Determination of Sucrose Levels in Infant Cereals Using the Agilent Cary 630 ATR-FTIR Spectrometer

Application Note

Food testing and agriculture

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Abstract

Cereal-based and sucrose-coated infant cereals were ground, and the sucrose, glucose, and fructose concentrations were determined with the Agilent Cary 630 Fourier transform mid-infrared, FTIR, instrument and compared to values measured by HPLC. The results indicate that use of the portable FTIR instrument was in line with the HPLC measurements, and that the Cary 630 is suitable for real-time analysis of sugar concentration in breakfast cereals.

Agilent Technologies

Introduction

Sucrose coating of breakfast cereal is used to improve taste and appearance, but due to health concerns, there is a need to measure and control the amount of sugar present in these cereals. Controlling the amount of sugar coating can be difficult during manufacturing, and this increases the need to measure sugar in the finished product. Chromatography or wet chemistry is often used for this purpose, however, these methods are time-consuming and require additional sample preparation. For this reason, there is substantial interest in methods for measuring sugar that eliminates some of the issues associated with traditional analytical methods.

This application note shows [1] that the Agilent Cary 630 FTIR Spectrometer equipped with a single-reflection diamond attenuated total reflectance (ATR) sample interface provides equivalent results to that of commonly used HPLC methods. The advantages provided by the Cary FTIR system relative to the chromatography method include:

- The speed of analysis is increased.
- No sample dilution or preparation other than grinding is required.
- The level of user experience required to obtain reliable answers is reduced.
- Samples do not have to be sent to a remote lab for analysis, that is, the spectrometer can be used where and when the measurement needs to be made.

This latter point is important since there is an increasing demand to analyze food ingredients close to the point of production, or where the product is received for further processing for authenticity and content. Midinfrared spectroscopy is a nondestructive, rapid screening tool for quality assurance, and Agilent FTIR systems such as the Cary 630 [2,3] or the Agilent 4500 fully portable FTIR [4] systems are valuable assets for use in the field, at point of reception, or on the manufacturing line for food stuff.

Experimental

Cereal samples (90 samples including plain, sucrose-coated, or flavored) were individually ground in a blender. Each powdered sample was placed directly on the FTIR spectrometer fitted with an Agilent diamond ATR sample interface (Figure 1). A sample press, which is part of the ATR interface, was used to ensure that the ground powders were in good contact with the sensor surface. Spectral data, consisting of 64 co-added interferograms, were collected in the 4,000–700 cm^{-1} region at 4 cm^{-1} resolution, and analyzed using a commercial multivariate analysis software package [5]. The spectra were normalized, and second-derivative transformed through a Savitsky-Golay second order polynomial filter with a 35-point window, prior to the partial least squares regression analysis.

Figure 1. An Agilent Cary 630 FTIR Spectrometer equipped with diamond ATR sample interface was used for the measurement of sugars in cereal.

Sucrose, glucose, and fructose levels were determined using a standard HPLC method on samples that had been ground, dissolved, and filtered.

Results and Discussion

The sugar levels present in the infant cereals, as determined by the HPLC reference method (Tables 1A,1B), varied from 0.5 to 18.1 g/100 g of cereal depending on the presence or lack of a sugar coating.

Table 1A. Average Glucose and Fructose Levels in Infant Cereal, Per 100 g of Cereal

Sugar	Amount (g)
Glucose	0.66 ± 0.32
Fructose	1.09 ± 0.51

Table 1B. Average Sucrose Levels in Sugar-coated and Uncoated Infant Cereals, Per 100 g of Cereal

	Uncoated cereal	Sugar-coated cereal
Sucrose	1.2 ± 0.7 g	11.8 ± 3.5 g

The midinfrared spectra of the powdered cereal samples (Figure 2) show a strong, broad absorbance at approximately 1,000 cm^{-1}, which arises from the contribution of both the sugar and corn starch components present.

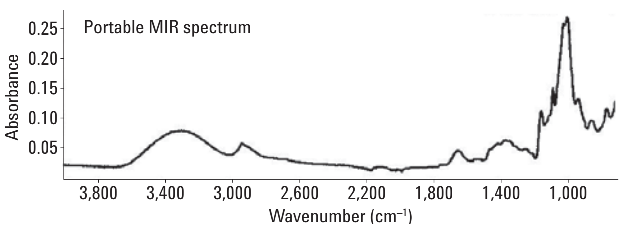

Figure 2. Mid IR spectrum of infant cereal. Strong absorption in the 1,200–900 cm^{-1} region is due to C-C and C-O stretching and C-O-H and C-O-C deformation. A characteristic band for sucrose at approximately 1,000 cm^{-1} is obscured by the strong absorption of corn starch.

