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Analytical Methods

Rapid and non-destructive analysis of apricot fruit quality using FT-near-infrared spectroscopy

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ABSTRACT

A non-destructive optical method based on near-infrared spectroscopy has been used for the evaluation of apricot fruit quality. Diffuse reflectance measurements (800–2500 nm), physical, physiological and biochemical measurements were performed individually on 877 apricot fruits from eight contrasted cultivars harvested at different ripening stages. Relationships between spectral wavelengths and quality attributes were evaluated by application of chemometric techniques based on partial least squares (PLS) on fruit set divided randomly into two groups: 598 fruits for calibration and 279 for validation. Good prediction performance was obtained for soluble solids and titratable acidity with correlation coefficients of 0.92 and 0.89 respectively and root mean square errors of prediction of 0.98% Brix and 3.62 meq 100 g^{-1} FW respectively. For the other quality traits such as firmness, ethylene, individual sugars and organic acids, the prediction models were not satisfactorily accurate due to the high error of calibration and prediction.

1. Introduction

Quality fruit is characterised on the basis of size, colour, firmness, surface appearance and important traits related to the taste such as soluble solids and acidity. Most instrumental techniques measuring these properties are destructive and involve a considerable amount of manual work. Therefore, there is a demand for new and rapid analytical techniques for assessing fruit quality.

Optical visible/near-infrared spectroscopy (400–2500 nm) or only near-infrared spectroscopy (800–2500 nm) have been tested for non-destructive evaluation of firmness, dry matter, soluble solids, acidity and other physiological properties of many fruits and vegetables including apple (Liu & Ying, 2005; Peirs, Schenk, & Nicolaï, 2005; Zude, Herold, Roger, Bellon-Maurel, & Landahl, 2006), tomato (Shao et al., 2007), sweet cherry (Lu, 2001), stone fruit as peach, plum or nectarine (Carlomagno, Capozzo, Attolico, & Distante, 2004; Golic & Walsh, 2006; Peiris, Dull, Leffler, & Kays, 1998), prune (Slaughter, Thompson, & Tan, 2003), mandarin (Gomez, He, & Pereira, 2006), kiwi (McGlone, Jordan, Seelye, & Martinsen, 2002), watermelon (Ito et al., 2002), avocado (Clark, McGlone, Requejo, White, & Woolf, 2003). On apricot, Carlini, Massantini, and Mencarelli (2002) have shown the interest of wavelength selection methods and PLS (partial least square regression) for the evaluation of soluble solids in cherry and apricot. The main advantage of visible/near-infrared spectrometry is that, once models are established, it allows a non-destructive and individual characterisation of fruits, with simultaneous prediction of several quality traits. This technique appears to be useful to assist breeders in the selection of genotypes in breeding programs. Moreover it offers the possibility to screen fruits 'on-line' and estimate fruit quality which open new objective of market segmentation and fruit valorisation in a fresh or processed market.

The objective of this study was to evaluate the potential of near-infrared spectroscopy as a non-destructive method to predict apricot quality traits such as firmness, soluble solids, titratable acidity, ethylene production, sugar and acid contents through the comparison with standard techniques. The evaluation was performed on a large diversity of fruits covering genetic factor and physiological stages which was expected to be representative of the variability observed on the market. Samples belonging to eight cultivars harvested at different stages of ripening were used as a calibration set. The prediction models for each quality parameters were developed with partial least square technique.

Abbreviations: FT-NIR, Fourier transform near-infrared; SSC, soluble solids content; TA, titratable acidity; RMSEP, root mean square error of prediction; LV, latent variables; RMSEC, root mean square error of calibration; RMSEV, root mean square error of validation.

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2. Materials and methods

2.1. Selection of apricot fruit samples

Eight apricot cultivars or hybrids, named 'Moniqui', 'Goldrich', 'Bergeron', 'Iranien', 'Badami', 'A3844', 'A3759' and 'A4034' were chosen for their contrasted fruit quality traits such as colour, taste, physiological behaviour. To obtain a wide range of fruit composition, apricot fruits were collected from June 22 d to August 2 d, during the maturation period. A total of 877 fruits were collected. Fruits came from two INRA experimental orchards (Amarine (Gard) and Gotheron (Drôme), South of France) for 'Moniqui', 'Goldrich', 'Iranien', 'Badami', 'Bergeron' and 'A4034', and from a traditional private orchard (Donzère, Drôme, South of France) for 'A3844' and 'A3759'. At each picking date and for each cultivar, around 40 fruits were harvested. Non-destructive measurements were performed the day of picking and conventional destructive measurements were carried out a few days later on frozen materials.

