Archivos de Zootecnia
Journal website: https://www.uco.es/ucopress/az/index.php/az/
ORAL COMMUNICATION

Predictive ability of FT-NIRS in the assessment of chemical composition of pork seasoned products

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Additional keywords

Tuscan Ham.
Nutritional facts label.
Meat quality.

SUMMARY

Conventional chemical analyses of meat cured products are time-consuming, expensive and destructive. Advantages of NIR spectroscopy are its speed, simplicity, low-cost and the possibility to determine a large number of different parameters simultaneously in a large number of samples. The aim of this study is to assess the ability of FT-NIRs to predict chemical composition of seasoned pig products. One hundred and two seasoned products were sampled (43 "Cuore di spalla", 26 Toscano Ham and 33 "Capocollo") and the following chemical components were determined: protein, intramuscular fat, ashes and fatty acid composition. NIR spectra were collected using a Thermo-Fisher Antaris II instrument. Partial least squares (PLS) regression was applied in the calibration and the validation models; the models were fully cross-validated using the "leave-one-out" method. Calibration and cross validation models were developed both for each product separately and grouping all the samples. Calibration correlation coefficients show satisfying values (minimum R2=0.73), while cross-validation correlation coefficients, despite being generally acceptable, show lower values (minimum R2=0.42). Best R2 was found for fat content (cross-validation R2= 0.95). Results, even if obtained on a reduced sample, show how FT-NIRs could be used in routine analyses of pig seasoned products.

Capacità predittiva della tecnologia FT-NIRs della composizione chimica di prodotti stagionati di suino

Parole chiave aggiuntive

Prosciutto Toscano.
Etichetta nutrizionale.
Qualità della carne

SOMMARIO

Le tradizionali analisi chimiche dei prodotti stagionati richiedono tempo e sono costose e spesso distruttive. La spettroscopia NIRs ha il vantaggio di essere di rapida esecuzione, semplice, poco costosa ed ha la capacità di determinare un grande numero di parametri simultaneamente su un grande numero di campioni. Lo scopo di questo lavoro è quello di stimare la capacità predittiva della tecnologia FT-NIRs della composizione chimica di prodotti stagionati di suino. Sono stati campionati duecentodue prodotti stagionati (43 "Cuore di spalla", 26 Prosciutto Toscano e 33 "Capocollo) e sono state determinate le seguenti componenti chimiche: proteina, grasso intramuscolare, ceneri e composizione degli acidi grassi. Gli spettri NIR sono stati acquisiti usando uno strumento Thermo-Fisher Antaris II. I modelli di calibrazione e validazione sono stati sviluppati usando una regressione PLS (partial least squares); i modelli sono stati validati usando il metodo "leave-one-out". I modelli di calibrazione e validazione sono stati sviluppati sia per ognuno dei prodotti singolarmente sia unendo tutti i campioni in un unico dataset. I coefficienti di correlazione in calibrazione mostrano valori soddisfacenti (minimo R2=0.73), mentre i coefficienti di correlazione in validazione, sebbene siano generalmente accettabili, mostrano valori più bassi (minimo R2=0.42). Il migliore valori di R2 sono stati trovati per il contenuto in grasso (R2= 0.95 in validazione). I risultati, anche se ottenuti su un numero ridotto di campioni, mostrano che la tecnologia FT-NIRs potrebbe essere usata in analisi di routine di prodotti stagionati di suino.

