APPLICATION NOTE

FT-IR Spectroscopy

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The Use of FT-IR Spectroscopy as a Technique for Verifying Maple Syrup Authenticity

Introduction

Although usually not thought of until pancakes or waffles are on the table, maple syrup is a serious business. It is one of the key crops where demand is greater than supply. Surprisingly, it takes 10 gallons of sugar maple tree sap to produce one quart of maple syrup. Because the syrup produced is only 1/40th of the actual sap yield, unscrupulous syrup suppliers are tempted to fraudulently adulterate their products with lower value commodities, in order to maximize their profit. Adulterants include cane syrup, high fructose corn syrup, beet syrup, and rice syrup. Infrared spectroscopy is shown here to be a fast and easy technique for detection and identification of these adulterants.

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Method

Samples of Grade A maple syrup, corn syrup, high fructose corn syrup, and rice syrup were analyzed on the PerkinElmer Frontier™ Fourier Transform Infrared (FT-IR) spectrometer from 4,000 to 650 cm^{-1}, using a three-bounce Universal Attenuated Total Reflectance (UATR) sampling accessory. Samples were scanned by placing a single drop directly onto the diamond crystal of the UATR. After scanning, the UATR was cleaned using isopropyl alcohol on a laboratory wipe. Seven replicate measurements were performed for each sample type using a fresh aliquot for each scanned sample. Additional dilutions of maple syrup with the adulterants were prepared and scanned in order to validate the method.

Spectra of maple syrup and two of the common adulterants are shown in Figure 1A and an expanded region of interest in Figure 1B.

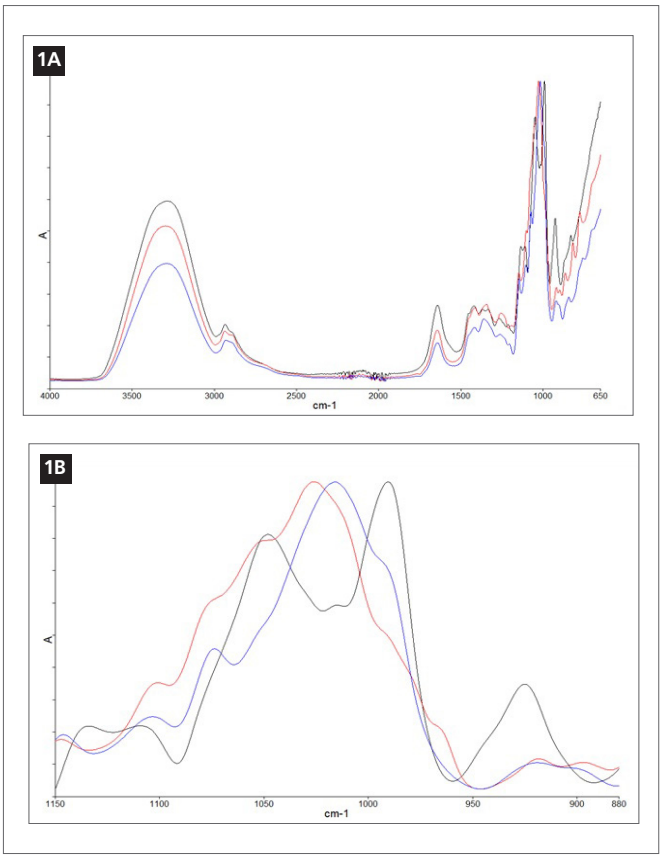

Figure 1A. FT-IR overlay of maple syrup and its common adulterants. maple syrup (black), high fructose corn syrup (red), rice syrup (blue). *Figure 1B.* Expanded spectral overlay of maple syrup (black), rice syrup (blue), and high fructose corn syrup (red), from 1150-880 cm^{-1}.

The spectra of these materials exhibit differences particularly in the spectral region from 1100 - 900 cm^{-1}.

A Soft Independent Model by Class Analogy (SIMCA) model was created to see if there was a measureable difference between the maple syrup and the adulterants. The SIMCA model is shown as Figure 2.