2.2. Near-infrared diffuse reflectance measurements

FT-NIR spectra were recorded on a multi-purpose analyser (MPA) spectrometer (Bruker Optics, Wissembourg, France) equipped with an integrating sphere to provide diffuse reflectance measurements and a TE-InGaAs detector. The MPA was completely software-controlled by the OPUS software Version 5.0 which was provided by Bruker Optics.

The NIR spectrum of each sample was obtained by taking the average of 32 scans. It was acquired between 800 and 2700 nm at 2 nm spectral resolution, with scanner velocity of 10 kHz and a background of 32 scans. The time required to achieve a spectral measurement was 30 s. The intact apricots were placed on an automated 30-position sample wheel, each position corresponding to an 18 mm diameter hole. Apricots were placed at each position with their stem–calyx axis horizontal. On each apricot, a diffuse reflectance spectrum was measured on two opposite sides, the first on the blushed side and the second on the un-blushed side.

2.3. Determination of quality traits using reference analyses

Fruit firmness was determined by the pressure (kPa) required to achieve a 3% deformation of fruit height with a multi-purpose texture analyser (Pénélaup®, Serisud, Montpellier, France). The skin colour (un-blushed and blushed sides) was determined using a CR-400 chromameter (Minolta, Osaka, Japan) and expressed in the CIE 1976 $L^*a^*b^*$ colour space (illuminant D65, 0° view angle, illumination area diameter 8 mm). Ethylene rate (nmol kg^{-1} h^{-1}) was measured by gas chromatography after 1 h of confinement in a jar (Chambroy, Souty, Jacquemin, Gomez, & Audergon, 1995). Then fruits were cut and frozen at −20 °C for a few days until biochemical analyses. Fruit pieces were homogenised with an Ultraturrax T25 equipment (Ika Labortechnik, Staufen, Germany) to obtain a puree. Soluble solids content was determined with a digital refractometer (PR-101 ATAGO, Norfolk, VA) and expressed in % Brix at 20 °C. Titratable acidity was determined by titration up to pH 8.1 with 0.1 N NaOH and expressed in meq 100 g^{-1} of fresh weight using an autotitrator (Methrom, Herisau, Switzerland). Sugars (glucose, fructose, sucrose) and organic acids (malic and citric acids) were quantified using an enzymatic methods with kits for food analysis (Boehringer Mannheim Co., Mannhein, Germany) and expressed in g 100 g^{-1} of fresh weight for sugars and meq 100 g^{-1} of fresh weight for acids. These measurements were performed with an automatic analyser BM-704 (Hitachi, Tokyo, Japan).

2.4. Statistical treatment of data

Several pre-processing techniques were tested and just the SNV (standard normal variate) correction was retained. Each spectrum was separately normalised to null mean value and unit variance.

Principal component analysis (PCA) was used to investigate sample spectra and eliminate the defective spectra (aberrant spectra due to a problem of acquisition). The PLS regression method was used to develop models for predicting the composition of apricot fruits. In PLS, both the spectral matrix X and the reference data in the data matrix Y are used for the calibration. The X and Y matrices are reduced to a few factors (latent variables) using all of the available information.

In order to carry out a validation test, the data set was randomly split into two subsets with 598 apricots (two thirds of fruits) used for calibration and 279 apricots (one third of fruits) used for validation. The spectral measurements used for validation might deviate from the calibration samples as they correspond to different fruits for each harvesting date and cultivars. For each quality trait, analysis of the NIR data involved 10 separate modelling/validation exercises. Each exercise used a different random split of the fruits. Repeating the analysis across 10 separate modelling/validation exercises makes possible to examine the stability of the model with variation of the fruit selection. In this work, different wavelength intervals were tested, starting with the full interval between 800 and 2400 nm (Fig. 1). The non-informative regions were tentatively purged and the resulting performances were estimated.

