APPLICATION BRIEF

FT-IR NIR Spectrometry

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Single NIR Measurement for the Detection of Adulteration and Measurement of Important Parameters in Cocoa Powders

Introduction

Cocoa powder is a product regularly used within the personal care, food, and beverages sectors. There have been reports indicating several health benefits of cocoa, from lowering blood pressure antioxidants to containing essential fatty acids, fiber, and minerals.

There have been cases of cocoa adulteration reported in Eastern Europe and across the world. In a recent case, samples contained 20% less cocoa compared to values listed on the labels, which was found to be fraudulent activity by the supplier.[1]

The initial method used to assess the quality of cocoa beans is the cut-and-taste test, although this is a subjective technique. Liquid chromatography methods are also often used for testing cocoa powder, but they are time consuming and can be complicated. A much faster and easier technique to verify the authenticity of cocoa is Near-Infrared (NIR) spectroscopy.

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NIR Methodology

Spectra of six cocoa brands and two adulterants (arrowroot and dark rye flour) were collected on a PerkinElmer Frontier™ NIR spectrometer in reflectance using the NIRA II sampling accessory at a spectral resolution of 16 cm^{-1} using 32 scans. These spectra were entered into PerkinElmer's Adulterant Screen™ in the Spectrum 10 software as a library of material (good) and adulterant spectra, as shown in Figure 1. Different lots of one of the cocoa samples were then spiked at concentrations of 10% and 20% weight/weight concentration of arrow root and dark rye flour to test the capability of Adulterant Screen™.

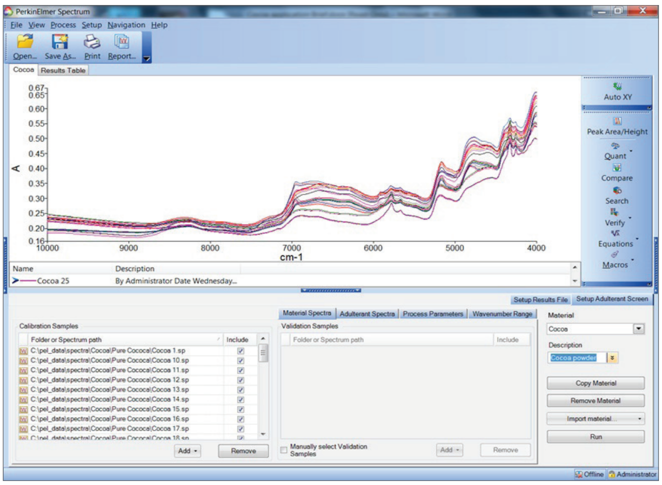

Figure 1. Adulterant Screen setup for cocoa powder showing cocoa powder material spectra.

Adulterants are commonly added to products at for financial gain. Adulterant Screen is able to correctly identify the adulterant used and estimate its concentration, as shown in Table 1, and can give an estimated level of detection of the adulterant, in this case <1%.

Table 1. Results from Adulterant Screen for spiked samples.

Sample Name	Adulterant	Level	Confidence	Material Fit
10% Dark Rye	Dark Rye Wholemeal Flour	0.10111	Likely	Abnormal
20% Dark Rye	Dark Rye Wholemeal Flour	0.18440	Likely	Abnormal
10% Arrow	Arrowroot	0.10105	Likely	Abnormal
20% Arrow	Arrowroot	0.17882	Likely	Abnormal

Adulterant Screen can be deployed using Spectrum Touch™ methods. This allows for an easy-to-use software environment for routine operators. A sample spiked with 1% Dark Rye Wholemeal Flour was tested using a Spectrum Touch method and the same Adulterant Screen. The resulting output in Figure 2 shows that the sample failed because adulterants were detected.

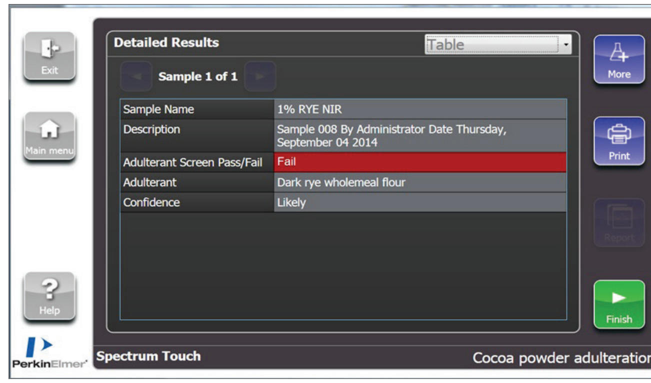

Figure 2. Spectrum Touch method deployment for an adulterated cocoa sample.

