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Use of visible and near infrared reflectance spectra to predict lipid peroxidation of light lamb meat and discriminate dam's feeding systems

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Abstract

Measurement of thiobarbituric acid reactive substances (TBARS) is a well-established method for determine lipid oxidation in meat. This assay, however, is time-consuming and generates undesired chemical waste. Dam's milk is the principal source of vitamins and provitamins that delay lipid oxidation of light lamb meat; these compounds are stored in the lamb's muscle tissue. Hence, lamb meat could be used to determine the origin of the dam's diet. The aim of this study is to evaluate Near-infrared reflectance spectroscopy (NIRS) as a tool for determining the lipid peroxidation of light lamb meat and differentiate the meat of light lambs according the diet of their dams during lactation (grazing alfalfa, lucerne, or fed a total mixed ration). NIRS using select wavelengths was able to detect the lipid oxidation of meat (TBARS method). NIRS can detect analytes at concentrations of parts per million. Moreover, the feed diets were discriminated successfully.

Keywords: NIRS; TBARS; VIS; diet; lactation; malondialdehyde

1. Introduction

Near-infrared spectroscopy has been used for many years in industry due to its recognized advantages as a rapid, clean and accurate tool. The most important application of near-infrared spectroscopy is the measurement of the chemical composition of raw materials in the agri-food industry. The dairy industry routinely uses infrared spectroscopy to analyze the chemical composition of milk. This technology is also widely used in the grain and livestock feed industry (Fernández-Ahumada, et al., 2013; Garrido-Varo, et al., 2005). Near-Infrared Reflectance Spectroscopy (NIRS) has also been used to analyze the chemical composition of meats (Denoyelle & Cartier, 1996; Olivan, de la Roza, Mocha, & Martinez, 2002; Prieto, Andres, Giraldez, Mantecon, & Lavin, 2006). Applications of NIRS to other meat quality characteristics are less well-developed. Even instrumental texture and sensory tenderness have been studied to determine if NIRS can be used as a reliable predictor, although predictions thus far have been inconsistent (Hildrum, et al., 1995; Hildrum, Nilsen, Mielnik, & Naes, 1994; Liu, et al., 2003; Ripoll, Albertí, Panea, Olleta, & Sañudo, 2008a).

One of the most important variables governing meat shelf life is lipid peroxidation. The carbon-carbon double bonds of polyunsaturated fatty acids allow the formation of lipid free radicals by reactive species. This generates a chain reaction of more lipid hydroperoxides and more lipid free radicals. Breakdown of lipid hydroperoxides produces malondialdehyde (Grotto, et al., 2009). In fact, thiobarbituric reactive substances (TBARS) are the standard method for detecting meat lipid oxidation by quantifying the amount of malondialdehyde present. This method, as with many others, is time-consuming and generates undesirable chemical products. For these reasons, an NIRS-based method to detect lipid oxidation would be of interest. The main challenge for using NIRS to measure lipid oxidation is that the malondialdehyde content in meat is less than 10 ppm, while the detection limit of NIRS often is considered to be 1000 ppm (Cen & He, 2007; Pasquini, 2003; Skoog, Holler, & Nieman, 1998). Thus, there is no literature to date that uses NIRS to estimate lipid

oxidation in lamb meat. There are examples of studies successfully detecting lipid oxidation in rabbit meat using the visible (VIS) spectrum (Cifuni, Contò, & Failla, 2016), as well as lipid oxidation in frozen fish muscles (Karlsdottir, Arason, Kristinsson, & Sveinsdottir, 2014).

Lipid peroxidation of light lamb meat is determined by the balance between anti- and pro-oxidant substances, such as vitamin E and carotenoids, and polyunsaturated fatty acids, respectively. The presence of these substances and the composition of polyunsaturated fatty acids are often related to the feeding system of the light lamb. Thus, these substances can be used to trace the diet of lambs (for example, grazing vs. concentrates). Some authors have proposed the use of spectral methods to achieve this authentication (Dian, Andueza, Jestin, Prado, & Prache, 2008; Prache, Cornu, Berdague, & Priolo, 2005; Priolo, Lanza, Barbagallo, Finocchiaro, & Biondi, 2003; Priolo, Micol, Agabriel, Prache, & Dransfield, 2002; Ripoll, Alberti, & Joy, 2012; Ripoll, Joy, Muñoz, & Albertí, 2008), primarily using the visible reflectance spectrum of subcutaneous fat. Counter to this approach, in Mediterranean areas, weaned light lambs are often fed concentrate with low amounts of vitamins and carotenes, and this does not increase the polyunsaturated fatty acid content of their meat. This indicates that the principal source of vitamins and pro-vitamins in lambs comes from the dams' milk. Furthermore, these substances are stored in the muscle, suggesting that not only subcutaneous fat but also meat could be used to determine the origin of the dam's diet.

