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Non-destructive analysis of fresh Iberian pork loins by near-infrared spectroscopy (NIRS)

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Abstract. A fiber-optic contact probe near infrared spectroscopy (NIRS) instrument is evaluated to predict fat, moisture and protein in intact Iberian pork loins. A total of 173 Iberian pig loin samples were analyzed. Spectra from intact loins were collected with a LabSpec®Pro A108310 spectrometer (Analytical Spectral Device) and from the ground meat with a FossNIRSystem instrument. Spectra attenuation was required for removing very signal noise areas in the intact loin spectra. As reference values were used the NIRS predictions of the sample set estimated with a robust calibration model for ground samples and analyzed in a FossNIRSystem instrument. Modified Partial Least Squared (MPLS) regression was performed and different spectra pretreatments and spectral regions evaluated. The range 450-2300 nm performed the best models for the instrument evaluated, showing a suitable potential of the NIRS instrument for on-line analysis of pork loins.

Keywords. Iberian pigs – Loins – NIR Spectroscopy – Fiber optic – Fat – Protein – Moisture.

Analyse non destructive d'échines fraîches de porc Ibérique au moyen de spectrospie à infrarouge proche (NIRS)

Résumé. *Un équipement spectroscopique à infrarouge proche (NIRS) doté d'une sonde de fibre optique de contact est évalué pour la prédiction de la graisse, de l'humidité et de la protéine dans des échines intactes de porcs Ibériques. Un total de 173 échantillons d'échines de porcs Ibériques ont été analysés. Les spectres ont eu besoin d'être atténués pour éliminer des aires ayant un signal bruyant. Comme valeurs de référence on a utilisé les prédictions NIRS de l'ensemble des échantillons estimées avec un modèle robuste de calibrage créé pour des échantillons hachés et analysés par un instrument FossNIRSystem. L'algorithme de régression des Moindres Carrés Partiels Modifiés (MPLS) a été utilisé pour évaluer différents pré-traitements et régions spectrales. L'intervalle 450-2300 nm a fourni les meilleurs modèles pour l'instrument évalué, en montrant le potentiel d'analyse en ligne de l'instrument NIRS pour l'échine de porc.*

Mots-clés. *Porcs Ibériques – Rein – Spectroscopie NIR – Fibre optique – Matières grasses – Protéines – Humidité.*

I – Introduction

Iberian pig products have a high rate consumer acceptance, leading to high prices in the market. Consumers assess the quality of those products based on the exceptional organoleptic, healthy and sensory characteristics, but guarantee a target meat product is a complex task that requires control procedures (Prieto *et al.*, 2009a). Fresh meat is a very heterogeneous product and the determination of the major chemical constituents, such as fat, moisture or protein, is interesting for labelling purposes or for preparing good mixtures to produce dry-cured or processed products.

Traditional wet chemistry to determine these parameters are time-consuming, tedious, costly and destructive methods. Nowadays, the meat industry has shown a great interest for new

technologies that enables fast, accurate, non-destructive quality analysis. Near-Infrared Spectroscopy (NIRS) has shown its potential to predict chemical composition in meat products (Prevolnik *et al.*, 2004; Prieto *et al.*, 2009a), even in on-line applications in the industry (Tøgersen *et al.*, 1999; González-Martín *et al*., 2002; Huang *et al*., 2008; Prieto *et al*., 2009b) resolving part of the demands of the industry. Most of the literature has used ground samples which require a sample preparation. In this study is evaluated a fiber-optic contact probe NIRS instrument to predict chemical composition (fat, moisture and protein) of intact Iberian pork loins.

II – Materials and methods

1. Sample set and NIRS measurements

One hundred and seventy three samples of Iberian pork loins were analyzed. The samples were taken from the beginning of the loin in the shoulder area of the animals. Two NIRS analyses were performed with different instruments and samples presentation.

A post-dispersive diode array scanning monochromator spectrometer LabSpec®Pro A108310 (Analytical Spectral Device-ASD Inc., Boulder, Colorado, USA) working from 350-2500 nm (1nm spectral resolution) in reflectance mode was used to analyze intact pork loin samples at the slaughter house, two hours post-mortem. The loins after being analyzed with the LabSpec®Pro were vacuum packaging and frozen at -20ºC to be stored. A FossNIRSystem 6500 equipped with a spinning module for standard circular cups working from 400-2500 nm (2 nm spectral resolution) in reflectance mode was used to analyze the same samples set in ground presentation. Before recording the NIRS spectra, the muscles were ground and homogenized by a vertical cutter mixer (Heidolp homogenizer DIAX 900). Two spectra per sample were measured in each case.