Information

Cronología del artículo.
Recibido/Received: 05.01.2017
Aceptado/Accepted: 01.07.2017
On-line: 15.01.2018
Correspondencia a los autores/Contact e-mail:
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INTRODUCTION

Nowadays consumers are concerned about environmental impact of animal husbandry, food safety and food quality, sensory proprieties and nutritional and health value (Zamora-Rojas, 2011). In niche markets highlighting the quality of products is fundamental; product label could be an ideal means to underline its proprieties. Traditional analyses are time consuming and expensive; pork industry needs on line, fast, accurate and low-cost methods for the quality control. Developing cheap, fast and not destructive analyses could be especially useful in niche product, with poor economic resource to set up a wide number of analyses. As reviewed by Monin (1998), near infrared spectroscopy (NIRS) is one of the most promising techniques for

large-scale meat quality evaluation. Unlike chemical analysis, NIRs is a non-destructive method, it requires a small amount of sample and produces reduced chemical waste (Park et al. 1998; Stuth et al. 2003; Andrés et al. 2005); furthermore, this technology can provide a multiple evaluation of constituents (Prevolnik et al. 2004). Farming activity in Tuscany is generally characterized by small farms, by not well-structured breeders' consortia, lacking organisation and economic resource; furthermore, Tuscan seasoned pork products supply is composed of a wide range of different products with a lot of local typical variations. In this contest it is difficult to set up and to routinely develop analyses of seasoned pork product based on wet chemistry methods. Our investigation is a preliminary study on three different seasoned Tuscan pig products: Cuore di Spalla (CS), Capocollo (CC) and Tuscan Ham PDO (TH). Tuscan ham is the most important product, obtained from swine improved breeds. Capocollo is a cured product, made from the muscle above the neck; it's a typical product characterized by a high quantity of intermuscular fat; ageing about three mounts. Cuore di Spalla is an innovative product, obtained from the shoulder muscles; shoulder is trimmed, rolled in swine bladder and aged for 5 months. This product is characterized by a relatively high percentage of intermuscular fat. The aim of the present work is to assess the ability of FT-NIRS to predict chemical composition of Tuscan seasoned pig products.

MATERIAL AND METHODS

One hundred and two samples were collected (26 TH, 43 CS and 33 CC); TH samples were collected at several ham factories, while CC and CS samples were products obtained by experimental animals reared by our department, slaughtered and cured in two different meat-processing plants. CS and CC samples were sliced transversely and once slice was minced to be analyzed; regarding TH one transversal slice and one longitudinal slice were obtained from the center of each ham. The two slices were minced together to obtain a representative sample of the whole ham. Reference values for all traits were determined in all products using wet chemistry methods. Moisture (by lyophilisation), intramuscular fat (ether extract), protein and ash contents were determined (AOAC, 2000). Total lipids were extracted according to Folch et al. (1957); fatty acid profile of total lipids were obtained using the modified technique of Morrison and Smith (Morrison &Smith, 1964). Fatty acids methyl esters were prepared by esterification and analysed by gas chromatography using a Varian 430 apparatus (Varian Inc., Palo Alto, CA, USA); saturated fatty acid, monounsaturated fatty acid, polyunsaturated fatty acid, omega-3 and omega-6 fatty acids were determined. Fatty acid composition and moisture content were determined only in TH and CS. Fresh (not frozen) samples were minced; spectra were acquired on a Thermo-Fisher Antaris II FT-NIRs instrument, using a cup spinner, a rotating plate that can permit to perform continuous scans of the sample. It's particularly important in irregular sample like minced meat to produce a representative spectrum of the entire sample. Each spectral measurement was obtained from 32 scans performed at a wave numbers resolution of 16 cm^{-1}. Spectral data processing was performed by using TQ Analyst software v.8.6.12 (Thermo Fisher Scientific Inc.). Multiplicative scatter correction (MSC) was applied to all spectrum, in order to eliminate optical interference (Martens et al. 1983), physical effects like particle size and surface blaze at spectra wavelengths (Maleki et al. 2007). Partial least squares (PLS) regression was applied in the calibration and the validation models using TQ Analyst software; the models were fully cross-validated using the "leave-one-out" method. Calibration and cross validation models were developed for each product separately and grouping all the samples; the purpose of merging the different data set was to increase the size of the data set in order to produce better calibration models.

Table I. Wet chemistry analysis results (Risultati delle analisi chimiche).