The aim of this study is to evaluate VIS-NIR reflectance spectroscopy as a tool for detecting lipid peroxidation of light lamb meat and discriminate meat of light lambs according the diet of their dams during lactation.

2. Material and methods

2.1 Experimental design and animal management

The 122 light lambs used in this study were all single reared male light lambs of the Rasa Aragonesa breed. Lambs were weaned at 45±1.8 days of age and fed with concentrates until slaughtering at a live weight of 22-24 kg.

Lambs came from two different experiments with slight management differences.

Experiment 1. Sixty lambs were fed with commercial concentrate after weaning and were slaughtered at a live weight of 23.0 kg ± 1.38.

Experiment 2. Sixty-two lambs were divided into three management groups during lactation:

- TMR (n=20), ewes and lambs were housed and received a total mixed ration (TMR) *ad libitum*.

- Sainfoin (n=21), ewes and lambs were rotationally grazed in sainfoin paddocks.

- Alfalfa (n=21), ewes and lambs were rotationally grazed in alfalfa paddocks.

After weaning, lambs were fed commercial concentrate and slaughtered at a live weight of 22.8 kg ± 1.19.

Lambs were slaughtered using standard commercial procedures, and carcasses were hung by the Achilles tendon and chilled 24 h at 4ºC in total darkness to preserve carotenoid pigments.

The Animal Ethics Committee of the Research Centre approved the experimental and slaughter procedures used in this study, which were performed in accordance with the European Union Directive 2010/63 on the protection of animals used for experimental and other scientific purposes (E.U., 2010).

2.2. Preparation of samples and packaging

After carcass chilling, M. *Longissimus thoracis et lumborum* (LTL) were extracted from the carcasses and sliced. The portion of loin from the 5th thoracic *vertebra* to the 6th lumbar *vertebra* was sliced into samples of 2.5 cm thickness. These samples were randomly assigned to trays and kept in darkness at 4ºC for 0, 2, 5, 7, 9, 12 and 14 d of storage; samples were then frozen at -20ºC until

collection of spectral data and TBARS analysis. A total of 632 samples were collected (Table 1).

2.3. Collection of NIR reflectance spectra

Samples for spectroscopy were thawed overnight at 6 ºC in darkness, and kept at room temperature 1 h before taking the spectra. The intermuscular fat covering of the LTL was eliminated, and the muscle was homogenized in a meat mincer Moulinex D-56 (Groupe SEB, France). The minced meat was inserted in a cylindrical cup with a quartz glass with an internal diameter of 35 mm and a depth of 10 mm. Reflectance (R) spectra were scanned and collected twice per sample with a NirSystems 6500 monochromator instrument (FOSS Iberia, S.A., Spain). Spectra were stored as $\log\left(\frac{1}{R}\right)$ and referenced to a ceramic disk (reference offset, $\log\left(\frac{1}{R}\right)$ = 0.09691). Immediately after spectral acquisition, minced samples were used for lipid oxidation analysis (TBARS).

2.4. Lipid oxidation analysis

LTL intramuscular lipid oxidation was measured with the procedure reported by Pfalzgraf, Frigg, & Steinhart (1995), following the protocol described by Ripoll, Gonzalez-Calvo, Molino, Calvo, and Joy (2013). The TBARS results are expressed in milligrams of malondialdehyde per kilogram of fresh meat.

2.5. Chemometrics and statistical analysis

The samples from both experiences were equally distributed to create the Calibration and the Validation sets. All samples from the same lamb were assigned together to one or another set. Calibration set joined 418 samples (66 %) from 82 lambs while Validation set joined 214 samples (34 %) from 40 lambs.

Mathematical treatments and pre-treatments such as scatter correction and derivatives, as well as gap and smooth segments, were investigated. Similarly, several spectral ranges and individual wavelengths were selected and tested to obtain the best calibration model. The results from the three best models are shown in results and discussed.

