Quality control meets chemometrics: a case study for the quantification of free phenol in syntans using FTIR-ATR spectroscopy

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Content

Chemometrics is a powerful statistical way to solve complex analytical issues and nowadays it is largely used in different industrial fields, as well as leather industry[1]. It is already known that quality control operators have to deal rapidly and efficiently with complex issues. Infrared spectroscopy is a *fingerprint* type technique frequently used in quality control laboratories because of its flexibility, fastness and cheapness. The mathematical interpretation of the spectral data using chemometric models allows to analyze simultaneously multivariate data set, such as infrared spectra. Although chromatographic methods are much sensitive, the application of chemometrics to vibrational spectroscopy ensures fast and cheap analyses, no sample treatment, and no specialist interpretation. In this work, a case study based on chemometrics applied to the characterization of chemicals for leather sector is proposed. The aim was the development of an innovative and reliable method that combines all the usefulness of spectroscopic techniques and multivariate statistics for the quantification of free phenol in phenolic syntans. The quantitative regression model was based on Partial Least Squares (PLS) calibration applied on IR analysis. PLS technique is based on the construction of a predictive mathematical model that allows calculating quantitative values related to experimental variables[1], where the variables are, in this case, the intensities of singular wavelengths of the spectrum. HPLC coupled with UV-Vis detector was used to quantify free phenol concentration on calibration standards, previous distillation of a sample solution. The raw liquid syntans were analyzed using FTIR-ATR (Attenuated Total Reflectance) equipped with diamond crystal, spectral range 650 cm^{-1} - 1800 cm^{-1}, and 4 cm^{-1} of resolution. The training set was built considering the spectra of 58 calibration standards (Figure 1) in a range of concentration between 500 ppm and 11000 ppm and the effectiveness of the model was calculated using a cross-validation method and external set of six test samples (Figure 2). Even though the low sensitivity of FTIR-ATR, a root mean square error of 188 ppm was calculated for the regression model.

1. R. G. Brereton, *Applied Chemometrics for Scientists*, John Wiley & Sons, LTD, Chichester, 2007

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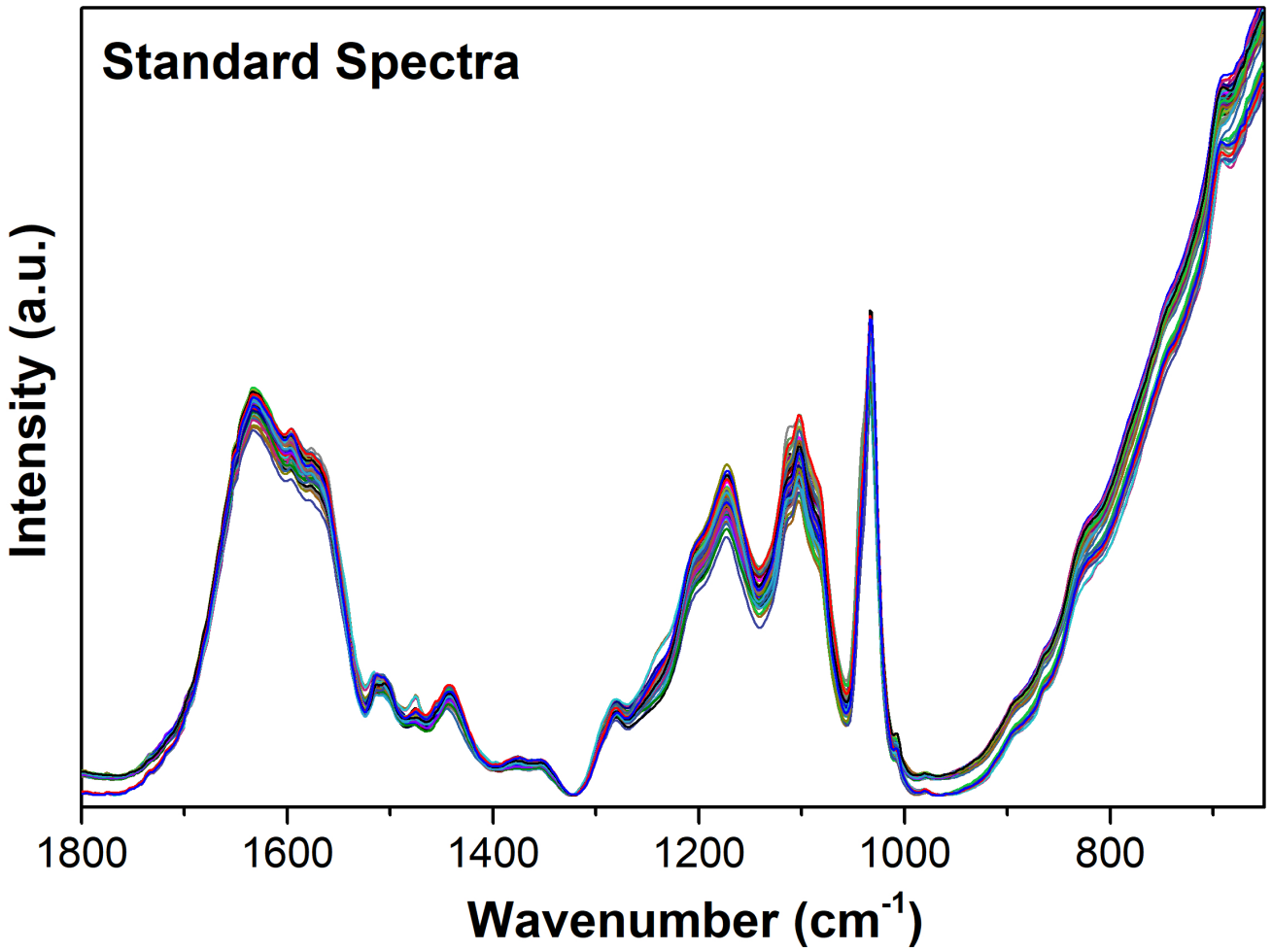

