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Feasibility of near-infrared spectroscopy to predict a_w and moisture and NaCl contents of fermented pork sausages

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ABSTRACT

The feasibility of near-infrared spectroscopy (NIR) for predicting parameters related to the drying process of fermented sausages (water activity (a_w), moisture, and NaCl contents) was assessed. A FT-NIR spectrometer and two spectra acquisition setups with contact and remote probes were tested. NIR calibration models were developed using 207 samples scanned between 12,000 and 4000 cm^{-1} (833–2500 nm) in reflectance mode. Partial least squares (PLS) regression was used to process spectra and develop calibrations. Predictive models for moisture, a_w and NaCl yielded 0.997, 0.988 and 0.974 determination coefficients in prediction (R^2_p) respectively with the on-contact probe method, and 0.998, 0.985 and 0.974 respectively with the remote measurement head method. Furthermore 0.675, 0.006 and 0.117 root mean square errors of prediction (RMSEP) were achieved with the contact probe method, and 0.622, 0.007 and 0.116 with the remote measurement head method. The results confirmed that NIR spectroscopy is an useful technique for predicting moisture and NaCl and suggest it could also be useful to predict a_w on the surface of fermented sausages. Both setups are appropriate for further on-line applications for monitoring drying processes in a non-destructive way with non-significant differences in the predictive accuracy.

1. Introduction

The quality of fermented sausages is related to both their composition and the manufacturing process. In order to produce fermented sausages of high quality it is important to maintain strict control during the drying and ripening processes. Deficient control during the drying process could result in texture problems such as crust formation (Ordóñez & de la Hoz, 2001). Several studies have related these problems with certain chemical parameters such as superficial water activity and moisture, and NaCl contents (Arnau, 1998; Ruiz-Ramírez, Arnau, Serra, & Gou, 2005; Serra, Ruiz-Ramírez, Arnau, & Gou, 2005). The on-line monitoring of water content and a_w at the surface of the product would be an useful technique to prevent crusting (Ruiz-Ramírez, Serra, Arnau, & Gou, 2005).

Near-infrared spectroscopy (NIR) is a rapid, low cost and non-destructive method of analysis widely used to predict the composition of meat samples (Kruggel, Field, Riley, Radloff, & Horton, 1981; Lanza, 1983) using fibre optic technology (Mitsumoto, Maeda, Mitsuhashi, & Ozawa, 1991) and on-line determinations (Isaksson, Nilsen, Tøgersen, Hammond, & Hildrum, 1996; Tøgersen, Isaksson, Nilsen, Bakker, & Hildrum, 1999). NIR spectrum has its origin in the different vibration modes of the molecules which are caused by their interaction with electromagnetic radiation absorbed at wavelengths between 750 and 2500 nm. The use of chemometrics allows the relevant information contained in the NIR spectra to be extracted and used in the development of calibration models that permit the prediction of the composition of meat samples.

Applications of this technique in the meat industry have been recently reviewed (Huang, Yu, Xu, & Ying, 2008; Prevolnik, Čandek-Potokar, & Škorjanc, 2004), revealing that NIR has the potential of predicting different attributes of meat quality quickly and accurately. On pork, several studies have been performed relating to the application of NIR spectroscopy to predict composition (moisture, fat and protein) of raw meat (Brøndum et al., 2000; González-Martín, González-Pérez, Hernández-Méndez, Alvarez-García, & Hernández-Andaluz, 2002) and of pork products such as fermented sausages (Gaitán-Jurado, Ortiz-Somovilla, España-España, Pérez-Aparicio, & De Pedro-Sanz, 2008; Ortiz-Somovilla, España-España, Gaitán-Jurado, Pérez-Aparicio, & De Pedro-Sanz, 2007). NIR spectroscopy has also been used to determine NaCl in pork sausages (Begley, Lanza, Norris, & Hruschka, 1984; Ellekjær, Hildrum, Næs, & Isaksson, 1993). To our knowledge, there are no reports in which NIRS has been used to determine a_w in any food product or superficial moisture and NaCl contents in fermented sausages.

The overall aim of the present work was to evaluate the feasibility of NIR spectroscopy to predict water activity, moisture and NaCl

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contents in the surface of fermented sausages during processing. Different spectra acquisition setups based on the use of two different light fibre technologies were evaluated.

2. Materials and methods

2.1. Sausage samples

In order to obtain samples with a wide range of variability, a trial with three sources of variation was designed (salt content, air relative humidity (RH) inside the drying chamber and sampling day). Sausages with three NaCl contents (10, 20 and 30 g/kg) were manufactured. Shoulders and bellies (70:30) were ground by passing them through a 6 mm plate. Ground meat was subsequently mixed for 3 min at 0 °C in a vacuum mixer with the starter (AF3 Saga, Kerry Bio-Science Americas, Hoffman Estates, USA) (0.25 g/kg), water (25 g/kg), sodium nitrite (0.15 g/kg), potassium nitrate (0.15 g/kg), sodium ascorbate (0.5 g/kg), dextrose (7 g/kg), black pepper (3 g/kg) and NaCl in the three different proportions mentioned above. The mixtures were stuffed into 80 mm diameter collagen casings (Fibran, Fibran S.A., Sant Joan de les Abadesses, Spain) and were then hung in a chamber for fermentation at 25 °C and 90–95% RH for 2 days until the pH decreased below 5.0. Thereafter, sausages from each salting treatment were divided into three groups and dried in three different drying chambers at 12 ± 0.2 °C, with an air velocity of 0.5 ± 0.3 m/s, at different relative humidities (low RH: 72.0 ± 2.2% for the first 42 days and 58.6 ± 3.4% for the 24 resting days; medium RH: 78.5 ± 2.7% for the first 42 days and 66.6 ± 2.8% for the 24 resting days; high RH: 90.8 ± 3.3% for the first 42 days and 85.5 ± 2.6% for the 24 resting days).

