Evaluation of sensory parameters of grapes using near infrared spectroscopy

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ABSTRACT

Grape seed and skin sensory parameters are important characteristics in making decisions concerning the optimal harvest time and producing high-quality red wines. The potential of near infrared spectroscopy to determine several sensory parameters of seeds and skins was evaluated. Taste (sourness), texture (astringency, tannic intensity, dryness and hardness), visual (colour) and olfactory (intensity and type of aroma) attributes were considered. Calibration models were performed by modified partial least squares regression. The differences between sensory analysis and NIRS analysis in external validation were lower in the case of seeds; they were between 4.5% for hardness and 8.7% for colour. For the skins, differences in external validation were between 9.8% for tannic intensity and 13.7% for astringency. The results obtained show that NIRS technology has considerable potential for predicting the above sensory attributes.

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1. Introduction

Currently, deciding on the optimal harvest time is a great concern for wineries. Physical and chemical changes take place during ripening and all of them must be controlled. A large number of routine analyses have been used, such as the determination of pH, acidity, density, and the weight of the berries. These allow the ripening process to be monitored but do not consider the sensory parameters. Sensory characteristics such as sourness, astringency and colour are affected by phenolic changes (Ferrer-Gallego et al., 2010a, 2011b; Ristic and Iland, 2005), which differ in grape skins and seeds. It is therefore crucial to evaluate both skins and seeds separately in order to decide on the optimal grape harvesting time (Ferrer-Gallego et al., 2012).

A descriptive sensory analysis of skins and seeds during ripening using a trained panel is a common and useful tool to evaluate the organoleptic characteristics of grapes (Le Moigne et al., 2008; Rousseau and Delteil, 2000; Rousseau et al., 2005). However, it is not possible to have a panel of tasters available throughout to ripening process to decide on the optimal harvest time, essentially because this is expensive; there are many samples to be evaluated, and there is insufficient time for such determinations to be carried out.

Monitoring the changes that occur during ripening could offer an important tool for decisions to be made about the optimal harvest time, but this would require having rapid and inexpensive analytical methods. Moreover, it would require a technique capable of considering physical and chemical parameters. This is because in any sensory analysis taste, visual, texture and olfactory attributes must be taken into account. Near infrared spectroscopy (NIRS) provides physical and chemical information and it has shown a good potential to predict different parameters in both food and agricultural products (Liu et al., 2010; Mlcek et al., 2010; Nicolai et al., 2007; Prevolnik et al., 2011). Among others, it has been used to determine the quality in intact tomatoes (Flores et al., 2009), table olives (Morales-Sillero et al., 2011) and pears (Ying and Liu, 2008) to characterise strawberries (Sanchez et al., 2012), to estimate the mango fruit firmness (Valente et al., 2009), etc. Regarding grapes, this technology has also been used to determine different parameters such as glycerol, soluble solids, dry matter, mineral elements, sugars, aromas and phenolic compounds, among others (Cozzolino et al., 2004, 2006, 2008; Fernandez-Novales et al., 2009; Ferrer-Gallego et al., 2010b, 2011a; Kemps et al., 2010; Smyth et al., 2008; Versari et al., 2008).

Using this technology, several authors have addressed the sensory attributes of different products such as chicory, cheese, dry-cured ham, beef, and oil olive (Francois et al., 2008; González-Martín et al., 2011; Ortiz et al., 2006; Ripoll et al., 2008; Sinelli et al., 2010).