2. Data modelling

Chemometric data treatment was performed using the software WinISI II ver 1.50 (Infrasoft International, Port Matilda, PA, USA). The Root Mean Squared (RMS) error statistic was used for spectral repeatability evaluation (Shenk and Westerhaus, 1995). The fat, moisture and protein composition of each sample was determined by NIRS using a robust model developed for ground Iberian meat in the range 1100-2500 nm (not published). These predictions were used as reference for further analysis.

Modified Partial Least Squared (MPLS) regression method (Shenk and Westerhaus, 1991) was used to develop calibration models for predicting fat, moisture and protein with intact pork loin samples analyzed with the fiber optic contact probe instrument. As spectral pre-treatments, Standard Normal Variate (SNV) plus Detrending (DT) (Barnes *et al.*, 1989) was used to remove the multiplicative interferences of scatter and two derivative mathematical treatments were performed: window-wise filtering (1,10,5,1) and (2,5,5,1) (ISI, 2000). The optimum number of model factors was selected by cross-validation using 4 groups.

Signal noise at the beginning and end of the spectral range was eliminated for the LabSpec®Pro spectra measurements. Two spectral regions were selected for performing the calibration models: 450-2300 nm and 1100-2300 nm.

The evaluation of the models was performed with different statistics: the standard error of calibration (SEC), the standard error of cross-validation (SECV) and the coefficient of determination for cross-validation (r^2).

III – Results

The collection of high-quality spectra is a crucial task to develop accurate calibration models. NIRS spectral repeatability using the RMS statistic can help to evaluate the quality of the spectra. In the case of the LabSpec®Pro, the RMS values exceeding the 90,000 µlog 1/R for comparisons of spectra form the same sample and location across the whole spectral range (350-2500 nm). By removing spectral areas with a very signal noise, i.e at the beginning (350-450 nm) and the end (2300-2500 nm) of the spectra, the RMS values were lowered to 86,115 µlog 1/R. Figure 1 shows the spectra collected from intact loins with the fiber-optic contact probe and it is observed the high signal noise over 2000nm. In the case of the FossNIRSystem, the RMS values are on the average of 9.941 µlog 1/R.

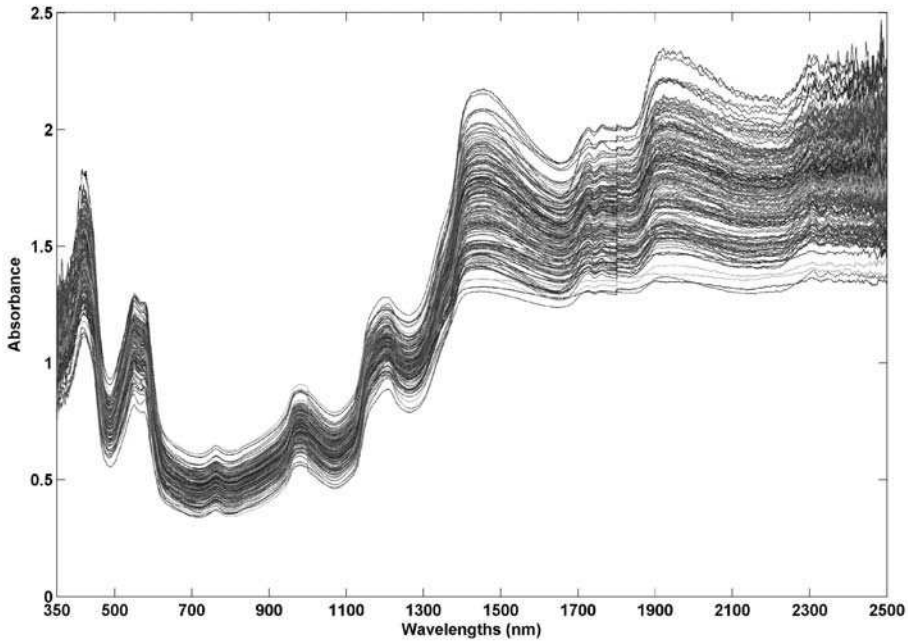

Fig. 1. Spectra collected from intact pork loins using a fiber-optic NIRS instrument from LabSpec®Pro.