The performance of the calibration models and the number of latent variables (up to a maximum of 15) were evaluated by the root mean square error of calibration (RMSEC), the root mean square error of prediction (RMSEP) and the correlation coefficient r between the predicted and the measured parameters. Acceptable models should have low RMSEC and RMSEP, high r and small differences between RMSEC and RMSEP. Large differences indicate the introduction of too many PLS factors (latent variables) in the model.

The pre-processing and calculations were carried out under SAISIR environment (Bertrand, 2007) and DESIR interface (Lecomte, 2007) in MatLab software package (version 7.2, MathWorks, USA).

3. Results and discussion

The general profile of the absorption spectra for apricot (Fig. 1) is very similar to that of other plant materials as mandarin (Gomez et al., 2006), apple (Liu & Ying, 2005) or tomato (Pedro & Ferreira, 2005). These spectra are in fact dominated by water absorption bands with overtone bands of the OH-bonds at 970, 1190 and 1450 nm and a combination band at 1940 nm (Nicolaï et al., 2007).

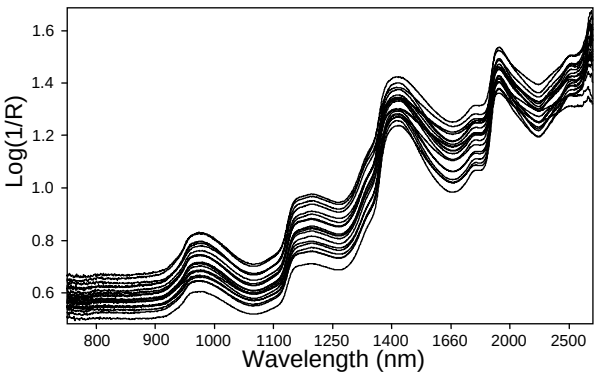


Fig. 1. Typical original spectra (Log(1/R)) for 20 apricot fruits issued from different cultivars and ripening stages.

A synthetic view of the observed physical, physiological and biochemical traits is presented (Table 1). According to the already known variability on apricot species (Audergon, Souty, Breuils, Reich, & Duffillol, 1989; Bureau et al., 2006; Gurrieri, Audergon, Albagnac, & Reich, 2001), the data set appeared representative of the expected variation on the related quality traits and well adapted for this study. The PC plot of PCA illustrates the large variability of the eight apricot cultivars chosen to cover most of the possible range of quality traits (Fig. 2). PC1 discriminated the fruits according to their ripening stages and PC2 was mainly representative of the variations in the colouration of fruit skin.

PLS predictions for several quality traits: pressure, ethylene, SSC, TA, and individual sugars and organic acids (sucrose, glucose, fructose, malic and citric acids) are summarised in Table 2.

3.1. Prediction of soluble solids content

A high correlation of calibration was found between the NIR spectra and the soluble solids content (SSC) values with a correlation coefficient (r) of 0.92 and a root mean square error of calibration (RMSEC) of 0.99% Brix (Table 2). When the model was applied to predict 279 other apricot fruits, the prediction results were similar with r of 0.92 and root mean square error of prediction (RMSEP) of 0.98% Brix giving a prediction error less than 8% of fresh weight (FW). The PLS model appeared to be robust with nine factors (latent variables) used in the calibration model for apricot.

The regression value obtained for the validation in this work was similar to those obtained on other fruits. Indeed, $r = 0.89$ and 0.95 were reported for the 'Sam' and 'Hedelfinger' cherry varieties respectively (Lu, 2001). In the same way, $r = 0.91$ on tomato 'Heatwave' (Shao et al., 2007) and $r \geqslant 0.92$ in kiwi (McGlone et al., 2002) have been observed. On the other hand, better results have been found on 'Satsuma' mandarin with $r = 0.96$ (Gomez et al., 2006), on 'Fuji' apple with $r = 0.97$ (Liu & Ying, 2005) and on prune with $r = 0.98$ (Slaughter et al., 2003). In these quoted references, RMSEP from 0.16% to 0.52% Brix were obtained giving a prediction error equal or inferior to 5% FW which were actually lower than 8% FW in the present work. The higher RMSEP observed here was in relation with the large phenotypic variability used in this study. The present study indeed deals with eight contrasted cultivars and different ripening stages. When the atypical 'Iranien' cultivar was removed from the calibration set, the prediction results were improved with RMSEP of 0.91% Brix (or 7.5% FW). But less variability in the calibration set increased the risk of the prediction models to be not representative for future measurements. In the same way, an accurate model has been obtained by pooling seven apple cultivars for calibration with RMSEP equal to 0.88% Brix and slightly higher than RMSEP of 0.80% Brix when the model was established specially for Golden delicious (Peirs, Tirry, Verlinden, Darius, & Nicolaï, 2003).

