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Miniature handheld NIR sensor for the on-site non-destructive assessment of post-harvest quality and refrigerated storage behavior in plums

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ABSTRACT

This study evaluated the feasibility of using a handheld micro-electro-mechanical system (MEMS) spectrometer working in the 1600–2400 nm range for the measurement of quality-related parameters (soluble solid content, firmness, variety and post-harvest storage duration under refrigeration) in intact plums. Spectroscopic measurements were also made for each fruit using a diode-array Vis–NIR spectrophotometer (400–1700 nm) for purposes of comparison. A total of 264 plums (*Prunus salicina* L.) cv. 'Black Diamond', 'Golden Globe', 'Golden Japan', 'Fortune', 'Friar' and 'Santa Rosa', received and stored at 0 °C and 95% RH for 9 days, were used to build calibration models using different spectral signal pre-treatments and the modified partial least squares regression method. The two NIR instruments evaluated provided good precision, although the diode-array instrument yielded slightly greater precision for soluble solid content; statistic values were $r^2 = 0.73$ and the standard error of cross validation (SECV) = 1.11% for calibration, and $r^2 = 0.68$ and the standard error of prediction (SEP) = 1.22% for validation. Firmness measurements were less precise in both instruments, though again slightly better in the diode-array instrument: $r^2 = 0.64$ and SECV = 1.77 N for calibration; and $r^2 = 0.61$ and SEP = 2.30 N for validation, respectively. The performance of the two instruments for classifying plums by variety and by refrigerated post-harvest storage duration (0, 6 and 9 days) was evaluated using partial least square-discriminant analysis. A total of 96.5 % of samples were correctly assigned to their variety, while 94.5 % of plums were correctly assigned to their refrigerated storage day. In general, promising results were obtained with both instruments, with similar levels of accuracy for the measurements for soluble solid content, variety and refrigerated storage duration; the prediction model developed using the diode-array spectrophotometer provided better results for firmness.

1. Introduction

Plums vary considerably in size, shape, flavor and color. Harvest date is currently determined by specific skin color changes in each variety. As the fruit matures, its color changes due to chlorophyll degradation, enabling the expression of yellow pigments (carotenoids) both in pulp and in skin (Srivastava, 2002); other pigments – anthocyanins – give rise to the purple color characteristic of the skin surface of many plum varieties (Crisosto and Mitchell, 2003). However, the measurement of fruit firmness is recommended for cultivars in which skin ground color is masked by full red or dark color development before ripening (Crisosto and Kader, 2000). The rate of softening also varies according to orchard and season, so firmness measurements should be taken to protect fruit integrity during the ripening process (Crisosto, 2004).

Since consumer acceptance of plums is determined not only by color but also by total soluble solid content (Lozano et al., 2007), measurement of firmness and sweetness at harvesting is also recommended in order to establish minimum quality standards.

At the same time, the plum industry, supermarkets and retailers require rapid, non-destructive analytical techniques for assessing internal quality characteristics in fruits, enabling on-site and in-line classification and certification of fruit based on quality criteria prior to harvest, in packaging plants and even in supermarkets. These techniques, as well as avoiding possible discrepancies between different batches and samples, reduce wastage to a minimum.

Since very little is known about the physiological processes taking place in plums during cold acclimation, these non-destructive analytical methods could also prove valuable for estimating changes in fruit quality during post-harvest refrigerated storage. Since they are highly susceptible to internal breakdown and softening, plums can only be stored for a short time (Plich, 2006; Larrigaudiére et al., 2009). In general, fruit at 20 °C loses almost 8 N per day, compared with less than 4 N per day when it is stored

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at less than 2 °C. When fruit reaches "ready to buy" firmness, the rate of softening slows (Crisosto and Parker, 2003). Plums will reach their "ready to eat" firmness of 2–4 lb (cheek) after 2–3 days at room temperature, this is the firmness range at which most consumers claim the highest satisfaction when eating tree fruit (Crisosto and Parker, 2003).

Near-infrared (NIRS) spectroscopy has been developed and used as a convenient non-destructive technique for the individual quantitative and qualitative characterization of many kinds of fruit, giving rise to a new approach to market segmentation and fruit evaluation in both fresh and processed markets (Bureau et al., 2008).

Previous research has demonstrated the potential of NIR spectroscopy for assessing soluble solid content (SSC), firmness and/or other physiological properties in stone fruits (Peiris et al., 1998; Lu, 2001; Slaughter et al., 2003; Carlomagno et al., 2004; Walsh et al., 2004; Golic and Walsh, 2006; Nicolaï et al., 2007; Saranwong and Kawano, 2007). Specific studies of plums (Onda et al., 1994; Abu-Khalaf and Bennedsen, 2002; Walsh et al., 2004; Paz et al., 2008) have focused mainly on the measurement of SSC and – to a lesser extent – on the prediction of firmness or acidity. Predictions vary considerably, due to the particular characteristics of each variety.

The prediction of plum shelf-life – a major commercial parameter – has not been widely addressed using NIRS technology. Only one published study (Mizrach, 2004) discusses the use of an ultrasonic method to determine plum ripeness during storage.