Figure 2 shows the average spectra of the samples set for the analysis performed for intact pork loins with LabSpec®Pro and for ground pork meat with the FossNIRSystem instrument in the 400-2300 nm range. It is observed strong similarities between both, although the quality of the spectra is different with a smoother pattern in the case of the ground meat, due to the sample presentation and instrument differences. The FossNIRSystem is a laboratory instrument working under controlled-conditions, while the LabSpec®Pro was working on-line in the industry. Ground samples presented a more clearly-defined spectrum with sharper peaks than the intact loins. However, both show different characteristic absorption peaks: around 1200, 1720-1760nm as areas of fat and water-related peaks at 1450 and 1940 nm (Williams and Norris, 1987; Osborne, Fearn and Hindle, 1993).

The spectra of the ground samples were used to predict fat, moisture and protein composition of the Iberian pork loins with a robust model developed for the FossNIRSystem working in the

1100-2500 nm range (not published) over different years and with different ground pork muscles in our research group. Table 1 shows the statistic of that model. The prediction obtained for each sample was used as reference value for developing calibration models for the determination of fat, moisture and protein composition in intact loins using the fiber-optic instrument.

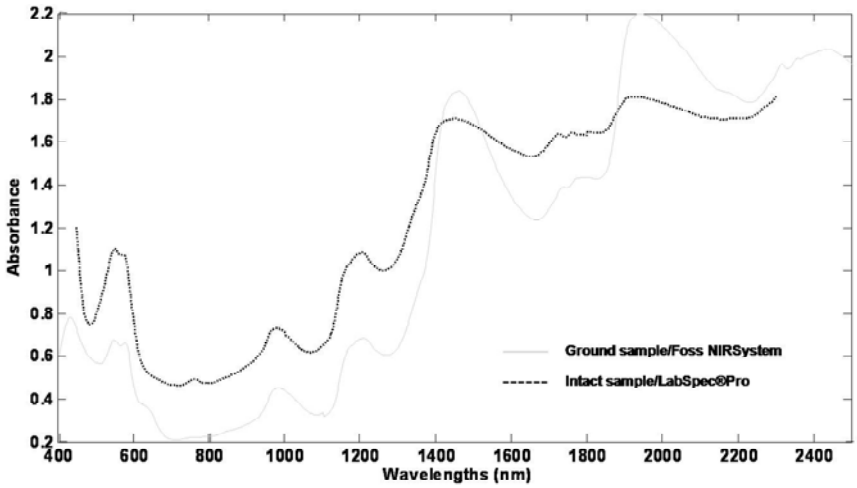

Fig. 2. Average spectra collected with the different instruments and samples presentation.

Table 1. Calibration statistics for the NIRS prediction using ground Iberian meat samples of fat, moisture and protein composition

Parameter	Pre-treatment	No. of samples	Nº of Principal Components	SEC (%)	r^2	SECV (%)
Fat	SNV + DT (2,5,5,1)	315	4	0.37	0.98	0.40
Moisture	SNV + DT (2,5,5,1)	315	5	0.47	0.96	0.51
Protein	SNV + DT (1,10,5,1)	324	7	0.48	0.91	0.49

Table 2 shows the statistics for the different parameters (fat, moisture and protein) studied of the samples set predicted by NIRS using the Table 1 models. Modified Partial Least Squared (MPLS) algorithm was used to develop calibration models for the prediction of the major chemical composition parameter in intact Iberian pork loins with a remote NIR reflectance fiber-optic contact probe instrument.

Table 2. Calibration statistics for the NIRS prediction using ground Iberian meat samples of fat, moisture and protein composition

	Training set (173 samples)			
Parameter	**Minimum**	**Maximum**	**Average**	**Standard Deviation**
Fat (%)	3.20	18.95	8.69	2.90
Moisture (%)	63.20	74.97	70.29	2.08
Protein (%)	17.16	21.95	19.68	0.90

The best calibration models obtained for predicting the three main chemical parameter in meat, measured with the fiber-optic contact probe in intact Iberian pork loin, are shown in Table 3 (i.e. after testing second and first derivative for two spectral range: 450-2300 nm and 1100-2300 nm).

