ATR-FTIR Spectroscopy for determination of free acidity, electrical conductivity, ash and total polyphenol content in honey

II International Symposium on BEE PRODUCTS 9-12 September 2012 - Bragança, Portugal

Ofélia Anjos[1,2]*; Fátima Peres[1]; Paulo Antunes[3]; José Rodrigues[4]

1: IPCB – Polytecnic Institute of Castelo Branco, Castelo Branco, Portugal;
2: CERNAS – Coimbra, Portugal;
3: CATAA – Agri-food Technological Support Center, Castelo Branco, Portugal;
4: IICT – Tropical Research Institute, Lisboa, Portugal.

Aim

This study compares the results of Fourier transform infrared spectroscopy using an attenuated total reflection window (ATR-FTIR) with physico-chemical parameters for different honey samples analyzed by standard methods.

MATERIAL AND METHODS

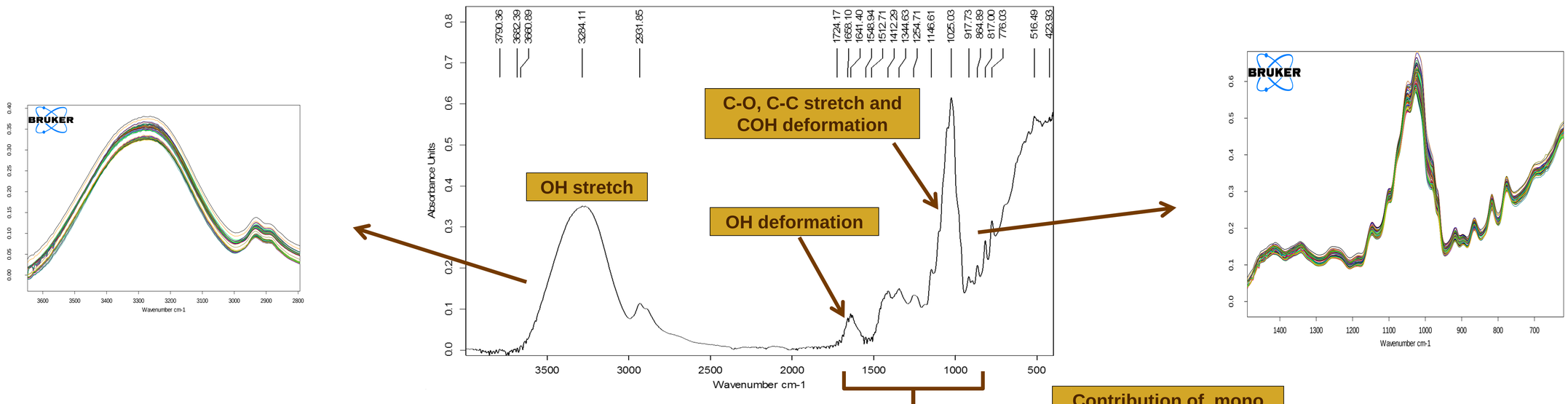
Different commercial honeys (50 samples), from different floral sources were tested for ash content, free acidity, electrical conductivity (methods adopted by the International Honey Commission) and total phenol content (determined by modification of the Folin–Ciocalteau method) and the results expressed as mg of gallic acid equivalents (GAE)/100 g of honey.

ATR - FTIR spectra were acquired with a Bruker FT-IR spectrometer (Alpha) using a diamond single reflection attenuated total reflectance (ATR) device and a zero filling of 2.

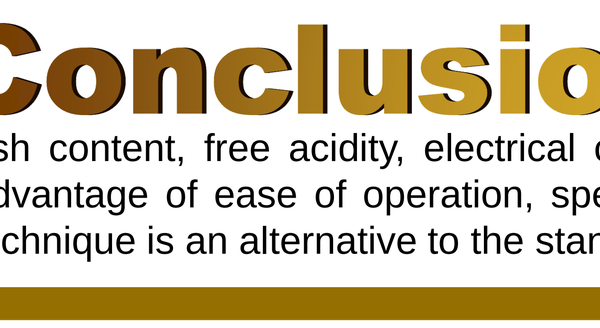
Duplicate spectra per sample were obtained with 32 scans per spectrum at a spectral resolution of 4 cm^{-1} in the wavenumber range from 4000 to 400 cm^{-1}. Principal component analyses (PCA) and partial least squares regression (PLS-R) modeling were performed using OPUS Quant 2 (Bruker Optics, Ettlingen, Germany).

RESULTS AND DISCUSSION

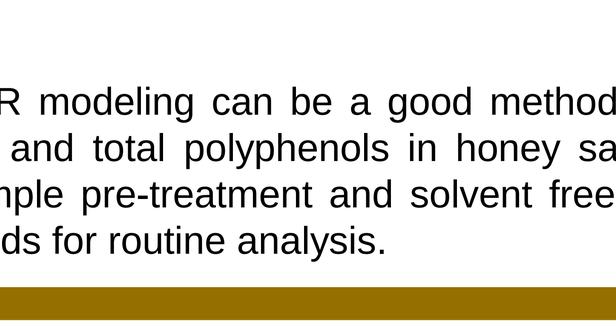
The mean values and standard deviation for analyzed parameters were: electrical conductivity – 451(mS/cm) ± 287,4; ash content – 0,18 % ± 0,17; Free acidity – 28,3 meq/kg ± 9,3; Total phenols content – 36,20 mg GAE/100g ± 23,9; which covers a high variability and gives a better calibration model.



ATR-FTIR spectrum of the average spectra from all honeys acquired from 4000 to 450 cm^{-1} with the identification of the most important bands used in the calibration models



Cross validation for electrical conductivity - Predict vs True



Cross validation for ash content - Predict vs True

Cross validation for free acidity - Predict vs True

Cross validation for total polyphenol content - Predict vs True

Table 1 - Cross validation and validation results of the calculated models for honey samples.

Parameters	Preprocess	Cross validation			Validation		
		r^2	RMSECV	RPD	r^2	RMSECV	RPD
Electrical conductivity	1stDerMSC	0.925	92.4	3.64	0.854	100	2.65
Ash content	1stDer	0.970	0.028	4.02	0.934	0.036	4.02
Free acidity	ConOff	0.824	4.6	2.4	0.605	4.7	1.61
Total polyphenol content	MinMax	0.844	8.14	2.53	0.803	9.44	2.3

1stDer – first derivative; 1stDerMSC – first derivative + multiplicative scatter correction; N – normalization; ConOff – constant offset validation; MinMax - minimum–maximum normalization; RMSECV – root mean square error of cross-validation; RPD - ratio of the standard deviation of the reference values of the validation samples

Conclusion

PLS-R modeling can be a good methodology to predict ash content, free acidity, electrical conductivity and total polyphenols in honey samples, with the advantage of ease of operation, speed, no sample pre-treatment and solvent free. Therefore, the technique is an alternative to the standard methods for routine analysis.

Instituto Politécnico de Castelo Branco
Escola Superior Agrária

Saber Tropical 125 anos IICT 1883-2008

CATAA CENTRO DE APOIO TECNOLÓGICO AGRO ALIMENTAR