Detection of bovine meat adulteration by UV-Vis, near infared and midinfrared spectroscopy

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Introduction

The identification of species origin in industrial meat products is important for economic, religious, quality, and safety reasons. The detection and qualification of food constituents is required in order to conclusively prove that food stuffs are fraudulent. The replacement products are often biochemically similar to the original materials, and hence their detection can be extremely difficult.¹ Various techniques have recently been developed for identifying meat species (both protein- and DNA-based methods), but the development of an easy, rapid and sensitive method is still needed. The aim of the work presented here was to investigate the capacity for ultra violet to visible (UV-Vis), near infrared (NIR) and mid-infrared (MIR) spectroscopies to detect bovine meat adulteration with turkey meat.

Materials and Methods

Eleven lots of beef and turkey meat from different animals were used. Each lot was separately minced and then used to prepare 7 duplicate mixtures (450 g each) of bovine meat added with different percentages of turkey meat, ranging from 5 to 50%. From each lot, 4 aliquots of pure bovine meat (0% adulteration) and 4 of pure turkey meat (100% adulteration) were also obtained, for a total of 242 samples.

The UV-Vis spectra (200–780 nm) were collected using a V-650 spectrophotometer (Jasco Europe, Cremella, Lecco, Italy) equipped with a horizontal sampling integrating sphere working in diffuse reflectance. The NIR and MIR spectra were recorded using an FT-NIR spectrometer (MPA, Bruker Optics, Ettlingen, Germany) fitted with an integrating sphere (12 500–3900 cm⁻¹) and an FT-IR spectrometer (VERTEX 70, Bruker Optics) equipped with an ATR cell (4000–700 cm⁻¹), respectively. All spectra were collected in duplicate.

Replicates of spectral data were averaged, pretreated (standard normal variate (SNV) correction and first and second derivatives) and processed with principal component analysis (PCA), linear discriminant analysis (LDA) and partial least squares (PLS) regression. Data elaboration was performed with The Unscrambler 9.8 (Camo Software AS, Oslo, Norway) and V-PARVUS.²

Results and Discussion

Spectral data ranges were reduced to remove confounding noise, leaving 220–700 nm for UV-Vis; 3749–10614 cm⁻¹ for NIR; 1008–2295 and 2642–3701 cm⁻¹ for MIR. PCA was performed for preliminary data examination. In the score plots, obtained from NIR pretreated spectra, a slight trend as a function of adulteration percentage was observed along the first principal component (PC1), with a clear distinction of the turkey meat samples (Figure 1). A total of 97% variance was explained by the first and the second PCs. Similar results were obtained for each spectroscopic technique.

Meat samples were classified according to adulteration percentage with LDA, which was applied to the UV-Vis, NIR and MIR spectra, after mathematical pre-treatments and selection of the 30 most relevant variables of each spectral region by the SELECT algorithm.³ The LDA and PLS results were validated using both a cross-validation procedure with five cancellation groups (5CV) and an external test set. For each dataset, 66 samples belonging to three different lots were used as an external test set (Table 1).

Reference paper as:

E. Casiraghi, N. Sinelli, M. Casale and C. Alamprese (2012).Detection of bovine meat adulteration by UV-Vis, near infrared and mid-infrared spectroscopy, in: Proceedings of the 15th International Conference on Near Infrared Spectroscopy, Edited by M. Manley, C.M. McGoverin, D.B. Thomas and G. Downey, Cape Town, South Africa, pp. 424-426.

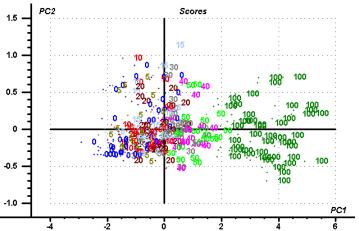


Figure 1. Score plot (PC1 vs. PC2) of NIR spectra after SNV correction. Sample identification by numbers refers to adulteration percentage.