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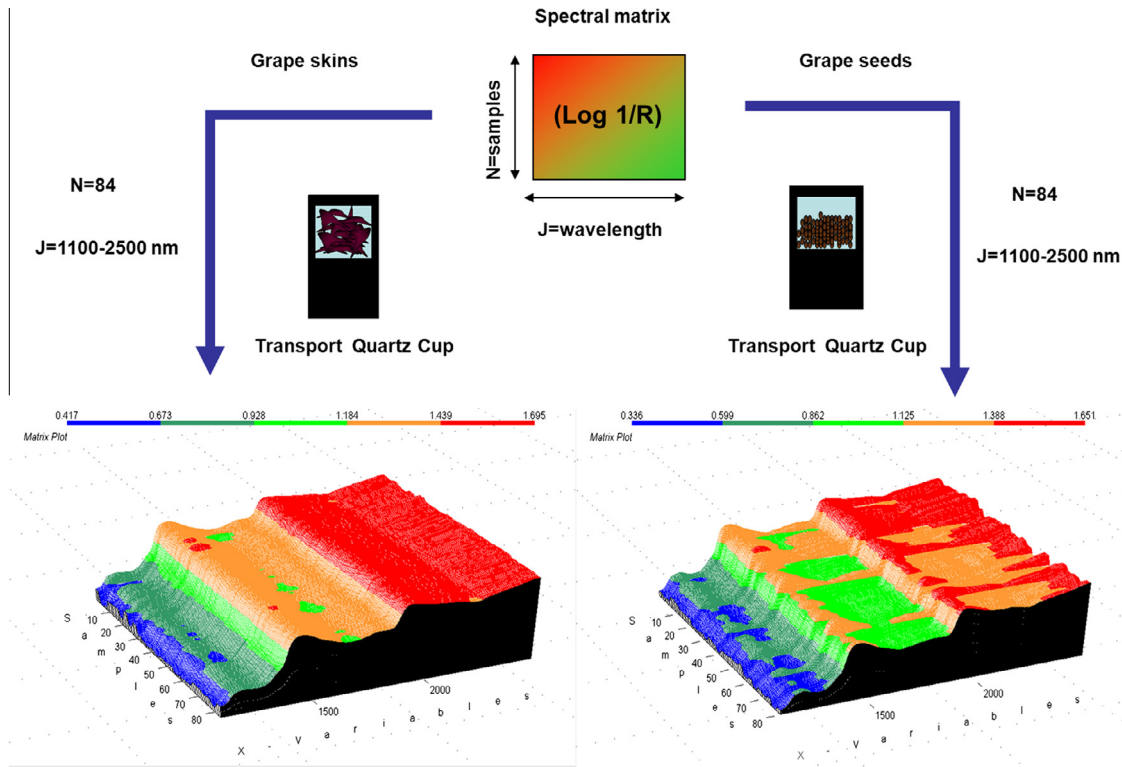E-mail address: escriban@usal.es (M.T. Escribano-Bailón).

Fig. 1. Spectra of the whole group (84 samples) in the NIR zone between 1100 and 2498 nm.

The aim of this study was to quantify, using NIR spectroscopy, some of the sensory parameters of grape seeds and skins collected during ripening. To our knowledge this is the first time that NIRS technology has been used for this purpose in the above matrices.

2. Materials and methods

2.1. Grape samples

Vitis vinifera L. cv. Graciano red grape samples were collected from two different vineyards. Samples were collected at different developmental stages from veraison (September 3rd) to over-ripeness (November 5th) in two different vintages (2008 and 2009) and two vineyards (located in different areas of the Rioja A.O.C.). In the case of 2008 vintage seven sampling data were taken into account for one vineyard and eight for the other one. In 2009 vintage the number of the sampling data taken into account was six and seven respectively. 150 berries (50 × 3) were collected randomly on each sampling date. Berries were collected from both sides of vines located in different rows within the vineyard. Edge rows and the first two vines in a row were avoided. Skins and seeds were separated manually and sensory analyses were performed. The remaining part of each sample was frozen and stored at −20 °C until NIRS analyses were carried out.

2.2. Panel training

The sensory panel included eleven members; some of them were oenologists who were experienced in this task. The other members were from our laboratory and were instructed to taste the samples. Four training sessions allowed us to recognise the perceived sensation, to evaluate intensity, and to practice the same number of chews. The aqueous solutions of quinine monohydrochloride dihydrate (0.025–0.05 g/L), aluminium potassium sulphate (0.3–0.9 g/L) and tartaric acid (0.06–2 g/L) were used as standards to carry out the training sessions related to bitterness, astringency and sourness respectively. Different tests were performed in the training sessions. The panellists were asked to state when they began to feel some stimulus and were requested to order it on an ascending scale. Some duo-trio tests were also performed and an aroma kit was used to carry out the olfactory training. The aroma kit allowed the panellist to recognise the different aromas released during the skin taste (herbal, fruity, jam, etc.).

