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PORTABLE RAMAN SPECTROMETER AS A SCREENING TOOL FOR CHARACTERIZATION OF IBERIAN DRY-CURED HAM

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Background

Acorn-fed or bellota dry-cured Iberian ham high commercial value has led to an increase in labelling fraud, both in terms of the pig feeding regime and racial purity.

To overcome this problem, recently, the Spanish “Iberian Pork Quality Regulation” was published. While this regulation set the criteria to classify Iberian ham in different commercial categories, it did not include any analytical method for its authentication.

Since its implementation, the annual production of “acorn-fed” Iberian hams has increased drastically. However, neither the extensive livestock nor the acorn-producing dehesa area were multiplied, which could be a sign of possible fraud in the Iberian ham sector.

For this reason, a standardized analytical method could help the authorities to fight fraud and provide guarantees to the final consumer.

Raman spectroscopy has been broadly used in studies of porcine meat quality control.

But it has no current application for the authentication of dry-cured meat

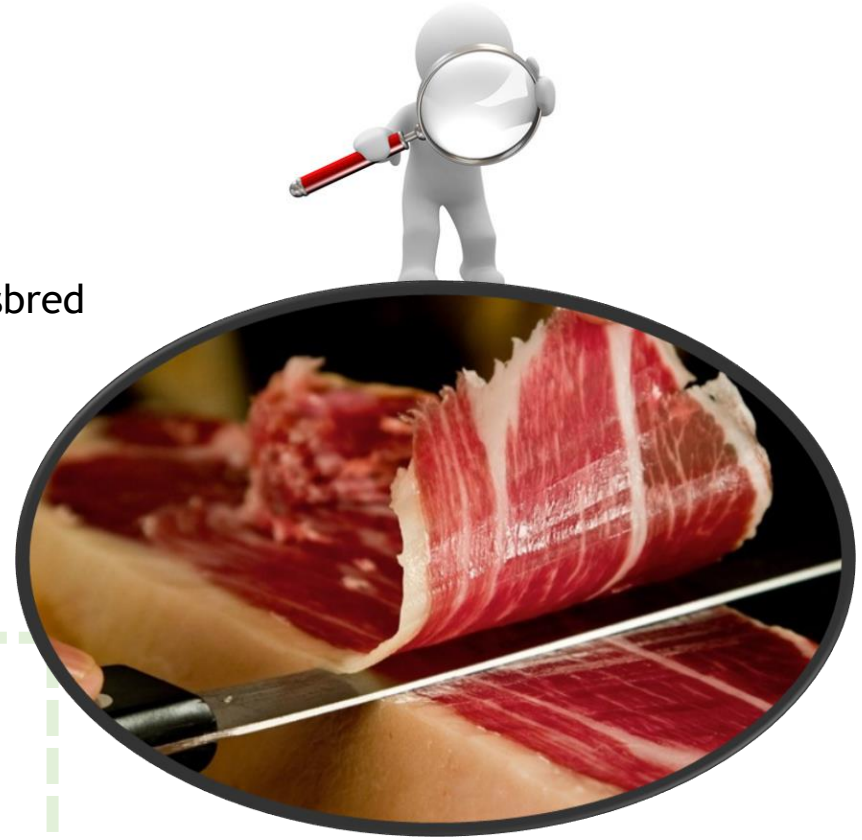
Alternative

Background

In the present study, one of the most representative meat products from the Iberian pig was analyzed with a Raman spectrometer: its **dry-cured ham**.

Four commercial categories

- Acorn-fed purebred Iberian
- Acorn-fed crossbred Iberian
- Free-range feed-fed crossbred Iberian
- Feed-fed crossbred Iberian