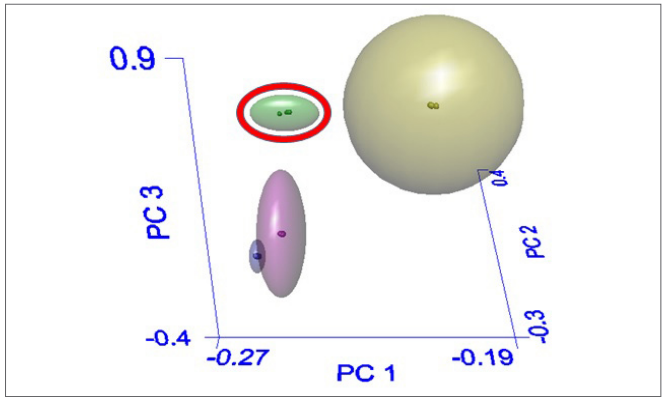

Figure 2. SIMCA model for the maple syrup and adulterants dataset (maple syrup highlighted).

There is good separation between maple syrup and the other adulterants, with a little overlap between the corn syrup and rice syrup. This model could be used to determine if the sample of interest is a maple syrup or not.

An Adulterant Screen™ method was set up using all of the maple syrup spectra as "Material Spectra" and single spectra of the adulterants as the "Adulterant Spectra". A Spectrum Touch™ method was set up incorporating the SIMCA model and the Adulterant Screen method into a simple user interface for the routine analyst. The model and method were tested using one of the diluted samples, 10% high-fructose corn syrup in maple syrup. The Spectrum Touch results screen is shown as Figure 3.

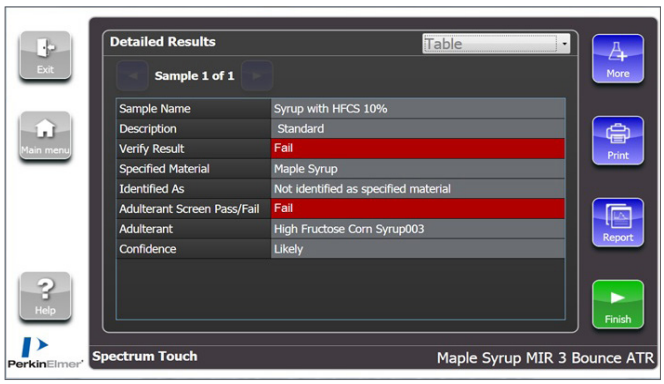

Figure 3. Spectrum Touch results screen.

This test sample fails both the SIMCA and the Adulterant Screen analysis. SIMCA indicates that this test sample does not conform to the maple syrup spectra in the model and Adulterant Screen states that adulteration is likely with high fructose corn syrup.

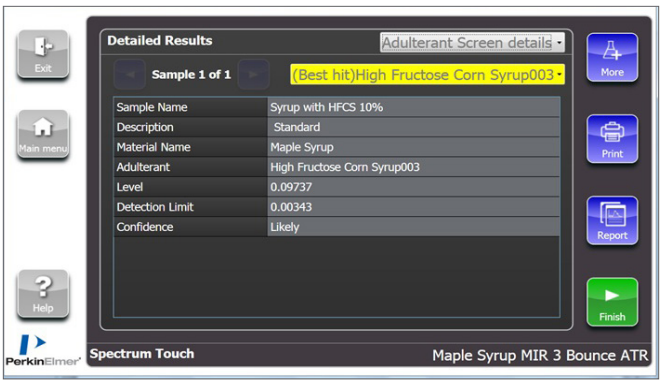

Figure 4. Spectrum Touch results screen highlighting the "Adulterant Screen details" view.

The Adulterant Screen results not only predict which adulterant is present, it will also predict how much of that adulterant is present and estimate its detection limit. This is achieved without the need for the lengthy process of preparing and measuring spectra of calibration standards. In this case, Adulterant Screen predicts a concentration level for the high-fructose corn syrup adulterant at 9.737%, very close to the actual concentration of 10%.

Conclusion

As maple syrup is a prime target for food fraud, there is a clear need to test for its authenticity. It has been demonstrated that utilizing an FT-IR empowered method with Adulterant Screen and SIMCA allows for the measurement of maple syrup quality and detects any adulterants that may be present. The advantage of Adulterant Screen is that it only requires base materials for the method and is fast and easy to use. This dramatically reduces the time required to develop a screening method as quantitative calibration development is not required. Additional adulterants can readily be added into Adulterant Screen without having to recalibrate the method.

References

- http://www.mi-maplesyrup.com/education/facts.htm
- http://tapmytrees.com/coprsap.html

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