2.3. NIRS analysis and chemometric techniques

The spectra of the grape seeds and skins were recorded with a Foss NIRSystem 5000 using a transport quartz cup (4.7 cm × 5.7 cm) with an optical pathway of 1.7 cm. Reflectance spectra were recorded in the ½-full mode, measuring close to

Table 1
Statistical overview of sensory values of grape skins and seeds.

	Mean	Minimum	Maximum	SD
Seeds				
Astringency	2.59	1.83	3.73	0.47
Colour	2.17	1.00	3.09	0.47
Hardness	2.89	1.50	3.82	0.65
Tannic intensity	2.68	2.00	3.50	0.45
Skins				
Sourness	1.97	1.10	3.00	0.55
Astringency	1.33	1.00	1.83	0.25
Dryness	1.54	1.00	2.73	0.52
Hardness	1.46	1.00	1.91	0.28
Tannic intensity	1.44	1.00	2.18	0.34
Aroma intensity	2.83	1.00	4.40	0.76
Type of aroma	2.26	1.50	3.00	0.38

Table 2
Calibration statistical descriptors[a] for the NIRS determination of sensory attributes in grape seeds.

Sensory attribute	Astringency	Colour	Hardness	Tannic intensity
Spectral pre-treatment	SNV 0,0,1,1	SNV 0,0,1,1	MSC 2,4,4,1	None 2,4,4,1
N	53	49	50	49
PLS Factors	3	7	7	7
Mean	2.59	2.17	2.89	2.71
SD	0.44	0.38	0.61	0.40
Minimum	1.28	1.03	1.06	1.52
Maximum	3.89	3.31	4.72	3.91
SEC	0.25	0.12	0.06	0.06
RSQ	0.665	0.895	0.990	0.977
SECV	0.27	0.15	0.17	0.13
VC[b] (%)	9.8	5.7	2.1	2.2

[a] N: number of samples; SD: standard deviation; SEC: standard error of calibration; RSQ: coefficient of determination; SECV: standard error of cross-validation.
[b] VC: variation coefficient (SEC/mean)·100.

1100–2498 nm in the IR zone. The spectra were recorded at intervals of 2 nm, recording 700 data points per sample. The cup was cleaned thoroughly to avoid contamination among samples. To minimise spectral error, all samples were analysed in triplicate. Fig. 1 shows the spectra of the whole group for (84 samples) in the NIR zone between 1100 and 2498 nm.

Spectral data were analysed by principal component analysis (PCA) which is a common tool used to obtain useful information about the latent structure of spectral data, sample distribution, and spectral outliers, and it is also important for creating cross-validation groups. The spectra were pre-treated with different techniques, including the standard normal variate (SNV), multiplicative scatter correction (MSC) and detrend (Dhanoa et al., 1995). Some mathematical treatments were also used to develop models (a, b, c, d), in which the first digit is the number of the derivative; the second is the gap from which the derivative is calculated; the third is the number of data points in a running average, or smoothing, and the fourth is the second smoothing. The modified partial least squares method (MPLS) was used to calculate the equations. MPLS is often more stable and accurate than the standard PLS algorithm. In MPLS, the NIR residuals at each wavelength, obtained after each factor has been calculated, are standardised (dividing by the standard deviations of the residuals at each wavelength) before calculating the next factor. When developing MPLS equations, cross-validation is recommended in order to select the optimal number of factors and to avoid over-fitting (Shenk and Westerhaus, 1995). T-outliers were identified by cross-validation. These were samples that had a high residual error when predicted by the model generated in this process. The final calibration was developed after removing these outliers.

Finally, validation errors were combined into a standard error of cross-validation (SECV). The ratio of the standard deviation (SD) to the SEP was used to quantify the performance of the model, called ratio of performance to deviation (RPD). The statistics used to select the most adequate equations were multiple correlation coefficients (RSQ) and SECV.

