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Determination of sucrose content in sugar beet by portable visible and near-infrared spectroscopy

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ABSTRACT

Visible and near-infrared spectra in interactance mode were acquired for intact and sliced beet samples, using two portable spectrometers for the spectral regions of 400–1100 nm and 900–1600 nm, respectively. Sucrose prediction models for intact and sliced beets were developed and then validated. The spectrometer for 400–1100 nm was able to predict the sucrose content with correlations of prediction (r_p) of 0.80 and 0.88 and standard errors of prediction (*SEP*s) of 0.89% and 0.70%, for intact beets and beet slices, respectively. The spectrometer for 900–1600 nm had r_p values of 0.74 and 0.88 and *SEP*s of 1.02% and 0.69% for intact beets and beet slices. These results showed the feasibility of using the portable spectrometer to predict the sucrose content of beet slices. Using simple correlation analysis, the study also identified important wavelengths that had strong correlation with the sucrose content.

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1. Introduction

Sugar beet is grown in a wide range of climatic conditions and in about 50 countries worldwide, including North America (United States and Canada), South America (Chile), Asia, North Africa (Morocco and Egypt), and most of Europe (Mosen, 2007). Sucrose content is the most important trait in sugar beet production, and it is made up of more than 99.5% in the final white crystalline sugar. Hence breeders and researchers are striving to achieve high content of sucrose in beet production, using genetic, molecular, and conventional breeding approaches.

Rapid measurement of sucrose content in sugar beets can assist breeders in selecting promising germplasms and help sugar beet growers and processors in determining the yield and quality of beets after harvest and during storage and processing. Many methods have been used to measure the sucrose content of beets, including polarimetry, enzyme-based spectroscopic assays, and high-performance liquid chromatography (HPLC) (McGrath & Fugate, 2012). Polarimetry is the generally accepted method used in commercial sugar beet processing factories. Newer generation polarimetric instruments can measure sucrose from dark and coloured samples of molasses without juice clarification (Singleton, Horn, Bucke, & Adlard, 2002). Enzyme-based spectroscopic assays utilise a series of enzyme-catalysed reactions to quantitatively couple sucrose reaction to the synthesis of a spectrally detectable compound (Spackman & Cobb, 2002). A relatively rapid and inexpensive enzymatic-fluorometric microtitre plate assay was developed for sucrose quantification (Trebbi & McGrath, 2004). The method provided accurate and sensitive sucrose measurements from the tissues of young sugar beet roots with a coefficient of determination (r^2) of 0.976, but it was less accurate for older, field-grown root tissues (r^2 = 0.605). Owing to its high sensitivity and specificity, HPLC is also used in the sucrose analysis of sugar beet. But the technique is time-consuming and labour intensive in sample preparation and sequential analysis (~12 min per sample) (Mulcock, Moore, Barnes, & Hickey, 1985).

Numerous studies have been reported in recent years on using visible and near-infrared (Vis/NIR) spectroscopy for the spectral region of 400–2500 nm for fast measurement of soluble solids content and other quality attributes of apple (Fan, Zha, Du, & Gao, 2009; Mendoza, Lu, & Cen, 2012), peach (Carlomagno, Capozzo, Attolico, & Distante, 2004), pear (Xu, Qi, Sun, Fu, & Ying, 2012), pineapple (Chia, Abdul Rahim, & Abdul Rahim, 2012), Satsuma mandarin (Gómez, He, & Pereira, 2006), sweet cherry (Lu, 2001), and many other fruits (Camps & Christen, 2009; Cayuela & Weiland, 2010; McGlone, Jordan, Seelye, & Martinsen, 2002; Paz, Sánchez, Pérez-Marín, Guerrero, & Garrido-Varo, 2009; Wang, Nakano, & Ohashi, 2011). NIR reflectance spectroscopy was used

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to determine sucrose content in ground soybean samples (Sato, Zahlner, Berghofer, Lošák, & Vollmann, 2012). NIR spectroscopy was reported for achieving excellent predictions of sucrose in chocolate, with a correlation coefficient of 0.997 or higher (Da Costa Filho, 2009). Vis/NIR spectroscopy was also reported for measuring the sucrose content of sugarcane. Mat Nawi, Chen, Jensen, and Mehdizadeh (2013) applied visible and shortwave near-infrared (Vis/SWNIR) spectroscopy to predict the sugar content of sugarcane based on skin scanning. They reported an r^2 value of 0.91 and a root mean square error of prediction of 0.721 °Brix. Roggo, Duponchel, and Huvenne (2004) measured the sucrose content of more than 2700 homogenised beet brei samples from 15 different factories using Vis/NIR (400–2500 nm) and reported excellent results with a standard error of prediction of 0.10%.

While previous Vis/NIR studies have showed promising results for sucrose measurement in beets, they needed to destroy and homogenise beet samples. In addition, these studies were carried out using expensive laboratory Vis/NIR instruments that cover the entire visible and near-infrared region (i.e., 400–2500 nm). No research has been reported on measuring the sucrose content of intact and sliced beets using spectroscopic instruments that only cover the Vis/SWNIR region of 400–1100 nm or a portion of the NIR region (i.e., 900–1600 nm). Spectral measurements for these two spectral regions offer considerable advantages over the full Vis/NIR region because they can be done more quickly and conveniently, using low-cost portable or handheld CCD-based (charge-coupled device) (400–1100 nm) or InGaAs-based (indium gallium arsenide) (900–1600 nm) spectrometers.

