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Analysis of adulteration in honey with standard sugar solutions and syrups using attenuated total reflectance-Fourier transform infrared spectroscopy and multivariate methods

Análisis de la adulteración en miel de abeja con azúcares estándar y jarabes utilizando espectroscopia de transformada de Fourier con reflectancia atenuada total y métodos multivariados

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Attenuated total reflectance-Fourier transform infrared (ATR–FTIR) spectroscopy and multivariate analysis were used to study honey intentionally adulterated with standard sugar solutions (glucose, fructose and sucrose), and also with cheap syrups (corn, inverted and cane sugar). By using the principal component analysis (PCA) method on pure and adulterated (0–100%) honey samples, the determination of the type of adulterant was realized in an easy way through the use of the two- and three-dimensional PCA score plots. From this analysis, a tetrahedral structure was obtained. Superior vertex is formed by the set of samples of pure honey, whereas the edges are formed by the adulterant sugars. Partial least squares method was employed to develop optimal calibrations for the six adulterants used, obtaining values of standard error of calibration in the range 0.377–0.583 and standard error of prediction in the range of 1.550–3.150 for the standard sugars, which suggest a good predictive capacity of the model employed in this study.

Keywords: honey adulteration; PCA-PLS; quantitative determination; FTIR spectroscopy

Se utilizó la espectroscopia de transformada de Fourier y análisis multivariado para la discriminación y cuantificación de miel intencionalmente adulterada con soluciones de azúcar estándar (glucosa, fructosa y sacarosa), así como también con jarabes baratos (maíz, invertido y caña). Mediante el uso del método de análisis de componentes principales (PCA) en miel pura y adulterada (0–100%), se realizó la determinación del tipo de adulterante en una manera fácil mediante el uso de las gráficas de resultados PCA en dos dimensiones y PCA en tres dimensiones. De este análisis se obtuvo una estructura tetraedral cuyo vértice superior está constituido por el conjunto de muestras auténticas, mientras que las aristas están constituidas por las adulteraciones de cada uno de los azúcares de referencia. Se aplicó el método de mínimos cuadrados parciales (PLS) para desarrollar calibraciones del contenido de los adulterantes utilizados, obteniendo valores de error estándar de calibración en el rango 0.377–0.583 y de predicción en el rango de 1.550–3.150 para azúcares estándar que sugieren una adecuada capacidad predictiva del modelo empleado en este estudio.

Palabras clave: adulteración de miel; PCA-PLS; determinación cuantitativa; espectroscopia FTIR

Introduction

Honey is a high-value foodstuff which is a target for adulteration. Sugar and water represent the main chemical constituents of honey (typically 80% carbohydrate and 17% water), whereas proteins, flavors and aromas, pigments, vitamins, free amino acids, and numerous volatile compounds constitute the minor components (Gonzalez, Marquez, Sanchez, & Gonzalez, 1998). Given its composition (mainly carbohydrates and water), honey is adulterated in the practice by cheaper, commercially available sugar syrups with similar composition. In several feasibility studies, the ability of near infrared spectroscopy (NIR) and middle infrared spectroscopy (MIR) to detect adulteration of authentic honey by either added corn syrup (CS) (Sivakesava & Irudayaraj, 2001a), beet invert syrup (BI) (Sivakesava & Irudayaraj, 2001b), sucrose syrup (SS) (García, 2003) or high fructose corn syrup (HFCS) (Gallardo, Osorio, Zuñiga, & Rivera, 2009) has been reported.

Fourier transform infrared (FTIR) spectroscopy offers a fast and non-destructive alternative to chemical measurement techniques for qualitative characterization (Kelly, Petisco, & Downey, 2006). The narrower bands of the fingerprint region reduce the problem of overlap, allowing the use of some simple mathematical treatments, such as calibrations of peak heights or areas plotted directly against concentration (Lichtenberg, Hedtke, & Bienefeld, 2002). However, in complex systems such as adulterated honey samples, the spectra of the individual components are very similar, and the effect of the overlap requires more sophisticated approaches. The multivariate methods such as principal component regression (PCR) (Tewari & Irudayaraj, 2004) and partial least squares (PLS) (Kelly, Downey, & Fouratier, 2004) provide a useful way to develop models that are able to predict several variables such as sugar contents or adulterant contents in honey or other food products.

Like an initial procedure before the application of PCR or PLS prediction methods, the principal component analysis

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