The software used was Win ISI® (v1.50) (Infrasoft International, LLC, Port Matilda, PA, USA). This software allows not only the spectral acquisition but also the data treatment.

Table 3
Calibration statistical descriptors[a] for the NIRS determination of sensory attributes in grape skins.

Sensory attribute	Sourness	Astringency	Dryness	Hardness	Tannic intensity	Aroma intensity	Type of aroma
Spectral pre-treatment	MSC 2,8,6,1	MSC 2,4,4,1	Detrend 2,8,6,1	SNV 2,4,4,1	None 2,4,4,1	MSC 2,4,4,1	Detrend 2,10,10,1
N	56	55	53	56	55	51	53
PLS Factors	7	5	7	6	5	4	7
Mean	1.54	1.46	1.41	1.97	1.33	2.25	2.74
SD	0.52	0.27	0.32	0.55	0.25	0.37	0.68
Minimum	0.00	0.64	0.44	0.33	0.59	1.15	0.69
Maximum	3.09	2.29	2.39	3.62	2.08	3.36	4.79
SEC	0.11	0.08	0.07	0.13	0.10	0.11	0.19
RSQ	0.952	0.906	0.948	0.942	0.851	0.906	0.925
SECV	0.22	0.19	0.17	0.36	0.20	0.21	0.37
VC[b] (%)	7.4	5.8	5.2	6.7	7.2	5.0	6.8

[a] N: number of samples; SD: standard deviation; SEC: standard error of calibration; RSQ: coefficient of determination; SECV: standard error of cross-validation.
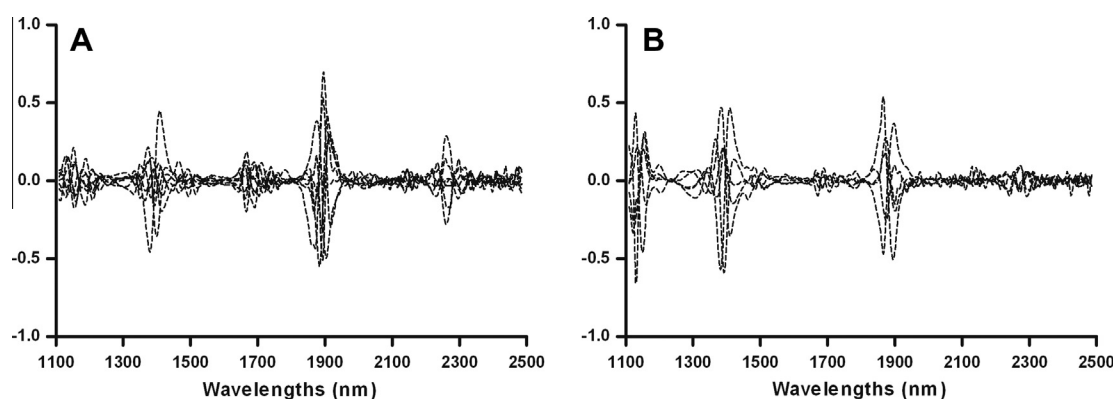[b] VC: variation coefficient (SEC/mean)·100.

Fig. 2. Tannic intensity: loading plot for grape seeds (A) and skins (B) MPLS models.

Table 4
Internal and external validation of NIRS models for sensory attributes of grapes.