3.2. Prediction of titratable acidity

The correlation between NIR measurement and TA for apricot was good with r equal to 0.88 and RMSEC of 3.73 meq 100 g^{-1} FW (Table 2). When the model was applied to predict 279 other apricot fruits, the prediction results were comparable with r of 0.88 and RMSEP of 3.62 meq 100 g^{-1} FW giving a prediction error of 15%. The PLS model seemed to be acceptable since 10 factors were used in the calibration model. Few works have been made on the prediction of the titratable acidity by using NIR spectroscopy. On 'Fuji' apples, Liu and Ying (2005) obtained a prediction model $r = 0.72$ and RMSEP = 0.0043 g 100 g^{-1} expressed in % malic acid in flesh giving a prediction error of 14%. On apples including seven cultivars as 'Jonagold', 'Elstar', 'Boskoop', 'Golden Delicious', 'Cox's Orange Pippin', 'Gala' and 'Braeburn', Peirs, Lammertyn, Ooms, and Nicolaï (2000) obtained a prediction correlation coefficient between 0.82 and 0.86 depending on the orchards and the years. On 'Tommy Atkins' mango, worse results in prediction ($r = 0.63$) were probably due to the low acidity of the fruit (Schmilovitch, Mizrach, Hoffman, Egozi, & Fuchs, 2000).

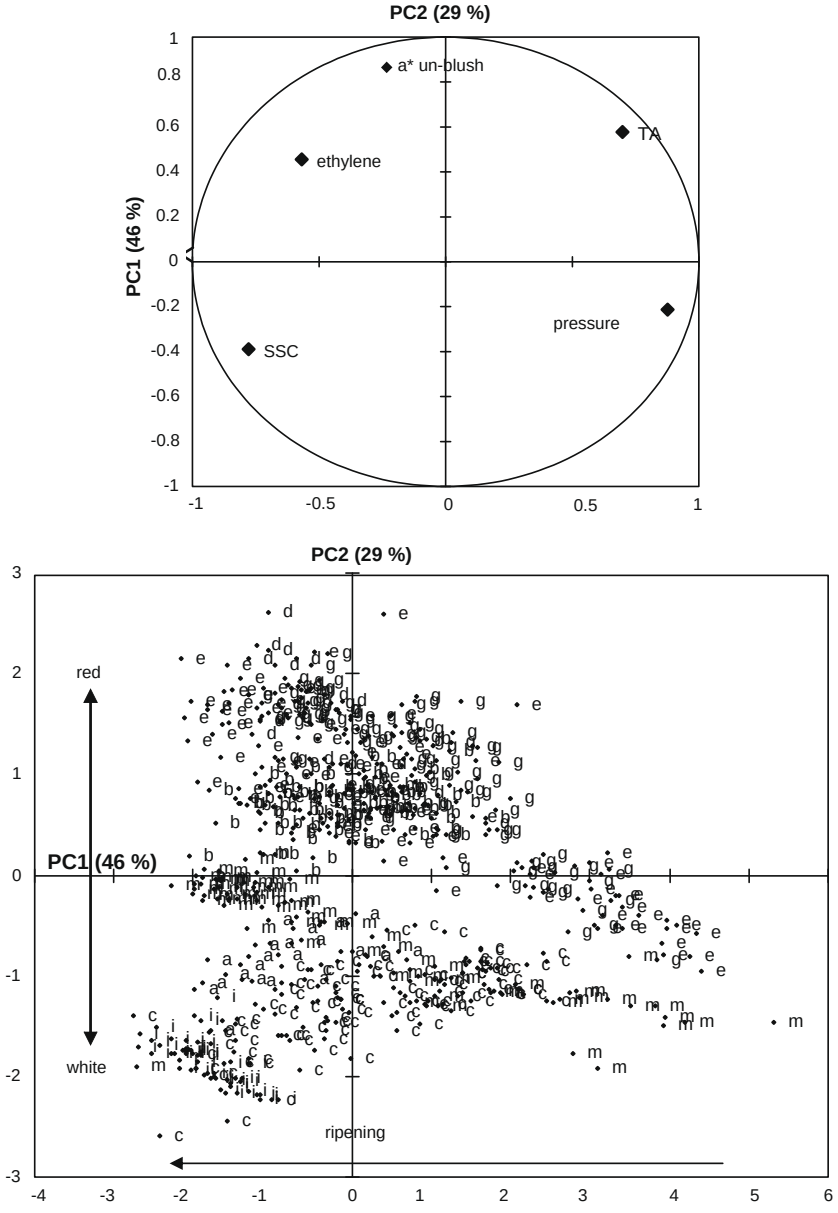
Fruits are known to be inhomogeneous. Indeed, radial and tangential gradients have been observed in apple, avocado and melon for firmness and biochemical traits (Duprat, Roudot, Grotte-Nicolas, & Roudot, 1991). It was thus important to check the prediction results according to the recording of spectra on two opposite sides of the fruit and especially on the un-blushed or blushed sides. The scatter plots with the ordinate and abscissa axes representing predicted and measured fitted values of SSC and TA allow the comparison of results obtained with spectra acquired on the blush or un-blushed side of the fruit (Fig. 3). There was a little difference between the two conditions but for SSC, the r and RMSEP were slightly better for spectra performed on the un-blushed side than for spectra performed on the blushed side. For the further measurements, we thus decided to acquire spectra only on the un-blushed face.