In order to obtain a representative set of samples, two sampling days during fermentation and seven sampling days during the drying process were selected, producing a total of 207 samples (3 NaCl contents × 2 fermentation sampling days × 3 sausages) + (3 NaCl contents × 3 drying conditions × 7 drying sampling days × 3 sausages). Surface samples (2 mm depth) were sliced lengthways, vacuum-packed and frozen at −20 °C. After thawing (12 h at 3 ± 1 °C), the samples were minced and homogenised using a commercial grinder, NIR scanned and chemically analysed.

To develop and validate the regression models, the samples were divided into two sets, a calibration set for modelling and a set for validation of the developed models. To define the two groups, the samples were arranged according to the experimental value of each parameter, and subsequently distributed alternately, one for calibration and another for validation. In this way, both sets were the same size and covered the whole range of chemical data.

2.2. Spectra acquisition

Spectra were recorded on a Fourier Transform NIR spectrometer model Matrix-F duplex (Bruker Optik GmbH, Germany). NIR spectra were collected using the OPUS™ software (Bruker Optik GmbH, Germany).

NIR reflectance spectra were collected over the 12,000–4000 cm^{-1} spectral region (corresponding to a wavelength interval of 830–2500 nm) using two spectral acquisition setups based on two light fibre optic probes (Fig. 1).

An on-contact probe IN 268–2 (Solvias AG, Switzerland) was used. This probe uses a bifurcated (Y-shaped) fibre optic bundle to illuminate the sample (four fibres input) and to collect the diffuse reflection (four fibres output). The measured spot size is 3 mm ø. Using this probe, spectra were acquired on-contact with the sample, moving the sample container at the same time as the scans were performed. Each spectrum was obtained from 32 scans performed at 16 cm^{-1} resolution.

A remote measurement head Q410/A Nema 4 (Bruker Optik GmbH, Germany) was also used. This system has an external illuminating source composed of four tungsten sources which illuminate the sample; the diffuse reflection is collected and guided via a four fibre optic cable to the spectrometer. The measured spot size was 10 mm ø. With this contactless system, spectra were taken with the head fixed at 17 cm from the sample. Each spectrum was obtained from 32 scans performed at a wavenumber resolution of 8 cm^{-1}.

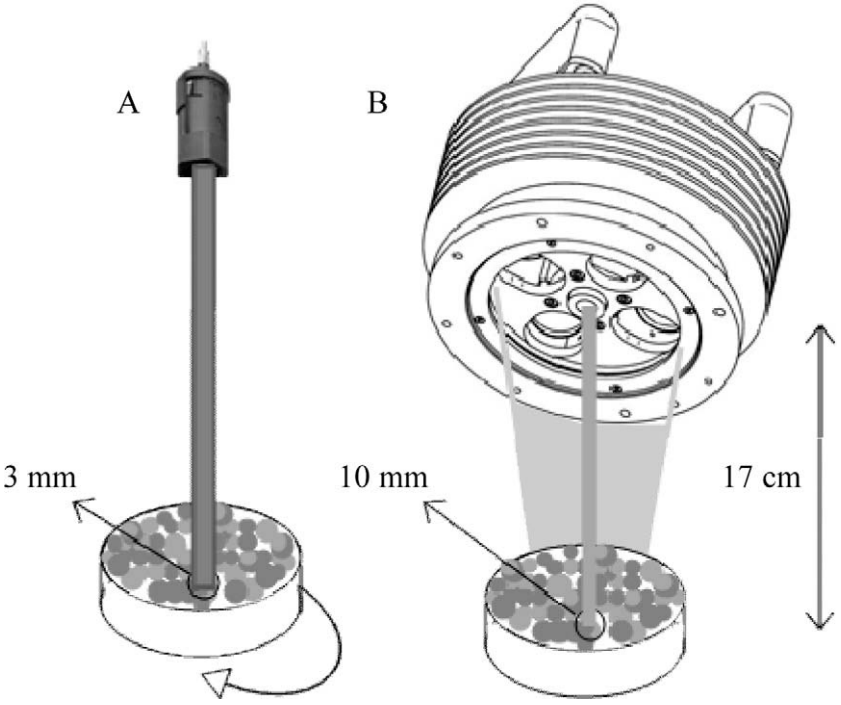

Fig. 1. Spectral acquisition setups. (A) On-contact probe IN 268–2 "movement". (B) Remote measurement head Q410/A "fixed".