Internal validation					External validation			
SEEDS	RPD[a]	RSQ[b]	SEP[c]	SEP[d] (C)	Differences (%)	SEP	SEP (C)	t-Student
Astringency	1.8	0.685	0.242	0.245	8.4	0.245	0.249	0.775
Colour	3.4	0.910	0.113	0.114	8.7	0.242	0.237	0.153
Hardness	10.9	0.992	0.056	0.056	4.5	0.224	0.225	0.375
Tannic Intensity	7.2	0.980	0.055	0.056	7.4	0.320	0.326	0.770
SKINS	RPD	RSQ	SEP	SEP (C)	Differences (%)	SEP	SEP (C)	t-Student
Sourness	4.9	0.958	0.105	0.106	11.1	0.200	0.195	0.140
Astringency	3.4	0.914	0.08	0.08	13.7	0.239	0.243	0.978
Dryness	4.8	0.955	0.068	0.068	10.3	0.170	0.172	0.550
Hardness	4.4	0.948	0.124	0.125	12.9	0.303	0.307	0.709
Tannic Intensity	2.8	0.864	0.09	0.091	9.8	0.155	0.158	0.865
Aroma Intensity	3.4	0.913	0.108	0.109	11.7	0.338	0.336	0.266
Type of aroma	4.0	0.935	0.172	0.174	10.2	0.391	0.394	0.455

[a] RPD: ratio performance deviation (SD/SEP).
[b] RSQ: coefficient of determination.
[c] SEP: standard error of prediction.
[d] SEP(C): corrected standard error of prediction.

3. Results and discussion

3.1. Sensory analysis

Several sensory parameters were evaluated in grape seeds and skins during ripening according to Rousseau and Delteil (2000). Taste (sourness), texture (astringency, tannic intensity, dryness and hardness), visual (colour) and olfactory (intensity and type of aroma) attributes were considered. The range, mean values, and the standard deviations of the sensory data, which were used as a reference analysis, are shown in Table 1. In the case of the sensory parameters that were evaluated in both matrices, the score range of seeds was always higher than in skin samples. Moreover, seeds always showed higher values of standard deviation in these sensory parameters. This suggests that seeds have a higher variation in these sensory parameters than skins. The highest standard deviation was obtained for the aroma intensity of skins and the smallest changes during ripening were found for skin hardness.

3.2. NIRS analyses and chemometric techniques

The quantitative descriptive analysis generated by the panellists (reference data) and the NIR spectral data were used to develop the calibration models. From the 84 samples (three samples of 50 berries collected at each sampling date), 66% were included in the calibration group (56 samples) and 33% of the samples (28 samples) in the external validation group. Initially, a spectral pretreatment (SNV 2,4,4,1) and a principal component analysis were applied to the raw NIR data of samples from the calibration group. The spectral variability explained was 99% in grape seeds and 97% in grape skins. For these, six and fourteen principal components were required respectively. Three spectral outliers were detected
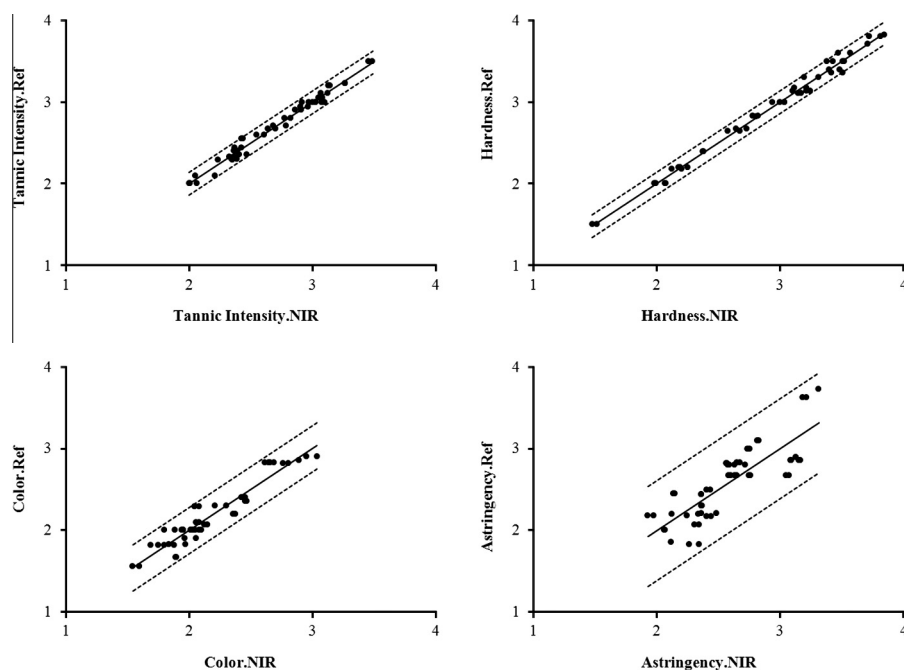