Because many of the data points in the spectrum were highly co-linear, they were compressed using only a few factors (Geesink, et al., 2003) to find the calibration equation. Compression was carried out using Partial Least Squares and Partial Component regressions. The performances of the different calibration equations obtained were determined from calibration and full cross validation. The root mean square errors (RMSEC and RMSCV), the coefficients of determination (R^2_c and R^2_{cv}) and the standard errors (SEC and SECV) of calibration and full cross validation, respectively; and the standard deviation of the laboratory (SD), the residual predictive value ($RPD = \frac{SD}{SEP}$) and the consistency ($C = \frac{SEC}{SEP} \cdot 100$) were used to test the accuracy of the calibration models (Barlocco, Vadell, Ballesteros, Galietta, & Cozzolino, 2006) and to choose the best model. A validation was performed with the best model and the root mean square errors of validation (RMSEP), the coefficients of determination validation (R^2_p), the standard errors of validation (SEP), the RPD and C were calculated.

Partial least squares discriminant analysis (PLS-DA) was performed to discriminate the lamb meat according to feeding during lactation using full cross validation. PLS-DA was applied as a classification method using dummy Y-variable values of 1 for the target category and 0 for the other categories being discriminated; applying this procedure separately to each category (TMR, Sainfoin, Alfalfa) thus produced three discriminant models. The results of PLS-DA were expressed as the percentage of correct classification events.

Chemometrics and spectral data management were carried out with Unscrambler X (Camo Software AS, Norway).

Statistical analysis of lipid oxidation was carried out using the MIXED procedure for repeated measures, based on Kenward-Roger's adjusted degrees of freedom solution using the SAS 9.3 statistical package (SAS Institute Inc., Cary, NC, USA) (SAS, 2002). The factors included were time of storage as a within-subject effect and a random animal effect as subject (experimental unit). The lowest Akaike Information Criterion (AIC) was used to choose the first-order autoregressive matrix of error structure. Least-square

means were estimated, and differences were tested with a t-test at P<0.05 level of significance.

3. Results and discussion

The statistics obtained for the lipid oxidation of samples used on calibration and validation sets are shown on Table 2. Both sets of samples are similar and ranged from close to zero to 3.6 mg of malonaldehyde per kg of meat. When a prediction equation has been developed, it is common practice to remove the outliers based on the Hotelling's T^2 or on the standardized Mahalanobis distance (H). However, studies usually do not give explanations about why these samples are out of the studied population. In the present study, some outliers were detected according Hotelling's T^2 at 95% confidence. However, we did not find evidence of failures in spectrum acquisition or determination of TBARS values. Hence, all samples were kept in the data set because they are representative of the meat from Rasa Aragonesa light lambs.

Figure 1 shows the evolution of TBARS according the time of display of meat. TBARS content increased as storage times became longer. The storage of meat over 14 d provides a wide variability in the reference values, which is essential for generating robust prediction models. Lipid oxidation at days 0 and 2 was similar (P>0.05). However, from day 2 on, lipid oxidation significantly incremented from day to day (P<0.05). Both the trend and the values of lipid oxidation are similar to those reported by several authors for light lambs of same breed, slaughtered at the same live weight, with similar feeding (Ripoll, et al., 2013; Ripoll, Joy, & Muñoz, 2011).

Mean spectra of each day of display are shown in Figure 2. As the time of display increases, the $\log\left(\frac{1}{R}\right)$ values through the whole spectrum decrease. A similar relation between the reflectance spectrum and TBARS has been reported for the NIR spectra of pork (Wu, Song, Qiu, & He, 2016) and the VIS spectra of rabbit (Cifuni, et al., 2016).

The three models that yielded the best statistics are shown in Table 3. These models were obtained using 1) the visible spectrum (VIS) from 400 nm to 700 nm; 2) a selection of wavelengths ranging from 400 nm to 1026 nm; and 3)

the whole spectrum (VIS-NIR) from 400 nm to 2500 nm. The optimal pretreatments of spectra were the same for the three models. Spectra were smoothed with a moving average, with a segment size of 3. Data were processed with a full multiplicative scatter correction (MSC) transformation to compensate for additive and/or multiplicative effects of spectral data. The most important quantitative factor in the NIR reflectance spectrum is the particle size of the sample, which is a multiplicative effect (Manley, McGill, & Osborne, 1994). Since log(1/R) coincidentally increases with higher wavelength in the reflectance spectra of foods and agricultural materials, the effect of particle size appears to be a function of wavelength (Osborne, 2000). In the present study, the log(1/R) of meat spectra is also a function of wavelength, and the MSC transformation improved the prediction model. The first-order Gap-Segment derivative (Hopkins, 2001) was applied with gap and segment sizes of 1. The optimal number of factors ranged from 6 to 9, controlling the risk of modelling spectral noise, which leads to overfitted models and bad predictions of unknown samples (Brereton, 2007). The VIS calibration model had slightly greater RMSEC and RMSECV and lower R^2 of both calibration and cross validation than the rest of the shown models. The calibration model using the selected wavelengths had similar RMSCV, R^2 of cross validation and RPD to the model using the whole VIS-NIR, but the former model had more consistency than the latter. Thus, the model developed with the selected wavelengths is considered the best.