The spectral data were processed using multivariate partial least squares. An independent external validation set of 20 additional samples shows high correlation for sucrose (> 0.95) when compared with data obtained by the HPLC reference method (Figure 3, Table 2).

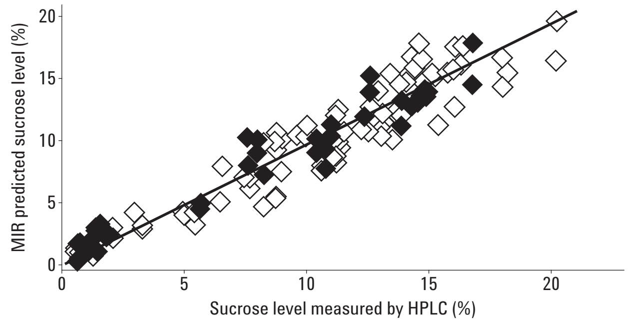

Figure 3. Estimated sucrose concentrations from FTIR spectra correlated with HPLC results through a multivariate PLS model. Black squares are the validation samples.

Table 2. Performance of Calibration and Prediction Model Generated from Spectra Obtained with the Agilent Cary 630 FTIR Spectrometer for Estimating Sucrose Concentration in Corn-based Infant Cereals

	Calibration model (n = 70)	Validation model (n = 20)
Sucrose range (%)	0.5–18.1	0.6–16.7
Number of cereal samples	70	20
Standard error of cross-validation in calibration model	1.46	1.27
Correlation coefficient	0.97	0.97
SD of reference data/SE of prediction	3.94	4.41

Similarly, FTIR results for total sugars present (glucose, fructose, and sucrose) compared to those obtained by the HPLC reference method also show very good correlation (Figure 4, Table 3).

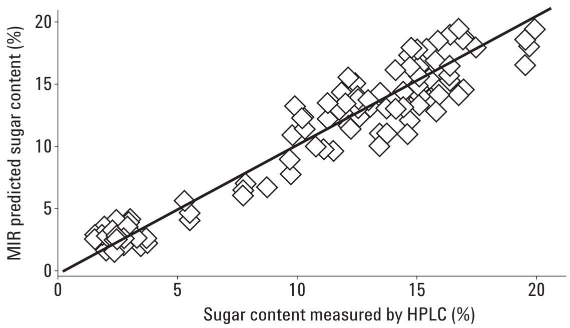

Figure 4. Calibration plot generated by comparing total sugars present by FTIR spectra correlated with the HPLC reference method results through a PLS method.

Table 3. Statistics for the Calibration Curve Generated by Correlating FTIR and HPLC Results

	Calibration model (n = 80)
Total sugars range (%)	1.4–19.9%
Number of cereal samples	80
Standard error of cross-validation in calibration model	1.56
Correlation coefficient	0.96
SD of reference data/SE of prediction	3.80

The compact Cary 630 and 4500 portable spectrometers use highly intuitive Agilent Microlab software. This software enables the PLS calibrations to be imbedded in a push button method that enables users of varied experience to obtain and report accurate results, including those users who have no understanding of the underlying chemometric models employed.

Conclusion

Midinfrared spectra of infant cereals recorded by the Agilent Cary 630 FTIR system yielded very good correlation with HPLC data for both sucrose and total sugars present. This is another demonstration of the value of compact Agilent FTIR spectrometers for routine analysis and quality control in the food industry. The method to measure sucrose in these cereal products by FTIR is far simpler than that required for chromatographic analysis. It is more rapid and avoids almost all of the sample prep required by HPLC. Because the level of technical expertise needed to get reliable results with the FTIR system is much less than that required for chromatography, the mid IR system can be used in a variety of settings and by personnel with all levels of training. In addition to the Cary 630 spectrometer, the availability a fully mobile, battery-powered Agilent 4500 portable FTIR enables analysts to make these measurements out of the lab and closer to the sample whenever required.

Acknowledgement

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References

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