3.3. Prediction of individual sugar content

In these apricots the predominant sugar was sucrose with an average of 5 g 100 g^{-1} FW for different cultivars and ripening

Table 1
Mean, standard deviation (SD) and range of the apricot quality traits in both calibration and validation sample sets (an example of a random split of the fruits)

	Calibration ($n = 598$)			Validation ($n = 279$)		
	Mean	SD	Range	Mean	SD	Range
Weight (g)	59.0	13.0	23–116	58.0	12.0	29–90
Pressure (kPa)	112.4	89.2	6–611	115.3	92.0	5–537
L^* blush	54.1	11.4	29–76	53.8	11.5	30–77
a^* blush	20.8	12.6	−9 to 43	20.6	12.7	−8 to 43
b^* blush	32.1	10.5	9–52	31.8	10.5	5–52
L^* un-blush	64.8	6.6	39–81	64.6	7.0	41–81
a^* un-blush	8.6	12.2	−13 to 41	8.5	12.4	−14 to 42
b^* un-blush	42.7	7.4	17–57	42.4	7.6	14–55
Ethylene (nmol h^{-1} kg^{-1})	297.5	844.2	1–6322	279.9	837.4	0–6120
Ethylene (ln(nmol h^{-1} kg^{-1}))	3.6	2.0	0.4–8.7	3.5	2.0	1.4–8.7
SSC (% Brix)	12.5	2.5	6.6–22.2	12.4	2.4	7.3–21.7
TA (meq 100 g^{-1} FW)	23.4	7.7	4.4–40.6	23.5	7.8	5.4–42.5
Glucose (g 100 g^{-1} FW)	2.1	0.7	0.6–5.1	2.1	0.7	0.7–5.1
Fructose (g 100 g^{-1} FW)	0.8	0.3	0.2–1.7	0.8	0.3	0.3–1.9
Sucrose (g 100 g^{-1} FW)	5.0	2.0	0.4–11.7	5.0	2.0	0.3–10.7
Citric acid (meq 100 g^{-1} FW)	16.0	9.4	0.1–41.5	16.4	10.0	0.3–44.4
Malic acid (meq 100 g^{-1} FW)	10.5	6.7	0–30.1	10.2	6.4	0.1–29.4



a: 'Badami', b: 'Bergeron', c: 'A4034', d: 'A3844', e: 'A3759', g: 'Goldrich', i: 'Iranien', m: 'Moniqui'.