Figure 1
FTIR-ATR spectra of the calibration standards. Spectra of no treated samples were collected in a spectral range between 650 cm^{-1} and 1800 cm^{-1} and resolution of 4 cm^{-1}.

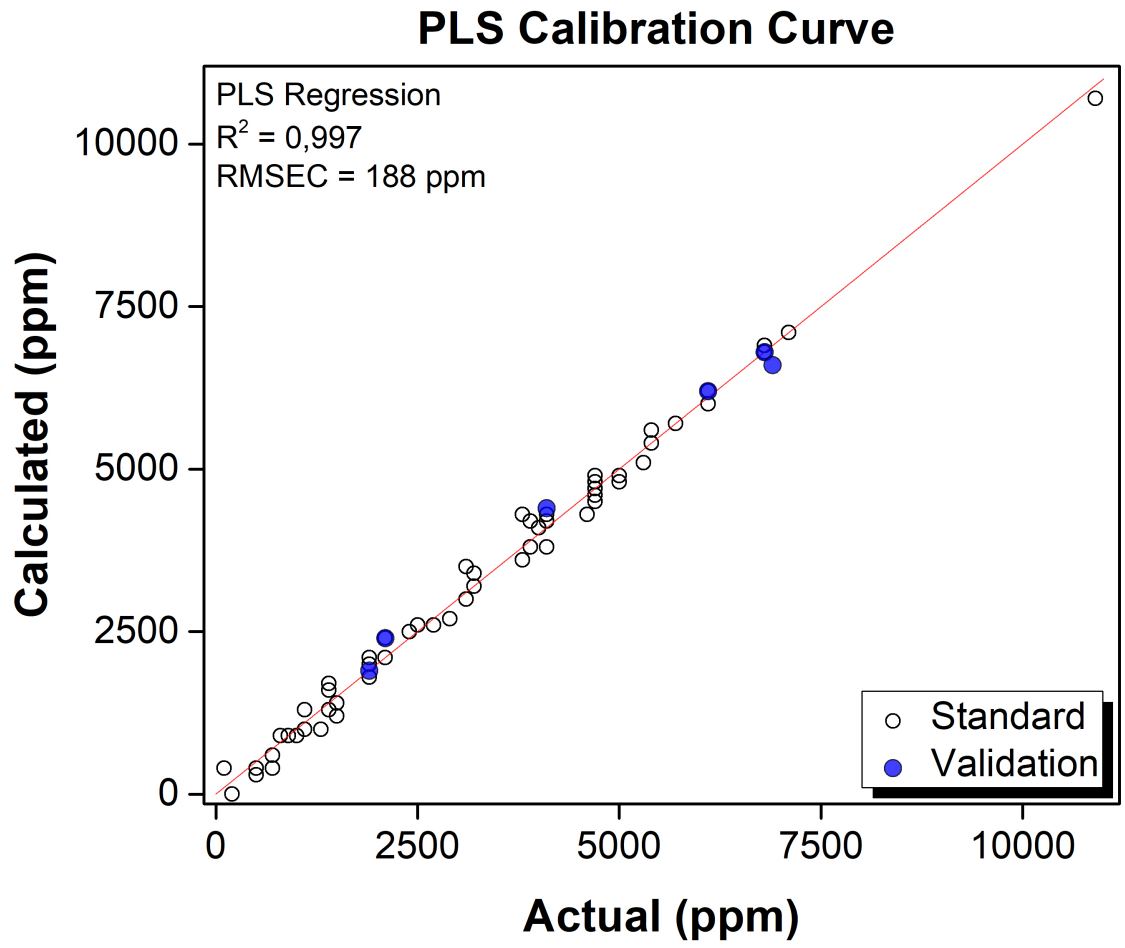

Figure 2
PLS calibration plot: white dots correspond to calibration standards and blue dots correspond to validation samples.

Keyword
chemometrics, syntans, spectroscopy