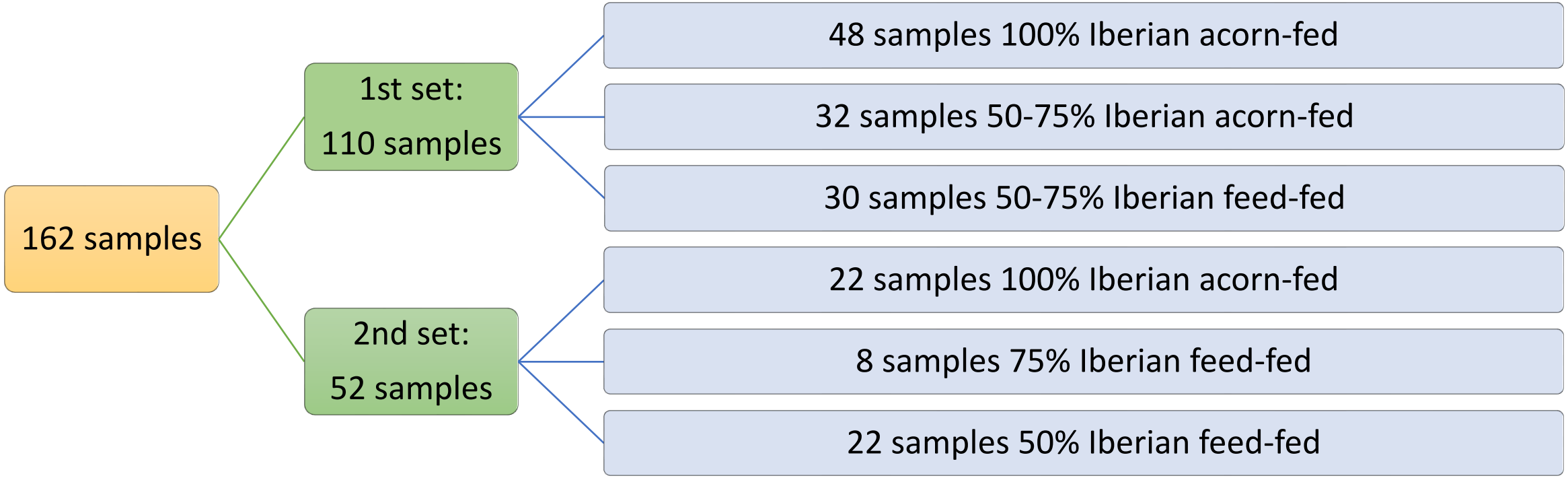


Similar characteristics and composition → Complex classification

Objectives

1) First objective of the present paper was to demonstrate the potential of a conventional **Raman** and portable device as a screening technique for dry-cured Iberian ham classification.
2) The second objective was to ensure the robustness of the method with an interlaboratory study, comparing the classification results of two similar Raman devices.

Materials and Methods I Samples



The first set of samples was analyzed using device A and the second set using device B

- Samples of **feed-fed ham** were produced in different parts of Spain and purchased in local markets or supplied by the protected designation of origin (PDO) company Jamones Ibéricos Dehesa de Campo Alto S.L. (Espiel, Córdoba, Spain).
- **Acorn-fed samples** were supplied by four companies from Los Pedroches (Córdoba) PDO and others from Salamanca, Zaragoza, Huelva and Badajoz.