Fig. 2. Principal component analysis (PCA) performed on a subset of representative apricot quality data: ethylene, colour (a^* of un-blushed side), pressure, soluble solids content and titratable acidity.

stages while fructose was the minor with an average of 0.8 g 100 g^{-1} FW. The correlation between NIR measurement and sugar contents for apricot ranged between 0.81 and 0.86 (Table 2). The RMSEC was between 0.16 and 1.15 g 100 g^{-1} FW and increased with the compound level in the fruit (Table 2). When the model was applied to predict 279 other apricot fruits, the prediction

Table 2
Results of NIR calibration and validation performance for non-destructive quality assessment of apricot fruits (Spectra performed on the un-blushed side of the fruit)

Quality traits	Wavelength range	Factors	Calibration (n = 598)		Validation (n = 279)	
	(nm)	(LV)	r	RMSEC	r	RMSEP
Pressure (kPa)	800–2700	10	0.80	54.61	0.74	60.27
Ethylene (ln(nmol h^{-1} kg^{-1}))	800–2700	11	0.86	1.05	0.82	1.13
SSC (% Brix)	900–2000	9	0.92	0.99	0.92	0.98
TA (meq 100 g^{-1} FW)	1100–2500	10	0.88	3.83	0.88	3.62
Sucrose (g 100 g^{-1} FW)	1400–2500	10	0.81	1.15	0.82	1.11
Glucose (g 100 g^{-1} FW)	1100–2700	11	0.86	0.37	0.87	0.37
Fructose (g 100 g^{-1} FW)	900–2000	10	0.84	0.16	0.81	0.17
Citric acid (meq 100 g^{-1} FW)	800–2700	10	0.88	4.48	0.88	4.53
Malic acid (meq 100 g^{-1} FW)	800–2700	11	0.82	3.72	0.77	4.17

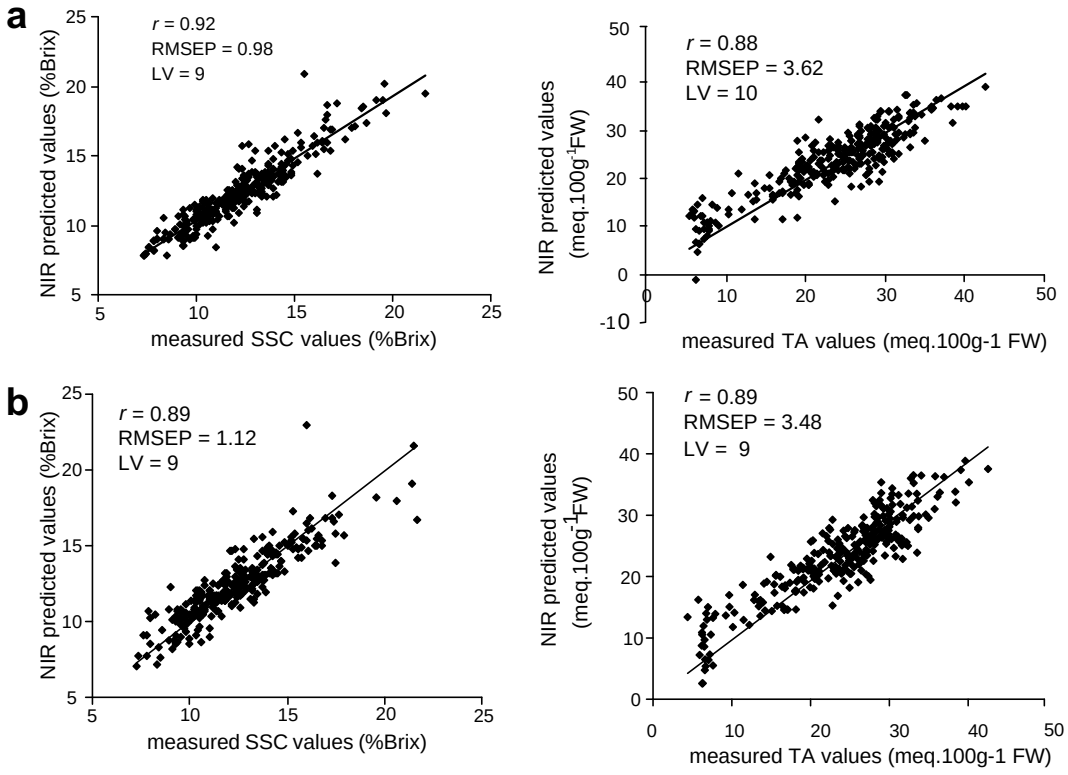


Fig. 3. NIRS prediction results of the established PLS models for SSC and TA of apricots fruit using spectra performed on the un-blushed side of the fruit (a) and spectra performed on the blushed side of the fruit (b).

results were comparable with *r* between 0.81 and 0.87 and RMSEP between 0.17 and 1.11 g 100 g^{-1} FW giving a prediction error between 17% and 22%. The best results were obtained for glucose present in apricot at an average level of 2 g 100 g^{-1} FW. In literature, an accurate prediction of individual sugars using FT-NIR technique has already been described but only in fruit juices (Rodriguez-Saono, Fry, McLaughlin, & Calvey, 2001).