Figure 3 shows the weighed regression coefficients of the model, with 8 factors, for the selected wavelengths. The greatest absolute values of the regression coefficients were found at 570 nm, 520 nm, 546 nm, 436 nm, 416 nm and the region from 600 to 722 nm. The latest factors (7 and 8) had high regression coefficients on the region from 924 nm to 930 nm. The former region of spectra (VIS) is associated with meat color and the latter with water loses. Malondialdehyde is a low molecular weight product formed by the decomposition of several lipid peroxidation products. Aldehydes absorb in the range from 2200 to 2700 nm (King & Vig, 1962); however, these bands are not used in the selected model. This is because the TBARS method quantifies the concentration of the MDA-TBA adduct (2 mol of TBA plus 1 mol of MDA). This

adduct is a cromatogen that absorbs mainly in the visible spectrum, and explain the importance of the VIS region. This phenomenon leads to some criticism of the TBARS method because other aldehydes and non-lipid materials present in biological samples such as meat may also form TBA adducts (Knight, Pieper, & McClellan, 1988). Thus, non-specific absorption bands could be related to these unknown adducts.

Figure 4 shows predicted vs. reference values of the regression model with the selected wavelengths applied to validation set. Reference values of TBARS are well distributed in the studied range, although there is a cluster of values close to zero. The RPD of the model was high (RPD=3.01) but the consistency of the model (C=88) was moderate. Sinnaeve, Dardenne, Agneessens, and Biston (1994) considered models adequate for analytical purposes if they had an RPD greater than 2.5 for forage calibrations. However, meat is a more complex matrix than dry forage because of the storage and preparation steps, as well as the higher water content. In that sense, Barlocco, et al. (2006) considered an RPD greater than 2 to be a good calibration for pork. Other authors also argue that 2 is an adequate limit for quantitative prediction and can be applied to meat and most agricultural materials (Mouazen, Saeys, Xing, De Baerdemaeker, & Ramon, 2005; Prieto, Roehe, Lavín, Batten, & Andrés, 2009; Williams & Sobering, 1996).

With regard to the dam feeding systems, Figure 5 shows the mean spectra of TMR, Alfalfa and Sainfoin treatments. The TMR spectrum had lower $\log\left(\frac{1}{R}\right)$ values than the other treatments. Meat spectra had the characteristic maximum in the Soret region at 420 nm (Moss, Millar, & Kilpatrick, 2000). They also showed local maxima at 540 nm and 580 nm, which result from oxymyoglobin's strong absorbance around those two wavelengths (Krzywicki, 1979). The NIR region is also typical, with an increase of $\log\left(\frac{1}{R}\right)$ until 1450 nm, followed by a local minimum at approximately 1650 nm and the greatest values from 1970 nm to 2500 nm; this is consistent with results from Ripoll, Albertí, Panea, Olleta, and Sañudo (2008b). Spectra of TMR had lower values of $\log\left(\frac{1}{R}\right)$, meaning that meat from lambs fed TMR was lighter than that from lambs fed grazing grass (Luciano, et al., 2009). The PLS-DA (Figure 6) correctly

classifies 97% of the TMR treatment, while Alfalfa and Sainfoin treatments were correctly distinguished in the 96% of all cases. The explained variance for each factor of the PLS-DA, which increases constantly until a plateau at 30 factors. The percentage of correctly classified TMR samples was higher than Alfalfa and Sainfoin from 1 to 18 factors. However, Alfalfa and Sainfoin were similarly classified regardless of the number of factors used in the PLS-DA.

Conclusions

Near-infrared spectroscopy is able to of estimate the lipid oxidation of light lamb meat, as measured using the TBARS method. NIRS is also able to detect analytes at concentrations of parts per million. Prediction models could be improved eliminating statistical outliers. However, the model could also be over fitted, leading to less reliability in measuring unknown samples. Light lamb meat from three different systems (indoor, alfalfa grazing and sainfoin grazing) of feeding during lactation were discriminated successfully, although grazing on alfalfa and sainfoin meat were more similar to one another, while TMR feed showed clearly distinct spectra.