Therefore, the objective of this research was to study the feasibility of Vis/SWNIR and NIR spectroscopy for predicting the sucrose content of intact and sliced sugar beets. The specific objectives were to: (1) measure spectra of intact and sliced sugar beets using two portable spectrometers operated in interactance mode, for the spectral regions of 400–1100 nm and 900–1600 nm, respectively; (2) develop statistical models for the spectral data to predict the sucrose content of intact beets and beet slices; and (3) identify important wavelengths that have strong contributions to the sucrose content prediction.

2. Materials and methods

2.1. Samples

Sugar beets used for this experiment were harvested from the Michigan Sugar Company's official variety trials in the experimental field of Michigan State University's Saginaw Valley Research and Extension Center at Frankenmuth, Michigan during the 2012 harvest season. The beet samples came from different commercial hybrid varieties, and they were stored in refrigerated storage at 4 °C for about 2 months prior to the experiment. Only those beets that did not show physiological deterioration (e.g., rot) and physical damage (i.e., cuts and bruises) were selected for the study. The samples were moved from cold storage within 24 h prior to the experiment and were washed to remove adhered soil. Spectral measurements were first made on intact beets using two portable spectrometers for the spectral regions of 400–1100 nm and 900–1600 nm, respectively (see details in the following section). Thereafter, the beet samples were cut into two sections in the direction that is approximately perpendicular to the crown-root axis. Spectral measurements were immediately made at the centre of the lower section of the beet. After the spectral measurements had been completed, wet lab chemistry analysis (i.e., HPLC) was performed to measure the sucrose content of each beet sample. A total of 398 beet samples were tested in the study.

2.2. Visible and near-infrared spectroscopic measurement

Two portable spectrometers were used for this research: a Vis/SWNIR spectrometer (Model LOE-USB; tec5USA Inc., Plainview, NY) for the spectral region of 400–1100 nm, and an NIR spectrometer (Model NIR 512L-1.7T1; Control Development Inc., South Bend, IN) for the spectral region of 900–1600 nm. Both spectrometers were operated in interactance mode (Fig. 1a). The sugar beet sample was illuminated with a broadband light source that was delivered from the 25-mm diameter ring of a lighting/detection probe, which was in contact with the beet sample. In the centre of the probe was a light detector covering an area of 11 mm diameter to receive the light that had travelled through the flesh tissue. A separating distance of 3.5 mm between the light illuminating ring and the detector ensured that only the light that travelled through the beet tissue would be received by the probe (Fig. 1b). A soft rubber sealing ring was used between the illuminating ring and the detector to block the illuminating light from entering the detecting area directly. In addition, a sponge ring (5 mm thick) was attached to the periphery of the probe to block ambient light.

For Vis/SWNIR measurements, the lamp power supply was set to 100 W and the integration time of the spectrometer was set to 575 ms. For NIR measurements, the lamp power supply was set to 200 W and the integration time was set to 4 s. Spectral measurements were made at a location approximately 10 mm from the crown end of each intact beet. For both spectrometers' measurements for intact beets, three scans were acquired from the same location of each beet sample, and they were then averaged for further analysis.

After spectral measurements for the intact sample had been made, the beet was sliced in the transverse direction (i.e., perpendicular to the crown-root axis) into two sections using a stainless steel kitchen knife. Spectral measurements were then made, using each spectrometer, from the lower section (toward the root side) of the beet. Two scans were taken at two locations that were approximately equidistant from the centre and the outside edge of each beet slice, and these values were then averaged. The integration times were 0.2 s and 1.5 s for Vis/SWNIR and NIR measurements, respectively. Thereafter, one cylindrical specimen of 50 mm in diameter and 10 mm in height (without skin) was taken from one of the two detection locations for the beet slice and subsequently used for sugar content analysis by HPLC. Reference spectra were acquired from a white Teflon disk in order to calculate the relative interactance of each beet sample.

2.3. Sucrose measurement by HPLC

Sucrose content was measured by means of high-performance liquid chromatography (HPLC) (Trebbi & McGrath, 2004). Each beet specimen of 30 g fresh weight was lyophilised until the pressure was <1 mTorr for at least 3 h and then ground to fine powder with mortar and pestle. Pulverised dried tissue (100 mg) was resuspended in 4 mL of 80% ethanol in a 5-mL fluted-cap tube (USA Scientific, Inc., Ocala, FL) and placed horizontally on an orbital shaker (50 rpm) at 40 °C for 16 h. The suspension was centrifuged at 3000*g* for 10 min to obtain the clarified ethanol sugar extract. An aliquot (1.0 mL) of clarified ethanol sugar extract was vacuum dried, the pellet was resuspended in 1.0 mL of high-resistivity water (18 MΩ cm^{-1}), and the solution was passed through a 0.22-μm nylon filter (Spin-X Centrifuge Tube Filter; Corning, New York, NY). The aliquot of water-resuspended sugar extract (1.0 mL) was used for HPLC analysis with a 6.5 mm × 300 mm Waters Sugar-Pak I carbohydrate column (WAT085188; Waters Co., Milford, MA). The mobile phase was 134 μM Na2CaEDTA at a constant flow of 0.5 mL min^{-1}, 90 °C, 12 min run time, and quantified with a Waters 410 differential refractometer held at 35 °C,