Materials and Methods I Instrumentation and data processing

Instrumentation

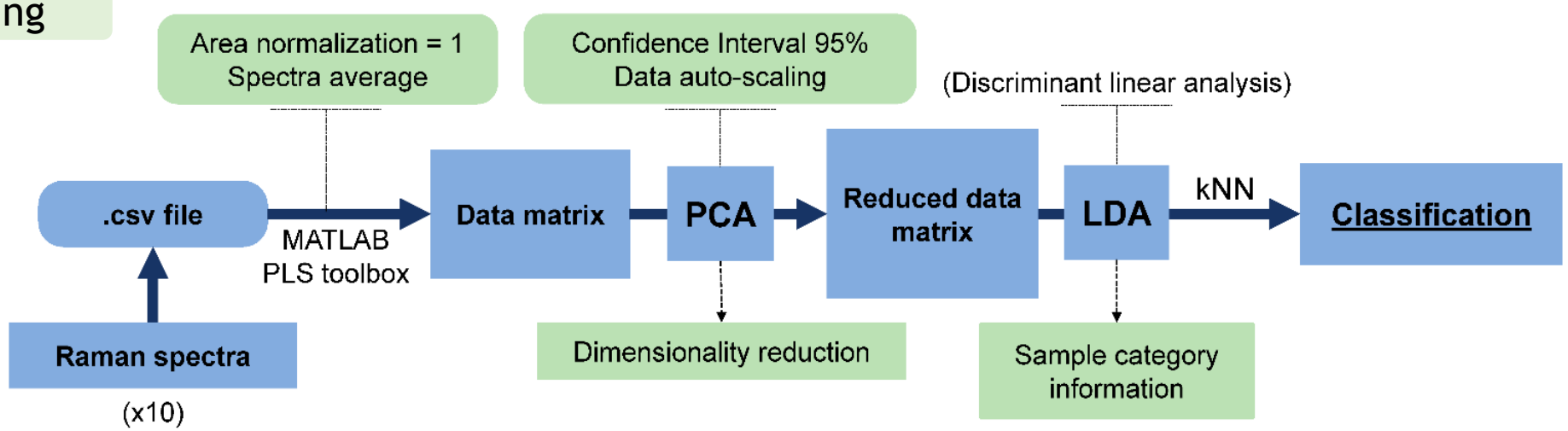
Device A - University of Córdoba

Device B - University of Murcia

i-Raman BWS415 portable spectrometer systems

- Laser with a wavelength of 785 nm and a maximum laser output power at the system's excitation port of 354 mW ± 15% HS system and 285 mW in the probe.
- Spectra were obtained in the range of 1–2035 cm^{-1} with a resolution of 4.5 cm^{-1}.
- The laser power at the probe was set to 10% of the maximum, which was 28.5 mW.
- The laser beam was focused on the sample through a 20×/0.40 NA objective.
- Spectra were recorded using a 500 ms acquisition time, a time average of 3 and a multiplier of 10.
- Data were collected by using BWSpec 3.27 software.

Data processing



Results I Classification of Dry-Cured Hams

1st set samples → Device A

Two sequential classification models

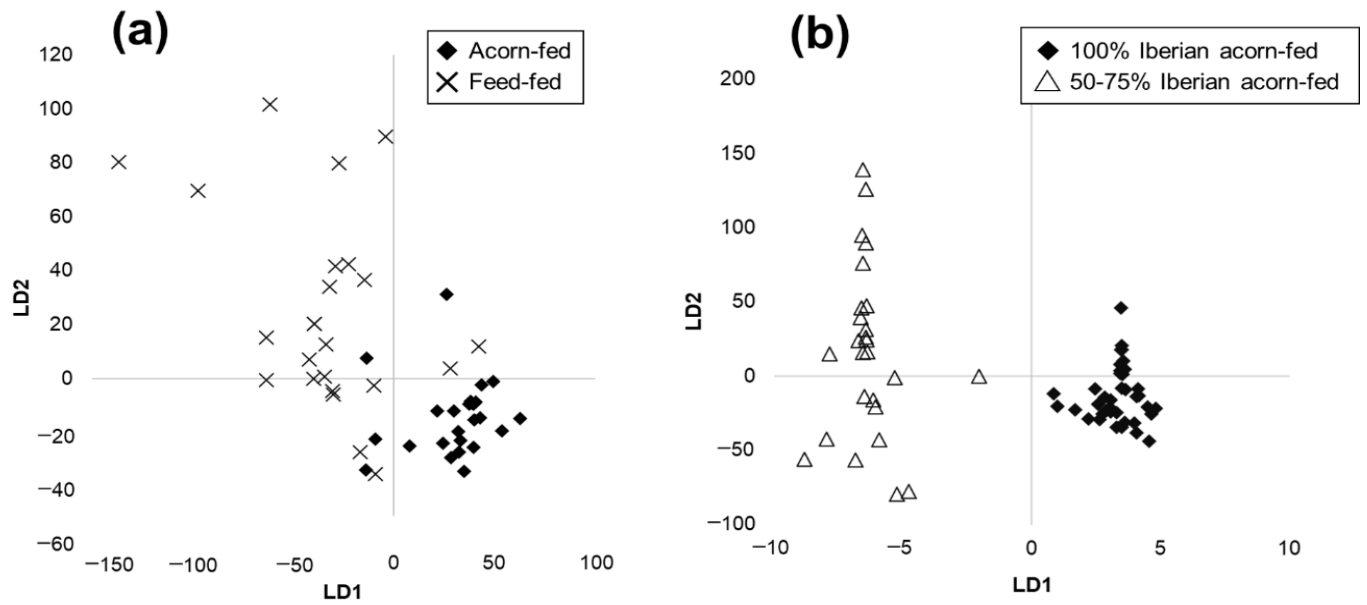
- Classification by feeding regime
- Breed discrimination

- A classification set (80% samples) was used to train the models and the remaining 20% of the data was used for model validation
- A PCA allowed us to reduce the dimensionality to 20 principal components (99% cumulative variance)
- Subsequently, an LDA calibration model was obtained and a kNN algorithm with k=3 was applied to obtain the percentage of correctly classified samples

Score plots of LDA calibration models using device A for:

(**a**) feeding regime

(**b**) breed discrimination



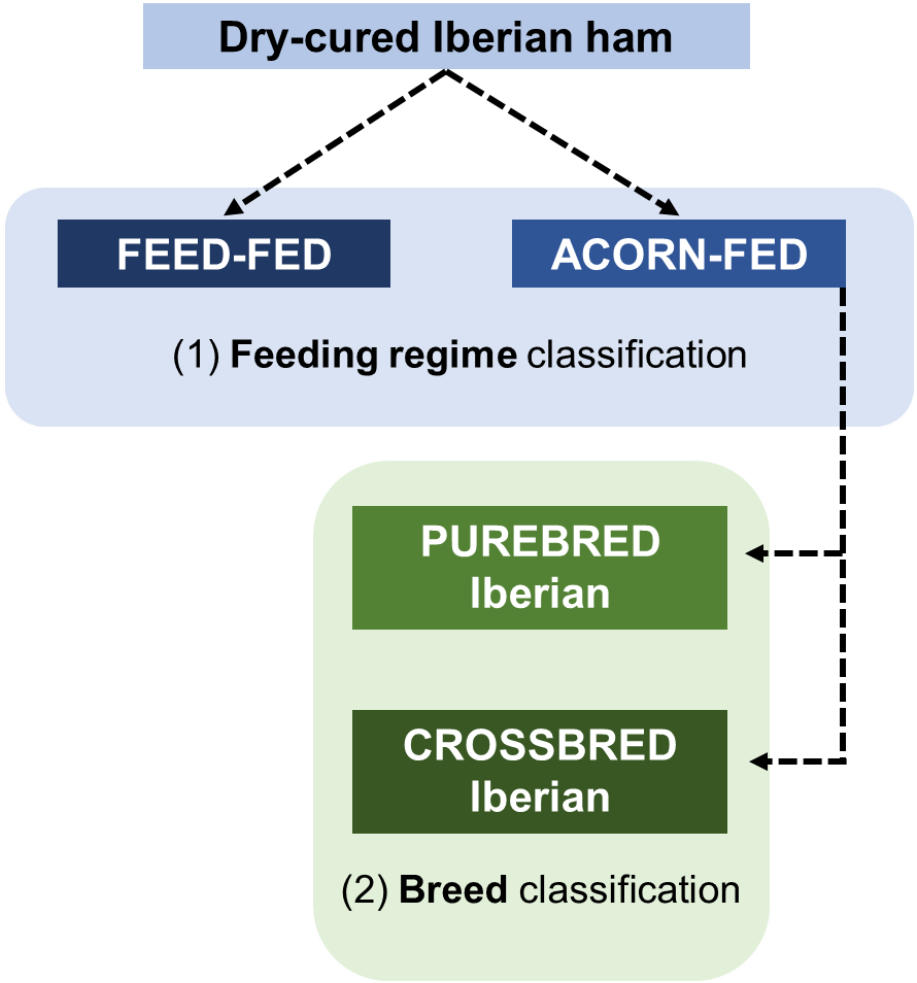
Results I Classification of Dry-Cured Hams

Feeding regime classification model

Feeding Regime		Total	% Correctly Predicted	Predicted Classes		
				Acorn-Fed	Feed-Fed	
Actual classes	Acorn-fed	6	83.3	5	1	Success 83.3%
	Feed-fed	6	83.3	1	5	

Breed discrimination classification model

Breed		Total	% Correctly Predicted	Predicted Classes		
				100% Iberian	50–75% Iberian	
Actual classes	100% Iberian	9	77.8	7	2	Success 86.7%
	50–75% Iberian	6	100	0	6	