3.4. Prediction of malic and citric acid content

Within the eight apricot cultivars at different ripening stages used in this work, the mean contents of malic and citric acids were 10 and 16 meq 100 g^{-1} FW respectively. Concerning the relationships between NIR measurement and malic and citric acid contents in apricot, *r* were equal to 0.82 and 0.88 and RMSEC were equal to 3.72 and 4.48 meq 100 g^{-1} respectively (Table 2). When the model was applied to predict 279 other apricot fruits, the prediction results were comparable with *r* of respectively 0.77 and 0.88 and RMSEP of respectively 4.17 and 4.53 meq 100 g^{-1} FW giving a prediction error of 40% and 28% respectively. In these cases, the error of calibration and prediction were not acceptable. In other conditions, on juice and in transmittance, better results have been obtained by Chen, Zhang, and Matsunaga (2006). Working on intact fruits presents some difficulties in comparison with juices. The first problem is related to the fruit inhomogeneity (Duprat et al., 1991). For example, in *Vitis vinifera* the main tartaric and malic acids are not evenly distributed in grapes and 70% of them are located in skin (Lamikanra, Inyang, & Leong, 1995). The second difficulty concerns NIR spectroscopy: the penetration of NIR radiation into fruit tissue decreases exponentially with the depth and the skin drastically reduces the light penetration (Nicolaï et al., 2007). The limited penetration of NIR radiation in apricot pericarp may explain the relatively poor predictions of individual acids.

3.5. Prediction of firmness and ethylene production

In our laboratory, we routinely measure the fruit firmness which is the pressure (kPa) required to deform the fruit of 3% of its height and the ethylene production (nmol kg^{-1} h^{-1}). This last feature allows the determination of the physiological stage of the fruits. The observed data between NIR measurement and these quality traits were too poor for allowing their analysis by NIR spectroscopy (Table 2). In fact RMSEC and RMSEP were greater than 48% and 30% for pressure and ethylene production respectively. In the literature, even for other species, no references have been found concerning the prediction of ethylene production, and only one for the prediction of respiration rate (Peirs et al., 2005). According to these authors, it was possible to predict the respiration rate with a RMSEP between 0.9 and 1.5×10^{-8} mol s^{-1} kg^{-1} depending on the cultivars. The RMSEP was 1.6×10^{-8} mol s^{-1} kg^{-1} with all cultivars in the calibration model. Concerning fruit firmness two main situations have been reported: on 'Sam' and 'Hedelfinger' cherries the NIR model appeared acceptable for predicting the compression force (Lu, 2001) and, on 'Golden Delicious' and ''Idared' apples, Zude et al. (2006) used the visible wavelength range from 500 to 730 nm to predict flesh firmness probably linked to a measure of maturity through the loss of chlorophyll in the fruit. On 'Satsuma' mandarin, in spite of acceptable correlation coefficient between NIR measurements and firmness (compression force) obtained, high standard errors of calibration and prediction were observed (Gomez et al., 2006). The present situation on apricot could be related to the large diversity as far as texture and firmness were concerned. Some further investigation could be inter-

estingly carried out in order to integrate such kind of co-variables in the analysis.

4. Conclusion

On intact apricot, near-infrared wavelength (800–2500 nm) could be used to accurately predict soluble solids and titratable acidity. But, the prediction of the other quality traits such as firmness, ethylene production, individual sugars and organic acids seemed not to be sufficiently accurate. In the future we plan to check the robustness of these established models on other apricot fruits, including different cultivars, harvested over different years and cultivated in different orchards. According to its non-destructive and rapid characteristics, the near-infrared spectroscopy appeared already as a suitable technique for screening quality parameters of a large number of apricots.

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