The samples were ranked based on the adulteration with turkey meat and they were assigned to one of the five following quality categories:

- 1 =no to very poor adulteration ($\leq 10\%$ adulteration with turkey meat);
- 2 = very poor to poor adulteration ($\leq 20\%$ adulteration with turkey meat);
- 3 = significant adulteration ($\leq 40\%$ adulteration with turkey meat);
- 4 = very significant adulteration (50% of adulteration with turkey meat);
- 5 = pure turkey meat (100% of adulteration).

Some classes were mixed along the first two LDA canonical variables when the entire spectral range was analysed (Figure 2). NIR and MIR techniques gave the best results, whereas UV-Vis proved to be inadequate for identifying alteration classes, particularly for classes with lower percentages of adulteration (classes 2 and 3). Pure turkey meat (class 5) is always well distinguished.

Table 1. LDA results:	percent	age of	correctly	classified	d samples	•	

	Class 1 (%)	Class 2 (%)	Class 3 (%)	Class 4 (%)	Class 5 (%)	Average (%)
UV-Vis <i>(</i> SNV+d1)						
Calibration	83.9	81.3	90.6	100	100	88.6
Cross validation	71.0	50.0	59.4	78.6	100	70.1
Prediction	75.0	33.3	33.3	83.3	100	65.2
NIR (SNV)						
Calibration	86.1	93.8	98.1	100	100	93.5
Cross validation	75.8	71.9	81.3	100	100	82.6
Prediction	54.2	83.3	50.0	50.0	100	69.7
MIR (SNV)						
Calibration	86.8	88.1	90.0	97.1	100	90.8
Cross validation	80.7	66.0	81.3	92.9	96.7	81.8
Prediction	54.2	66.7	75.0	66.7	100	68.2

Class1, 0–10% adulteration; class 2, 15–20% adulteration; class 3, 30–40% adulteration; class 4, 50% adulteration; class 5, 100% adulteration.

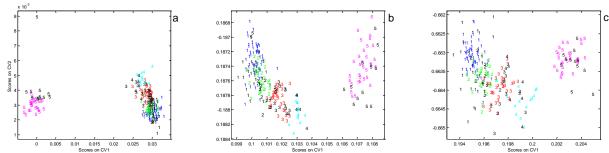


Figure 2. LDA score plots (CV1 vs. CV2) of UV-Vis (a), NIR (b), and MIR (c) spectra. Sample identification by numbers refers to adulteration class; test set samples are indicated in black.

PLS regression analysis was carried out in order to predict the percentage of adulteration with turkey in the bovine meat. Results for the three spectroscopic techniques are reported in Table 2; before analysis, all

spectra were pretreated with SNV and first derivative. PLS regression models showed that spectroscopic techniques could predict the percentage of adulteration of bovine meat with turkey meat. Also in this case, the best results were obtained with NIR and MIR spectroscopy. As an example, the predicted versus the measured response (percentage of adulteration) for the NIR spectral test set is shown in Figure 3.

Table 2. PLS results.

	UV-Vis	NIR	MIR
	<i>(</i> 9 LV <i>)</i>	<i>(</i> 5 LV <i>)</i>	(10 LV)
RMSECV (%)	8.48	6.41	6.47
RMSEP (%)	15.63	8.37	7.60

LV=latent variables considered in model construction

RMSECV = root-mean-square error of cross validation

RMSEP = root-mean-square error of prediction

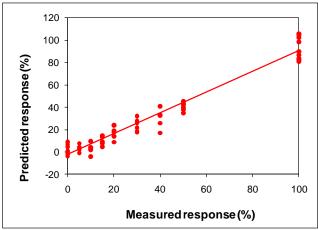


Figure 3. Predicted versus measured response (percentage of adulteration) for the test set NIR spectra.

Conclusion

The potential for spectroscopic techniques to easily and rapidly identify meat adulteration was demonstrated. In particular, NIR and MIR techniques, combined with the use of the appropriate chemometric strategies, could be effective tools in predicting the percentage of bovine meat adulteration with lower grade substitutes, such as turkey. Spectroscopy techniques could be an inexpensive alternative to protein- and DNA-based methods commonly used for species identification, particularly in a preliminary screening step.

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Reference paper as: