Quantitative and qualitative NIRS analysis of acorns (*Quercus, Sp*)

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Introduction

The "dehesa" is an agrosilvopastoral system characteristic of Southwest of Europe. The predominant tree type is the *Quercus sp*. From October to February these trees provide acorns that are consumed directly by the Iberian pigs during the fattening period (between 80–160 kg). This food provides meats of excellent quality and a high market price.

The production and characteristic of acorns are very variable, depending on factors such as species, individual and numerous agriculture–environmental factors, such as the attack of plagues. Table 1 show the chemical composition of the acorns obtained by several authors.

Table 1. Chemical composition of dehulled acorn.

Author	Species	Moisture	Ether extract	Crude protein
Mazuelos *et al.,* (1966)[1]	*ns*	24.8–36.2	7.0–11.6	5.93–6.62
Ramirez (1994)[2]	*Q. ilex*	35.8–45.4	sd	sd
Cava *et al.,* (1997)[3]	*Q. ilex*	40.21	6.33	4.21
Tejeda (1999)[4]	*Q. ilex*	35.2	5.8	5.5
Sánchez (2002)[5]	*Q. suber*	31.56–50.67	0.70–7.60	2.11–4.70

The importance of acorns in the feeding of the Iberian pig, the slowness and difficulty of carrying out the determinations, as well as the variability in the composition, require the characterisation of acorns to be carried out using fast techniques.

The objective of the present work is to demonstrate the viability of near infrared (NIR) technology for determining quantitative and qualitative attributes of acorn fruits.

Material and methods

Samples and reference data

Forty one samples belonging to Q*uercus ilex* corn and forty eight from Q*uercus suber* species were used. These samples were collected from the sierra de Aracena (Huelva, Spain) from November 99 to January 2000. Samples were manually dehulled and the kernels ground in a horizontal cleaver processor.

Moisture, ether extract, crude protein and fatty acids were analysed according to the Official Method of the B.O.E. [6]

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NIR analysis

Acorn samples were analysed by reflectance in a Foss-NIRSystems 6500 monochromator equipped with a transport module. It was used two types of cups: a small ring cup and a quarter rectangular cup (Shenk and Westerhaus, 1995).[7] Spectra were collected between 400 and 2500 nm by repacking of two subsamples using the ISI NIRS 3 software ver. 3.11 (Infrasoft International, Port Matilda, PA, USA).

Data treatment

Modified partial least square (MPLS) regression equations were developed to predict moisture, fat, crude protein and fatty acid composition (%C16:0, %C18:0, %C18:1, %C18:2, %C18:3 and %C20:0). Before this regression step, a principal component analysis (PCA) was developed to evaluate the sample set, considering as spectral outliers those samples with Mahalanobis distance (*H*) higher than 3 (Shenk and Westerhaus, 1991).[8]

PCA and MPLS regression equations were obtained by using WinISI software ver. 1.50. The cross-validation method was selected to evaluate the calibration equations developed. The statistics calculated to compare the different calibration results were *SECV* and r^2.

Results and discussion

Qualitative analysis

Figure 1 show the spectra means of each group of acorns obtained with the two sample cups used. It can bee seen the great similarity of the spectral pattern for acorns scanned in the two cups. However, for the visible region (400–700 nm), differences between species (*ilex* vs *suber*) are higher to those that exist between the capsule type (circular vs rectangular). Also can be observed differences between species in the area of the near infrared in both types of capsules.

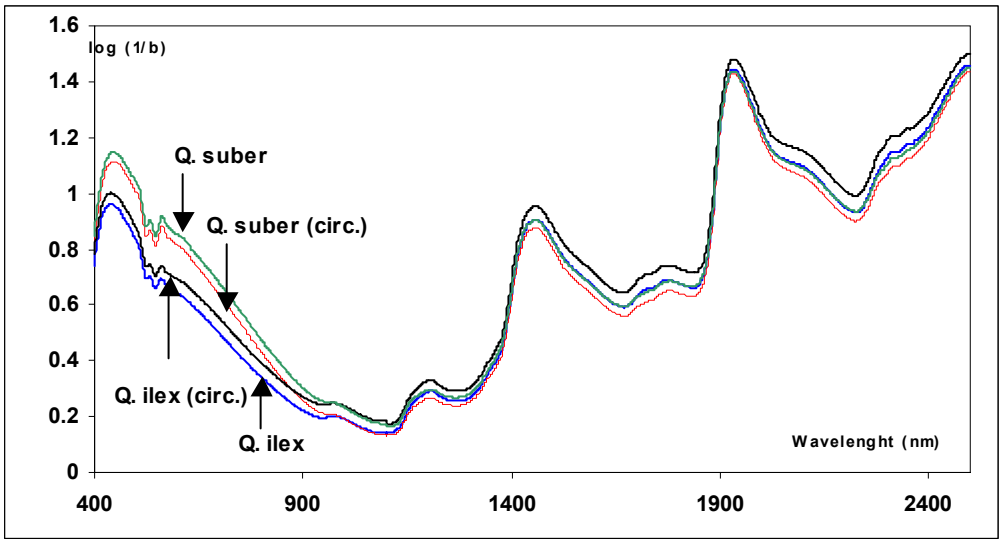

Figure 1. Spectra of ground acorns.

The principal component analysis carried out from the spectra recommended to use of 22 and 19 PCs for the development of models with circular and rectangular capsule respectively, explaining 98.93% and 99.06% of the variance.

Figure 2 show *Q. ilex* and *Q. suber* groups according to the first two principal components. A very similar distribution is observed in both types of capsules; the range of variation of the first PC was only bigger in the rectangular capsule than in the circular capsule. When the samples are represented as a function of the first and third PC, (Figure 3) it is appreciated that six samples of *Q. ilex* located in the area of *Q. suber*, are distanced to rest of group of *Q. suber*. This could be due to singularities of those samples, well that corresponds to a hybrid, well to a composition different.

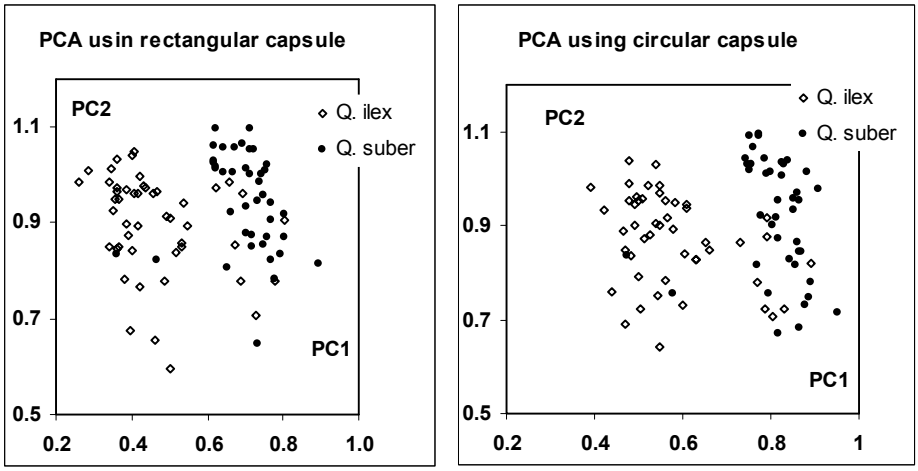

Figure 2. PC1 *versus* PC2 scores for the two acorns groups samples scanned in the rectangular and circular cups.