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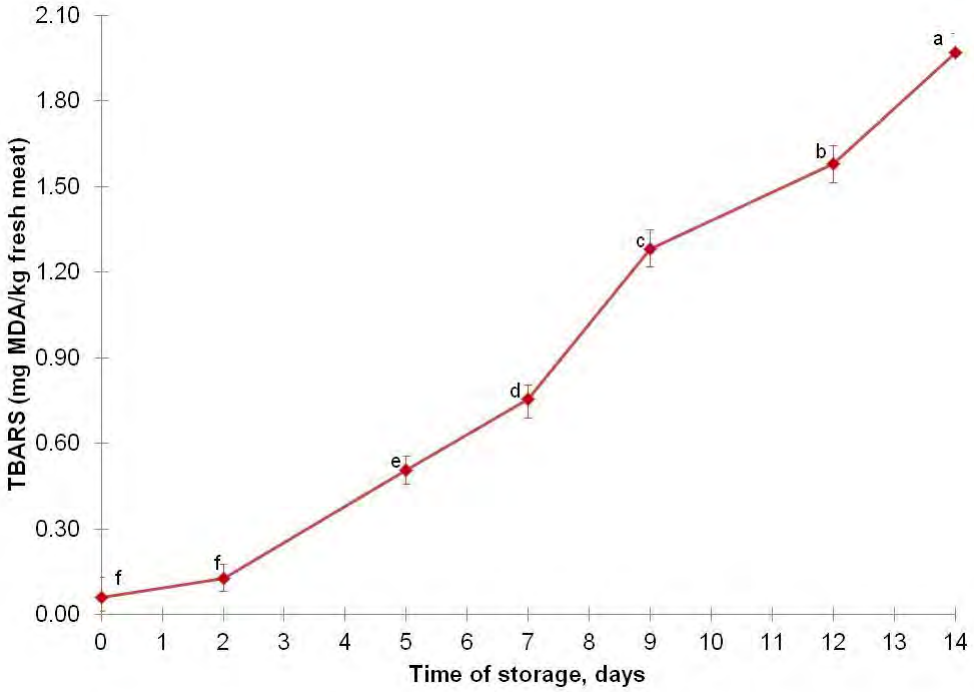
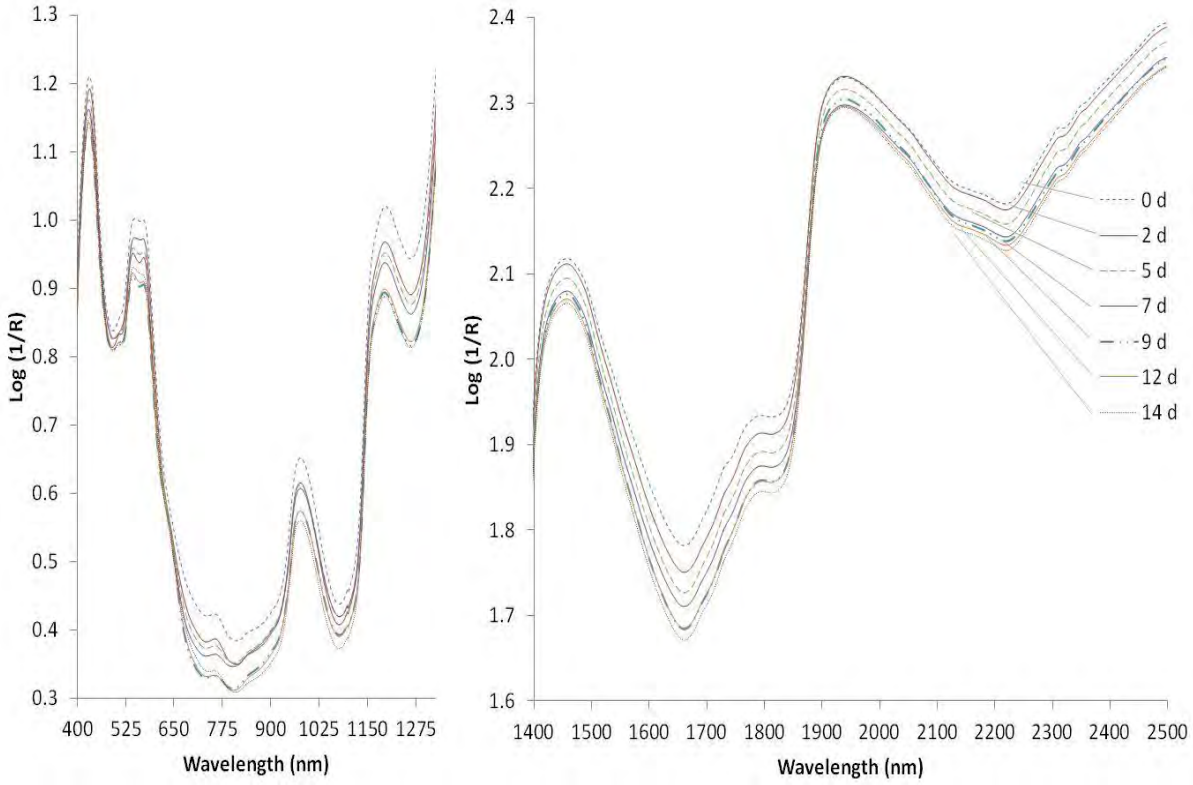
Figure 1. Lipid oxidation of the whole data set through time of storage.