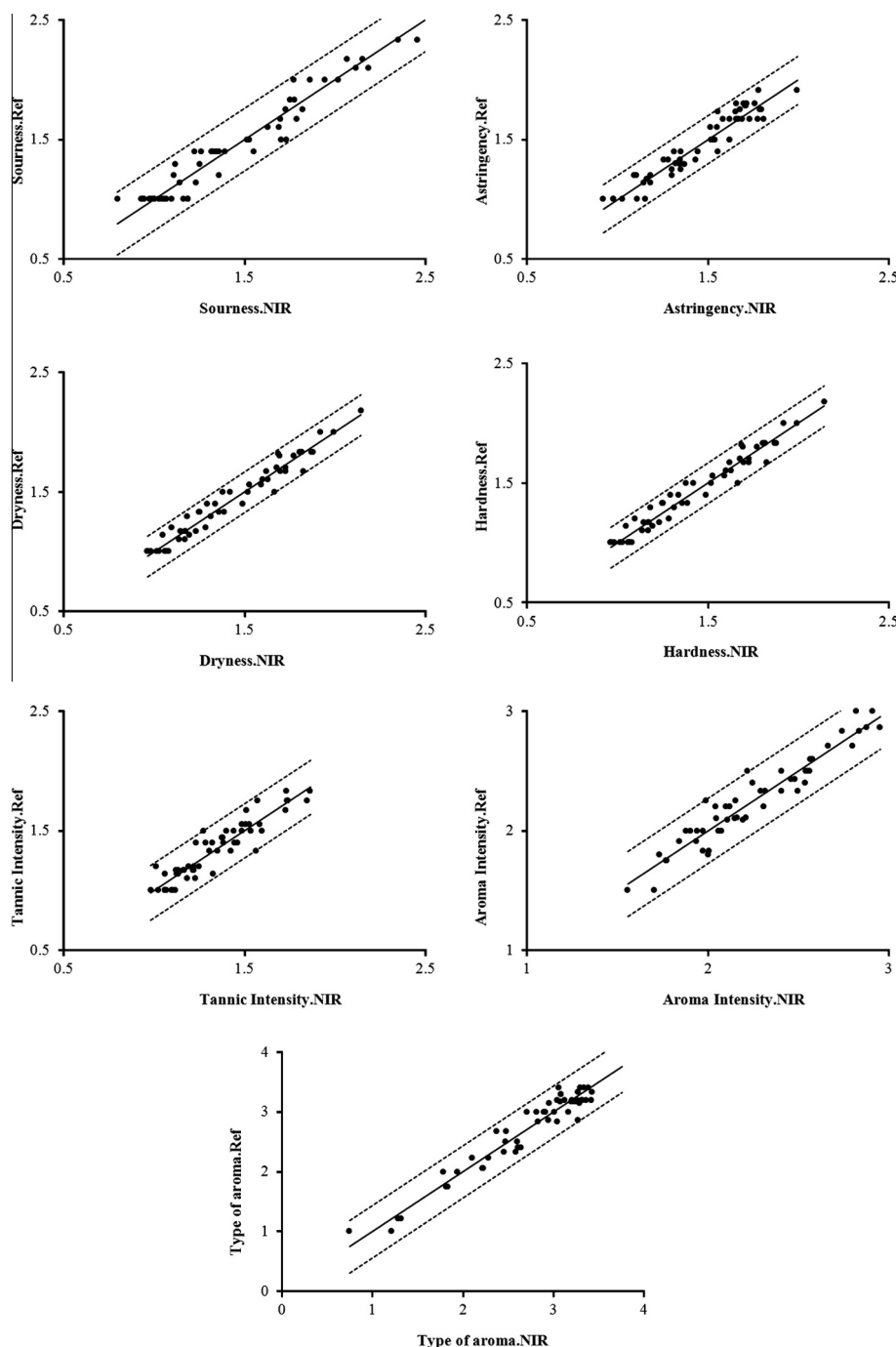**Fig. 3.** Comparison of NIRS predicted values with sensory analysis of grape seeds. Internal validation.