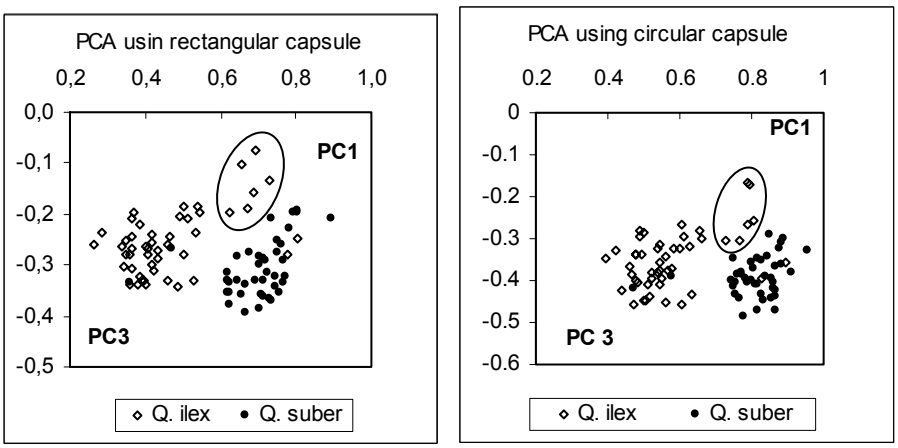

Figure 3. PC1 *versus* PC3 scores for the two acorns groups samples scanned in the rectangular and circular cups.

Quantitative analysis

In both types of capsules (circular and rectangular) were developed calibration equations for the prediction of moisture, ether extract and protein percentage, also of the main fatty acids of the acorn. The r^2 and *SECV* values of the equations obtained with each capsule type were very similar.

For the same mathematical treatment of the spectral information, the equations obtained with rectangular capsule had bigger r^2 values and smaller *SECV* values that those obtained using the circular capsule.

Table 2 show the statistics of NIR equations to predict different parameters of composition on acorn, according to mathematical treatment which better r^2 and *SECV* values were obtained. These values were very similar.

Table 2. Statistics of NIR equations to predict different parameters of composition on acorn according to the capsule type.

Parameter	Derivative	Type of cup	Mean	Range	*SECV*	r^2
Moisture	0,0,1,1	Rectangular	41.13	35.63–46.30	0.48	0.96
	0,0,1,1	Circular	40.95	35.63–46.30	0.57	0.94
Fat	2,10,5,1	Rectangular	4.60	1.20–8.30	0.58	0.92
	0,0,1,1	Circular	4.48	1.20–8.30	0.59	0.92
Crude Protein	1,4,4,1	Rectangular	3.15	2.21–4.70	0.21	0.83
	2,10,5,1	Circular	3.16	2.42–4.70	0.19	0.85
C 16:0	1,4,4,1	Rectangular	14.34	9.80–18.20	1.14	0.65
	0,0,1,1	Circular	14.31	9.80–18.20	1.01	0.71
C 18:0	1,4,4,1	Rectangular	2.53	1.00–4.80	0.44	0.85
	0,0,1,1	Circular	2.53	1.00–4.80	0.46	0.83
C 18:2	1,4,4,1	Rectangular	14.58	11.40–20.20	1.30	0.63
	0,0,1,1	Circular	14.45	9.20–20.20	1.33	0.65
C 18:3	0,0,1,1	Rectangular	0.98	0.50–2.10	0.13	0.91
	1,4,4,1	Circular	0.98	0.50–2.10	0.17	0.85
C 20:0	2,10,5,1	Rectangular	0.35	0.20–0.60	0.05	0.84
	2,10,5,1	Circular	0.35	0.20–0.60	0.05	0.81

For moisture, protein and ether extract percentage all the equations had r^2 values superior at 0.8, while in the determination of fatty acids composition only exceed this value the calibration equations of C18:0, C18:3 and C20:0; r^2 values for other fatty acids oscillated between 0.6 and 0.7.

Conclusions

Any influence of capsule type is appreciated in the quantitative or qualitative results. However, the use of the rectangular cup improved the accuracy of the quantitative calibrations.

The qualitative analysis of principal components shows clearly the differences between *Q. ilex* and *Q. suber*.

NIR technology allows to estimate accurately moisture, protein and ether extract percentage, as well as the percentage of certain fatty acids.

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