Most NIRS studies of plums have been performed using either laboratory instruments or portable diode-array instruments – the latter allowing on-line measurement, though in a narrow region of the near-infrared, generally between 800 and 1100 nm, and always below 1700 nm, in order to ensure that these instruments are cost-competitive; this probably limits the development of applications providing sufficient predictive capacity for determining more complex quality parameters. No previously-published studies have focused on the use of miniaturized, handheld near-infrared instruments based on a MEMS (micro-electro-mechanical system). This type of sensor offers significant advantages in terms of size, weight, robustness, spectral range and low-cost manufacturing processes (Coates and Ramani, 2006). The reflected light in this system is collected and recombined (using a regular fixed grating) onto a single photodetector, a major cost advantage compared with diode-array instruments (Day et al., 2005). This kind of instrument has been used successfully for pre-harvest and post-harvest analysis of nectarines (Pérez-Marín et al., 2009).

The aim of this study was to compare the performance of calibration models obtained using two NIRS instruments, one of which is highly suited to field measurement (MEMS-based spectrophotometer) and the other better suited to on-line use in the packing house (diode-array spectrophotometer). Specific objectives were: (1) to establish correlations between NIRS measurements and the major physiological quality indices relating to plum internal quality (SSC and firmness), comparing calibration models obtained with different instruments, different spectral treatments and different wavelength ranges; (2) to assess the application of partial least squares-discriminant analysis to spectral data as a means of classifying plums according to variety and post-harvest refrigerated storage duration.

2. Materials and methods

2.1. Fruit samples

A total of 264 plums (*Prunus salicina* L.) cv. 'Black Diamond', 'Fortune' 'Friar', 'Golden Globe', 'Golden Japan', and 'Santa Rosa' harvested between June and September 2007 were evaluated. All varieties were received in 6 batches of 44 plums. In order to determine product post-harvest storage duration, batches were stored at 0 °C and 95% RH for 9 days. The product was kept under these conditions throughout the trial period, with samples drawn for analysis at 6 and 9 day intervals. The raw material sample (0 days' storage) served as control.

Prior to each measurement, the fruit sample was left at room temperature to allow the near-surface fruit temperature to rise to, and stabilize at, the laboratory temperature of 20 °C.

2.2. Determination of physical–chemical quality parameters

Firmness (penetration test) and SSC were determined using traditional destructive tests. Firmness was measured based on the resistance of the fruit flesh to penetration by a puncture probe. The Magness–Taylor technique was used to measure the maximum force required to pierce the plum to a depth of 10 mm with an 8 mm diameter probe. The measurement was carried out in a Universal Testing Machine (Model 3343 Single Column, Instron Corporation, Norwood, MA, USA). Crosshead speed was 0.0016 m/s (100 mm/min); skin was removed with a knife for M–T measurements; a 1000 N load cell was used for these firmness measurements. The firmness of each individual fruit was measured at two positions around the equator, approximately 180° apart, and perpendicular to the stem-calyx axis, and flesh firmness was calculated as the average of two measurements per fruit and expressed in Newton (N).

Next, two longitudinal (from stem end to calyx end) wedges were removed from each fruit, pressed through cheesecloth, and the SSC of the juice was measured with a temperature-compensated refractometer (model ATC-1, Atago Co., Tokyo, Japan).

2.3. Spectrum collection

Spectra were collected on all fruit in reflectance mode ($\log 1/R$) using two NIR instruments: (1) a handheld micro-electro-mechanical system (MEMS) spectrophotometer (Phazir 2400, Polychromix Inc., Wilmington, MA, USA), and (2) a diode-array VIS–NIR spectrophotometer (Perten DA-7000, Perten Instruments North America, Inc., Springfield, IL, USA).

The Phazir 2400 is an integrated near-infrared handheld analyzer that incorporates all the essential components to deliver on-site applications. These include a MEMS-based DTS NIR spectrophotometer and a tungsten light source for illuminating the sample in the near-infrared region. The reflected light is collected and measured by a single InGaAs photodetector, and the instrument has no moving parts. The spectrophotometer scans at 8 nm intervals (pixel resolution 8 nm, optical resolution 12 nm), across a range of near IR wavelengths (1600–2400 nm). Two spectral measurements were made with this instrument from both sides of the equator on each intact fruit, with a measurement time of 1–2 s. The two spectra were averaged to provide a mean spectrum for each fruit.

NIR spectra of intact plums were also captured using a Perten DA-7000 parallel diode-array Vis–NIR spectrophotometer (Perten Instruments North America Inc., Springfield, IL, USA). This instrument does not use any moving parts in the optics, making it very stable and suitable for on-line measurement, providing fast non-contact measurement (1–3 s). Plums were placed centrally upon the fruit holder, with the stem-calyx axis horizontal, and were irradiated from above by the light source while they rotated; the spectrophotometer scanned at 5 nm intervals, across a range encompassing the entire visible (400–780 nm) and near IR (780–1700 nm) wavelength ranges. Two separate spectral measurements were made on each plum, rotating the sample through