protein and 54% for SNF. Moreover, the demonstrated ability of sharing calibration models among several units is essential for implementation of portable instruments for in-situ analysis to provide indicators of milk composition at farm level.

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1. Introduction

In the near future more and more dairy farms will uptake sophisticated Precision Livestock Farming (PLF) by sensors systems to support farm management. PLF is a combination of developing animal sensing (sensors) tools and decision-making process at the farm level. These precision systems include an instantaneous knowledge of dairy milk composition; this information can be used as a tool to guarantee milk quality and safety. It also has the potential to support animal feed suppliers, human-food retailers and

other players along the supply chain to make better choices. The current challenge for PLF is the integration of the technology in the farm but not only to the pioneering farms (Halachmi, 2015). Banhazi, Babinszky, Halas, and Tschärke (2012) outlined the potential role that PLF can play in ensuring that the best possible management processes are implemented on livestock farms increasing farm profitability and quality of milk products for consumers.

A new, alternative model for labour-efficient dairy production is emerging. Part of this trend in automation, robotic milking - an example of Precision Dairy Management (PDM) - reduces labour requirements and minimize food safety risks (Rodenburg, 2012; Bewley et al., 2015). However, in order to fully exploit the potential of this changing trend in dairy management, specific technologies should be considered together with the most widespread as,

* Corresponding author.

E-mail address: broza@serida.org (B. de la Roza-Delgado).

¹ Present Address: CETEMAS (Forest and Wood Technology Research Center), Pumarabule, Carbayín, s/n, 33936 Asturias, Spain.

Abbreviations

FNS	Foss NIRSystem 6500 monochromator	PLF	Precision Livestock Farming
FTIR	Fourier Transform Infrared	R^2_{cv}	Coefficient of Determination in Cross-Validation
GH	Global H	RMS(C)	Root Mean Square of Differences Corrected for the Bias
INIA	National Institute for Agricultural and Food Research	SD	Standard Deviation
MBM	Meat and Bone Meal	SECV	Standard Error of Cross-Validation
MEMS	Micro-Electro-Mechanical System	SED	Standard Error of Difference
MP	Microphazir™ NIRS Instrument	SEP	Standard Error of Prediction
MPLS	Modified Partial Least Square	SERIDA	Regional Institute for Research and Agro-food Development
MP-SERIDA	Microphazir™ NIRS Instrument- Regional Institute for Research and Agro-Food Development	SNF	Solids-Non-Fat
MP-UCO	Microphazir™ NIRS Instrument- University of Cordoba	SNV	Standard Normal Variate
NH	Neighbor Distance	SNVD	Standard Normal Variate plus Detrend
NIRS	Near Infrared Reflectance Spectroscopy	st1	Cloning set comprising 1 sample (the sample closest to the center of the population)
PDF	Precision Dairy Feeding	st10	Cloning set comprising 10 samples
PDM	Precision Dairy Management	TMR	Total Mixed Ration
		UCO	University of Cordoba

electronic radio frequency identification systems, robotic milking and calf-feeding systems, cameras, microphones, etc. These technologies allow control with precision as feed quality as the final product, milk, which could include under the term of Precision Dairy Feeding, (PDF). Taking into account that feed cost represents the most significant item of the total costs in milk production, and that in recent years, the volatility of the prices of cereals and flour protein, has been recurrent in world markets, it makes necessary to use alternative rations, as far as possible, trying to introduce raw materials of low cost, and the greatest possible use of local resources and by-products, often based on a total mixed ration (TMR) that combines all ration ingredients into a single feed mix. This complicates the nutritionist roles, who must formulate rations with many raw materials, even with nutritional value and composition little known to them, maintaining quality and assessing milk safety. This situation of fragility of the dairy sector at the global level is causing, innovative nutritionists to look for alternatives such as NIRS instruments to be used as a necessary tool in PDF. There are numerous works in the NIR literature applying NIRS technology to milk analysis (reviewed by Holroyd, 2013). They have shown that it is possible to obtain high or moderate accuracy and precision in calibration models to predict the main chemical constituents. Papers dealing with the application of NIR to liquid milk can be split into several areas that involve; the determination of milk composition, authentication of cow feeding regimes and geographic origin of milk, including milk classification, calibration robustness, industrial applications and the measurement of milk microbiological content.

A high percentage of water content in samples to analyze could interfere with NIRS analyses. Water content in fresh milk is one of the major contributors to the variation in the NIR spectra due to the strong absorption bands of O–H groups in the NIR region, which can create a critical interference in quantitative analysis. Most of the research milk works are carried out using homogenized and dried samples (DESIR method) (Núñez-Sánchez et al., 2016).

The use of NIRS technology on-farm, for the analysis of forage and TMR has been demonstrated scientifically and there are some commercial solutions developed, such as a NIR Analyzer installed directly on the self-propelled mixer wagon or in the shovel of the front loader. It is able to predict dry matter for each ingredient during the loading phase recalculating automatically the quantity to load to maintain a consistent ration (<https://www.dinamicagenerale.com/Media/Default/Catalogues/>

[PrecisionFeeding-ENG-LOW.pdf](#), Dimamica Generale, 2015). However, research about the employment of portable NIRS sensors, susceptible to use for the on-site control of milk obtaining information on individual cow state is very limited or almost non-existent (Kawasaki et al., 2008; dos Santos, Lopo, Páscoa, & Lopes, 2013). Therefore, it is urgent and important, to get scientific information about the potential of portable NIRS instruments for the analysis of raw milk, existing currently in the market.

The challenge facing this applied research is that the instruments more consolidated in the market, are not designed for this specific purpose of analyzing complex liquids such as milk. In terms of spectral characteristics and physico-chemical properties, it is necessary to show their adaptation and feasibility for the analysis of quality of raw milk.

The main goals of the present work are to develop a new methodology based on use of hand-held portable NIR spectrophotometer for the analysis of fat, protein and solids-non-fat (SNF) in raw milk. Further we will evaluate the transferability of the developed methodology and calibration models to a second portable NIRS unit. Finally we will study the alternative of sharing prediction models among several units as essential tool for implementation of portable NIR instruments for in-situ analysis to provide indicators of milk composition at farm level.

2. Material and methods

2.1. NIR instruments and analysis methods

- 1) A Foss NIRSystem 6500 monochromator (FNS). This is an at-lab instrument, working in a wavelength range between 400 and 2500 nm, equipped with transport module under controlled environmental conditions (temperature $24\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$, relative humidity $50\% \pm 10\%$). This instrument was used as a qualitative reference instrument to optimize sampling strategy and to evaluate the loss of spectra performance using portable instrument with small scanning window and narrow wavelength range. Spectra were collected using a liquid opaque quartz cuvette, reusable, with a 17 mm pathlength (C17) and an aluminum backside (FOSS. Ref US-ISIH-0398) for trans-reflectance measurements, combining reflectance and transmittance together into a single mode. The spectra data were recorded in reflectance mode ($\log 1/R$) with ISI scan software (Infrasoft International Inc., Port Matilda, PA, USA). Each sample

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