Means with different letters are different (P<0.05).

Figure 2. Plot of the mean spectrum of light lamb meat according to the day of display.



Spectra have been selected and scale adjusted in the ranges from 400 nm to 1325 nm and from 1400 nm to 2500

Figure 3. Weighted regression coefficients of the first eight factors of regression model with selected wavelengths (400-1026 nm).

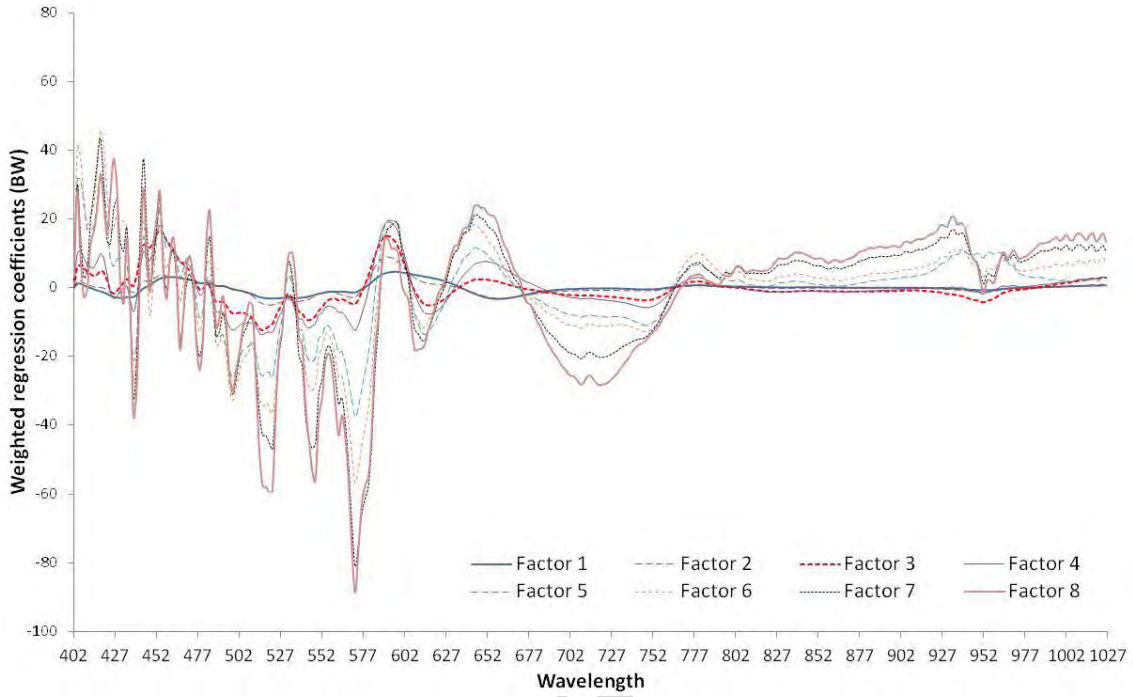
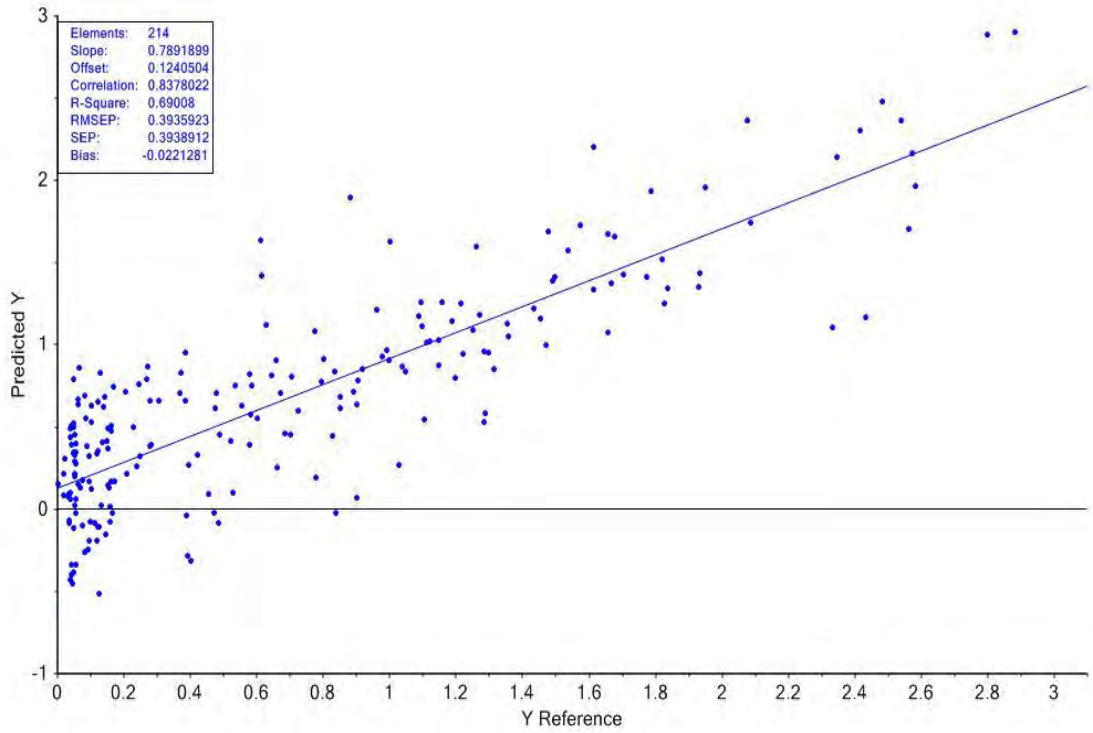
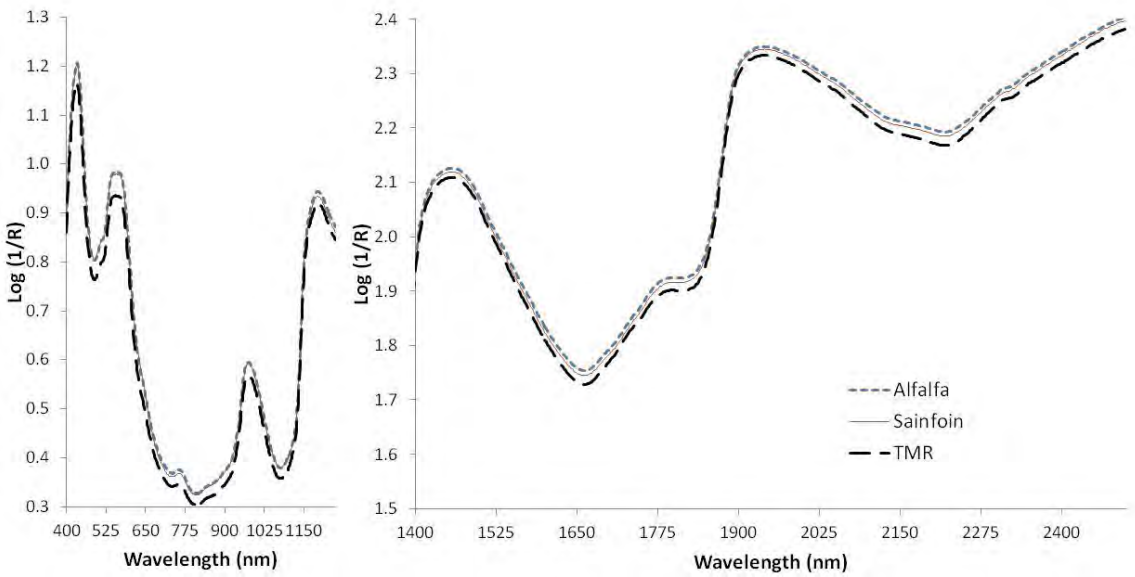


Figure 4. Predicted vs. Reference values of the regression model with selected wavelengths (400-1026 nm).



RPD=3.01; Consistency=88.

Figure 5. Plot of the mean spectrum of light lamb meat according to the diet used during fattening.



