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Food Fraud - Adulteration Control on Quebec Maple Syrup

Food fraud for economic reasons can affect all value-added foods. Although it is a very old practice, with the liberalization of the markets, this type of fraud is becoming increasingly worrying and affects producers who see part of their income drop as much as consumers who find themselves cheated and left with a falsified product.

Maple syrup, a natural sweetening agent that contains value-added compounds, can also be the target of adulteration through the addition of cheap sugars. Official methods to counter maple syrup adulteration have been developed since the 1980s (e.g. AOAC Official Method 984.23). However, since these methods cover a limited number of adulterants, fraudsters can easily circumvent them and use other types of adulterants. Thus, during the last decade, a new analytical approach has been adopted with the development of non-targeted analytical methods. The principle of non-targeted analytical methods is based on screening a very large number of authentic samples and chemometrics in order to construct a fingerprint specific to the product to be characterized. The success of this approach requires recognized scientific expertise and a perfect knowledge of the factors that influence the natural composition of the product. In fact, even with state-of-the-art instruments (e.g. NMR), the samples used to build the reference model must cover all the variables that affect the composition of the product to guarantee the reliability of the results and avoid false positives that will penalize producers instead of fraudsters.

In addition, according to the AOAC programme on food authenticity and fraud, non-targeted analytical methods should be used in combination with targeted analytical methods to provide better protection against adulteration. In reality, non-targeted analytical methods (e.g. NMR FoodScreener) will only be sensitive to detect adulteration with exogenous sugars that are not part of the maple syrup composition (e.g. adulteration with rice syrup - maltotriose). While for adulteration with sugars that are also present in maple syrup, the use of targeted analysis methods is essential.

At the Centre ACER, several research projects to develop methods for analyzing maple syrup adulteration have been conducted in recent years. In 2014, they led to the development of SpectrAcer™, the first non-targeted analysis system dedicated to maple syrup. This system uses Raman spectroscopy as a technique to detect adulteration. The analysis is performed using a laser that is sent to the syrup sample. The different chemical molecules that make up maple syrup (water, sucrose, glucose, fructose, etc.) will interact specifically with the laser radiation. The reflected portion of the laser is then detected and analyzed by a spectrometer. The spectra collected are then compared using advanced modelling techniques to those of a database of several thousand known and characterized samples

(e.g.: pure maple syrup, maple syrup deliberately adulterated with different percentages of foreign sugars).

In Quebec, adulteration is routinely verified during the grading and inspection of maple syrup delivered to warehouses by producers. Currently, this verification is carried out by 3 SpectrAcer™ systems installed in designated centralized laboratories. Since 2016, more than 200,000 samples have been analyzed by the SpectrAcer™ system. The number of samples tested per year is constantly increasing and will be in the order of 70,000 samples in 2020. This number makes Quebec the only place in the world where so many barrel maple syrup samples are inspected for adulteration.

When a maple syrup sample is declared positive for SpectrAcer™ adulteration detection, it is sent to the Centre ACER laboratory for further analysis. It should be noted, however, that this type of positive adulteration case is rare and that the few samples declared positive at inspection are subject to detailed laboratory checks in order to rule out the effect of certain sources of interference.

The Centre ACER's laboratory performs in-depth analyses to complete the verification of samples analyzed at SpectrAcer™ or to test samples submitted directly by maple syrup processors as part of their quality control program. The techniques used in the laboratory mainly include carbon isotope signature analysis and sugar profile analysis. In addition, the Centre ACER's notoriety as a research centre dedicated to maple products and which analyzes thousands of maple syrup samples each year gives it a perfect knowledge of the chemical composition of maple syrup. Its access to a large number of authentic samples coming directly from producers allows it to regularly validate product specifications.

On the other hand, the Centre ACER also ensures that it continues to offer its clients the best service available worldwide by following up on adulteration alerts that could affect the product and by updating the list of potential adulterants. In addition, major investments have been made to acquire the most recent technologies for the analysis of adulteration, whether for targeted analysis by liquid chromatography coupled to the isotope ratio mass spectrometer (LC-IRMS) or non-targeted analysis by liquid chromatography coupled to the high-resolution mass spectrometer. The LC-IRMS method is currently being validated and will be available in the coming months. It aims to determine the specific isotope signature of each of the sugars instead of the isotope signature of the whole product (bulk). This will certainly improve the sensitivity of this analysis compared to the current "AOAC-984.23" method.

Concerning the LC-HRMS, it is the latest generation of high-resolution spectrometer systems (resolution > 120K). This high-performance system simultaneously separates and detects the exact mass of the components of the product to be tested. Its mass accuracy of less than 1 ppm will allow significant advances in the identification of unknown adulterants during non-target analysis. The LC-HRMS method project has been postponed due to the current situation related to the COVID-19 pandemic and will be launched in the coming year.

Example of Current Methods for the Analysis of Maple Syrup Adulteration

Non-targeted analysis methods	Targeted analysis methods
• SpectrAcer™ (Raman spectroscopy[1-2]) • NMR Food Screening (RMN spectroscopy[3])	• AOAC-984.23 (carbon isotope signature analysis for whole product (bulk) • LC-RI or LC-MS (sugar profile)

Upcoming methods for the analysis of maple syrup adulteration

Non-targeted analysis methods	Targeted analysis methods
LC-HRMS (high-resolution mass spectrometry)	LC-IRMS (carbon isotope signature analysis for specific compounds)

References:

1. Maple syrup quality control BENEFITS FROM ADVANCES IN TECHNOLOGY, Canadian Food Insights-2014.
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3. https://www.bruker.com/products/mr/nmr-food-screening.html?gclid=EAIaIQobChMI2p22ycrU6gIVEIrICh3-ggeKEAAYASAAEgKP3_D_BwE.