Fiber-optic NIR technique and Aquaphotomics for detecting high fructose corn syrup adulteration of honey

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Objective

Characteristics of unifloral False Acacia (*Robinia pseudoacacia*) honey (liquid due to the high fructose content, very light colored and flavored) may easily be spoiled, negatively influencing market growth by damaging consumer confidence. Large scale honey adulteration appeared on the world market in the 1970s when high-fructose corn syrup (HFCS) was introduced by the industry [1]. Aquaphotomics considers water as a multi-element system that can be well described by its multi-dimensional near infrared (NIR) spectra. Since moisture is present in most of the natural samples, this analytical approach using water through its perturbations in different environments as a mirror of the sample itself can be applied in many fields very effectively [2]. The goal of the present study was to build an applicable NIR model for screening Robinia honey by using fiber-optic probe and to describe its spectral characteristics using recent findings of auqaphotomics.

Materials and Methods

Pure Robinia honey samples were collected from four geographic regions of Hungary, in different periods of False Acacia blossom in 2012. Liquid, cleaned, sterile, ion exchanged and filtered isoglucose syrup (HFCS) produced in closed process at high temperature contained 40% fructose and 18.7% moisture. All samples were stored in dark glass holders at room temperature (25°C) until analysis. Honey samples were diluted with HFCS in random concentrations (n = 40, HFCS concentration range = 1–40%, mean±SD = 22.3±12%). Pure and adulterated honey samples placed into glass holders were scanned at room temperature (25°C) using a FOSS NIRSystems 6500 spectrometer (FOSS NIRSystems, Inc., Laurel, MD, USA) equipped with an OptiProbe fiber optic immersion sampling unit, with 2mm layer thickness. Transflectance spectra (1100–1800 nm) were recorded with a spectral step of 2 nm as an average of 32 scans. Log(1/R) spectra acquisition was performed with the VISION 2.51 software (FOSS NIRSystems, Inc., Laurel, MD, USA). All samples were scanned ten times and all recorded spectra were stored for data analysis. Recorded spectra were exported from VISION in NSAS file format and The Unscrambler 9.7 (CAMO Software AS, Oslo, Norway) was applied for data processing. Scanned samples were tested for dry matter content by means of conventional method of drying at 103°C for 4 hours.

Results and Discussion

Moisture of pure honey samples was 12.8±0.65%, thus, strong negative correlation (r=-0.82) was found between adulteration level and measured dry matter content of mixtures. Smoothed and standard normal variance (SNV) corrected NIR spectra were used for calibration on adulteration level by means of partial least squares regression (PLSR). Model performed on the entire spectral region resulted perfect calibration with four latent variables (R^2cal = 0.981, RMSEC = 0.018). Cross-validation was applied by leaving one honey out of four (R^2val = 0.882, RMSEV = 0.046). The validation results did not weaken considerably when the PLSR model was developed only on the first overtone region of water (1300-1600nm) (Figure 1). The achieved regression vectors accentuated the water bands playing important role in the detection of HFCS content. The spectral characteristics of honey and HFCS showed significant differences in H-bonds of water and these differences dominated the PLSR models. Results show that the extent of adulteration can be predicted through the water spectral pattern of the honey mixtures. Quick, user friendly, non-destructive NIR spectroscopy combined with the aquaphotomics approach can help to describe important structural characteristics of honey, and provide possibility for accurate detection of a dominant form of adulteration.

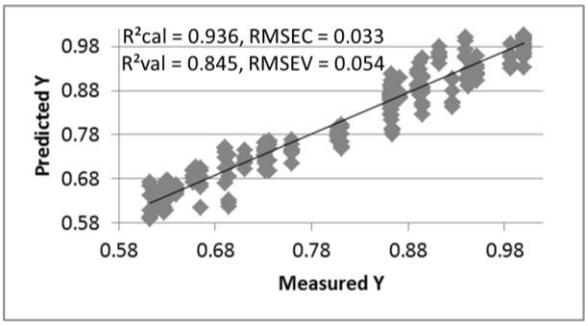

Figure 1: Results of NIR calibration on HFCS content of honey samples using 1300-1600nm spectral interval

References

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