Fig. 4. Comparison of NIRS predicted values with sensory analysis of grape skins. Internal validation.

upon applying the Mahalanobis distance in the case of seeds and there were not any outliers in the case of skins. Those outliers were excluded from the analysis. The risk of errors in the equations under practical conditions is very low using the standardized H-statistic Mahalanobis distance during routine analysis (Shenk and Westerhaus, 1995).

Calibration models were obtained by modified partial least squares regression (MPLS), using the sensory data, as reference values, and the corrected spectral data. Up to twenty different mathematical treatments were tested to correct scattering. The optimum number of PLS factors used for the calibration in each matrix (Tables 2 and 3) was obtained by cross-validation. Samples with high residual values (T criterion ⩾2.5) were excluded from

the group. Validation errors were combined in the standard error of cross-validation (SECV). The statistical parameters of the calibration equations obtained are shown in Tables 2 and 3, indicating the number of samples used after the samples for spectral (H-statistic) or chemical reasons (the T criterion) had been eliminated. Moreover, the best mathematical treatment for each sensory parameter, standard deviation, standard error of calibration, coefficient of determination, variation coefficient and the concentration range are shown. After outliers had been excluded, an internal validation was carried out using samples that belonged to the calibration group.

The spectral regions between 1100–1358 nm and 1800–2100 nm showed important contributions to the loadings of grape

seeds models and are mainly related to second overtones of C–H and second overtones of the bonds present in the COOH groups, respectively (Fig. 2a). In the case of grape skins, the spectral regions close to 1140 and 1320 nm presented important contributions to the loadings of the aforesaid models and are mainly related to combination bands of the –OH functional group, symmetric and anti-symmetric stretching. This wavelength region is also related to C–H aromatic second overtones and C–H third overtones (Fig. 2b) (Osborne et al., 1993; Siesler et al., 2002). Fig. 2 shows the loading plot for tannic intensity as example and similar results were obtained for the remaining sensory parameters. The aforementioned wavelengths have already been reported in the determination of phenolic compound not only in grape seed (Ferrer-Gallego et al., 2010b) but also in grape skins (Ferrer-Gallego et al., 2011a) and the relationship between phenolic compounds and sensory parameters of grape seeds (Ferrer-Gallego et al., 2010a) and grape skins (Ferrer-Gallego et al., 2011b) has also been previously reported. Therefore a tentative assignation of the chemical basis for the parallel findings could be suggested.

The ratio of performance to deviation (RPD) was used to indicate the prediction capacity of the models (Table 4). The RPD values obtained indicated that the performance of the calibration models was adequate. Ideally, the RPD value should be greater than 2.5, although in cases which the reference standard deviation is lower than unity the RPD value may not reach this value, as is the case of the astringency of seeds (Williams and Sobering, 1993). Fig. 3 and 4 show the comparison of predicted NIR values versus the sensory analysis of grape seeds and skins in the internal validation, respectively.

An external validation was also performed to check the predictive ability of the method by applying NIRS spectroscopy to the 28 samples included in the external validation group. The Student t values obtained indicate that there were no significant differences between the NIRS technology and the sensory analyses. The differences between the sensory analyses and NIRS in external validation were lower in the case of seeds. They were between 4.5% for seed hardness and 8.7% for colour. In the case of skins, the differences were between 9.8% for tannic intensity and 13.7% for astringency.

4. Conclusions

NIRS technology seems to have good potential for the prediction of several sensory parameters such as taste, texture, visual and olfactory attributes in grape seeds and skins. The results obtained in the present study are comparable to those obtained by the panellists. Thus, the technology described could offer a rapid way to estimate sensory analysis and therefore to decide on the optimal harvest time. Nonetheless, a comprehensive study should be made in order to evaluate not only other grape varieties, but also other factors such as different production areas, etc.

Acknowledgments

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