The NIR spectra of the samples also have the capability of determining the fat and dry-mass contents of cocoa samples. Cocoa 1 and 6 contained 22.6 g and 2.3 g of fat per 100 g of cocoa powder high-fat, respectively. The different fat levels are apparent in the spectra shown in Figure 3, particularly in the first overtone region of the C-H stretch, just below 5,900 cm^{-1} and the combination region at about 4,300 cm^{-1}.

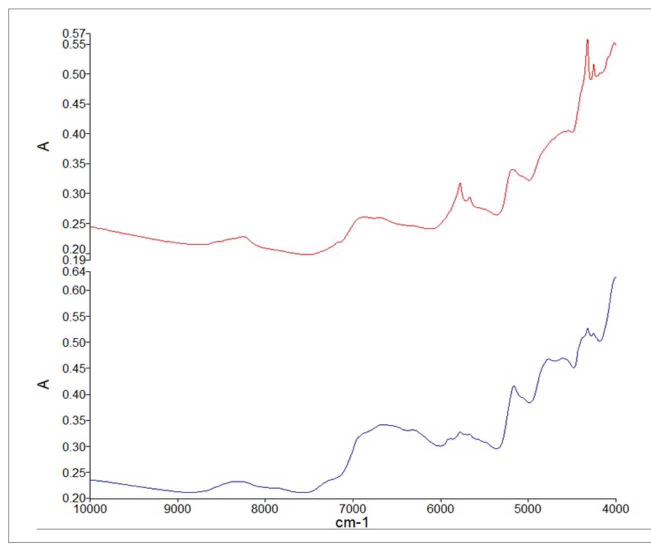

Figure 3. NIR spectra of high fat cocoa (top) and low-fat cocoa (bottom).

The spectral differences can be further highlighted by applying the second derivative to the spectra as shown in Figure 4. Applying the second derivative to the spectra will remove any broad baseline slope and offset due to the different scattering properties of the powders. These spectra show clear differences between the low- and high-fat cocoa powders and would form the basis of any quantitative measurement.

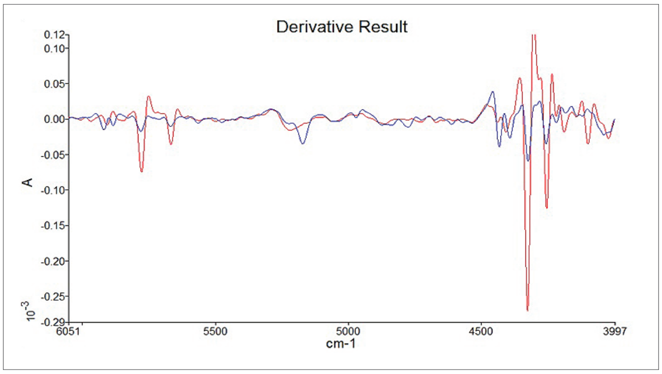

Figure 4. 2nd derivative of high-fat cocoa (red) low-fat cocoa (blue).

The dry-mass content of the powders can be measured in the combination region of the spectrum just above 5200 cm^{-1}. The second derivative spectra of a series of cocoa powders with very small variation in the dry-mass content are shown in Figure 5.

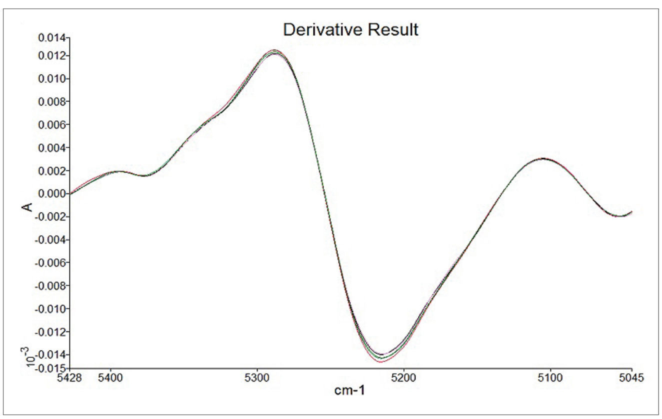

Figure 5. Variation in dry-mass content of cocoa powders.

This spectral region would form the basis for a quantitative measurement of the dry-mass content of the cocoa powders.

Conclusion

A single NIR measurement of cocoa powders can allow easy measurements for adulteration of the material and, with calibrations, also allow for determination of fat and dry-mass content within cocoa powder. Adulterant Screen allows for a fast, easy, and low-cost method for screening adulterants within cocoa powder. New adulterants can be added into the method simply by measuring the spectrum of the pure adulterant. This results in an easier method for the detection of adulteration in cocoa powder.

Reference

1. Czech Agriculture and Food Inspection Authority: Press Release Polish cocoa adulteration classes in Kaufland 04/25/2012

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