Spectra have been selected and scale adjusted in the ranges from 400 nm to 1250 nm and from 1400 nm to 2500

Figure 6. Explained variance of the PLS-DA and percentage of rightly discriminated samples for each factor included in the partial least square discrimination analysis.

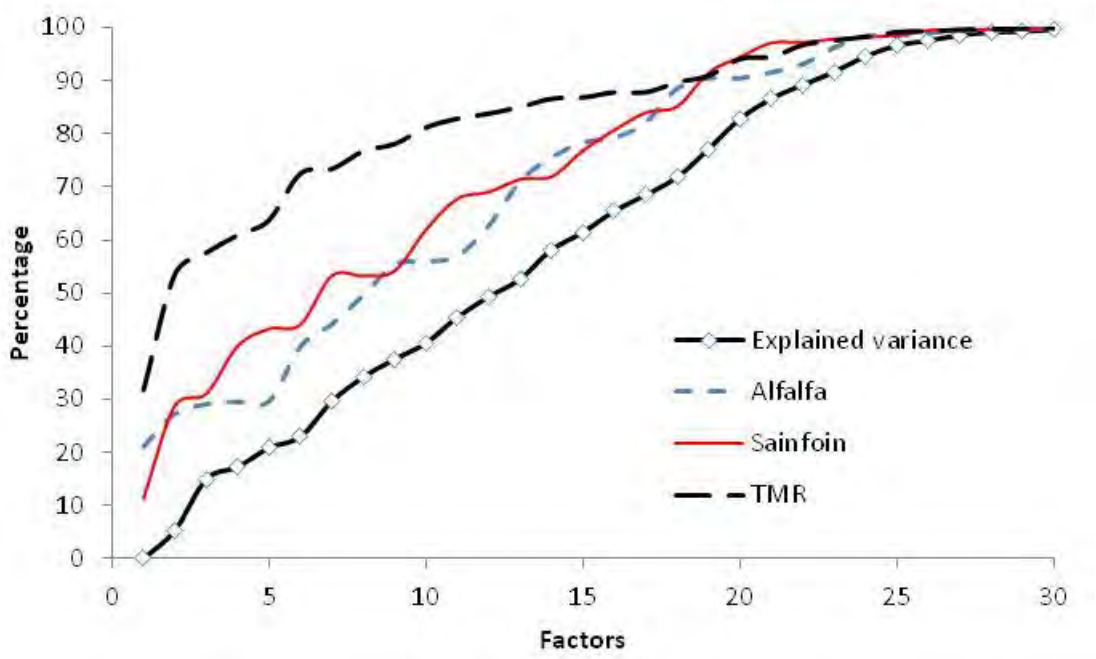


Table 1. Meat samples and spectra used per experiment and time of storage.

Time[a]	0	2	5	7	9	12	14	Total
Exp. 1	60	60	60	60	6	7	7	260
Exp. 2	0	62	62	62	62	62	62	372
Total	60	122	122	122	68	69	69	632

[a] Time of storage in darkness at 4ºC after sampling, days.

Table 2. Statistics of the lipid oxidation of calibration and validation sets.

	Calibration set	Validation Set
n of samples (%)	418 (66 %)	214 (34%)
n of lambs	82 (67 %)	40 (33%)
Mean	0.829	0.693
Median	0.530	0.465
Maximum	3.595	2.882
Minimum	0.025	0.003
Standard deviation	0.843	0.709

Table 3. Calibration and full cross validation statistics of three models according wavelengths used to predict the lipid oxidation of light lamb meat.

Wavelength interval (nm)	VIS (400-700)	Selected wavelengths (400-1026)	VIS-NIR (400-2500)
Optimal number of factors	7	8	9
Calibration set			
Slope	0.800	0.832	0.854
Offset	0.165	0.139	0.121
R^2_c	0.80	0.83	0.85
RMSEC	0.376	0.345	0.322
Cross validation set			
Slope	0.784	0.815	0.820
Offset	0.178	0.153	0.148
R^2_{cv}	0.77	0.80	0.80
RMSECV	0.406	0.376	0.374
Consistency	93	92	86
RPD	2.07	2.24	2.25

RMSEC, root mean square error of calibration; RMSECV, root mean square error of cross validation R^2, coefficient of determination; RPD = SD/SECV; Consistency = SEC * 100/SECV; SD, standard deviation of laboratory; SEC, standard error of calibration; SECV, standard error of cross validation

Highlights

- NIRS was useful to estimate the lipid oxidation of light lamb meat
- NIRS also proved able to detect analytes at concentrations of ppm
- Diets during lactation were discriminated using meat spectra