Table 3. Calibration statistics of fat, moisture and protein composition obtained for the NIRS prediction with a remote fiber-optic probe using intact Iberian loin samples

Parameter	Range (nm)	Pre-treatment	No. of samples	No.º of Principal Components	SEC (%)	r^2	SECV (%)
Fat	450-2300	SNV + DT (1,10,5,1)	173	4	1.42	0.68	1.65
Moisture	450-2300	SNV + DT (2,5,5,1)	168	4	0.83	0.59	1.25
Protein	450-2300	SNV + DT (1,10,5,1)	171	4	0.51	0.56	0.59

As expected, models developed using intact loins are less accurate (Table 3) than those obtained with ground samples (Table 2). Intact loins showed larger sample heterogeneity than ground samples; freezing/drawing of the ground samples can affect mainly the moisture parameter and the quality of the spectra is different between instruments and sample presentation. Nevertheless, the models showed the possible potential of a fiber-optic contact probe NIRS instrument for analyzing chemical composition of intact pork loins. Moreover, an on-line analysis enables instantaneous and without sample presentation measurement providing important industrial advantages for quality and process control. However, there are several topics that require further studies such as the sampling optimization of intact meat products or the choice of the best instrument measurement parameters for an adequate spectra collection.

IV – Conclusions

The on-line NIRS instrument evaluated in this study for the quantitative chemical composition prediction of intact Iberian pig loins shows a very promising result enabling several practical advantages of the analysis. Further work is required in order to develop more accurate models.

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References

Barnes R.J., Dhanoa M.S. and Lister S.J., 1989. Standard normal variate transformation and detrending of near infrared diffuse reflectance spectra. In: *Applied Spectroscopy*, 43: 772-777.

González-Martín I., González-Pérez C., Hernández-Méndez J., Alvarez-García N. and Hernández-Andaluz J.L., 2002. On-line non-destructive determination of proteins and infiltration fat in Iberian pork loin by near infrared spectrometry with a remote reflectance fiber optic probe. In: *Analytica Chimica Acta*, 453: 281-288.

Huang H., Yu H., Xu H. and Ying Y., 2008. Near Infrared spectroscopy for on/in-line monitoring of quality in foods and beverages: a review. In: *Journal of Food Engineering*, 87: 303-313

ISI, 2000. *The complete software solution using a single screen for routine analysis, robust calibrations and networking*. Infrasoft International Sylver Spring MD, USA.

Osborne B.G., Fearn T. and Hindle P.H., 1993. Practical NIR spectroscopy with applications in food and beverage analysis. In: *Food and Beverage Analysis*. (pp.11-35). B.G. Osborne, T. Fearn and P.H. Hindle (Eds*.)*. Longmand, Essex.

Prevolnik M., Candek-Potokar M. and Skorjanc D., 2004. Ability of NIR spectroscopy to predict meat chemical composition and quality - a review. In: *Czech Journal of Animal Science,* 49 (11): 500-510

Prieto N., Roehe R., Lavín P., Batten G. and Andrés S.. 2009a. Application of near infrared reflectance spectroscopy to predict meat and meat products quality: A review. In: *Meat Science*, 83: 175-186

Prieto N., Ross D.W., Navajas E.A., Nute G.R., Richardson R.I., Hyslop J.J., Simm G. and Roehe R., 2009b. On-line application of visible and near infrared reflectance spectroscopy to predict chemical-physical and sensory characteristics of beef quality. In: *Meat Science*, 83: 96-103.

Shenk J.S. and Westerhaus M.O., 1991. Population structuring of near infrared spectra and modified partial least squares regression. In: *Crop Science*, 31: 1548-1555.

Shenk J.S. and Westerhaus M.O., 1995. *Analysis of agriculture and food products by near infrared reflectance spectroscopy*. Monograph, NIRSystems.

Tøgersen G., Isaksson T., Nilsen B.N., Bakker E.A. and Hildrum K.I., 1999. On-line NIR analysis of fat, water and protein in industrial scale ground meat batches. In: *Meat Science*, 51: 97-102.

Williams P.C. and Norris K.H. (Eds.). 1987. *Near-infrared technology in the agricultural and food industries*. St. Paul, MN: American Association of Cereal Chemists.