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Characterization of Green Coffee Cultivars by NIR, HPLC and Chemometrics

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Abstract

The target of this work was to study and characterize, both qualitatively and quantitatively, samples of green coffee. In particular, there were taken in considerations two aspects of product interest: on one hand, the possibility of verifying the botanical origin of the product, focusing on differentiation between samples of the species Arabica and of Robusta analysed by NIR (whole grain and powder) and HPLC-DAD (considering two different types of extraction of analytes: infusion with water and extraction with solvent); on the other hand, it was investigated whether the coupling of chemometrics methods of subsequent resolution of co-eluting signals enabling it to provide an accurate and precise method for the simultaneous quantification of caffeine and chlorogenic acid by HPLC-DAD analysis, using a relatively rapid gradient, which therefore did not lead to the complete separation between the samples at the level of the chromatographic system.

Introduction

The ability to check whether a coffee sample belonged to the species Arabica or Robusta through the coupling of chemometric methods of classification, both discriminant (PLS-DA) and modeling (SIMCA), and instrumental fingerprinting was first investigated using near infrared spectroscopy. In this regard, the near-infrared spectroscopy followed by subsequent chemometric signal processing, is a fast approach, as well as economic, non-invasive and non-destructive. From an environmental point of view, moreover, it is zero impact, as the sample can be analyzed as received without any pre-treatment and therefore without the use of solvents that must be successively disposed. The NIR spectroscopy offers a viable alternative to liquid chromatography, as regards the coffee industry, where, by arranging a large number of samples to be analyzed, to ensure quality to the consumer, there is the need to resort to analytical techniques that provide reliable data quickly.

At the same time, it is also studied the possibility to characterize the botanical origin of the samples through the HPLC analysis, considering two different types of extraction of analytes from the matrix (infusion with water and extraction with solvent).

Materials & Methods

Samples. For this work were analyzed samples of green coffee of Arabica and Robusta, of different geographical origins, coming from three different Italian Customs for a total of 37 samples, 23 of which are Arabica and 14 Robusta.

NIR analysis. NIR analysis was carried out on a Thermo Nicolet FT-NIR 6700 spectrometer, equipped with an integrating sphere and an InGaAs detector (Thermo Scientific, Walton, MA). The spectra were acquired at a nominal resolution of 4 cm^{-1} in the range 4000-10000 cm^{-1}, averaging 82 scans; All available samples were used for the spectroscopic analysis, using both the whole grain that the powder obtained using an electric grinder.

HPLC analysis: For chromatographic analysis was used a ThermoQuest chromatographic system, model "Spectrasystem", equipped with a P4000 pump and DAD UV6000LP detector (Thermo Fisher Scientific, Waltham, MA); a Phenomenex column "Kinetex 5u C18 100A" with 5 μm particles, inner diameter of 4.6 mm and length of 25 cm and a 4mm guard column with the same characteristics of the column (Phenomenex, Torrance, CA, USA). The chromatograms were acquired by operating a

scan from 200 nm to 400 nm, with wave length intervals of 2 nm. For all injections it has been worked with a 6-way valve full-loop, 20-L.

Only 24 samples of green coffee were used for chromatographic analysis, they were treated with two different extraction procedures, one with water and one with solvent. The extracts were analyzed both as they are and diluted, thus obtaining a total of 24 chromatograms for the infusion such as, 24 for the diluted infusion, 24 for the extract with a solvent such as and 24 for the extract with solvent diluted.

Results

The pretreated NIR spectral data were Analysed by PLS-DA and by SIMCA obtained good results. The PLS-DA algorithm was applied to the pretreated data with SNV and first derivative obtaining a correct classification percentage equal to 100% respectively for both varieties, both for the whole grain and powder samples(Figure 1).

As regards the models constructed with the SIMCA algorithm is observed that the one relative to the Arabica class model has resulted in a sensitivity of 100% and a specificity of 90% for the whole grain and in a 94.28% sensitivity and in a 100% sensitivity for powder samples; the model for the class Robusta presented slightly less good performance, with a sensitivity of 87.50% and a specificity of 60% for the same state and sensitivity 75% and specificity of 94.14% for the ground.
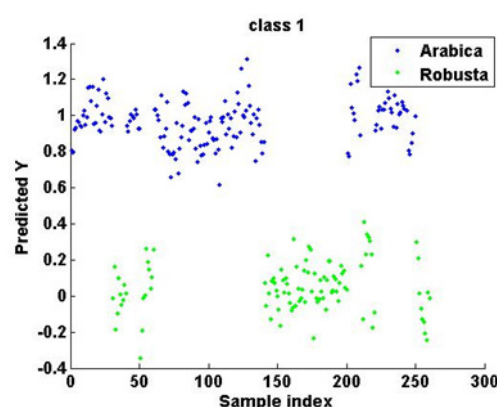

Figure 1: Graphical representation of predicted y values for Arabica (class 1)

The classification obtained using chromatographic data gave similar results to those obtained with NIR spectroscopy.

Regard the quantitative analysis it was possible, through the use of the algorithm MCR, obtain an almost complete resolution of co-eluting chromatographic peaks. The well resolved peaks were used for the next calibration phase. In particular, starting from the standard peaks and the internal standard, it is the calibration curves are constructed for the caffeine and chlorogenic acid, and have been used to calculate the concentrations of the two analytes in the coffee samples.

Conclusions

The results show that the use of different chemometric methods for the optimization of the experimental procedures and the processing and interpretation of spectroscopic and chromatographic data for qualitative and quantitative purposes, proves a valid strategy, versatile and very promising in the quality control of optical samples of coffee, in particular as regards the green coffee

References

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