APPLICATION BRIEF

Near-Infrared Spectroscopy

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Use of NIR Spectroscopy and Adulterant Screen for the Detection of Common Adulterants in Milk

Introduction

The value of milk on the open market is linked to its protein content, and standard methods for protein analysis rely on a simple nitrogen assay, with the protein concentration inferred from the nitrogen content. Consequently, the addition of chemicals rich in nitrogen, such as urea, can artificially increase the apparent protein content and thus the price demanded. Urea occurs naturally in milk and is typically present at levels of about 0.02% - 0.05%. Higher levels of urea in milk are present only in cases of adulteration. Cane sugar is another known milk adulterant used to increase its carbohydrate content and weight. This allows extra water to be added into the milk without detection from a standard lactometer test for milk quality.

NIR spectroscopy coupled with PerkinElmer's Adulterant Screen™ is shown here to be capable of detecting adulterants intentionally or accidentally added to milk.

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Method

Spectra of a variety of milk samples were collected on a PerkinElmer Frontier™ NIR spectrometer in transflectance using the NIRA II sampling accessory. The set of samples was selected in order to cover adulteration within a broad range of different types of milk, and included full fat, semi-skimmed, skimmed, lactose-free, and organic varieties. These spectra were defined in Adulterant Screen as our set of Material Spectra and represented "good samples." A spectrum of cane sugar, urea, and a spectrum of a 10% aqueous urea solution were measured as adulterants.

A full-fat milk sample was spiked with urea to give a urea concentration of 1% w/w. The spectra of the milk sample and the urea-spiked sample are shown in Figure 1.

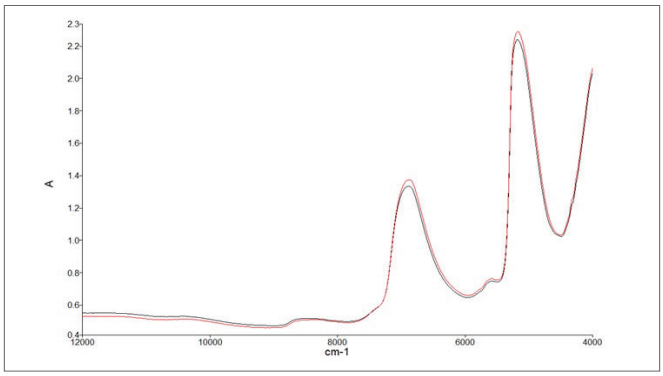

Figure 1. Spectra of whole milk (red) and milk spiked with 1% urea (black).

Although the spectra appear to be very similar, the application of a second derivative function shows that there are clear differences in the spectral region associated with urea absorptions as shown in Figure 2, thus allowing for the detection of urea in an adulterated milk sample.

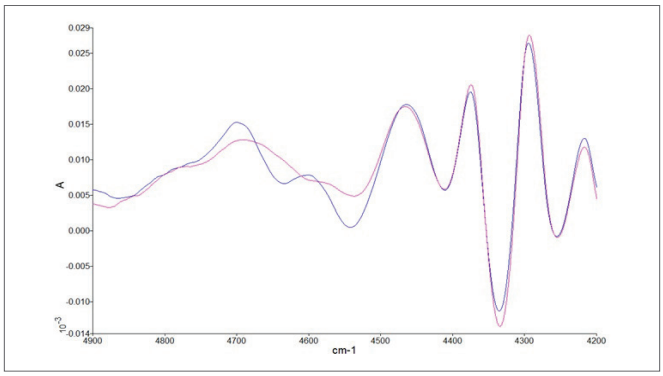

Figure 2. Second derivatives showing differences between milk (purple) and sample spiked with urea (blue).

The normal process for finding adulterants simply requires the measurement of a sample of the adulterant to provide a reference for subsequent comparison with the sample spectra. However, in the case of urea, the infrared spectrum changes significantly in the presence of water, resulting in the urea adulterant being incorrectly determined. The spectra of urea powder and urea solution (with the water subtracted) are shown in Figure 3.