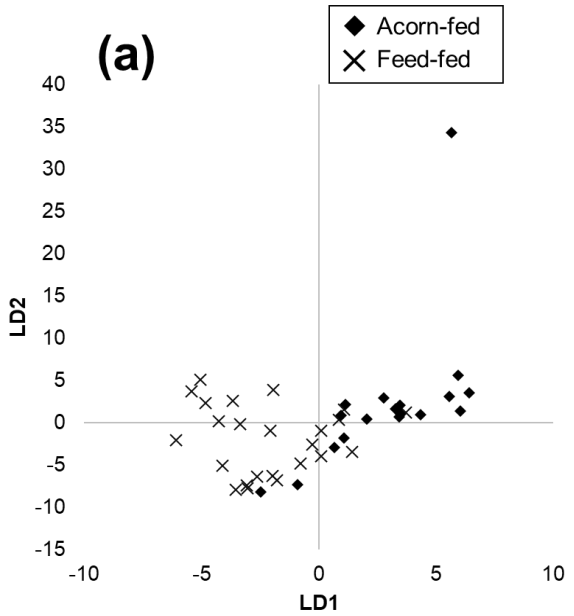


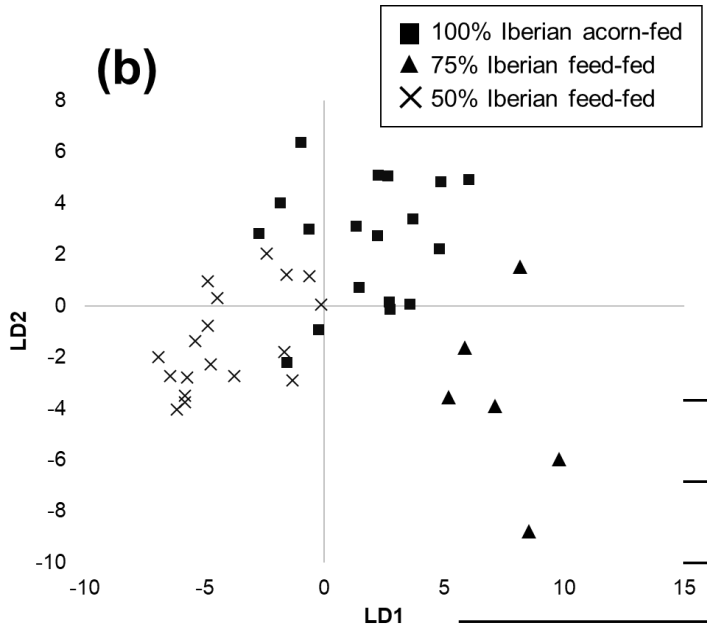
Procedure for Iberian ham classification in the industrial setting using the developed Raman method.

Results I Interlaboratory Study

2nd set samples → Device B

- Feeding regime discrimination
- Ternary differentiation





Score plots of LDA calibration models for:

(a) feeding regime discrimination

(b) ternary differentiation

Feeding Regime		Total	% Correctly Predicted	Predicted Classes		
				Acorn-Fed	Feed-Fed	
Actual classes	Acorn-fed	4	100	4	0	Success 70.0%
	Feed-fed	6	50.0	3	3	

Ternary		Total	% Correctly Predicted	Predicted Classes			
				100% Iberian Acorn-Fed	75% Iberian Feed-Fed	50% Iberian Feed-Fed	
Actual classes	100% Iberian acorn-fed	4	100	4	0	0	Success 90.0%
	75% Iberian feed-fed	2	100	0	2	0	
	50% Iberian feed-fed	4	75	1	0	3	

Conclusions

- The displayed results demonstrated that Raman spectroscopy could be used as a fast in situ screening tool to authenticate the quality of commercial dry-cured Iberian ham.

- A first Raman instrument was employed to classify 110 samples of dry-cured Iberian ham obtained from pigs with different breed purities and feeding regimes. The LDA chemometric models obtained with the Raman signal allowed a categorization according to the pig's breed and feeding regime with no need for the isolation of additional bands. **This approach was employed for the first time in dry-cured meat.**

- Moreover, an interlaboratory study carried out with an additional Raman device and the analysis of 52 additional samples ensured the robustness of the validated percentages of classification, which on average ranged between a promising **80.0-90.0% of success.**

- In the near future, Raman spectroscopy could serve as a useful screening technique for fast in situ analyses (5 min approximately) for the detection of labelling fraud within this product with high added value. Despite the usefulness demonstrated, to ensure the applicability of the methods in routine analysis, further studies could be necessary.

Acknowledgments

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