

FT-NIR Spectrometry

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Rapid Testing for Adulteration of Yogurt Candy using Near-infrared Spectroscopy and Adulterant Screen

Introduction

Melamine is an adulterant commonly found in milk, as it can increase the nitrogen content. Therefore, its apparent protein content, resulting in a better market

price. Melamine adulteration can be fatal, as was highlighted in 2008 when six infants died due to melamine adulteration in milk powder and thousands were sickened in China. Consequently, there have been stricter regulations globally and improved testing methods including the use of the PerkinElmer DairyGuard™ instrument for powdered milk testing.

However, cases of melamine adulteration are still appearing in other products. This year in Guangdong Province, China, 25 tons of yogurt candy tablets were seized as they were found to contain melamine. What follows is a description of a near-infrared (NIR) testing method of yogurt candy for melamine adulteration.

Experiment

Four different flavors of commercially-available yogurt candy were purchased (peach, cherry, blueberry, and tropical). The samples of yogurt candy for testing were ground into a powder and placed in a Petri dish. Spectra were collected on a PerkinElmer Frontier™ NIR spectrometer in reflectance using the sample cup spinner on the NIRA II sampling accessory at a spectral resolution of 16 cm⁻¹ using 32 scans. Several replicate samples of the yogurt candies and the spectrum of pure melamine were added into Adulterant Screen™ as “Material” and “Adulterant” spectra, respectively. A sample of a mixture of the different yogurt candies was prepared using equal amounts of each flavor. The NIR spectra of melamine and the mixture of yogurt candies are shown in Figure 1.

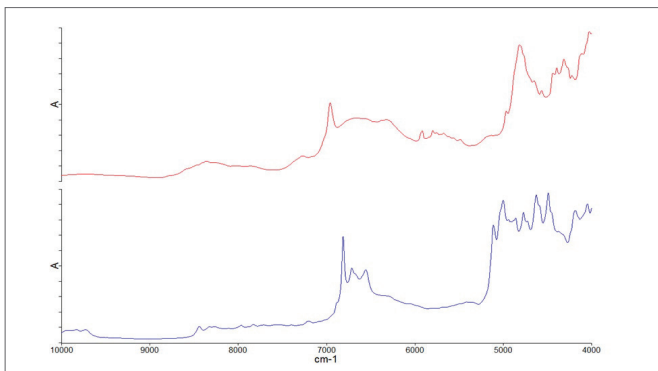


Figure 1. NIR spectra of melamine (blue) and mixture of yogurt candies (red).

The mixture of yogurt candies was spiked with melamine at 9.8%, 1% and 0.2% w/w levels and the spectra measured.

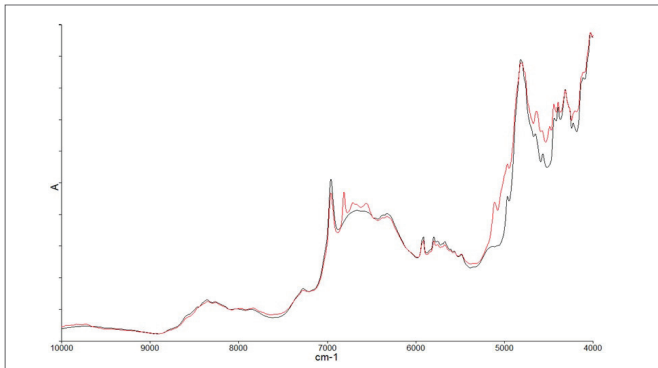


Figure 2. Spectra of yogurt sample with 9.8% melamine (red) and yogurt (black).

Figure 2 shows spectral features related to melamine present in the yogurt sample containing 9.8% melamine. The Adulterant Screen method was used to predict the presence and level of melamine in the samples, shown in Table 1. The close correlation between the measured amount and the predicted amount of melamine in the yogurt candy indicates that this Adulterant Screen method can be used to accurately predict the level of adulterant contamination.

Table 1. Adulterant Screen results for spiked samples.

Sample Name	Adulterant	Estimated Level (%)	Confidence
0.2% Melamine	Melamine	0.275	Likely
1% Melamine	Melamine	1.358	Likely
9.8% Melamine	Melamine	10.004	Likely

The Adulterant Screen method generates residual spectra from the analysis; showing the residual spectrum before adding in the adulterant spectrum, and the residual spectrum after adding in the adulterant spectrum. The spectral bands in the residual spectrum should decrease with the addition of the adulterant. Any remaining residual features are not accounted for by the method. Adulterant Screen allows for multiple adulterants to be detected and their concentrations estimated. The residual spectra from the analysis of the 0.2% melamine sample are shown in Figure 3.

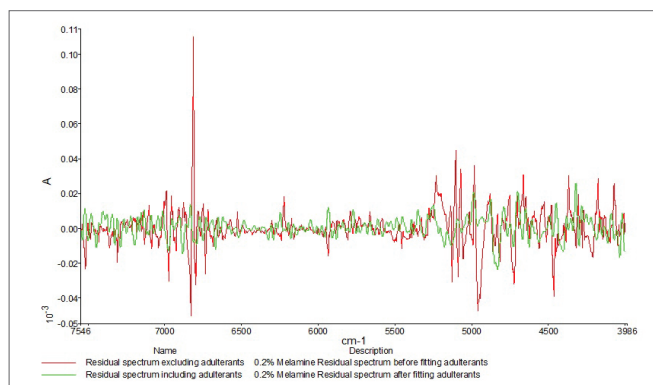


Figure 3. Residual spectra from Adulterant Screen for the 0.2% Melamine sample. Excluding adulterants (red), Including adulterants (green).

The addition of melamine into the Adulterant Screen model significantly reduces the residual spectrum, indicating the presence of melamine as an adulterant.

Conclusion

NIR spectroscopy with Adulterant Screen is a quick and simple method for detecting melamine adulteration in yogurt candy. The software is able to accurately predict the concentration level of melamine and identify any new adulterants. Rapid deployment of the method can be achieved for yogurt candy and similar products.