TRAIT	CS				TH				CC			
	(n.=43)				(n.=26)				(n.=33)			
	Aver	S.Dev	Min	Max	Aver	S.Dev	Min	Max	Aver	S.Dev	Min	Max
Moisture[1]	44.7	4.92	30	55.1	56	6.83	50.7	71.6	---	---	---	---
Ashes[1]	18.4	4.35	10.8	25.2	16.6	1.24	14.3	19.3	12.1	1.73	9.4	15.9
Intr. Fat[1]	22.4	6.66	12.5	36.6	11.9	2.68	6.94	16.9	37.6	5.98	27.9	50.7
Protein[1]	58.5	4.65	47.4	69	70.1	3.07	62.5	76	49.7	7.02	31.6	61.2
SFA[2]	7.71	2.38	4.21	14.1	1.82	0.58	0.81	3.72	---	---	---	---
MUFA[2]	8.74	2.59	5.22	17.7	2.59	0.78	1.09	4.79	---	---	---	---
PUFA[2]	1.59	0.526	0.77	2.93	0.95	0.168	0.64	1.38	---	---	---	---
OMEGA 3[2]	0.07	0.035	0.02	0.16	0.07	0.02	0.04	0.13	---	---	---	---
OMEGA 6[2]	1.52	0.49	0.75	2.78	0.88	0.15	0.6	1.24	---	---	---	---

[1]Espresso come percentuale (expressed as percentage).
[2]Espresso in as g/100g (expressed as g/100g).

Table II. Calibration statistics for proximate analysis (Risultati calibrazione analisi centesimale).

	Ashes				Protein Content			
Product	RMSEC	R2	RMSECV	R2	RMSEC	R2	RMSECV	R2
CS	0.46	0.99	2.92	0.73	1.39	0.93	3.03	0.61
TH	0.26	0.98	0.59	0.88	0.42	0.98	1.58	0.72
CC	0.24	0.99	1.32	0.64	2.11	0.94	3.49	0.83
Merged data set	0.57	0.99	1.93	0.87	2.85	0.94	3.55	0.89
	Fat				Moisture			
Product	RMSEC	R2	RMSECV	R2	RMSEC	R2	RMSECV	R2
CS	1.12	0.98	3.25	0.87	0.84	0.98	1.70	0.93
TH	1.60	0.79	2.09	0.62	0.55	0.93	1.21	0.65
CC	2.01	0.94	3.39	0.82	---	---	---	---
Merged data set	2.39	0.96	3.03	0.95	0.79	0.99	1.71	0.95

RESULTS AND DISCUSSION

Table I shows the results of chemical analyses. All products show a high variability both for proximate analysis and fatty acid composition and the result was expected given the nature of the products. A high variability could be useful to create good calibration models but it's important that this variability would be uniformly distributed around the range of values. On the contrary an excessive variability and a reduced number of samples could represent an obstacle in obtaining passing calibration models. Statistics results of the best calibration and cross validation models obtained by NIRs analysis for proximate analysis are shown in **Table II**. Calibration statistics are generally good, both for R^2 and RMSE. In cross validation model, R^2 and RMSECV are always lower than those reported for the previous models. Moisture shows a root mean square error in cross validation of 1.21 and 1.70 for TH and CS respectively; similar values were found by Prevolnik et al. (2011). The use of full data set slightly improved R^2 whereas RMSE remained unvaried. Regarding ashes and protein content TH shows the lower value of RMSECV (0.59 and 1.58 respectively), certainly higher than that found by Gaitan-Jurado et al. (2008). Using full data set didn't produce substantial improvements. Intramuscular fat content shows high RMSE for all products, without any enhancement in merging data. TH alone shows the best cross validation statistics (R^2=0.79 and RMSE=1.6), but worse than what found on "Kraškipršut" by Prevolnik et al. (2011). **Table III** shows the results of accuracy of NIRs for fatty acid determination. Fatty acid composition, was performed only in CS and TH. All calibration models have R^2>0.73 and RMSE values were similar to those found by Fernández-Cabanás et al. (2011) in Spanish Iberian pork dry-cured sausages. Unfortunately, cross-validation statistics fall down with lower R^2 and higher RMSECV. Due to the reduced number of samples TH presented the worst values (in particular R^2) with higher RMSECV and the use of full data set did not improve the R^2 of the model. In conclusion, our cross-validation models can't be considered ready for use; a greater number of samples could be useful to obtain better fatty acid cross-validation models as found in other studies (Pérez-Juan et al. 2010; Fernández-Cabanás, 2011). **Table IV** shows RPD (ratio of prediction to deviation) values (Williams et al. 1995), this parameter is an indicator of the goodness of the cross-validation models. An RPD value lower than 1.5 indicates that the model is unusable, between 1.5 and 2.0 indicates that the model has the potential to distinguish between high and low values, and between 2.0 and 2.5 that the prediction is possible. RPD values be-