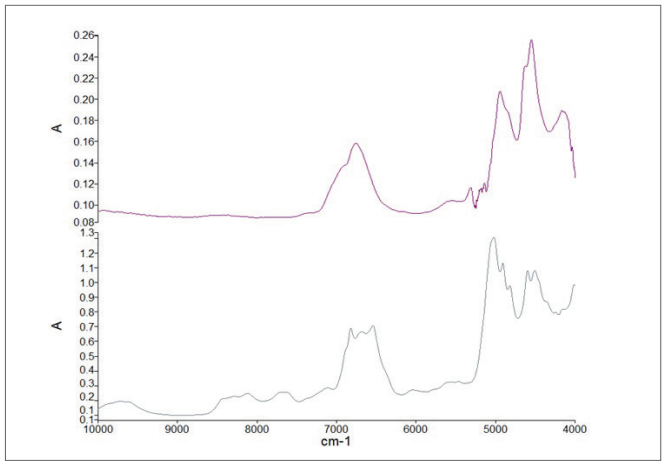

Figure 3. Spectra showing differences between 10% urea solution with water subtracted (top) and urea powder (bottom).

The urea solution spectrum with water subtracted is a more representative spectrum of urea in aqueous samples, such as milk, and should be used as the adulterant spectrum. This spectrum was then normalized to represent a 100% urea standard and added to the list of adulterants for this method. A spiked full-fat milk sample could then be checked for adulterants using Adulterant Screen. The result for the spiked sample is shown as Figure 4.

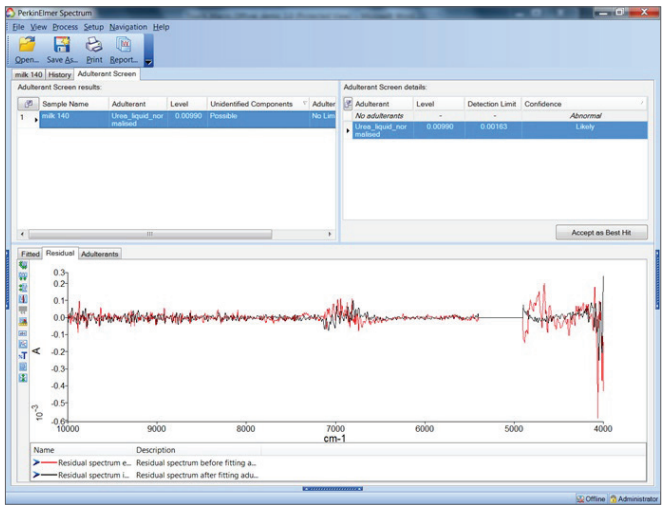

Figure 4. Adulterant Screen result for full-fat milk sample spiked with 1% urea.

The Adulterant Screen result that appears here shows the spectral residual when the unknown is tested against the model for the "good spectra" and the improvement achieved in reducing the residual when the spectrum of the adulterant (urea) is added. Adulterant Screen will also generate an estimated concentration of the adulterant and a detection limit. The estimated concentration of urea in this sample is 0.990%, very close to the known 1% concentration.

A different full-fat milk sample was spiked, but this time with sugar to give a 10% and 20% w/w of sugar. Adulterant screen was applied, and the results are shown in Table 1.

Since the cane sugar spectrum was measured in reflectance on the powder and the milk measurement is performed in transflectance on the liquid there are differences between the expected and observed levels of the adulterant. Therefore, the adulterant spectrum for sugar was normalized based on a known 10% sample. The limit of detection for cane sugar as estimated by the software is 3.5%.

Conclusion

Adulterant screen has been shown to be an effective method in detecting the adulteration of milk. Normalization of adulterant spectra may be required for some samples due to spectral changes that occur in solution. Nevertheless, NIR with Adulterant Screen is a fast and simple technique to use for the detection of adulterants. Additional adulterants can be readily added to the method by simply measuring the spectrum of the pure adulterant; thus providing a dynamic platform for adulterant screening.

Table 1. Adulterant screen results for milk spiked with sugar.

Sample Name	Adulterant	Level	Confidence	Material Fit
10% sugar	Cane sugar	0.10529	Likely	Abnormal
20% sugar	Cane sugar	0.21032	Likely	Abnormal

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