Table III. Risultati calibrazione composizione acidica (Calibration statistics for fatty acid composition).

	SFA				MUFA							
Product	RMSEC	R2	RMSECV	R2	RMSEC	R2	RMSECV	R2				
CS°	0.012	1	1.01	0.88	0.013	1	1.23	0.83				
TH	0.036	0.99	0.304	0.5	0.13	0.96	0.4	0.46				
Merged data set	0.16	0.99	1.64	0.88	0.17	0.99	1.32	0.91				
	PUFA				OMEGA3				OMEGA6			
Product	RMSEC	R2	RMSECV	R2	RMSEC	R2	RMSECV	R2	RMSEC	R2	RMSECV	R2
CS	0.007	1	0.306	0.79	0.012	0.93	0.02	0.74	0.006	0.99	0.29	0.79
TH	0.004	0.99	0.11	0.42	0.006	0.86	0.011	0.49	0.07	0.73	0.07	0.73
Merged data set	0.06	0.99	0.25	0.85	0.16	0.99	1.44	0.9	0.05	0.99	0.22	0.87

Table IV. Ratio of prediction to deviation RPD (Rapporto tra deviazione standard e errore in calibrazione RPD),

TRAIT	CS (n.=43)			TH (n=26)			CC (n=33)		
	S.Dev	RMSECV	RPD	S.Dev	RMSECV	RPD	S.Dev	RMSECV	RPD
Moisture	4.92	1.70	2.89	6.83	1.21	5.64	---	---	
Ashes	4.35	2.92	1.49	1.24	0.59	2.12	1.73	1.93	0.90
Intr. Fat	6.66	3.25	2.05	2.68	2.09	1.28	5.98	3.39	1.76
Protein	4.65	3.03	1.53	3.07	1.58	1.94	7.02	3.49	2.01
SFA	2.38	1.01	2.36	0.58	0.30	1.91	---	---	---
MUFA	2.59	1.23	2.11	0.78	0.40	1.95	---	---	---
PUFA	0.53	0.31	1.72	0.17	0.11	1.53	---	---	---
OMEGA3	0.04	0.02	1.75	0.02	0.01	1.82	---	---	---
OMEGA6	0.49	0.29	1.69	0.15	0.07	2.14	---	---	---

tween 2.5 and 3.0 and above 3.0 indicate the predictive capability of the model is excellent (Goldshleger et al. 2013), Some traits show not suitable values (<1.5), on the contrary a few traits show better values. Moisture shows better RPD values (2.89 and 5.64 for CC and TH respectively) but also protein content and some fatty acid RPD values can be considered a good starting point for further analysis.

CONCLUSIONS

NIR spectroscopy proved to be a suitable method for assessment of moisture, ashes, protein, intramuscular fat contents and fatty acid composition. Acceptable prediction accuracy together with the simplicity of measurements is an advantage for industrial, particularly on-line use, for which further studies are required. Our study was performed on three Tuscan pork seasoned product characterized by a high variability within and between products. Obtained calibration models are not enough robust to be applied on a routine analysis scheme, but can be considered a good starting point for further analysis, confirming the ability of FT-NIRs in prediction of swine meat quality. An enlargement of the number of samples will be necessary to set up more fitting calibration models.

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